

Applied whispering gallery modes on ZnO nanorods coated glass for humidity sensing application

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In this letter, a humidity sensor is demonstrated by applying a whispering gallery mode (WGM) from a microsphere resonator onto the ZnO nanorods coated glass surface. The diameter of the microsphere was 234 μm and the glass surface was coated with ZnO nanorods using the hydrothermal method at growth duration of 12 h. A significant response to humidity level ranging from 35%RH to 85%RH has been observed with the sensitivity of 0.014 2 nm/%RH. The proposed humidity sensor has successfully employed to enhance interaction between the whispering gallery mode evanescent and surrounds analyte with the assistance of ZnO nanorods coated glass.

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Optical micro resonators (OMRs) are dielectric structures that can sustain whispering gallery modes (WGMs). It confined and circulates the electromagnetic waves with minimal reflection losses and exceptionally high-quality (Q) factors. They can be simply fabricated by using various micro resonator geometries such as micro-discs, microspheres, and micropillars^[1]. Thus, OMRs play a ubiquitous role in applications such as optical filters, micro-lasers, cavity quantum electrodynamics, photonic devices, and sensing applications. WGMs are highly dependent on total internal reflection at the external cavity interface. When there are large refractive index (RI) contrasts between the host and the cavity, radiative losses could be minimized due to strong confinement of the WGMs which leads to improvement of Q factors^[2]. The evanescent wave of the WGMs is sensitive to the binding of the chemical molecules attach to the resonator's surface and the changes of the surrounding's refractive index^[3].

The humidity sensing happens due to interaction between the WGMs evanescent components with the ambient analyte. The sensing measurement could be characterized by amplitude changes (absorption) and phase

changes (change of refractive index). These changes would lead to a direct monitoring technique^[1,4].

Microsphere resonator (MsR) has been selected for this work among all the OMRs. It is the simplest three-dimensional WGMs resonator with a diameter of 10–100 μm which was firstly proposed by F-Vollmer in 2002^[5]. Since its first introduction, it has become one of the favorite choices for microcavity based sensors. It has a higher Q factor as compared to other types of WGMs resonator reaching up to 10^{10} . It is particularly suitable for low-concentration molecular detection^[6]. It can be fabricated by simply melting the tip of silica glass fiber. Despite the easy fabrication process, MsR differs from the planar waveguide ring resonator which can support complex modes with radial, equatorial, and polar field dependencies. It also has very low total optical loss resulting in exceptionally high Q values with narrow resonant-wavelength lines and high energy density^[1].

This work exploited the WGMs in MsR and changeable RI of ZnO nanorods for humidity sensing. It employed a MsR coupled to a tapered fiber also known as microfiber. The WGMs of the MsR are excited by the evanescent coupling of light using microfiber. It operates

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based on the interaction between the evanescent light and the ZnO-nanorods as a sensitive coating material. The change in the surrounding humidity varies the transmitted output power^[7]. ZnO was selected as a sensitive material because it exhibits reactive surfaces to allow water chemisorption at room temperature. Thus, better sensing response would occur if more water molecules adsorbed on the material's surface. It provides a larger surface area with the same volume as compared to bulk material^[8]. However, typical directly coated nanomaterials on the microfiber have proved to be complicated to handle during the synthesis process which could deteriorate the performance of the microfiber^[9,10]. Therefore, we have integrated MsR and ZnO nanorods coated glass surfaces to solve this issue for the first time to our knowledge. It has superiority in terms of reducing hassle to access the interaction of the ZnO coating layer and the evanescent wave from the MsR for sensing. The hybrid structures are capable to maximize the access of evanescent waves and open enormous opportunities for sensing applications due to their unique features such as high sensitivity, high linearity, compactness, and stable measurement.

The ZnO nanorods were grown onto microscope glass substrates (Heathrow Scientific LLC, USA) using the hydrothermal synthesis process as described in our previous work^[11]. It has undergone several procedures such as seeding process, annealing process, and growing process. In the experiment, the growth duration was chosen at 12 h based on work in Ref.[12]. Uniform growth and high density could change the optical response of the nanostructures in term of attenuation coefficients and scattering. Forward scattering would dominate with high density ZnO nanorods which will increase the humidity sensing response^[13].

WGMs in the MsR with a diameter size of 234 μm are excited by evanescent coupling using microfiber with 6 μm waist diameter size. To ensure good coupling efficiency, the gap between the microfiber and the MsR needs to be as close as possible. The MsR was carefully and gradually placed onto a microfiber that lay on the ZnO nanorods coated glass in direct contact (Fig.1) until the WGMs resonance was displayed in the output transmission spectrum^[14]. Subsequently, WGMs resonances were observed at the output transmission spectrum by means of an optical power meter that connected to the computer. If the resonance non-existence, the MsR needs to be repositioned on the microfiber. Thus, the microfiber needs to be directly contacted with the MsR surface to ensure the most stable and efficient coupling conditions. When the MsR is exposed to the variation of surrounding %RH, the effective refractive index would be changed. This leads to a change of coupling efficiency of the MsR, which in turn changes the transmission spectra.

Field emission scanning electron microscopy (FESEM) and energy dispersive X-ray (EDX) analysis were performed to investigate the morphology of the ZnO nanorods structure and determine the chemical composition of

the coating layer, respectively. In the sensing experiment, the coated glass surface was positioned directed upwards inside a controlled chamber (22 cm \times 12 cm \times 12 cm) and the microfiber was laid on it. The WGMs transmission spectrum was obtained by sweeping tunable laser source (TLS) within the operating wavelength range between 1 500 nm and 1 600 nm in a wavelength resolution of 0.001 nm. The transmission mode was acquired using an optical power meter (OPM) and displayed on the computer as shown in Fig.2. The resonance wavelength value was identified from the power spectrum, and the value was fixed during the humidity sensing experiment. Thus, light source emitted at 1 550 nm center wavelength was used during the sensing experiment. Sodium hydroxide (NaOH) was employed to increase the relative humidity level inside the controlled chamber. According to ASHRAE Standard 55 (2004), the acceptable %RH range for an indoor place is between 30%RH and 60%RH. This work focuses on investigating measurement between 35%RH to 85%RH due to our equipment capability. This range is still adequate to ensure thermal comfort conditions for humans and sustain the indoor air quality level^[15].

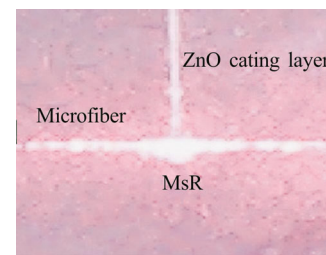


Fig.1 Microfiber with a diameter of 6 μm and MsR with a diameter of 234 μm

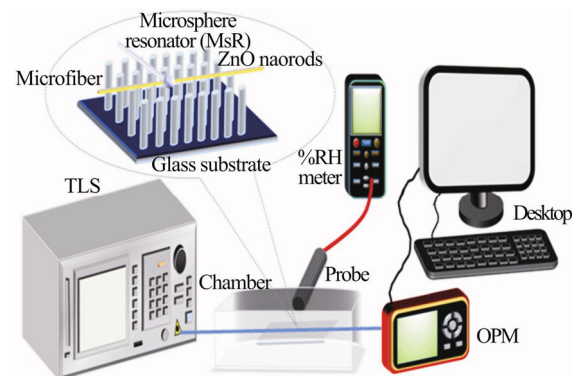


Fig.2 The experimental setup of humidity sensing

FESEM images of ZnO nanorods coated glass surface at 5.00k \times magnification are shown in Fig.3(a). A vertically grown ZnO nanorods could be observed on the surface of the glass. It has uniform growth and high density with an average of 28.7×10^{12} nanorods/m². The elements on the coated glass surface were confirmed to comprise of only zinc and oxygen elements as shown in Fig.3(b).

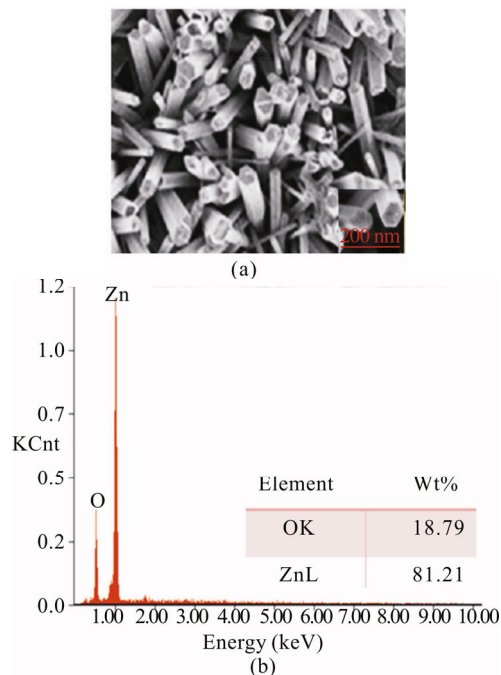


Fig.3 (a) Morphology of ZnO nanorods coated glass surface at magnification of 5.00kx; (b) EDX element analysis presenting zinc and oxygen peaks

Fig.4 shows the transmitted WGMs from MsR coupled with ZnO nanorods coated glass (MsR-ZnO) structure. Clear WGMs resonance wavelength has been identified from the transmission spectrum at around 1 550 nm. The Q-factors were calculated based on a formula of $Q=\lambda/\Delta\lambda$ where λ is resonance wavelength and $\Delta\lambda$ is the full width at half-maximum from the transmission spectrum. The corresponding Q-factor calculated is 6.46×10^6 for a resonance wavelength at 1 550.56 nm. The dips magnitude is around -46 dBm as shown in Fig.4. Ultrahigh Q factor of WGMs resonators would lead to high energy density, very narrow resonant wavelength lines, longer cycle times for the photons in the cavity and enhanced interactions between the light and the external environment^[6]. Thus, when there is a slight external environmental change, higher sensitivities and lower detection limits could be obtained as compared to other WGM dips. Q-factor highly depended on the loss characteristic of the structure, which causes a leakage of optical energy from the resonator. The losses are contributed by the scattering, roughness of the surface, absorption due to molecular resonance, surface contamination, energy coupling along the fiber taper and radiation due to boundary curvature during propagation^[3].

The wavelength shift towards humidity sensing was investigated and the results are presented in Fig.5. Fig.5(a) shows the output spectrum from the straight microfiber, which was laid onto the ZnO nanorods (SmF-ZnO). It is shown that the peak spectrum shifted from 1 530.16 nm to 1 530.72 nm with the increase of humidity from 35% to 85%. Fig.5(b) shows the resonance wavelength with the incorporation of MsR, which increases monotonically when the %RH increases. It

shifts from 1 550.05 nm to 1 550.75 nm when it was exposed to the humidity level from 35% to 85%RH. The amount of shift was 0.7 nm, which is larger than that of SmF-ZnO (0.56 nm). Fig.5(c) shows the trend line response in terms of wavelength shift. MsR-ZnO has better sensitivity with 0.0142 nm/%RH as compared to SmF-ZnO with 0.009 7 nm/%RH. The MsR-ZnO also produces linearity of 99.8% which is better than SmF-ZnO with 98.1%. When WGMs confined inside the resonator, some energy will spread in the form of the evanescent field to the surrounding medium. When there are outer environment changes inside the evanescent field, it will be transformed into changes in the modes^[16]. The interaction between the mode field and the analyte will cause the effective RI increase due to analyte that attached to the micro-resonator surface which increases the sensitivity of the sensor. MsR has a dielectric resonators in circular structures support the electromagnetic surface oscillations and evanescently coupled to the nearby waveguide. It occurs in MsR when highly angular momentum modes were excited by trapping light inside dielectric structure and repetition of near-total internal reflections. This generates some transmission dips due to confining light inside the resonator. The dip at the resonant wavelength in the transmission spectrum was caused by the light out-coupled into the tapered fiber. MsR will exhibit resonance wavelength shifts as the RI of the sphere changes which enable the MsR to trace amount of biological and chemical changes. WGMs resonances are greatly influenced by the refractive index of the surrounding resonator medium. If an appropriate coating material is applied to it. It interacts with the evanescent wave of the WGMs field when surround analyte aggregates at the resonator's surface and resulting in wavelength shift or a change in the Q factor^[17]. This make the MsR-ZnO has better sensitivity as compared to the SmF-ZnO. Fig.6 shows the repeatability of the proposed sensor. It shows almost the same value in each trial and exhibit good reproducibility.

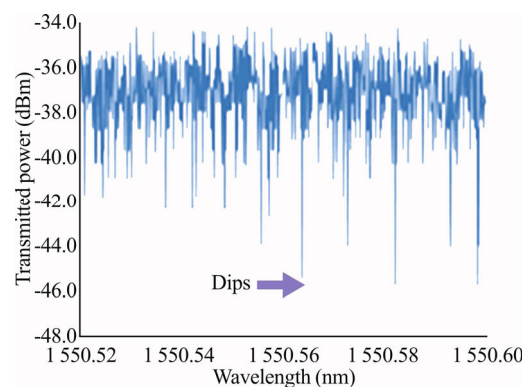


Fig.4 WGMs transmission modes of MsR-ZnO coupled on microfiber with 6 μm diameter where Q-factor estimation of 234 μm diameter silica microsphere is around 6.46×10^6

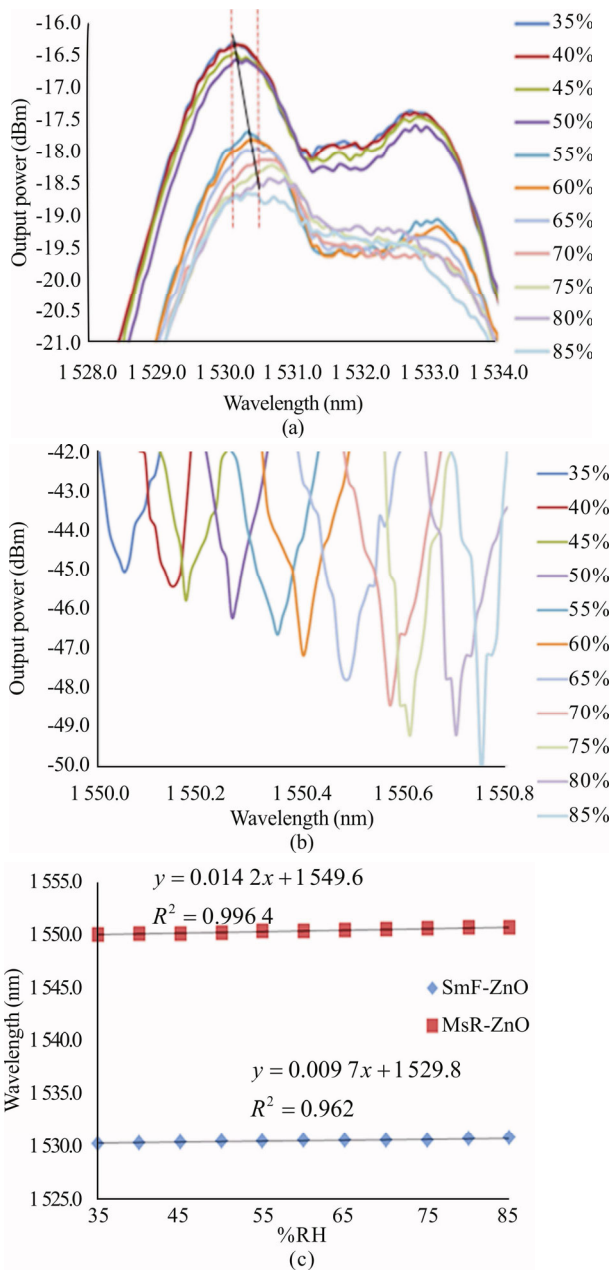


Fig.5 Wavelength shift towards humidity sensing for (a) SmF-ZnO and (b) MsR-ZnO; (c) Comparing of wavelength response with humidity

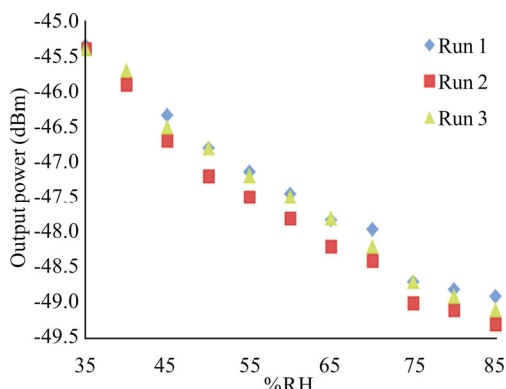


Fig.6 Repeatability of the proposed sensor

In conclusion, the microsphere laid on ZnO nanorods coated glass has shown better sensing performance as compared to straight microfiber laid on the same coated glass. MsR-ZnO produce better sensitivity of 0.0142 nm/%RH as compared the SmF-ZnO with 0.0097 nm/%RH respectively. It also has better linearity with 99.8% as compared to the SmF-ZnO with 98.1%. The WGMs of the MsR are excited by evanescent coupling of light using microfiber which leads to changes of transmitted output powers when exposed to different humidity level concentrations.

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