Northumbria Research Link

University**Library**

Agarose coated spherical micro resonator for humidity measurements

ARUN KUMAR MALLIK[1,*] **DEJUN LIU,**[1] **VISHNU KAVUNGAL,**[1] **QIANG WU,**[2] **GERALD FARRELL,**[1] **AND YULIYA SEMENOVA**[1]

[1]*Photonics Research Center, Dublin Institute of Technology, Kevin Street, Dublin 8, Ireland*
[2]*Department of Physics and Electrical Engineering, Northumbria University, Newcastle Upon Tyne, NE1 8ST, UK*
[*]*d13125763@mydit.ie*

Abstract: A new type of fiber optic relative humidity (RH) sensor based on an agarose coated silica microsphere resonator is proposed and experimentally demonstrated. Whispering gallery modes (WGMs) in the micro resonator are excited by evanescent coupling using a tapered fiber with ~3.3 μm waist diameter. A change in the relative humidity of the surrounding the resonator air induces changes in the refractive index (RI) and thickness of the Agarose coating layer. These changes in turn lead to a spectral shift of the WGM resonances, which can be related to the RH value after a suitable calibration. Studies of the repeatability, long-term stability, measurement accuracy and temperature dependence of the proposed sensor are carried out. The RH sensitivity of the proposed sensor depends on the concentration of the agarose gel which determines the initial thickness of the deposited coating layer. Studies of the micro- resonators with coating layers fabricated from gels with three different Agarose concentrations of 0.5%, 1.125% and 2.25 wt./vol.% showed that an increase in the initial thickness of the coating material results in an increase in sensitivity but also leads to a decrease of quality factor (Q) of the micro resonator. The highest sensitivity achieved in our experiments was 518 pm/%RH in the RH range from 30% to 70%. The proposed sensor offers the advantages of a very compact form factor, low hysteresis, good repeatability, and low cross sensitivity to temperature.

References and links

1. F. Vollmer, D. Braun, A. Libchaber, M. Khoshsima, I. Teraoka, and S. Arnold, "Protein detection by optical shift of a resonant micro cavity," Appl. Phys. Lett. **80**(21), 4057–4059 (2002).
2. S. Arnold, M. Khoshsima, I. Teraoka, S. Holler, and F. Vollmer, "Shift of whispering-gallery modes in microspheres by protein adsorption," Opt. Lett. **28**(4), 272–274 (2003).
3. W. Fang, D. B. Buchholz, R. C. Bailey, J. T. Hupp, R. P. H. Chang, and H. Cao, "Detection of chemical species using ultraviolet microdisk lasers," Appl. Phys. Lett. **85**(17), 3666–3668 (2004).
4. T. Ling and L. J. Guo, "A unique resonance mode observed in a prism-coupled micro-tube resonator sensor with superior index sensitivity," Opt. Express **15**(25), 17424–17432 (2007).
5. E. Krioukov, D. J. W. Klunder, A. Driessen, J. Greve, and C. Otto, "Sensor based on an integrated optical microcavity," Opt. Lett. **27**(7), 512–514 (2002).
6. V. S. Ilchenko, P. S. Volikov, V. L. Velichansky, F. Treussart, V. Lefèvre-Seguin, J.-M. Raimond, and S. Haroche, "Strain tunable High-Q optical microsphere resonator," Opt. Commun. **145**(1-6), 86–90 (1998).
7. N. Lin, L. Jiang, S. Wang, Q. Chen, H. Xiao, Y. Lu, and H. Tsai, "Simulation and optimization of polymer-coated microsphere resonators in chemical vapor sensing," Appl. Opt. **50**(28), 5465–5472 (2011).
8. P. R. Story, D. W. Galipeau, and R. D. Mileham, "A study of low-cost sensors for measuring low relative humidity," Sens. Actuator. Biol. Chem. **25**(1–3), 681–685 (1995).
9. A. Raichur and H. Pedersen, "Fiber optic moisture sensor for baking and drying process control," Proc. Food Processing Automation IV, Am. Soc. Agric. Eng. 180–189 (1995).
10. Z. Chen and C. Lu, "Humidity sensors: a review of materials and mechanisms," Sens. Lett. **3**(4), 274–295 (2005).
11. H. E. Posch and O. S. Wolfbeis, "Optical sensors, 13: fibre-optic humidity sensor based on fluorescence quenching," Sens. Actuators **15**(1), 77–83 (1988).

12. F. J. Arregui, I. R. Matias, K. L. Cooper, and R. O. Claus, "Simultaneous measurement of humidity and temperature by combining a reflective intensity-based optical fiber sensor and a fiber Bragg grating," IEEE Sens. J. **2**(5), 482–487 (2002).
13. J. M. Corres, I. R. Matias, M. Hernaez, J. Bravo, and F. J. Arregui, "Optical Fiber Humidity Sensors Using Nano structured Coatings of SiO$_2$ Nano particles," IEEE Sens. J. **8**(3), 281–285 (2008).
14. M. Hernáez, C. R. Zamarreño, I. R. Matías, and F. J. Arregui, "Optical fiber humidity sensor based on surface plasmon resonance in the infra-red region," JPCS **178**, 012019 (2009).
15. M. Yinping, L. Bo, Z. Hao, L. Yuan, Z. Haibin, S. Hua, Z. Weihua, and Z. Qida, "Relative humidity sensor based on tilted fiber Bragg grating with polyvinyl alcohol coating," IEEE Photonics Technol. Lett. **21**(7), 441–443 (2009).
16. A. Gastón, F. Pérez, and J. Sevilla, "Optical fiber relative-humidity sensor with polyvinyl alcohol film," Appl. Opt. **43**(21), 4127–4132 (2004).
17. S. Acikgoz, B. Bilen, M. M. Demir, Y. Z. Menceloglu, Y. Skarlatos, G. Aktas, and M. N. Inci, "Use of polyethylene glycol coatings for optical fiber humidity sensing," Opt. Rev. **15**(2), 84–90 (2008).
18. C. Barian, I. Matias, F. Arregui, and M. Lopez-Amo, "Optical fiber humidity sensor based on a tapered fiber coated with agarose gel," Sens. Actuator. Biol. Chem. **69**, 127–131 (2000).
19. K. J. Lee, D. Wawro, P. S. Priambodo, and R. Magnusson, "Agarose-gel based guided-mode resonance humidity sensor," IEEE Sens. J. **7**(3), 409–414 (2007).
20. N. M. Doliba, S. L. Wehrli, A. M. Babsky, N. M. Doliba, and M. D. Osbakken, "Encapsulation and perfusion of mitochondria in agarose beads for functional studies with P-NMR spectroscopy," Magn. Reson. Med. **39**(5), 679–684 (1998).
21. O. Iglesias, A. Garcıa, M. Roques, and J. L. Bueno, "Drying of water gels: determination of the characteristic curve of agar–agar," Dry. Technol. **11**(3), 571–587 (1993).
22. J. Mathew, Y. Semenova, G. Rajan, P. Wang, and G. Farrell, "Improving the sensitivity of a humidity sensor based on fiber bend coated with a hygroscopic coating," Opt. Laser Technol. **43**(7), 1301–1305 (2011).
23. Q. Wu, Y. Semenova, J. Mathew, P. Wang, and G. Farrell, "Humidity sensor based on a single-mode hetero-core fiber structure," Opt. Lett. **36**(10), 1752–1754 (2011).
24. G. Brambilla, V. Finazzi, and D. Richardson, "Ultra-low-loss optical fiber nanotapers," Opt. Express **12**(10), 2258–2263 (2004).
25. T. A. Birks and Y. W. Li, "The shape of fiber tapers," J. Lightwave Technol. **10**(4), 432–438 (1992).
26. H. Kurihara, S. Torigoe, M. Omura, K. Saito, M. Kurihara, and S. Matsubara, "DNA fragmentation induced by a cytoplasmic extract from irradiated cells," Radiat. Res. **150**(3), 269–274 (1998).
27. M. L. Gorodetsky, A. A. Savchenkov, and V. S. Ilchenko, "Ultimate Q of optical microsphere resonators," Opt. Lett. **21**(7), 453–455 (1996).
28. B. E. Little, J.-P. Laine, and H. A. Haus, "Analytic Theory of coupling from tapered fibers and half-blocks into Microsphere Resonators," J. Lightwave Technol. **17**(4), 704–715 (1999).
29. Y. Miao, K. Zhang, Y. Yuam, B. Liu, H. Zhang, Y. Liu, and J. Yao, "Agarose gel-coated LPG based on two sensing mechanisms for relative humidity measurement," Appl. Opt. **52**(1), 90–95 (2013).
30. J. Mathew, Y. Semenova, and G. Farrell, "Experimental demonstration of a high-sensitivity humidity sensor based on an Agarose-coated transmission-type photonic crystal fiber interferometer," Appl. Opt. **52**(16), 3884–3890 (2013).
31. I. M. White and X. Fan, "On the performance quantification of resonant refractive index sensors," Opt. Express **16**(2), 1020–1028 (2008).

1. Introduction

Whispering gallery mode (WGM) based micro resonators have attracted significant attention from researchers for applications in various photonic devices and sensors due to their ultra-high quality factors (Q), low absorption loss and easy and inexpensive fabrication methods. A significant amount of research work has been carried out on various micro resonator shapes, such as microspheres [1,2], microdisks [3] and microtubes [4]. Inside such a circular-shaped resonator the light propagates in the form of whispering gallery modes as a result of total internal reflection. Due to their extreme sensitivity to the size of the resonator and also to the refractive indices of the resonator and the surrounding medium, WGMs can be used for sensing of various parameters such as molecular adsorption [2], refractive index [5] and stress [6]. Polymer coated spherical microresonators have been previously developed for chemical vapor sensing [7]. When an optical microresonator is coated with a thin layer of polymer, WGM frequency shift occurs when the polymer undergoes a change in refractive index or thickness. This change can be induced for example by adsorption from the local environment by quantities such as the ammonia vapor [7].

Measurement of humidity is important in a wide range of applications, including meteorology, agriculture, food industry, clinical medicine, manufacturing, civil engineering

and many other fields [8–10]. Several types of fiber optic humidity sensors have been proposed and demonstrated to date, including a scheme based on fluorescence [11], fiber Bragg gratings [12], interferometry [13] and surface plasmon resonance [14]. Many of these sensing techniques involve using humidity sensitive coatings or gels on the surfaces or end faces of the optical fiber. For example, hydrogel polymers, such as polyvinyl alcohol (PVA) [15,16], polyethylene glycol [17] and Agarose [18], have been previously proposed and studied as coating materials for optical fiber based humidity sensors. Such polymer coatings have the advantage of good reproducibility and long term stability. The coatings usually swell physically and experience a refractive index change in response to changes in relative humidity (RH). For example, Barian et al. [18] demonstrated a tapered fiber coated with Agarose gel for humidity sensing based on the refractive index change of Agarose, which is linear over a wide range of RH values [19].

In this paper we propose a new type of fiber optic humidity sensor based on an Agarose coated silica microsphere resonator. WGMs are excited in the Agarose coated microsphere by coupling evanescent light to and from a tapered fiber. In this case the tapered fiber has a ~3.3 µm waist diameter. Fabrication of the microsphere and the fiber taper is an inexpensive process and involves commercially available fibers. Agarose gel is well known as a hygroscopic polymer commonly used in biological research [20,21], with an advantage of better long-term stability compared with materials used in [22] and [23]. Three sensor samples based on the silica microspheres dip-coated with Agarose gel of different concentrations (0.5, 1.125 and 2.25 wt./vol. %) are experimentally investigated. A detailed study of the sensor in terms of its sensitivity, repeatability, long-term stability and measurement accuracy is reported.

2. Experimental setup and sensor fabrication

2.1 Fiber taper fabrication

Tapered fiber with a waist diameter of ~3.3 µm was prepared from a standard single-mode telecommunications optical fiber (SMF-28 Corning) with core and cladding diameters of 8.3 and 125 µm respectively. For the experiments, a 1.5 m length of this fiber was used and a 3 cm-long section at the center was stripped off its coating by a mechanical stripper. The stripped part was cleaned with isopropyl alcohol and then fixed horizontally between two computer controlled XYZ translational stages. The tapered fiber was then fabricated by means of the customized micro-heater brushing technique described in [24]. A ceramic micro heater (CMH-7019, NTT-AT) was used to heat the fiber up to approximately 1300°C, making the silica material soft enough for tapering. A customized PC program allowed for an accurate control of the diameter, the length and the shape of the fabricated tapers [25]. In our experiment the tapered waist diameter is approximately 3.3 micron, the waist length is around $L_1 = 2$ mm and the full taper length is circa $L_2 = 12$ mm (as shown in Fig. 1). The fabricated fiber taper was then fixed on a glass slide at a height of ~5 mm from the slide surface using two drops of UV curable epoxy (Norrland). The total length of the fiber between the cured epoxy droplets was $L_3 = 4$ cm.
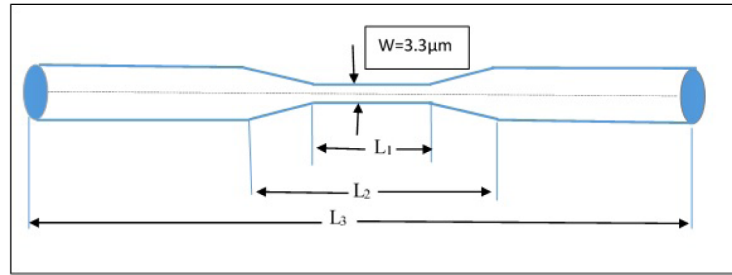
Fig. 1. Schematic diagram of the tapered fiber used for coupling light into the microsphere. The waist diameter W = 3.3 μm, waist length L_1 = 2 mm, full taper length L_2 = 12 mm and the length of the taper between the cured epoxy droplets on the microscope slide L_3 = 4 cm.

2.2 Microsphere fabrication

The microsphere for our experiments was fabricated at the tip of a standard single mode fiber SMF-28. A small length of fiber (~3.8 cm) was stripped from one end, cleaned with isopropyl alcohol and cleaved. The cleaved end of the fiber was placed inside a fusion splicer (Sumitomo Type-36). A series of electric arc discharges was then applied to the cleaved end of the fiber so that the tip of the fiber was gradually melted, assuming a spherical shape as the glass softens due to surface tension. The diameter of the sphere increases with the number of arcs applied. In our experiment we used a silica microsphere with a diameter of 171 μm (Fig. 2). A variety of microsphere diameters were fabricated but it was found that for our purposes the diameter had little effect on the sensing performance.
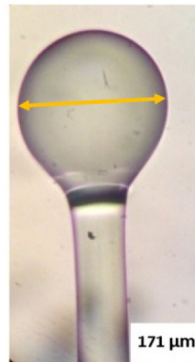

Fig. 2. Optical microscope image of the microsphere with the diameter of 171 μm used in the experiment.

2.3 Agarose gel coating preparation

Agarose is a complex sugar (polysaccharide) derived from the seaweed. Agarose gel is porous in nature and is used widely to separate, identify and purify nucleic acid in the field of bio chemistry [26]. It is usually sold in a form of white powder soluble in water at 34-38°C. In our experiment Agarose powder, obtained from Sigma Aldrich (A6013), was dissolved in water, in proportions of 0.5, 1.125 and 2.25 wt./vol.%. The solutions then were heated up to 65°C. To dissolve the Agarose in distilled water, the beaker containing the mixture was placed on a heater combined with a magnetic stirrer. Microsphere samples were dipped into the hot Agarose solution and pulled out very fast. When the Agarose solution cools down and reaches room temperature, it polymerizes to form hydrogel and will not assume a liquid form again unless it is heated above the melting point. All coated microspheres were left to dry for 48 hours at room temperature.

2.4 Experimental set-up for RH measurements

Figure 3 shows the experimental set-up for the humidity sensor characterization. The system consists of a broadband light source (Thorlabs S5FC1005S), polarization controller, optical spectrum analyzer (OSA, Advantest Q8384) and a temperature controlled humidity chamber (ETS 5503). The fiber taper fixed on a glass slide is placed inside the humidity chamber. The environmental chamber consists of ~0.11 m^3 air tight space with an inlet and an outlet. The upper inlet is made for the entry of the moist air into the chamber and the outlet is used for dragging the moist air to the dehumidifier through a pump. The dehumidifier box contains anhydrous calcium sulfate which absorbs moisture from the air. The compressed dry air is pumped to regulate the RH in the chamber. The humid air was obtained by bubbling dry air into the ultrasonic humidification system (ETS5462) controlling the ratio of humid air and compressed dry air in the chamber to achieve the required value of RH. This data is fed to the humidity controller through the reference sensor head. The chamber controller system allows for independent setup of both temperature and humidity inside the chamber. The accuracy of the chamber is ± 2% RH. Each humidity measurement was recorded five minutes after the RH level reached a certain set value to allow the humidity level to stabilize throughout the chamber.
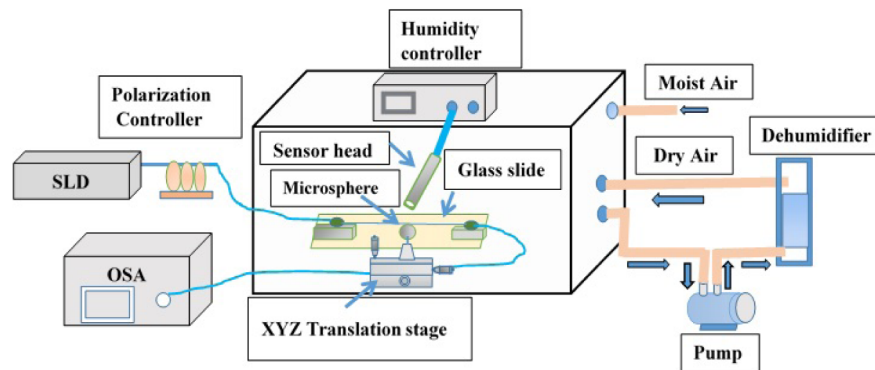

Fig. 3. Experimental set-up used for RH measurement using spherical microresonator.

2.5 Sensor characterization

The coated microsphere was placed in contact with the tapered fiber inside the humidity chamber. Light from the broadband superluminescent light source (SLD) operating in the wavelength range 1530-1570 nm was launched into the fiber taper and the corresponding transmission spectrum was observed at the taper output by means of the OSA. The wavelength resolution of the OSA was 10 pm. The microsphere, mounted on a XYZ nano-positioning stage, was gradually and carefully brought in direct contact with the tapered fiber until the WGM resonances were clearly observed in the transmission spectrum of the fiber taper. The polarization controller was also adjusted manually to achieve maximum light coupling efficiency. The humidity chamber was then closed and left to stand for one hour in order to stabilize the RH and temperature throughout its volume. In order to eliminate the effect of temperature variations, the temperature of the humidity chamber was set to a constant 25 °C (close to room temperature) throughout the entire RH measurement cycle.

3. Results and discussion

3.1 Spectrum characterization

Figure 4 illustrates a typical fiber taper transmission spectrum achieved with the Agarose coated microsphere. As can be seen from the figure, WGM resonances are clearly observed in the spectrum. The value of the free spectral range (F.S.R) determined from the graph (3.141

nm) can be used to estimate the microsphere diameter or its effective refractive index (RI) based on the approximate formula [27]:

$$F.S.R \approx \frac{\lambda_0^2}{\pi D n_{eff}} \qquad (1)$$

where λ_0, n_{eff} and D are the resonant wavelength, the effective refractive index of the fundamental WGM for the microsphere and the microsphere diameter, respectively. Effective RI can be calculated by solving the wave equation described in [28] considering the RIs of silica (1.4682) and the Agarose layer (1.336). This numerical calculation resulted in the $n_{eff} =$ 1.4321 for this case, so that resulting from the Eq. (1) microsphere diameter is 170.83 μm, which is in a good agreement with the result of the microscopic measurement (Fig. 2).

The Q-factors for the microsphere before and after its coating with an Agarose layer were estimated from the experimental spectra by measuring the full width at half maximum (FWHM) of the WGM resonance near 1551 nm (inset in Fig. 4 illustrates the corresponding WGM resonance and its Lorentz fitting in the spectrum of the microsphere before its coating with Agarose). The FWHM for an uncoated microsphere was 14.47 pm at the wavelength of 1557.3493 nm. The corresponding value of the Q- factor was calculated as 1.076×10^5. After application of the Agarose coating on the microsphere surface the Q-factor decreased to 1.56×10^4. Such a decrease in the Q-factor is likely due to scattering caused by the surface roughness and higher absorption and scattering within the Agarose layer.
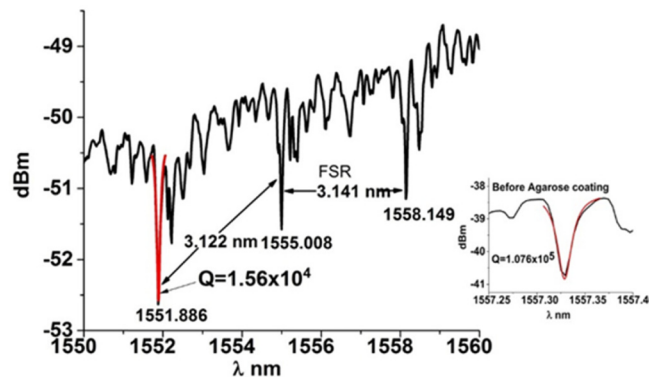

Fig. 4. Transmission spectrum recorded with an Agarose coated microsphere with a diameter of 171 μm.

3.2 RH dependency

We recorded a series of transmission spectra for the sensor at different RH values in the range from 35% to 81% RH. The corresponding results for the microsphere coated with 2.25% concentration Agarose solution are shown in Fig. 5(a). As can be seen from the figure, an increase in the surrounding RH leads to a red shift of the WGM spectrum. This could be explained by the fact that when the surrounding RH increases, the Agarose coating absorbs more water from the environment which leads to an increase of the effective refractive index of the coating and also to an increase of the coating thickness, with both factors contributing to the spectral shift of the WGMs. Figure 5(b) illustrates the shift of a selected WGM resonant wavelength versus RH at a constant temperature of 25°C. Two RH ranges within which the dependencies are almost linear but have different slopes can be identified as follows: lower RH range (38.7-65.7%) with lower sensitivity and a higher RH range (65.7-81%) with higher sensitivity. The corresponding RH sensitivity values estimated from the graph are 17.51 pm/%RH and 53 pm/%RH.

It can also be noticed from Fig. 5(a) that for the RH values above 70%, losses in the transmission spectrum increases rapidly. The main reason for such high losses may be the mechanical stress and resulting bend experienced by the tapered fiber due to accumulation of the water molecules on its surface. As mentioned in the previous section, the fiber taper was stretched in the air 5 mm above the microscopic glass slide inside the chamber so that accumulation of a quantity of water on the surface of the fiber taper may cause a slight sagging of the fiber taper. Another possible reason for the increased loss is the increase of absorption by the micro resonator material due to the change of the Agarose layer properties at high RH. To further investigate the influence of the high RH levels on the sensor performance, an additional experiment was carried out with the same tapered fiber but with an uncoated microsphere of the same diameter in the RH range from 28% to 92%. The results are presented in Fig. 5(c) where it can be seen that the overall loss increases by circa 5 dB when the RH changes from 69% to 92%. A small (~36 pm, equivalent to 0.56 pm/%RH) wavelength shift is also observed, possibly due to stress induced onto the fiber by the water weight.
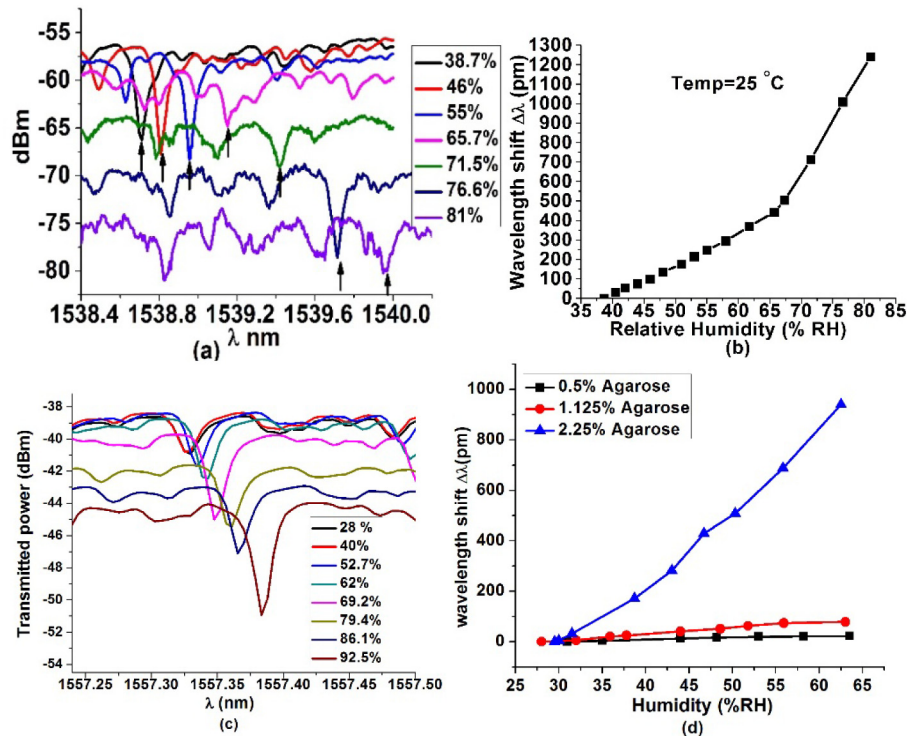

Fig. 5. Experimental results for the WGM resonator based on a 171 μm diameter microsphere coated with 2.25% Agarose gel coupled with a 3.3 μm tapered fiber in the range of humidity from 35% to 81% RH at a constant temperature of 25 °C. (a) WGM spectra at different RH levels; (b) wavelength shift of the WGM spectrum versus RH; (c) humidity response of the WGM spectrum for an uncoated microsphere resonator; (d) spectral shift versus RH for the sensors based on the same diameter microsphere coated with the Agarose solutions of different concentrations.

3.3 Coating thickness dependency

It should be noted that the average transmission loss level also depends on other factors, such as the tapered fiber waist diameter and the coating thickness on the surface of the microsphere. To investigate the effect of coating thickness, three different solutions with Agarose concentrations of 0.5%, 1.125% and 2.25 wt./vol. % were prepared and applied to

the microspheres of the same diameter (171 μm). Figure 5(d) shows that the RH sensitivity of the sensor depends significantly on the concentration of the Agarose solution used to form a coating layer. As can be seen from Fig. 5 (d) the highest RH sensitivity is achieved with the 2.25% Agarose solution in the range of RH from 25% to 65%.

The influence of the thickness of the polymer coating on the RH sensitivity of such a microsphere resonator can be analyzed as follows. It is known that the Q factor of an uncoated micro resonator is determined by the total loss including radiation loss, scattering loss from the surface irregularities and by material absorption, which can be expressed by Eq. (2) [27]:

$$\frac{1}{Q_{in}} = \frac{1}{Q_{rad}} + \frac{1}{(Q_{abs})_{silica}} + \frac{1}{(Q_{abs})_{agarose}} + \frac{1}{Q_{s.s}} + \frac{1}{Q_{coupling}} \qquad (2)$$

where $1/Q_{rad}$ denotes intrinsic radiative (curvature) losses of the resonator, $1/(Q_{abs})_{silica}$ and $1/(Q_{abs})_{agarose}$ denote the material absorption loss within the silica microsphere and the polymer layer, respectively, $1/Q_{s.s.}$ denotes scattering losses due to the microresonator surface inhomogeneities and $1/Q_{coupling}$ - losses due to coupling with the fiber taper. $1/Q_{rad}$ can be neglected in our case since this term vanishes exponentially for the resonators with $D/\lambda \geq 15$ [27]. The absorption limited Q factors can be calculated as $(Q_{abs})_{silica} = 2\pi n_s/\lambda_R \alpha_s$ and $(Q_{abs})_{agarose} = 2\pi n_a/\lambda_R \alpha_a$, where α_s, n_s and α_a, n_a are optical attenuation coefficients per unit length and refractive indices of silica and Agarose layer respectively. Since optical attenuation within the Agarose layer is several order of magnitude higher than that in silica, so the term $1/(Q_{abs})_{agarose}$ will be much higher than $1/(Q_{abs})_{silica}$. When the polymer layer thickness increases, greater portion of light energy is distributed within the polymer and thus the change in the polymer's RI (n_a) in response to changing RH leads to a higher RH sensitivity of the micro resonator. However, as the thickness of the polymer layer increases, the larger material absorption loss in the polymer compared to silica contributes to an overall decrease of the Q factor for coated resonators. Another significant contribution to the overall losses is due to scattering on the surface inhomogeneities, which strongly depends on their rms size and correlation length. In practice the total Q factor for the Agarose coated resonators considered in this study is limited by the last three terms of Eq. (2).

To further analyze the influence of the coating layer thickness, we carried out a series of experiments for a silica microsphere of a fixed diameter (100 μm) which was coated with 2.25 wt./vol.% Agarose gel applied multiple times through repeated coating cycles, forming coatings with progressively larger thickness values. The WGM spectra of the coated sphere were recorded after one, two, three, four, five and six coating cycles respectively using the same setup and the same tapered fiber at a constant temperature (23 ± 0.4°C) and humidity (66%RH). The spectra were further analyzed to estimate the corresponding Q-factors, as illustrated in Fig. 6 (a, b).

The quality factor can be calculated as $Q = \lambda_{res}/\Delta\lambda_{FWHM}$, where λ_{res} is the resonance wavelength and $\Delta\lambda_{FWHM}$ is the FWHM of the resonant lobe. We estimated the FWHM as 33.3 pm and 51.52 pm at the wavelengths of 1545.261 nm and 1545.035 nm, respectively. The corresponding Q factor values are thus estimated as 4.64×10^4 for the microsphere after the first coating cycle and 2.99×10^4 for the same microsphere after two coating cycles were carried out. As illustrated in Fig. 6 (c), the Q-factor of the sphere gradually decreased with each consequent coating cycle.
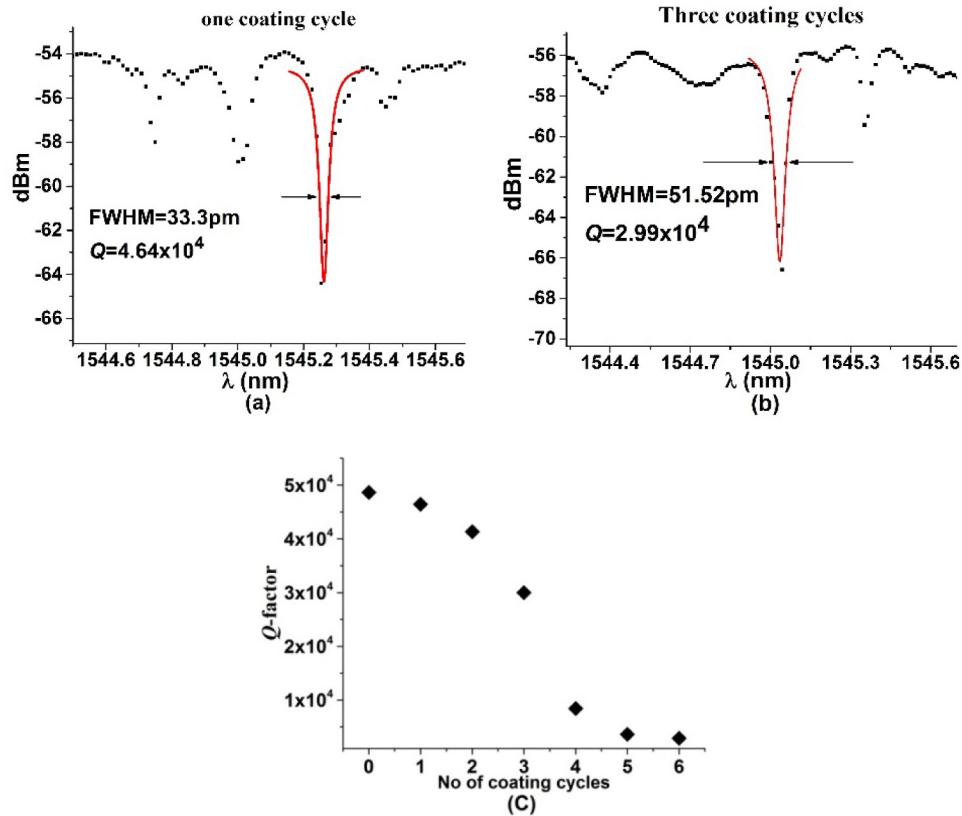
Fig. 6. Experimental spectra and Lorentzian fitting for a 100 μm diameter microsphere coated with 2.25% Agarose hydrogel: a) after one coating cycle; b) after three coating cycles; c) Q-factor versus the number of coating cycles.

The RH sensitivity of the sensors based on the same silica microsphere with different thicknesses of the Agarose coating was also investigated in the RH range from 30% to 70% RH and the results are presented in Fig. 7(a). The resonance wavelength shift over the entire humidity range was 0.41 nm for the sensor with a single coating, 0.964 nm for the sensor coated twice and 1.54 nm for the same sensor after three coating cycles. Although the thickness of the Agarose gel could not be controlled accurately, these results demonstrate that sensitivity gradually increases with the increase of the Agarose layer thickness. The maximum sensitivity in our experiments was observed for the sensor coated six times and was estimated at 518 pm/%RH which is significantly higher than that of 10 pm/%RH for the sensor with single coating as illustrated in Fig. 7(b).

This result is also, to the best of our knowledge, the highest sensitivity reported for Agarose coated fiber optic sensors, including that of 114.7 pm/%RH in the range 25%-96% RH for an Agarose- coated long-period grating (LPG) reported in [29] and for an Agarose-coated photonic crystal fiber interferometer previously reported by our group in [30], where for the same number of coating cycles the sensitivity was ~200 pm/%RH in the range of 30-90%RH.
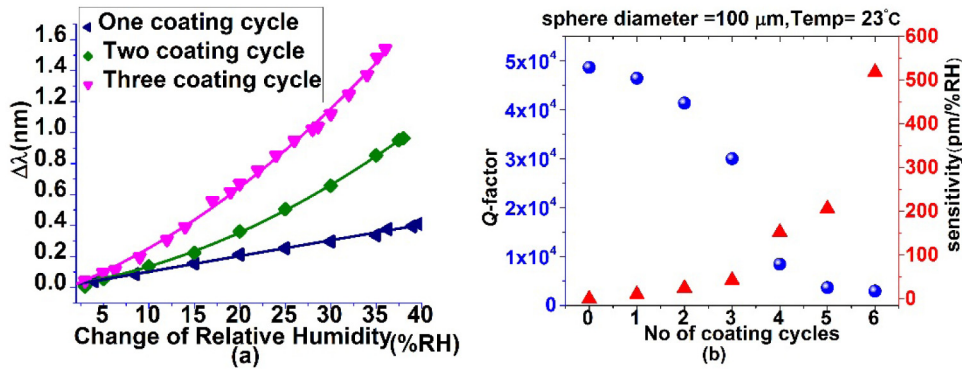
Fig. 7. (a) RH response of the sensor based on a 100 µm diameter microsphere with a different number of Agarose layers: (b) *Q*-factor and estimated RH sensitivity for the 100 µm diameter microsphere with a different number of Agarose coating layers.

3.4 Sensor characterization

The detection limit of the proposed sensor is calculated as the ratio of sensor resolution (*R*) to the sensitivity of the sensor (*S*). The sensor resolution depends upon the different sources of noise involved in the measurement and determined by the Eq. (3) [31]:

$$R = 3\sqrt{(\sigma^2_{amp-noise} + \sigma^2_{temp-induced} + \sigma^2_{spect-res})} \qquad (3)$$

where

$$\sigma_{amp-noise} \approx \frac{\Delta\lambda}{4.5(SNR^{0.25})}$$

σ is the standard deviation of resulting spectral variation and $\Delta\lambda$ is the full-width half maximum of the mode amplitude derived from the Q factor by $Q = \lambda/\Delta\lambda$. The Q-factor of the 171 µm coated microsphere calculated from the transmitted spectrum was 1.69×10^4 and the FWHM calculated by fitting the resonance dip with Lorentz equation was 91.06 pm at $\lambda = 1537.164$ nm. We assumed the SNR (Signal to Noise Ratio) of the system is approximately 60 dB, then the $\sigma_{amp-noise}$ is calculated as 0.636 pm. We also assumed the standard deviation due to temperature stabilization is 10 fm [28]. The spectral resolution of the optical spectrum analyzer used for our experiment is 10 pm ± 3%. The error in determining the position of the resonant mode is uniformly distributed between −0.3 pm to + 0.3 pm and the resulting standard deviation of $\sigma_{spect-res}$ is 0.2236 pm. The overall sensor resolution is $R = 2.0227$ pm. The detection limit of a 171 µm diameter coated microsphere is 1.15×10^{-1}%RH in the humidity range 38.7 −65.7%RH and 3.816×10^{-2}%RH in the humidity range 65.7-81%RH. It should be noted that the detection limit also depends on the nature of the optical source, where a narrow line width source will improve the detection limit [31]. However, such an improvement is not included or assumed here as in our case we used a broadband source.

3.5 Sensor stability

To test the stability of the sensor, RH measurements were carried out using the same sensor sample after a time interval of seven days in the RH range from 35% to 65% at a constant temperature. The sample used in this experiment was a 171 µm diameter microsphere coated with a 2.25% Agarose concentration gel. It can be seen in Fig. 8 (a), that the performance of the sensor is quite stable over one week with small fluctuations only. Figure 8 (b) illustrates studies of the hysteresis characteristics using data from humidification-dehumidification cycle for the three sensors based on the 171 µm diameter microspheres coated with hydrogel films

with Agarose concentrations of 0.5, 1.125 and 2.25 wt./vol.%. It can be seen from the figure that all the three sensors demonstrate very small or no hysteresis.
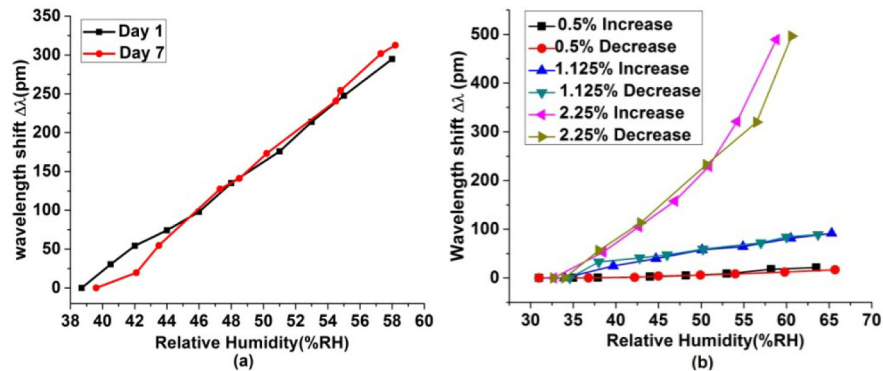

Fig. 8. (a) Relative humidity responses of the sensor, measurements recorded seven days apart; (b) studies of the sensor hysteresis: humidity response for the 171 µm microspheres coated with different Agarose concentration coatings during RH increase-decrease cycle at 25°C.

Finally, to investigate the effect of temperature on the RH sensor performance, the humidity in the chamber was set to constant at 41% RH. The temperature was then gradually raised from 16 °C to 27 °C. The wavelength shift of the WGM spectrum with temperature is shown in Fig. 9. It can be noted that the temperature sensitivity of the sensor is small compared to its RH sensitivity. For a 171 µm diameter sensor coated with 2.25% of Agarose hydrogel it is estimated as ~6 pm/°C in the temperature range of 16 −27 °C.
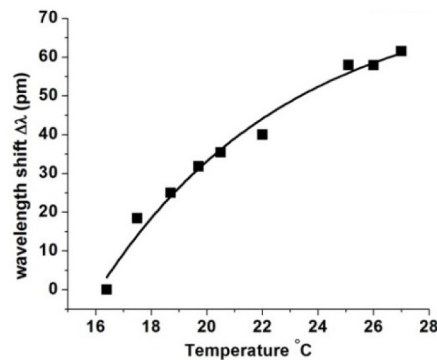

Fig. 9. Resonance wavelength shift versus temperature for the 171 µm microsphere coated with 2.25% wt./vol. Agarose solution at 41% RH.

4. Conclusion

A novel type of relative humidity sensor based on a whispering gallery mode microresonator has been proposed and experimentally demonstrated. WGMs are excited in the silica microsphere dip-coated with an Agarose gel evanescently coupled to a tapered fiber. Changes in the refractive index and thickness of the Agarose coating arising due to changes in the surrounding relative humidity lead to a spectral shift of the WGM resonances which can be related to the RH value after a suitable sensor calibration. The RH sensitivity of the proposed sensor depends on the concentration of the Agarose gel which determines the initial thickness of the coating deposited on the microsphere. Studies of the coating layers fabricated from gels with three different Agarose concentrations of 0.5%, 1.125% and 2.25 wt./vol.% and also by application of coating with the same Agarose concentration through repeated coating cycles showed that an increase in the thickness of the coating material results in an increase in

sensitivity but also leads to a decrease of quality factor of the micro resonator. The highest sensitivity achieved in our experiments was 518 pm/% RH in the RH range from 30% to 70%. The proposed sensor offers the advantages being very compact in nature, whilst also demonstrating low hysteresis, good repeatability, and a relatively low cross sensitivity to temperature.