Tapered Fiber Bragg Grating Sensor Coated with Zinc Oxide Nanostructures for Humidity Measurement

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***Abstract*—A new approach to measure relative humidity changes by using fiber Bragg grating (FBG) is presented. Etching method is used to fabricate an FBG taper, which is then coated with zinc oxide (ZnO) nanostructures. The performance of the ZnO coated FBG is compared to its uncoated version and the experimental results produced a sensitivity of 2.51 pm/% and 1.36 pm/% respectively. The results demonstrate the ability of ZnO coatings in enhancing the performance of the sensor for the measurement of relative humidity.**

***Index Terms*—Fiber Bragg Grating (FBG); Humidity Sensor; Tapered FBG; Zinc Oxide; ZnO Nanostructures.**

I. Introduction

The humidity is one of the main aspects of environment as it is an indication of moisture level which can affect industrial and biological elements of life. It is important to detect, monitor and control the humidity level in order to ensure human comfort and attain good surrounding atmosphere for industrial and domestic applications [1]. Humidity control is crucial in wafer processing in semiconductor industries and in humidity-sensitive medical applications such as respiratory equipment, sterilizers, incubators and pharmaceutical processing [2].

Numerous methods of measuring relative humidity (RH) have been presented over the years by growing studies on humidity sensing. An instrument called psychrometer was used as the simplest method for detecting humidity [3]. Another method developed later by researchers was by using hygrometric sensors which detect changes in physical and electrical properties of sensitive elements when exposed to different humidity conditions [4]. The concept of this type of RH sensing is the physical and chemical absorption of water [5]. Additionally, another possible method for the implementation of humidity sensing is by using fiber optic sensors. Research on fiber optic sensing has intensified due to its advantages such as electrically passive operation, immunity to electromagnetic interference (EMI), lightweight, small size, high sensitivity and multiplexing capabilities [6]. Diverse techniques available in applying fiber optics for humidity sensing either as extrinsic or intrinsic sensors [7] include direct spectroscopic, evanescent wave and in-fiber grating [8]. The in-fiber grating has become a favorable option for intrinsic fiber optic sensors in recent years. Based on the grating period, it can be categorized into two main classes which are fiber Bragg grating (FBG) and long period grating (LPG). The characteristics of FBG which are corrosion resistant and durable raises interest in its development as a viable sensing approach [9]. The method used to write gratings on FBG is by exposing the core to ultraviolet light through a phase mask [10]. FBG sensor has emerged as a useful sensing element for diverse applications and is frequently used for detecting parameters such as strain, temperature [11], and electromagnetic field [12].

In applying FBG for humidity sensing, Tao et al. [13] presented studies on humidity sensor made from a multimode fiber (MMF) taper coated with polyvinyl alcohol (PVA) cascaded with an FBG. The optical power is regulated twice by the multimode fiber taper (MFT) to enhance the sensitivity of the humidity sensor. However, the fabrication process is more complicated with the addition of the MFT. Fuxin et al. reported an experimental study on humidity sensing by coating an FBG with 10 layers of polyimide resin [14]. Polyimide coating is known to be moisture-sensitive and thus able to increase the sensitivity of the sensor towards humidity but the multi-layer coating again complicates the fabrication process. Sandra et al. on the other hand, experimented coating an FBG with organo-silica hybrid material known as di-ureasil [15]. Similar to polyimide, the di-ureasil coating acts as a transducer layer that expands in the presence of humidity, inducing thermal or mechanical actuation in the FBG. While the di-ureasil layer exhibited a much better enhancement in sensitivity compared to polymer-based solution, it is a time-consuming fabrication process. The synthesis of di-ureasil took around 23 hours to prepare while the coating process alone took more than 40 hours. With the advancement of nanotechnology over the past few decades, studies on one-dimensional materials preceded the field of nanoscience and nanotechnology [16]. Due to its extremely high surface-to-volume ratio characteristic, one-dimensional nanostructures exhibit better sensitivity than conventional materials. Among the earliest discovered, zinc oxide (ZnO) is a significant functional material widely applied as sensing materials [17, 18].

Here, we present a simple and low cost FBG sensor coated with ZnO nanostructures for the measurements of relative humidity. This sensor offers a less complex alternative to the previous works in terms of its fabrication. Due to the usage of the hydrothermal method in synthesizing ZnO nanostructures on the FBG, the process is relatively easy and energy efficient since the method uses temperatures that are less than the boiling water temperature and does not require complex vacuum environment.

II. EXPERIMENTAL

Figure 1 shows the flowchart of the overall process used for the design of an FBG sensor with ZnO coating for humidity detection. The process consists of three phases which are FBG etching, ZnO nanostructures synthesis and humidity sensing.

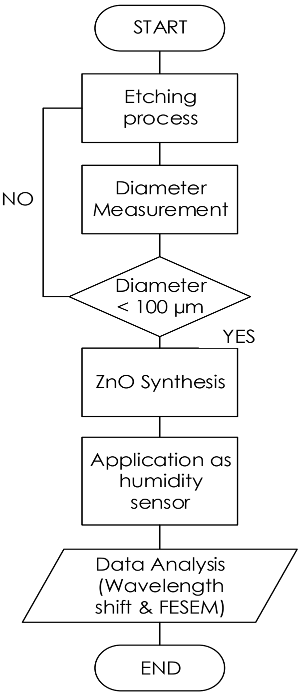

Figure 1: The overall process of designing an FBG-based humidity sensor

A. Etching Process

A hydrofluoric acid (HF) solution with a concentration of 46% is used to etch the fiber on the side of its grating. This is achieved by immersing the grating part in the HF solution for an hour. After an hour, the etched part of the FBG is rinsed with deionized water (DI water) to remove the residual etchant. Figure 2 illustrates the etching process of the FBG. An optical microscope is used to measure the diameter of the etched FBG. The diameter value after the first etching is measured to be 103 µm. The etching process is repeated to obtain a smaller diameter. After the second etching, the FBG is measured to be 84.74 µm. Figure 3 displays the microscopic image of an etched FBG after the second etching process.

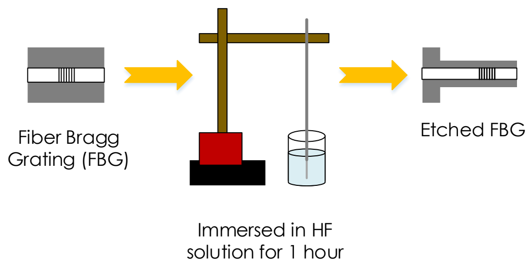

Figure 2: Etching process of an FBG

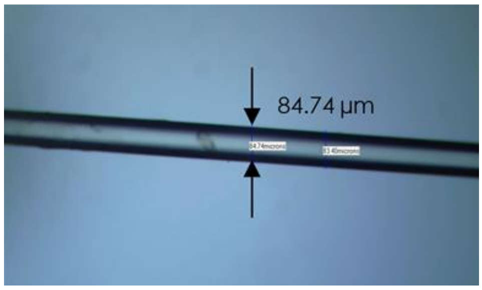

Figure 3: Microscopic image of the taper of an etched FBG

B. ZnO Nanostructures Synthesis

The second phase of the fabrication process is to coat the etched part of the FBG with ZnO nanostructures using hydrothermal method as reported in [19]. The synthesis of the ZnO nanostructures involved two major steps, namely the seeding and growing process.

Seeding process: A seeding solution made from 0.008776g of zinc acetate [$Zn(CH_3COO)_2{\cdot}2H_2O$] (Grade AR, Friendemann Schmidt) in 40ml of ethanol [C_2H_6O] (95%, HmbG) is prepared and stirred in a sonic bath for 5 minutes. 60ml of sodium hydroxide [NaOH] (Merck) with a molarity of 2M is then added to the solution. The etched FBG is placed on a hot plate at 90℃ and the seeding solution is dropped uniformly on the etched part and is slowly dried. The drop and dry process are repeated for 10 times and the fiber is finally left to anneal for 2 hours on the hot plate at 90℃. Figure 4 shows the step by step process of the seeding process. After the completion of the seeding process, a seeded layer is formed on the fiber which serves as the nucleation site for the growth of ZnO.

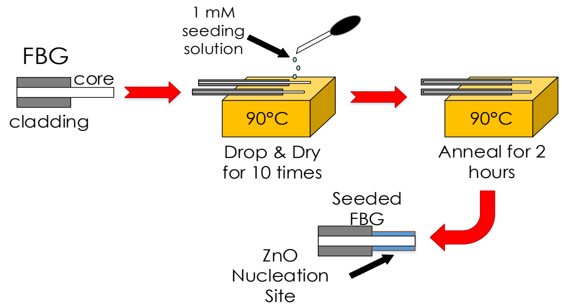

Figure 4: Microscopic image of the taper of an etched FBG

Growth process: In order to grow ZnO nanostructures on the FBG, 2.37976g of zinc nitratehexahydrate [$Zn(NO_3)_2{\cdot}6H_2O$] (98%, Sigma-Aldrich) combined with 1.12152g of hexamethylenetetramine [$C_6H_{12}N_4$] (99%, Sigma-Aldrich) are dissolved in 400 ml of deionized (DI) water. The solution is again stirred using a sonic bath to form a homogenous growth solution. 20 ml of 2M NaOH solution is also added into the solution. Next, the seeded FBG is placed vertically in a beaker containing the growth solution and left for 5 hours in an oven at 90̊C. The growth solution is replaced with a new batch of solution to maintain a constant rate of ZnO growth before being inserted back into the oven for another 3 hours. The growth process of ZnO nanostructures is illustrated in Figure 5. Altogether, the growth process took eight hours to complete and finally the fiber is taken out and rinsed with DI water. To analyze the ZnO nanostructures growth on the FBG, FESEM is used to view the image of the nanostructures. Figure 6 represents the FESEM image of the ZnO nanostructures grown on the etched FBG.

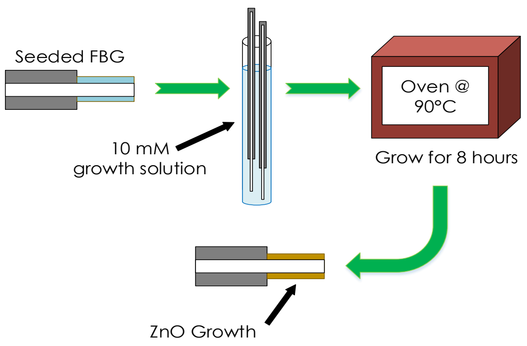

Figure 5: Growth process of ZnO nanostructures on FBG

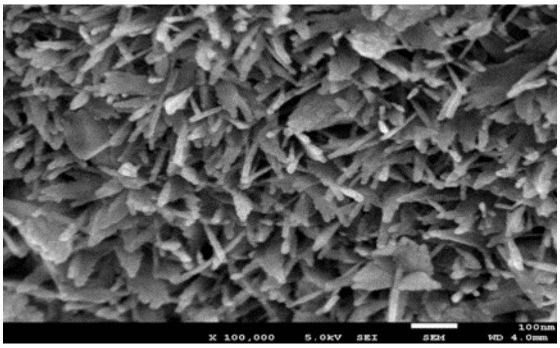

Figure 6: ZnO nanostructures grown on etched FBG

C. *Experimental Setup*

Figure 7 illustrates the experimental setup used for measuring relative humidity. The FBG is illuminated with an amplified spontaneous emission (ASE) from an erbium doped fiber amplifier (EDFA) as a broadband light source. It is placed inside a sealed chamber equipped with a dish filled with saturated salt solution. The reflection spectra is obtained by connecting the ASE to an optical spectrum analyzer (OSA). In the experiment, the performance of the proposed sensor is investigated for various relative humidity levels. The relative humidity inside the chamber is varied from 55% to 80%. The humidity level is measured using an omega RH-21 C temperature-relative humidity meter.

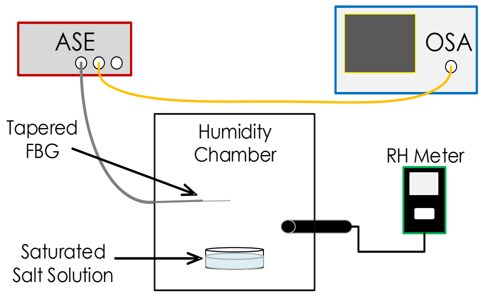

Figure 7: Experimental setup for the FBG-based humidity sensor

III. Results And Discussion

In the experiment, the peak reflectivity at 1547.16 nm corresponds to an unetched part of the FBG. After the first etching, the peak reflectivity shifted to 1547.18 nm, and further shifted to 1547.24 nm after the second etching, as depicted in Figure 8. The resolution of the OSA is set to be 0.05 nm for all the measurements of the relative humidity level.

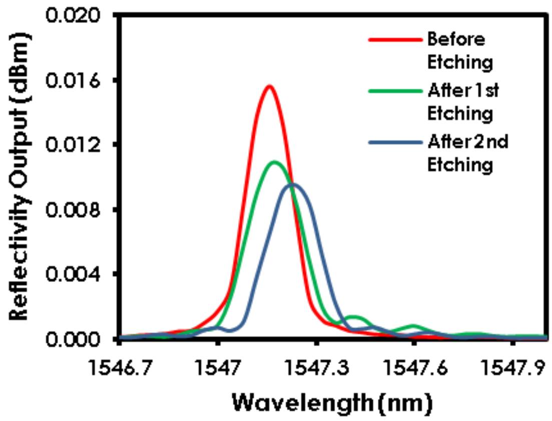

Figure 8: Spectra of FBG before etching (red), after 1st etching (green) and after 2nd etching (blue)

The tapered uncoated FBG is first tested to sense different relative humidity levels. The measured reflection spectrum of the uncoated tapered FBG with relative humidity levels varying from 55% to 80% in steps of 5% is presented in Figure 9. Table 1 lists the wavelength shift of the increasing relative humidity level acquired from the spectra at a reference point of 0.022 dBm. The spectrum wavelength at 1546.9684 nm has shifted a total amount of 35.3 pm to the left corresponding to the humidity level of 55% to 80%.

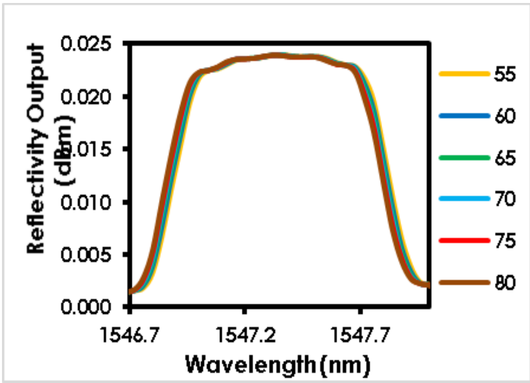

Figure 9: Spectra of the uncoated FBG with relative humidity levels varying from 55% to 80%

Table 1
The Wavelength Shift of the Uncoated FBG for 55% to 80% Relative Humidity Levels

Relative Humidity (%)	Wavelength (nm)	Wavelength Shift (pm)
55	1546.9684	0
60	1546.9553	13.1
65	1546.9475	20.9
70	1546.9413	27.1
75	1546.9368	31.6
80	1546.9331	35.3

Figure 10 shows the reflection spectrum of the FBG after being coated with ZnO nanostructures. Here, it can be observed that the total amount of wavelength shift which corresponded to the increase in the humidity level has increased, as quantitatively tabulated in Table 2. The wavelength of 1545.6958 NM experienced a 61.5 pm shift within the range of 55% to 80%.

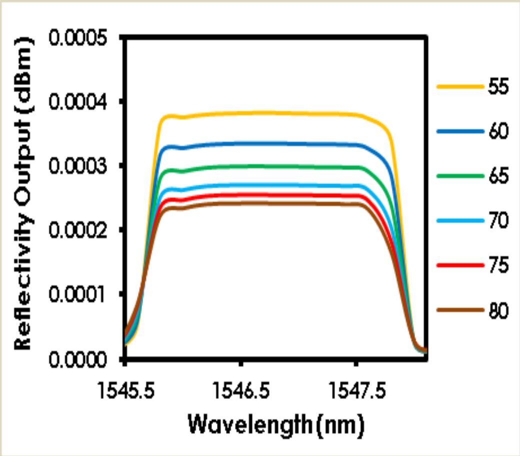

Figure 10: Spectra of FBG coated with ZnO nanostructures with relative humidity levels varying from 55% to 80%

Table 2
The wavelength shift of ZnO nanostructures coated FBG for the 55% to 80% relative humidity range

Relative Humidity (%)	Wavelength (nm)	Wavelength Shift (pm)
55	1545.6958	0
60	1545.7072	11.4
65	1545.7193	23.5
70	1545.7338	38
75	1545.7462	50.4
80	1545.7573	61.5

A graph of wavelength shift against relative humidity is plotted as in Figure 11 to evaluate the performance of the sensor in regards to the wavelength shifting. The ZnO nanostructures coated FBG experiences a larger wavelength shift and higher slope linearity with a sensitivity of 2.5086 pm/% and linearity of 99.9%, compared to 1.3611 pm/% and 94.6% for its bare counterpart. Overall, the ZnO nanostructures coating managed to increase the sensitivity of the sensor by 85%. Similarly, the resolution of the sensor improved from 3.10% to 1.95% with the ZnO nanostructures coating (Table 3). This is mainly due to the dependence of Bragg wavelength shifting on the applied strain and temperature change of the FBG. The absorption of water molecules by ZnO nanostructures generate more strain, triggering a larger shifting of the Bragg wavelength. Hence, coating the outer surface of the FBG with humidity sensitive ZnO nanostructures enables the sensor to be more responsive towards humidity.

IV. Conclusion

We report the experimental results of a novel FBG sensor coated with ZnO nanostructures for relative humidity change measurement within the range of 55% to 80%. The sensitivity of the sensor almost doubles in the presence of ZnO nanostructures coating compared to the tapered bare FBG and shows improvement in the value of its resolution, indicating the capability of the ZnO nanostructures coating in enhancing the performance of the sensor. An applicable low-cost and easily fabricated relative humidity sensor based on the FBG has been investigated in this work.

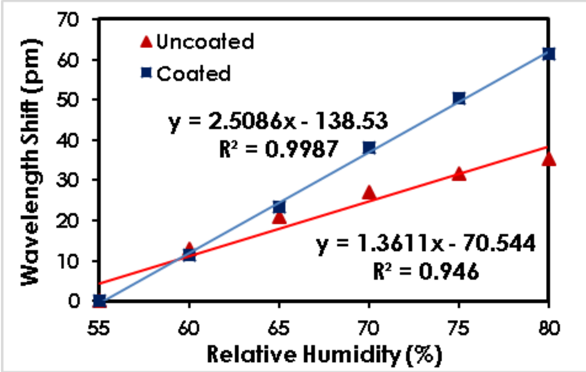

Figure 11: The wavelength shift against relative humidity for uncoated and ZnO nanostructures coated FBG within the range of 55% to 80% relative humidity

Table 3
The performance comparison between the uncoated and ZnO nanostructures coated FBG-based humidity sensor

Parameter	Uncoated	Coated
Sensitivity (pm/%)	1.36	2.51
Linear Range (%)	55 - 80	55 - 80
Linearity (%)	More than 94%	More than 99%
Standard Deviation (pm)	4.22	4.88
Resolution (%)	3.10	1.95

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