Enhancement the sensitivity of humidity sensor based on an agarose infiltration reflection-type photonic crystal fiber interferometer

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Abstract
Photonic Crystal Fiber Interferometers (PCFIs) are widely used for sensing applications. This work presents the fabrication and study the characterization of a relative humidity sensor based on a polymer-infiltrated photonic crystal fiber that operates in a Mach-Zehnder Interferometer (MZI) reflection mode. The fabrication of the sensor only involves splicing and cleaving Photonic Crystal Fiber (PCF) with Single Mode Fiber (SMF). A stub of (LMA-10) PCF spliced to SMF (Corning-28). In the splice regions. The PCFI sensor operation based on the adsorption and desorption of water vapour at the silica-air interface within the PCF. The sensor shows a high sensitivity to RH variations from (27% RH - 95% RH), with a change in its reflected power and the position of the interference peaks is found to be shifted that the interference pattern with a 100 nm span can be observed with high humidity sensitivity of (8.49 pm /%RH) is achieved with compact (4mm) PCF length . The sensor has the advantages for suitable for monitoring humidity in microenvironments. The repeatability, long-term stability, measurement accuracy. Wide humidity range. The response time of the sensor is found to be 1.4 sec for a change in RH of 50 %RH. The fast response time suggests that the sensor can potentially be used as a human breath rate monitor in a clinical situation.

Key words
Agarose, humidity measurement, Mach-Zehnder Interferometer (MZI) reflection mode.

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Introduction
The Photonic Crystal Fiber (PCF) surface (silica) is hydrophilic, so when it is exposed to humid air, the water vapor adsorbed by the PCF surface, and the amount of water vapor adsorbed PCF surface at room temperature is a function of relative humidity. The ratio of the percentage of water vapor present in air at a particular temperature and pressure to the maximum amount of water vapor the air can hold at that temperature and pressure is the relative humidity (RH), the presence of air holes in PCF provides a possibility of light propagation in air, or instead of that gives the ability to inject gases/liquids into the air holes. This provides a good controlled interaction between light and matter leading to novel sensing applications that cannot get it with conventional optical fibers [1, 2]. Measurement of RH is required in a range of areas, including meteorological services, high energy physics applications, chemical and food processing industry, civil engineering, air conditioning, horticulture, and electronic processing. Given the wide range of industrial humidity sensors available [3], optical fiber humidity sensors, by comparison to their conventional electronic counterparts, offer specific advantages such as small size and low weight, immunity to electromagnetic interference, corrosion resistance, and the potential for remote operation. A wide range of RH sensing techniques based on optical fibers has been reported to date including fiber bends [4], long period gratings [5, 6], fiber Bragg gratings [7, 8], fiber Fabry–Perot cavities [9], side-polished fibers [10], cladding-removed fibers [11], plastic optical fibers [12], surface plasmon resonances [13], heterocore fibers [14], and tapered fibers [15]. Recently, it was proposed to use a photonic crystal fiber interferometer (PCFI) operating in reflection mode for RH sensing with the benefits of simplicity, low cost, and an end-type probe. The sensor head fabrication was also simple since it involved only cleaving and splicing. The sensor showed a shift in its interference pattern due to the adsorption and desorption of water vapor with respect to ambient humidity. But the length of the sensor was relatively long at 4.5cm and its humidity measurement resolution was small. In this paper, we demonstrate a new PCFI based RH sensor with a focus on achieving an improved sensitivity, a very significantly reduced length, and a wider humidity range. The sensitivity of the sensor is improved by infiltrating the microholes of the PCF with a hygroscopic material. There are a number of possible hygroscopic materials. Agarose offers a wide operating humidity range with a simple solution preparation procedure and a consistent product with few impurities and limited ethical concerns, it is also a stable material with respect to temperature, agarose shows a linear change in its RI for a wide RH range [16]. Agarose is a polymer made up of subunits of the sugar galactose. Agarose has an added advantage of low material degradation compared with the materials we used earlier in [4] and [14]. Since agarose is soluble in hot water, the solution preparation procedure is easy. Agarose also has a good adhesion to silica and easily forms a thin coating film on silica fiber. All these factors make it a suitable choice as a hygroscopic material for the fiber-optic humidity sensor considered here. The selected hygroscopic polymer agarose shows a linear change of its refractive index (RI) over a wide humidity range [17]; hence, a wide measurement range for the sensor is also achieved. A detailed
study of the sensor performance in terms of its sensitivity, repeatability, long-term stability, and measurement accuracy is reported. We also study the response time of the sensor in this paper and it is found to be less than two second, which is better or comparable to the existing electronic or fiber-optic RH sensors.

Operating principle of the sensor
The excitation and recombination of modes can be carried out by the hole collapsed region of the PCF. A microscopic image of the PCFI and a schematic of the excitation and recombination of modes in the PCFI are shown in the inset of Fig. 1. The fundamental single mode fiber (SMF) mode begins to diffract when it enters the collapsed section of the PCF. Because of diffraction, the mode broadens; depending on the modal characteristics of the PCF and the nature of the hole collapsed region, the power in the input beam can be coupled to the fundamental core mode and to higher order core modes [16-18], or to cladding modes of the PCF[19]. The modes propagate through the PCF until they reach the cleaved end from where they are reflected. Since the modes propagate at different phase velocities, in a certain length of PCF the modes accumulate a differential phase shift. The phase velocities and phase difference are wavelength dependent; therefore, the optical power reflected by the device will be maximum at certain wavelengths and minimum at others [20]. When the reflected modes reenter the collapsed region, they will further diffract, and because the mode field of the SMF is smaller, the core acts as a spatial filter and picks up only a part of the resultant intensity distribution of the interference pattern in the PCF. A regular interference pattern in the reflection spectrum of PCFI suggests that only two modes are interfering in the device. In order to provide a good RH sensitivity to our device, we have infiltrated a small region of the PCFI with agarose. The effective RI of the cladding mode depends on the RI of the agarose material infiltrated into the microholes of the PCF. The RI of the agarose increases with an increase in the ambient RH [15], which in turn changes the modal propagation constant of the cladding mode. As a result, a phase change is induced between the interfering core and cladding modes, which in turn causes the shift of the interference pattern. The RH level can be measured by monitoring the humidity-induced changes in the interference fringes in the reflection spectrum of the PCFI.

![Fig. 1: Schematic of the humidity sensor system.](image-url)
Experimental

The PCF (LMA-10) designed for an endless single-mode operation is used; it has six layers of air holes arranged in a hexagonal pattern around a solid silica core, the fiber has core diameter of 10 μm, voids with diameter 2.5 μm, pitch of 8.07 μm and the outer diameter of 125 μm. These dimensions of the PCF alignment and splicing with SMF with splicing machine and due to mode-field diameter (MFD) mismatch compared to other PCFs, the loss was minimize. During the splicing process and due to surface tension, the voids of the PCF collapse within a microscopic region (~ 250 μm) near the splice point, as shown in the Fig. 1.

Humidity sensor based on reflection type of the PCFI has been proposed. First, the coating of a stub of PCF (LM A-10) and conventional optical fiber (Corning, SMF-28) are removed by using a mechanical stripping. Then, the second step is cleaving the PCF and SMF, which is done by fiber cleaver, and third step is cleaning the fibers. Then, the stub of PCF (LM A-10) is spliced to conventional optical fiber by a conventional splicing machine. After fusion splicing, the PCF is cleaved using a standard fiber cleaving machine so that the end of the PCF behaves as a reflecting surface, then the microholes of the PCF are infiltrated with agarose by immersing the tip of the PCF in a hot agarose solution. The solution is prepared by dissolving 1wt% agarose in distilled water. To dissolve the agarose in distilled water, the beaker containing the mixture is placed on a heater combined with a magnetic stirrer. The temperature of the heater is set to 65 °C and at the same time the mixture of agarose and water inside the beaker is stirred until the agarose is completely dissolved. Then keeping the solution at the same temperature, the open end of the PCF is immersed in this solution for about 30 sec. The outer portion of the fiber is wiped using a dry lens cleaning tissue immediately after the fiber is pulled out from the solution. When the infiltrated mixture is cooled, the gel polymerizes, and once the gelling point is reached (<30 °C) the mixture assumes its hydro gel form and will not take a liquid form again unless it is heated and reaches its melting point (>60 °C). The initial infiltration length depends on capillary forces, the length of the PCFI, and the temperature. At a constant temperature, it is the balance between the capillary forces and the forces exerted by the pressure of the air inside the silica holes. In our case, the air inside the microholes compresses as a result of the sudden cooling from 65 °C to room temperature of 23 °C after the fiber is taken out of the solution, and this results in an increase in the infiltration length inside the microholes of the PCFI. Fig. 1 shows the schematic of the humidity sensor based on PCFI, light source (1550 nm) is launched into the interferometer through the Fiber Optic Circulator (FOC), and light that reflected from the cleaved end is fed to power meter or the Optical Spectrum Analyzer (OSA). The fabricated sensor response to humidity variations is studied at room temperature and normal atmospheric pressure by putting it in an environmental chamber, which is a cuboid-shape sealed chamber, fabricated from Polyvinyl chloride (PVC) plastic. It consists of dry/wet air flow system that can vary the internal humidity in the chamber (27 %RH – 95 % RH), there are three fans (the first fan is pumps a dry air form container containing a silica gel, the second fan pumps a wet air from container containing distilled water and heater (70 watt), and the last fan is in the surface of chamber to expel the air). A calibrated electronic humidity
(X T9007-8 temperature & humidity control instrument) is used for monitoring the humidity and temperature inside the chamber.

**Results and discussion**

It has been studied an uninfiltrated PCFI that be observed PCF with length (4.5 cm) show the higher sensitivity (5.89 pm/RH%) [21], the sensitivity calculated from the linear fitting versus wavelength curve to RH (27 %-85 %).

in this paper demonstrated that there is no wavelength shift and no decreasing in reflection power when increase the RH >90 %. This is because water has a hydrogen-bonded network (ice-like), which grows up as the relative humidity increases from 0% to 30 %. The liquid water structure starts appearing in the RH range of 30-60 %, while the structure of ice-like continues growing to saturation [22] as shown in Figs. 2 and 3.

![Fig. 2: Change in the reflection spectrum of a PCFI with length 4.5 cm with respect to different ambient relative humidity values.](image)

![Fig. 3: Change in the reflection power of a PCFI with length 4.5 cm with respect to different ambient relative humidity values.](image)

The agarose-infiltrated in order to improve the humidity sensitivity of PCFI. An increase in the RH level increases the RI of the agarose and this change in RI changes the propagation constant of the cladding mode. The
resulting phase change between the interfering modes in turn results in a shift of the interference pattern as a function of ambient RH. When humidity increases, the interference pattern shifts to higher wavelengths. Therefore, the reflected power decreases for the sensor in the observed wavelength region, experiments were carried out using infiltrated PCFIs with different PCF lengths (4.5 cm, 3.5 cm, 2.5 cm, 1.5 cm, 4 mm), Fig. 4 and 5 show the relation between PCF lengths with reflection power and wavelength shift respectively.

Furthermore it is observed that the interference spectrum shifted to higher wavelengths with an increase in ambient RH at optimum length 4 mm with sensitivity 8.49 pm/RH% as shown in Fig. 6.

A microscope image of the fabricated sensor is shown in the inset (upper) of Fig. 1. The size of the sensing element is 4mm in length and 125 μm in diameter; the length includes both the 250 μm hole collapsed region and approximately 220 μm agarose infiltrated region. As seen from Fig. 7, when the length of the PCF is less than 5 mm, the period will be greater than 100 nm. Therefore, for a small sensor length, only a narrow subperiodic part of the interference pattern with a 100 nm span can be observed, when each line represents humidity value. In our experiment, we have selected a compact length of 4 mm for the sensor head. An additional advantage of choosing a compact length is that if the length of the PCF section of the interferometer is large the spectra observed may be unduly perturbed by vibrations and airflow currents, this is because bending or lateral strain of the
PCFI causes a shift in the interference pattern that is not related to humidity and this phenomenon becomes more significant as the PCFI length increases[4].

Fig. 7: OSA Graphics peaks shift of PCFI with respect humidity of the length 4 mm.

For a sensor head with a short length of 4 mm, we have observed that the spectrum is very stable when subjected to vibrations and air flow and concluded that an uninfiltreated PCFI with a compact length has only a weak sensitivity to RH changes. Fig. 8 and Table 1 show the relation between PCF lengths with sensitivity of device.

Table 1: The sensitivity of the sensor with different PCF lengths.

| PCF length (cm) | Sensitivity pm/ %RH |
|-----------------|---------------------|
| 0.4             | 8.49                |
| 1.5             | 7.76                |
| 2.5             | 6.60                |
| 3.5             | 5.88                |
| 4.5             | 5.48                |

Fig. 8: The relationship between PCF lengths and sensitivity of the sensor.
To calculate the rise time of the sensor, PCFI (with specific length 4 mm which is the length that shows the higher sensitivity to relative humidity variations) is exposed to an environment with rapid changes of the RH. First, keep the RH in the chamber a 40 % RH, and then rapidly increase the humidity of the chamber to 90 %. (at room temperature and normal atmospheric pressure). The measured rise time of the sensor is shown in Fig.9, the sensor has a fast response to humidity variations and the estimated response time from 10 % to 90% of the signal maximum) is about 1.4 sec when the RH changes from 50 % to 90 % at wavelength =1550nm.

Fig. 9: Response time of the sensor.

Conclusions
We have demonstrated a novel RH sensor based on a polymer-infiltrated PCFI Compared to the existing optical-fiber-based RH sensors, it proposed in this paper has the advantages of a very compact length, high resolution, low cost, wide humidity range and ease of fabrication. The end-type sensor head configuration offers advantages in terms of operating in environments, which demand a compact probe-type sensor and also reduced system complexity as only one interconnecting fiber is needed. Since the size of the actual sensor head is in millimeters, the reported sensor is suitable for sensing humidity in microenvironments, these advantages suggest a practical motivation to investigate disposable polymer-infiltrated PCFI for RH control. The sensor shows a high sensitivity of 8.49 pm/RH% in the range 27–95 % RH, The sensor response is repeatable and shows good long term stability, the observed fast response time of 1.4 sec suggests that the sensor can potentially be used as a human breath rate monitor in a clinical situation. In our study, we have used the polymer agarose, use of other hygroscopic materials may result in potentially improved sensor performance and opens a field for further research with this type of sensor head.

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