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Investigation of Relative Humidity Sensing Using Tapered No-Core Fiber Coated With Graphene Oxide Film

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ABSTRACT A microfiber interferometer based on a single-mode tapered-no-core single-mode (STNCS) fiber structure coated with a thin layer of graphene oxide (GO) was proposed and demonstrated as a relative humidity (RH) sensor. The STNCS fiber structure has a strong evanescent field, which can sensitively response to changes in the surrounding refractive index. The moisture-sensitive material GO was used to form the film on the no-core fiber by a dip impregnation method. The effects of GO concentration and the tapered waist diameter on RH sensitivity were studied experimentally. The experimental results showed that a thinner waist diameter provides higher RH sensitivity. When the concentration of GO solution is 0.01 mg/mL, the STNCS fiber structure with a waist diameter of 2.9 μm performs better in RH sensing; a high sensitivity of 0.461 nm/%RH in the range of 40-98% was achieved. The sensor has good stability with wavelength fluctuations of $\pm$0.009 nm and $\pm$0.008 nm over 60 mins at RHs of 50% and 80%, respectively. The proposed fiber optic humidity sensor has a high sensitivity over a wide measurement range and offers good stability showing promising potential application in the field of RH sensing.

INDEX TERMS Humidity sensor, graphene oxide, no-core fiber, microfiber interference.

I. INTRODUCTION

The measurement of relative humidity (RH) is important in the fields of industrial and agricultural production, meteorology, heritage conservation, storage, aerospace, and so on [1]–[3]. Compared with the traditional electrical humidity sensor, the optical fiber sensor has the advantages of fast response, small size, anti-electromagnetic interference [4] and it can realize distributed measurement. In recent years, optical fiber sensors coated in a moisture-sensitive film have widely attracted the attention of researchers. A variety of optical fiber sensors with different structures have been proposed, such as a fiber Bragg grating coated with polyimide [5], plastic optical fibers coated with hydroxyethyl cellulose [6], a long-period fiber grating coated with a polymer composite film [7], a side-polished fiber coated with TiO_2 [8], a photonic crystal fiber coated with agarose and GO [9], [10], a no-core fiber (NCF) coated with an agarose-gel film [11], and a small-core fiber coated with poly(ethylene oxide) (PEO) [12]. The humidity sensing mechanism can be divided into two categories: a light intensity sensitive mechanism and a phase-sensitive mechanism. The refractive index (RI) or the thickness of the film on the surface of the optical fiber will change with various environmental humidity levels due to the hydrophilic and hydrophobic effects. The small aspect ratio of most moisture-sensitive films lead to poor penetrability, which prevents water molecules from penetrating the film. GO is an important derivative

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of graphene and has a larger aspect ratio than other existing moisture-sensitive materials [13]. The two-dimensional structure of graphene means its carrier density is particularly sensitive to the ambient humidity. It can quickly absorb water molecules to form nanoscale molecular films of water [14]. The effective RI of the GO nano-film changes with the humidity of the surrounding environment, which will result in a change in spectral characteristics of an optical fiber interferometric sensor [15]–[17]. Several optical sensors based on the interaction between graphene and the evanescent wave of an optical waveguide have been proposed [18], [19]. A splitting ratio-adjustable Mach-Zehnder interferometer (MZI) coated with GO achieves a high humidity sensitivity of 0.263 dB/%RH over a RH range of 35% to 85% [20]. A RH sensor based on a GO coated few-mode fiber MZI shows humidity sensitivity of 0.191 and 0.061 nm/%RH over the RH range of 30-55% and 55-95%, respectively [21]. The sensors described above, however, are limited by either low sensitivity or high cost.

In this paper, a highly sensitive microfiber interferometer coated with GO for sensing RH was designed, and a preliminary demonstration was performed. The microfiber interferometer was based on a single-mode tapered-no-core single-mode (STNCS) fiber structure. The structure has a strong evanescent field, which can sensitively respond to changes in the surrounding RI. The RH sensing characteristics of the sensor modified by different concentrations of GO (0.001, 0.01, 0.1, 1 mg/mL) and the influence of the waist diameter of the tapered NCF on the RH sensing sensitivity were analyzed. The stability and the temperature response were also investigated experimentally. The proposed optic fiber RH sensor is low cost, flexible, highly sensitive and stable, which implies great potential commercial application in such fields as biomedicine and industrial production.

II. PRINCIPLE AND SIMULATION

The schematic of the sensing structure is shown in Fig.1. A section of the NCF is fused spliced between two single-mode fibers (SMFS). When the input light is injected from the lead-in SMF into the NCF, multiple modes are excited and propagate in the NCF section. When the NCF is tapered to small diameters, the evanescent wave will be enhanced, resulting in the increased RI sensitivity of the fiber sensor [22], [23].

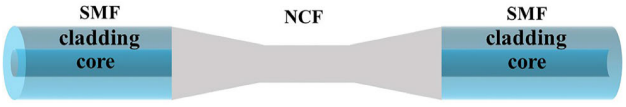

FIGURE 1. Schematic diagram of the single-mode tapered-no-core single-mode structure.

Numerical simulations based on a 2D model were conducted by the beam propagation method (BPM). In the simulation, the diameter and length of the tapered waist were 3.1 μm and 8 mm, respectively. The length of the taper transition zone was approximately 3.55 mm. The core and cladding diameters of SMF were 8 and 125 μm, with a corresponding RI of 1.4682 and 1.4628, respectively. Figure 2 (a) shows the distributions of the optical field and the normalized optical intensity propagating along the STNCS fiber structure at 1575.28 nm. The simulated transmission spectrum in the wavelength range from 1565 nm to 1580 nm is presented in Fig.2 (b).

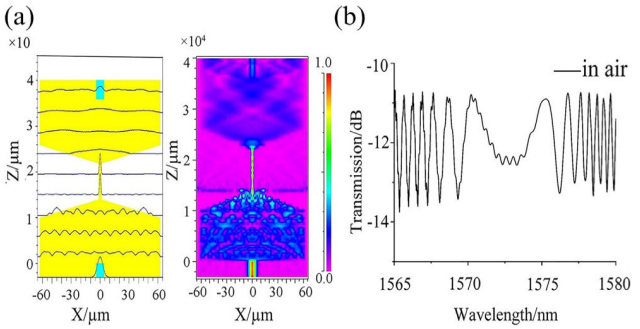

FIGURE 2. (a) The distributions of optical field and the normalized optical intensity propagating along the STNCS structure with a tapered waist diameter of 3.1 μm at 1575.28 nm. (b) The simulated spectrum response of the STNCS structure.

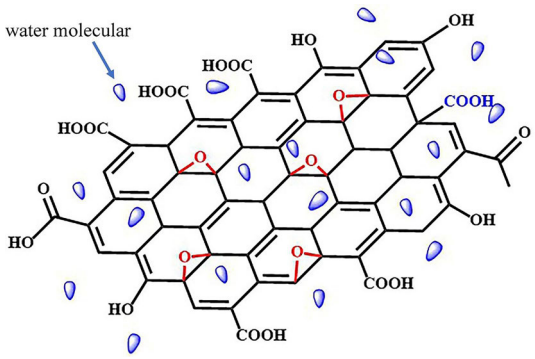

FIGURE 3. Molecular structure model of GO after water absorption.

GO structurally links a large number of hydrophilic groups in a pseudo-2D lattice. It is an ideal sensor material due to its large specific surface area, good dispersion, moisture-sensitivity and that fact that it can be easily to be modified. Water molecules act as active carriers on the outer surface of the film. Figure 3 shows the molecular structure of GO after water absorption [24]. Blue droplets indicate the water molecular. As a result, the effective RI of the GO nano-film changes with the humidity of the surrounding environment, which provides the basis for the sensing of humidity reported here.

III. FABRICATION AND EXPERIMENT

A section of 20 mm NCF was fused spliced between two SMFs by a commercial fusion splicer (FSM-80S, Fujikura). The NCF section of the sensor was heated and pulled to form the tapered region using a fiber optic puller

(OC2010, Nanjing Jilong optical communication Co. Ltd) by the hydrogen-oxygen flame brushing technique. To enable the optical fiber sensor interact with the ambient water molecules, a RH sensitive GO film should be coated on the surface of the STNCS. According to the analysis of the relevant literatures [24], [25], the GO film coating process used in this paper was listed as shown below: (1) the fiber optic sensor was cleaned with alcohol before coating; (2) GO was dispersed in water by sonication and heated to 70 °C to prepare a stable suspension of GO crystallites; (3) the fiber sensor was then impregnated with GO solutions for 0.5h; (4) to obtain a uniform and solid GO nano-film, the sensor was dried in a drying oven at 30 °C for 4h. The fabrication process and a scanning electron microscope (SEM) image of a tapered waist fiber with a GO coating are shown in Fig.4.

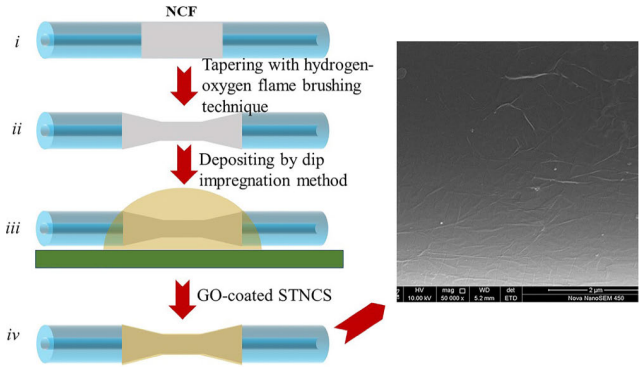

FIGURE 4. The fabrication process (left) and a SEM image of GO-coated STNCS (right).

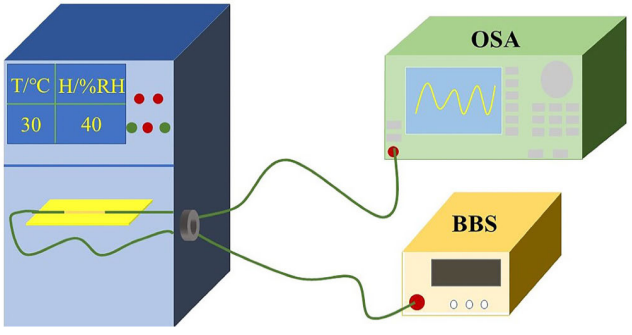

FIGURE 5. Schematic diagram of the experimental setup.

Figure 5 shows a schematic diagram of the experimental setup. When the incident signal propagates from the input SMF into the NCF, a percentage of the evanescent field transmitted around the NCF surface increases significantly, increasing the RI sensitivity of the fiber sensor. The variation in the optical properties caused by the ambient humidity was recorded by an optical spectral analyzer (OSA, Yokogawa AQ6370D). A humidity range from 40% to 98% was provided by a constant temperature and humidity chamber (ST-80L, Xiamen Yishite Instruments Co.Ltd). A broadband light source (BBS, ASE-C-30-B, Hefei Max-ray Photonics) provides a measurable wavelength range of 1530-1610 nm.

Four different concentrations of GO (0.001, 0.01, 0.1, 1 mg/mL) solution were prepared for the humidity sensing experiment using an STNCS structure with a waist diameter of 2.9 μm. The STNCS fiber structure was firstly coated with low concentration GO solution (eg. 0.01 mg/mL) and tested for RH measurement. The coated STNCS RH sensor was then washed three times with deionized water (10 mins each time) before coating with higher concentration GO solution (eg. 0.1 mg/mL), and the process repeated from low to high concentration of GO solution. In order to verify that three times' washing is enough to remove the existing GO film, a sensor coated with GO concentration of 0.1 mg/mL was selected to test its RH sensitivity after each wash and the spectral responses to RH after each wash are shown in the Fig. 6. It can be seen that after three times of wash, the RH has limited influence on the sensor structure, indicating that three washes is enough to remove the existing coating of GO.

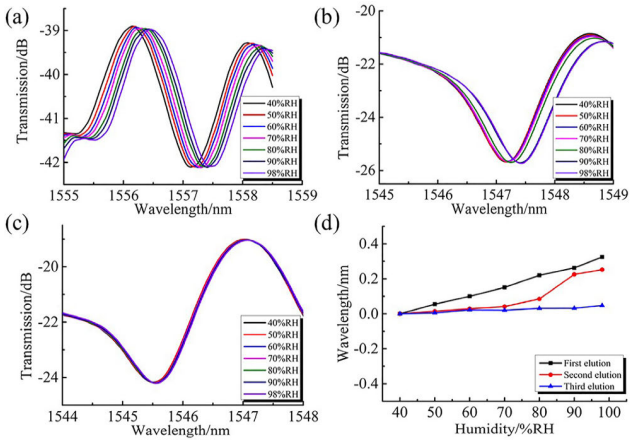

FIGURE 6. The transmission spectral response of the STNCS sensor after each elution. (a) first elution (b) second elution and (c) third elution. (d) The RH response of the sensor after each elution.

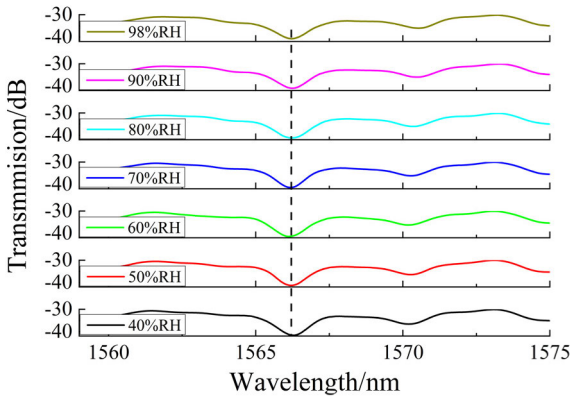

FIGURE 7. The spectral response of the STNCS sensor without coated GO showing no response over a range of RHs, 40 – 98%.

IV. RESULTS AND DISCUSSION

The response to the RH of the STNCS structure without GO coating was firstly studied and shown in Fig.7. There is

no clear wavelength shift can be observed, indicating that the STNCS fiber structure without GO coating is insensitive to RH.

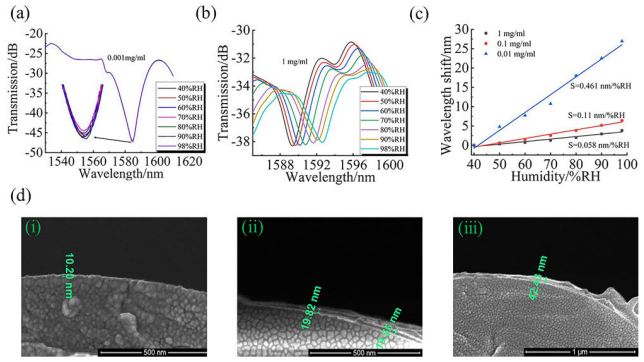

FIGURE 8. The recorded transmission spectra of the STNCS sensor coated with (a) 0.001 mg/ml and (b) 1 mg/ml GO; (c) the RH sensitivity of the STNCS structure coated with different GO concentrations; (d) SEM images of the STNCS coated with GO (i) 0.01 mg/mL (ii) 0.1 mg/mL (iii) 1 mg/mL.

The spectral response of the fiber sensor to the different RHs was observed and shown in Fig. 8, starting from the lowest (0.001 mg/mL) to the highest (1 mg/mL) GO concentration. The RH sensing characteristics of the sensor modified with different concentrations (0.001, 0.01, 0.1, 1 mg/mL) of GO were analyzed. It can be seen from Fig. 8 (a) that when the concentration of GO solution is 0.001 mg/ml, the sensor is insensitive to RI variations, and the spectrum remains almost unchanged. Fig.8 (b) shows the transmission spectrum of the sensor coated with 1 mg/mL GO. It can be seen that the spectrum moves to a longer wavelength as the RH increases from 40% to 98%. Fig.8 (c) shows that the sensor demonstrated linearity between the wavelength shift and the RH variation. A sensitivity of 0.461 nm/%RH can be reached in the RH range of 40-98% when the concentration of GO is 0.01 mg/mL, which is higher than that of most humidity sensors reported [21], [26].

The thickness of GO nanofilms depend on the concentration of GO solution [27]. The film thickness was measured using field emission scanning electron microscope (FESEM, FEI Nano SEM450∗). SEM images of STNCS coated with GO concentrations of 0.01, 0.1 and 1 mg/mL are shown in Fig.8 (d), indicating thicknesses of 10.20, 19.82 and 42.43 nm respectively. We were able to infer that the thickness of the GO film plays an important role in the sensitivity of the STNCS RH sensor. The thickness of the GO nano-film needs to be within a range; neither too thick nor too thin. When the thickness of the GO film is thin, the change in refractive index caused by different RHs is too small to effect a response from the sensor. When the GO film is thicker, the permeability of water molecules is limited, and the RH sensitivity of the STNCS optical sensor is subsequently decreased [28].

We take the parameter figure of merit (FOM) which defined as the ratio of the sensitivity S to the full width at half-maximum FWHM to evaluate the quality of the sensor. The FOMs of corresponding fiber sensors coated with three different GO concentrations (0.01 mg/ml, 0.1mg/ml and 1mg/ml) are 0.2545, 0.0914 and 0.0230, respectively. It means that the sensor coated with thinner film provides higher quality in certain range of coated film thickness.

To study the influence of the waist diameter on humidity sensing, three STNCS structures with different waist diameters were fabricated, each coated with 0.01 mg/mL GO. The different waist diameters were obtained by adjusting the parameters of the fiber optic puller.

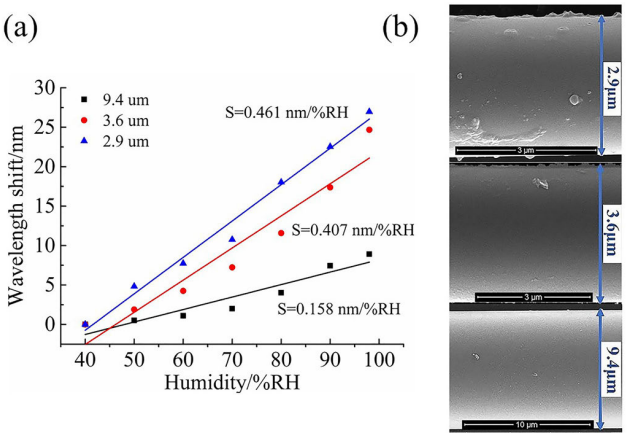

FIGURE 9. (a) The RH sensitivity and (b) SEM images of STNCS structure with waist diameters of 2.9 μm, 3.6 μm, 9.4 μm.

Figure 9 clearly shows that the RH sensitivities increased as the taper waist diameter decreases. The RH sensitivity is optimal at 0.461 nm/%RH for an STNCS fiber structure with a waist diameter of 2.9 μm, and the sensitivity decreases to 0.158 nm/%RH at a waist diameter of 9.4 μm.

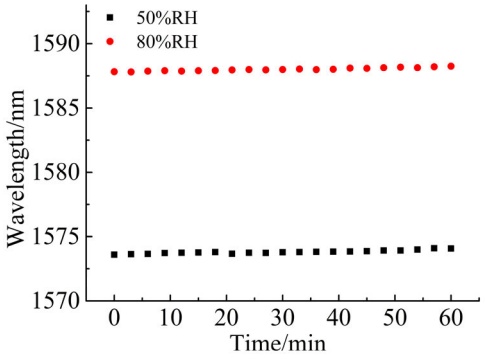

FIGURE 10. Stability of the proposed sensor at RHs of 50% and 80%.

The stability of the proposed sensor was investigated under RHs of 50 and 80% at 30 °C. As shown in Fig.10, the wavelength remains relatively stable at a fixed RH; fluctuations of ±0.009 nm and ±0.008 nm were observed over 60 mins at RHs of 50% and 80% respectively. The small wavelength fluctuation that was observed may be due to vibrations within the temperature and humidity chamber during the RH adjustment and/or arise from the instability of the BBS.

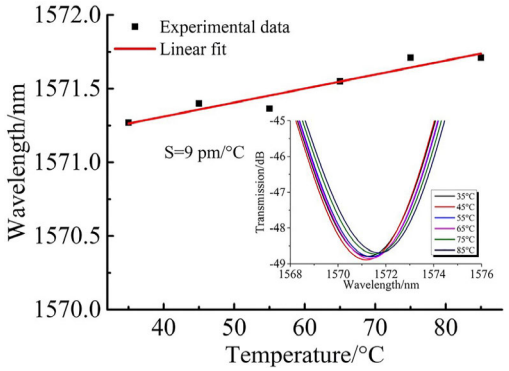

FIGURE 11. The temperature sensitivity of the STNCS structure.

In addition, the temperature response of the proposed sensor structure was tested after coating with GO as shown in Fig. 11, where a sensitivity of 9 pm/°C was achieved at temperature range from 35 to 85 °C. This result indicates that although there is a cross-sensitivity of temperature to RH, the influence of the temperature is limited [29].

TABLE 1. Performance of other RH sensors reported.

Structure	Material	Measurement Range(%RH)	Sensitivity (nm/%RH)	Ref.
SNCS	Agarose-Gel	30-75	0.149	[11]
Few-mode MZI	GO	30-55 55-95	0.191 0.061	[21]
Side-polished SMF	GO	85-97.6 32-85	0.915 0.145	[25]
Micro-knot resonator	GO	0-80	0.01	[30]
Waist-enlarged taper SMF	GO/PVA	25-80	0.193 dB/%RH	[24]
Small core fiber	PEO	80-83 83-95	4.3 0.5	[12]
SNCS	HEC-PVDF	40-90	0.196 dB/%RH	[31]
SMF-HCF probe	PVA/GODS	11-81.34	0.117	[32]
STNCS	**GO**	**40-98**	**0.461**	**This paper**

Table 1 summarizes the performance of different sensors reported in the literature. A small core fiber coated with PEO has a maximum RH sensitivity of 4.3 nm/%RH at a very narrow RH range 80∼83%, however with nonlinear wavelength shift over a wide RI range 40∼95% [12]. A side-polished SMF structure coated with GO shows the maximum sensitivity of 0.915 nm/%RH, but the RH range is only 85∼97.6% [25]. The Microfiber-Knot resonator provides data across a wide RH range but with a low sensitivity of 0.01 nm/%RH [30]. Compared to the sensors in Table 1, the STNCS structure in this paper shows a high sensitivity across a wide range of RH. In addition, it has the advantages of a simple structure, easy fabrication, and low cost.

V. CONCLUSION

In this work, a highly sensitive GO coated microfiber interferometer based on an STNCS fiber structure for the measurement of RH was proposed and investigated. The RH sensing characteristics of STNCS fiber structures coated with different concentrations of GO (0.001, 0.01, 0.1, 1 mg/mL) and of different waist diameters (2.9, 3.6, 9.4 nm) were studied experimentally. Combined with SEM images, the results demonstrated that a decreasing GO nano-film thickness gave a greatly increased RH sensitivity. The thinner waist diameter of the STNCS fiber structure provided a higher RH sensitivity. A maximum RH sensitivity of 0.461 nm/%RH in the range of 40-98% was achieved using a structure with a waist diameter of 2.9 μm. The wavelength fluctuations were only $\pm$0.009 nm and $\pm$0.008 nm over 60 mins at RHs of 50% and 80%, respectively. Compared to other RH sensors, this structural design offers the advantages of higher sensitivity, better stability, and a simpler structure, which make it attractive for practical applications in the fields of biochemical analysis and medicine.

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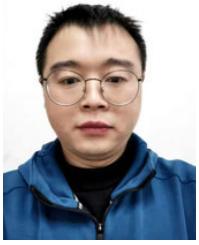

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