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Agarose coated micro-bottle sensor for relative humidity detection

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The whispering gallery mode (WGM) based micro-bottle resonator (MBR) sensor has been proposed and demonstrated for relative humidity measurement by using an agarose gel as a transducer. MBR was fabricated by using a soften-and-compress method to form a 190 μm bulge bottle. The micro-bottle was optically excited by a 3 μm tapered fiber and it exhibits a resonance with a Q factor in an order of 10^5. The agarose coated MBR produces a good sensing response towards humidity with the sensitivity of 0.107 dB/%RH and linearity of 99.614%. The agarose hydrophilic nature and its changing porosity and refractive index with increasing relative humidity made the coated MBR structure to be more sensitive than the uncoated structure. This humidity sensor has a simple fabrication and is showing good sensitivity, linearity, resolution, response time and operational in a wide humidity range.

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Accurate, real-time measurements of relative humidity are vital to many industrial applications. For instance, process, quality, and safety controls rely on accurate relative humidity measurements in pharmaceutical factories and semiconductors manufacturing[1,2]. Traditional relative humidity sensors have limitations such as susceptibility to electromagnetic interference, and limited operation in hazardous environments[3-5]. Fiber optics provides a valuable choice for overcoming these challenges. Fiber optics immunity to electromagnetic interference, ability to operate in hazardous environments, and lower cost make them an attractive option for designing practical relative humidity sensors[2-6].

Likewise, optical micro-resonators (OMRs) are captivating more attention in recent years due to their wide range of applications. Recently, OMR based optical sensors were reported for humidity, temperature, and formaldehyde concentration measurements[7-10]. OMRs also show great promise as building blocks for add-drop filters, laser, and other optics devices[11,12]. OMRs provides large evanescent fields and strong temporal and spatial light confinement and thus suitable for various sensing applications. The optical confinement increases the quality factor of the resonator and thus enhances the performance and lifetime of the devices[13,14]. Up to date, many types of OMRs have been proposed such as micro-bottle, loop, knot, and micro-ball resonators. These OMRs show great promise as a modern optical sensor device due to their simple fabrication, high quality factor, and lower intrinsic losses[14-19].

Micro-bottle resonator (MBR) is a type of OMRs fabricated by altering the shape of a regular optical fiber cable to have a bottle-like bump in the middle. The electromagnetic field existing around the MBR makes it a viable choice for sensing applications[16,19,20]. For instance, MBR was used to sense humidity with shown sensitivity of 0.048 7 dB/%RH, in the range of 40%—80%RH[7]. Additionally, MBRs were used to sense temperature in the range of 40—100 °C and showed sensitivity of 0.014 9 dB/°C[10]. MBR was also used to sense liquid formaldehyde with sensitivity of 4.397 dB/% for concentrations of 1%—5%[21].

Coating the optical sensing element with materials that respond to changes in ambient relative humidity has been shown to improve sensitivity[22-24]. Agarose was chosen as the coating for this sensor for several reasons: the refractive index changes with variations in relative humidity, simple fabrication, easier application process,

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affordability, and lower toxicity[25-27]. Previously, agarose has been used to coat a polished plastic fiber for humidity sensing[27]. In this paper, a relative humidity sensor was proposed and demonstrated using an agarose-coated micro-bottle resonator. It employed a tapered optical fiber to couple light into or out of MBR. The response of this sensor with the change in relative humidity was investigated in a temperature-controlled environment.

Using a standard SMF-28 and applying the soften-and-compress method we fabricated the MBR element[20]. An intact length of fiber is placed in a manual splicing machine (Furukawa Electric Fitel S178A). Then, this length was heated by electric arcs and pushed forward from each end to compress and form a bulge. The size of the MBR bulge is decided by the number of the arcs applied. The MBR characteristic may be defined by various parameters such as the bottle diameter (D_b), the stem diameter (D_s), and the neck-to-neck length (L_b)[7,28]. Our sensor probe dimensions are: D_b=190 μm, D_s=125 μm and L_b=180 μm. Next, to optically guide light through the sensor we used a tapered microfiber with a waist diameter of 3 μm. The SMF-28 fiber was subjected to flame brushing to obtain this desired diameter[29]. Fig.1(a) shows the magnified image of MBR structure with a dimension of D_b=190 μm, D_s=125 μm and L_b=180 μm. Fig.1(b) shows the image of the prepared microfiber with a waist diameter of 3 μm. The microfiber was attached to the MBR structure to assemble the sensing probe as shown in Fig.1(c).

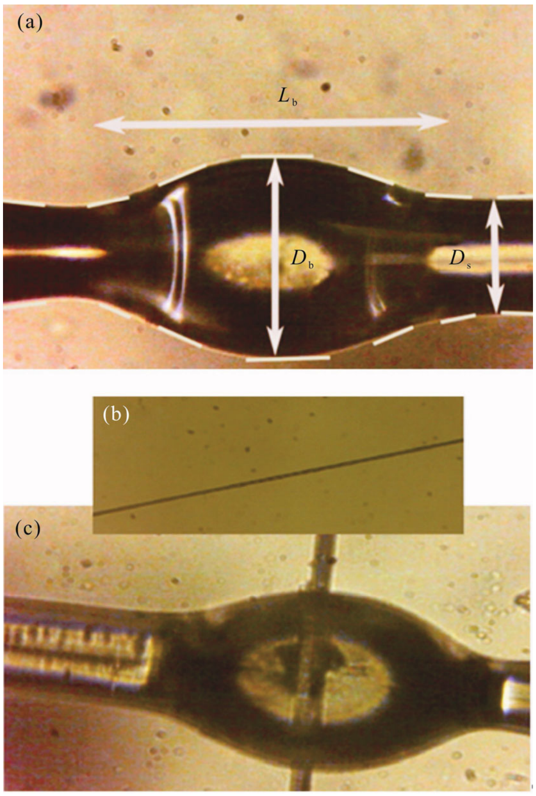
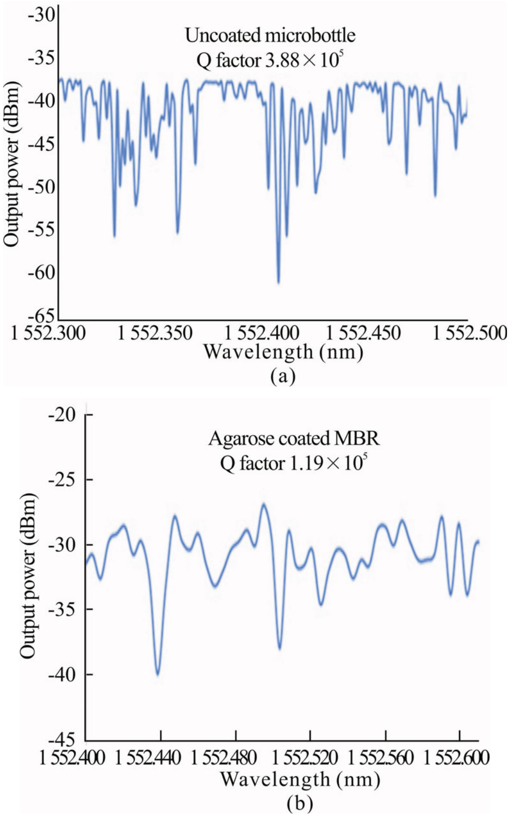


Fig.1 The magnified images of (a) MBR structure, (b) microfiber, and (c) assembled sensor probe

Field Agarose powder from Sigma Aldrich (No.A6013) was dissolved in distilled water to make a 0.5% solution. The MBR was coated by this solution by dipping the element in a Petri dish filled with the solution. Then the MBR was secured on the tapered fiber and left 24 h to fully dry[30]. The tapered region allows the evanescent mode to reach the coated MBR and excite it. To characterize the MBR, a tunable laser source (TLS) was used. The TLS (ANDO AQ4321D) was set to provide light in the range of 1 550—1 560 nm in increments of 0.001 nm. The TLS ability to provide this fine resolution and wide range of tunable light was the reason why the TLS was chosen over a traditional ASE. The light output power was detected using an optical power meter (THORLAB PM100D) and recorded in a computer[8]. The transmission spectral characteristics of the MBR without and with agarose coating are shown in Figs.2(a) and (b), respectively. The Q-factor is calculated by dividing the resonant peak wavelength by the difference in wavelength before and after the resonance peak[17,31]. The Q-factor is obtained at 3.88×10^5 and 1.19×10^5 for the MBR without and with agarose coating. The Q-factor measurements were conducted in relative humidity of 35% RH. The Q-factor reduces with the coating due to the reduction of refractive index contrast at the bottle region, which in turn increases the leaky modes. Insertion losses were incurred due to non-adiabatic tapering of the coupling microfiber.



(a)

(b)

Fig.2 WGM transmission modes for MBR (a) without and (b) with agarose coating

To study the MBR response to changes in relative humidity the setup shown in Fig.3 was used. Both the MBR and the microfiber were placed in a locked chamber. Inside the sealed chamber, relative humidity was continuously measured by a hygrometer (RS 1365). Both the temperature and atmospheric pressure were maintained at 25 °C and 1.0 atm[15,17]. The power output changes with variations in relative humidity were recorded by the optical power meter for both MBR sensor probes without and with agarose coating. Light from the TLS is passed through one end of the fiber. At the other end, light is received by the OPM connected to a computer that continuously records the light output power. Using saturated salts put in the sealed chamber; we varied the relative humidity inside the locked chamber and continuously monitored the changes in the light output power. The TLS wavelength was set at the peak resonant wavelength for the MBR[32].

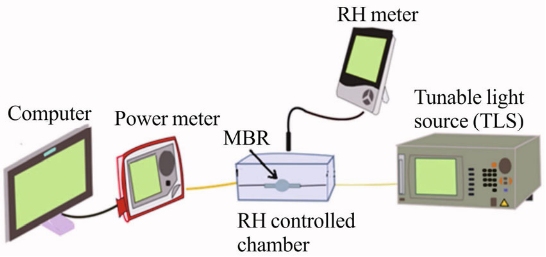


Fig.3 Experimental setup for the proposed sensor for measurement of relative humidity using MBR with and without agarose gel

The change of transmitted power with the variation of relative humidity is investigated for both the uncoated MBR and the agarose coated MBR at constant temperature at 25 °C and the results are summarized in Fig.4. As shown in the figure, the sensor performance is significantly improved with the use of agarose coating on the sensing element of the MBR. Agarose is a hydrophilic polymer material, which has a net structure containing many pores with an average diameter of around 100 nm. When the MBR coated with the agarose gel is exposed to humidity, water molecules enter the pores due to the capillary forces. The air in the pores is replaced by the water molecules, which has a higher refractive index. This increases the average refractive index of the coating, reduces the index contrast between the micro-bottle and surrounding, and causes the resonance wavelength to shift in higher magnitude and thus improves the sensing performance.

The sensitivity of the proposed humidity sensor improved with the agarose gel coating, which functions as a transducer to increase the variation of the resonant leaky modes in the MBR structure. This improvement is a result of the agarose gel hydrophilic nature, which allows the refractive index of agarose to change with the relative humidity[2,33]. It was previously reported that the refractive index of agarose gel increases from 1.52 to 1.54 with a change of relative humidity from 20% to 80%. The high porosity of agarose allows absorbing moisture and changing the light propagation characteristics in the WGM resonator. The agarose swelling caused by its hydrophilic nature alters the refractive index of the coating with changing relative humidity and modulates the light passing in the sensor. The change in refractive index causes more losses in the sensing element, thus the power output decreases with increasing relative humidity as shown in Fig.4[26,33,34]. Fig.5 shows how increasing relative humidity affects the WGM of the sensor. The water particles absorbed into the agarose gel coating and the resulting increase in the refractive index causes changes in the resonant dips.

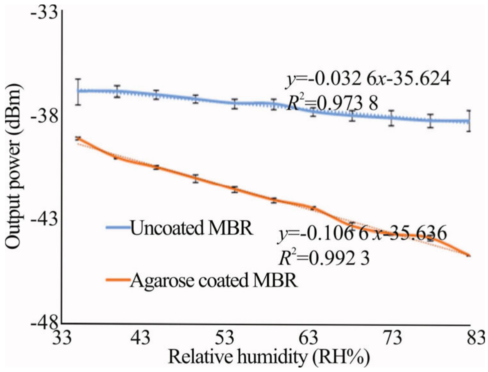


Fig.4 Output power against RH for bare MBR and agarose coated MBR

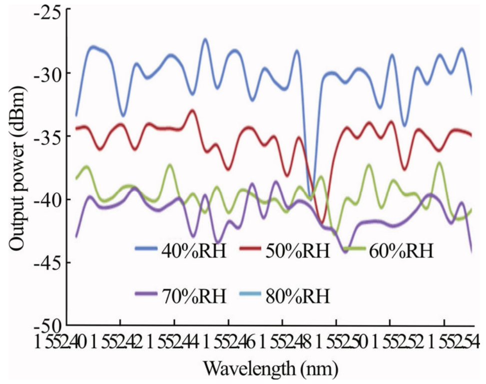


Fig.5 WGM transmission modes for the agarose coated MBR at different levels of relative humidity

The response time and the recovery time of both uncoated MBR and agarose coated sensors are also investigated as shown in Figs.6(a) and (b), respectively. The test was conducted by exposing the agarose coated MBR probe in a chamber where the humidity can be varied from 35%RH to 85%RH. The temperature and atmospheric pressure were fixed at 25 °C and 1.0 atm during the experiment. The response time was calculated when the humidity was changed from 35%RH to 85%RH while the recovery time was measured when the humidity is reduced from the maximum 85%RH to 35%RH. It was observed that the response time is in range of

7.4—14.8 s and the recovery time is in a range of 8.0—10.9 s for our agarose coated sensor. Meanwhile, the uncoated MBR had a response time ranging between 61—74 s and a recovery time in the range of 18—27 s.

The stability of the agarose coated sensor was studied by recording the output power of the sensor continuously for a few hours. The chamber was stabilized at 65%RH, 25 °C , and 1.0 atm, Fig.7(a) shows the variations of the sensor output as compared with the recorded relative humidity using the chamber's relative humidity sensor with accuracy of ±2%RH, the variations in output power with time at 65%RH were <0.1 dBm. The variations of the sensors output for the rest of the relative humidity levels were calculated to have the standard deviation of 0.093 dBm for the agarose coated sensor as shown in Tab.1. The resolution of the agarose coated sensor was estimated as 0.869%RH.

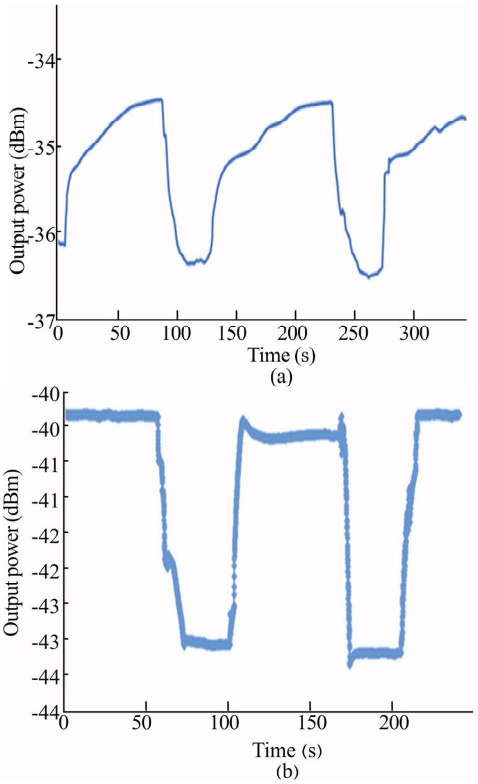
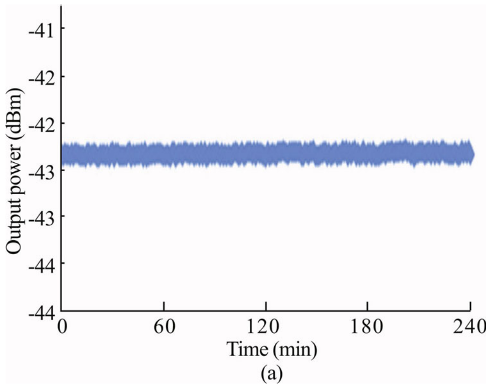


Fig.6 Response and recovery time for (a) the uncoated MBR and (b) the proposed agarose coated MBR sensor



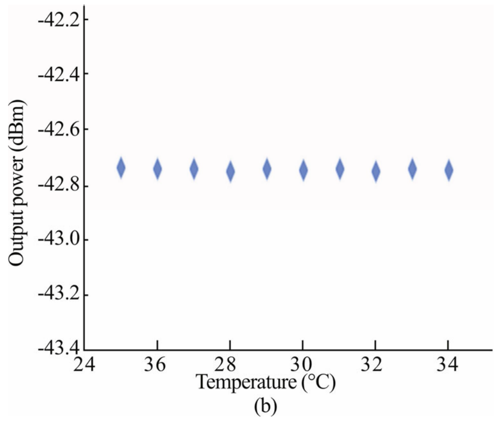


Fig.7 The variation of output power against (a) time and (b) temperature

To study the effect of temperature on the agarose coated structure, the power output of the sensor was continuously recorded in the 25—34 °C range and relative humidity level was fixed at 60%RH as shown in Fig.7(b). The lower limit at 25 °C was decided by room temperature and the cooling limitation of the chamber. The upper limit 34 °C was chosen to make sure that the experiment was conducted outside of the gelling range of the agarose coating 36—42 °C[34]. It is observed that the temperature variation in the range of 25—34 °C had little effect on the output power of the agarose coated sensor.

According to Refs.[34—36], the temperature cross-sensitivity of the silica fiber is rather low due to the mode index changes with temperature at a very close rate while most energy resides inside the silica fiber. Therefore, the proposed sensor is almost insensitive to ambient temperature due to the self-compensated of the thermal optic effect[35]. For instance, Liang et al[36] reported that temperature does not heavily influence the sensitivity of the proposed sensor. There is only a tiny attenuation of the WGMs when the different temperature was applied. The linearity error due to temperature was approximately only 3.5%[34,36].

Tab.1 summarizes the sensing performances of both coated and uncoated MBR samples. The agarose coated MBR produces a good sensing response towards humidity with the sensitivity of 0.107 dB/%RH and linearity of 99.614%. This performance is much better than that of the uncoated MBR, which exhibits the sensitivity of only 0.033 dB/%RH and linearity of 98.681%. In addition, the %RH resolution was found to be a factor of 9.5 times better for the coated MBR as opposed to the uncoated MBR. The standard deviation of 0.093 dB was obtained with the coated sensor. This value is much better as compared to the uncoated sensor with 0.303 dB. These results indicate that agarose hydrophilic nature and its changing porosity and refractive index with increasing relative humidity made the coated MBR structure to be more sensitive and performing the sensing with better linearity and resolution.

Tab.1 Sensing parameters of the sensors

Parameters	Uncoated MBR	Coated MBR
Average standard deviation (dB)	0.303	0.093
Resolution (%RH)	9.181	0.869
Sensitivity (dB/%RH)	0.033	0.107
Linearity (%)	98.681	99.614

In conclusion, an MBR based humidity sensor has been fabricated and tested. It used agarose gel as a transducer for humidity as it is easy to handle and inexpensive. The soften-and-compress method was used to fabricate the MBR. A tunable laser source was launched into the agarose coated MBR structure via 3 μm tapered fiber to excite the WGM. Both agarose-coated and uncoated MBR had Q-factors in the order of 10^5. The performance of the coated and uncoated MBR as humidity sensor was investigated by measuring the power output change with varying relative humidity. The agarose coated MBR showed a higher sensitivity of 0.107 dB/RH% and better stability and resolution compared to the uncoated MBR due to the hydrophilic nature of the agarose porous coating.

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