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Ethanol Gas Sensor Based on a Hybrid Polymethyl Methacrylate–Silica Microfiber Coupler

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***Abstract*—Gas sensors have broad applications for either industrial process control or biological detection. We developed a versatile and compact optical hybrid coupler gas sensor composed of a polymethyl methacrylate (PMMA) microwire and a silica microfiber. The light propagating in the microfiber interacts with the polymer microwire. When the hybrid coupler is exposed to different concentrations of ethanol vapor, the refractive indices of the polymer and the surroundings are changed, resulting in shifts in the resonant wavelength. The experimental sensitivity for ethanol vapor is 0.65 pm/ppm.**

***Index Terms*—Couplers, Hybrid optical sensor, Microfabrication, Microsensors.**

I. Introduction

THE detection and measurement of gases is an important task in various fields of industry, environment, and biochemistry. There are different kinds of gas sensors, including electrical and optical gas sensors. On the one hand, traditional electrical gas sensors can be classified according to their working principles. Some perform the electrochemical oxidation or reduction of gases in order to distinguish their composition and concentration, and are convenient to detect hazardous gases [1-3]. Some are sensitive to the thermal conductivities of different gases and they are usually employed in high-temperature environments [4]. Some are based on semiconductors whose electrical conductivities change when exposed to different gases [5-7]. On the other hand, optical sensors—in particular, fiber gas sensors—have been paid significant attention owing to their many desirable properties such as high flexibility, ease of integration, multiplexing capability, and strong immunity to electromagnetic interference. Generally, fiber gas sensors employ optical detection techniques such as spectroscopy [8-11] and refractive index measurement [12-15].

As there is an urgent need for device miniaturization and compactness, significant progress has been made in micro-/nano-optics in recent years with the improvement of microfabrication techniques. Especially, microfibers have received immense attention because of their low loss, large evanescent field, strong confinement, configurability, and robustness [16]. However, monotonous materials have limited their application and retarded their development.

Polymers are another kind of widely used materials. Apart from their mechanical flexibility, easy processing, and low cost, polymers offer various attractive advantages for gas sensing applications [17]. For instance, gas molecules can be either selectively adsorbed on their surfaces or diffused into the polymer matrix [18], which may be unrealizable with other materials such as metals, semiconductors, or silica. Moreover, polymers are hospitable to various functional dopants, which may enhance the sensitivity to specific gases and contribute to the improvement of the device performances.

Some research groups have already focused on other materials and have achieved small-volume, low-dimensional microfiber/nanowire gas sensors [19-23]. However, to the best of our knowledge, hybrid microfiber coupler sensors have not been reported yet. Herein, we demonstrate an optical coupler by closely aligning a polymer microwire and silica microfiber for realizing the detection of small concentrations of ethanol gas with good sensitivity.

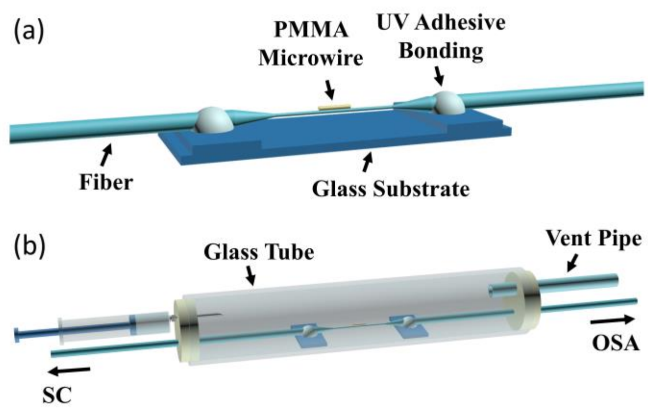

Fig. 1. Schematic diagram of (a) hybrid microfiber coupler and (b) experimental device; SC, supercontinuum; OSA, optical, spectrum analyzer

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In this paper, we develop a versatile, easily fabricated vapor sensor based on a polymethyl methacrylate (PMMA)–silica microfiber coupler. We choose PMMA because it is easy to be prepared and processed, and has low toxicity. The sample is illustrated in Fig. 1. It is composed of a silica microfiber and a PMMA microwire, both of diameter approximately 1.2 μm. When the coupler is exposed to ethanol vapor, the PMMA microwire interacts with the vapor and hence, the effective refractive index is changed. Consequently, the resonant wavelength undergoes a corresponding redshift. Compared with other kinds of gas sensors, the proposed optical hybrid coupler gas sensor is much easier to fabricate and integrate. The construction is simple and micro-/nano-waveguides of various materials can be selected for different functions.

II. Sensor Fabrication

PMMA microwires were fabricated by directly drawing from a polymer solution [24]. First, 100 mg of PMMA pellets (Mw 35 000, Sinopharm) was dissolved in 2.0 g Anisole (98.0%, Lingfeng). Subsequently, the mixture was stirred at room temperature to form a uniform solution with high viscosity. In the drawing process, a fiber probe with a sharp tip, whose diameter was hundreds of nanometers, was used to transfer a tiny droplet of the solution onto a glass slide, followed by drawing a wire out of the droplet rapidly, as shown in Fig. 2(a). The solvent evaporated instantaneously, leaving a PMMA microwire on the glass slide. The microwires usually had diameters ranging from hundreds of nanometers to several micrometers, and they could be easily cut off using a tungsten probe. Their diameters were determined by the drawing velocity and the viscosity of the solution. We could obtain a thinner wire from a viscous solution at a higher drawing velocity (1-2 m/s). The process is repeatable because the velocity and viscosity can be well controlled.

The microfiber was fabricated from a standard telecom optical fiber (Corning, SMF-28) using the flame brushing method [25]. It was comprised of two conical transition regions: a central uniform waist region and two input/output ports. The PMMA microwire was first picked up from the glass slide using a fiber probe immobilized on a high-precision 3D translational stage, as illustrated in Fig. 2(b), and thereafter aligned on the waist of the silica microfiber under a microscope. Owing to the Van der Waals' force, the PMMA microwire could cling very tightly to the microfiber. Figure 2(c) illustrates the optical micrograph of the sample, which was further examined using a scanning electron microscope (SEM), as shown in Fig. 2(d). The length of the coupling region was 175 μm.

Figure 2(e) illustrates the typical coupling spectrum of the coupler. We chose a UV-curable adhesive (EFiRON UVF PC-375, Luvantix) to fix the coupler to a substrate. A supercontinuum source (NKT, K91-120-02) was utilized to characterize the transmission spectrum of the sample. The signal light was injected into one of the pigtails of the coupler. An optical spectrum analyzer (OSA, Yokogawa, AQ6370C) was used to record the output spectrum ranging from 1450 to 1650 nm. To investigate the function of the PMMA microwire,

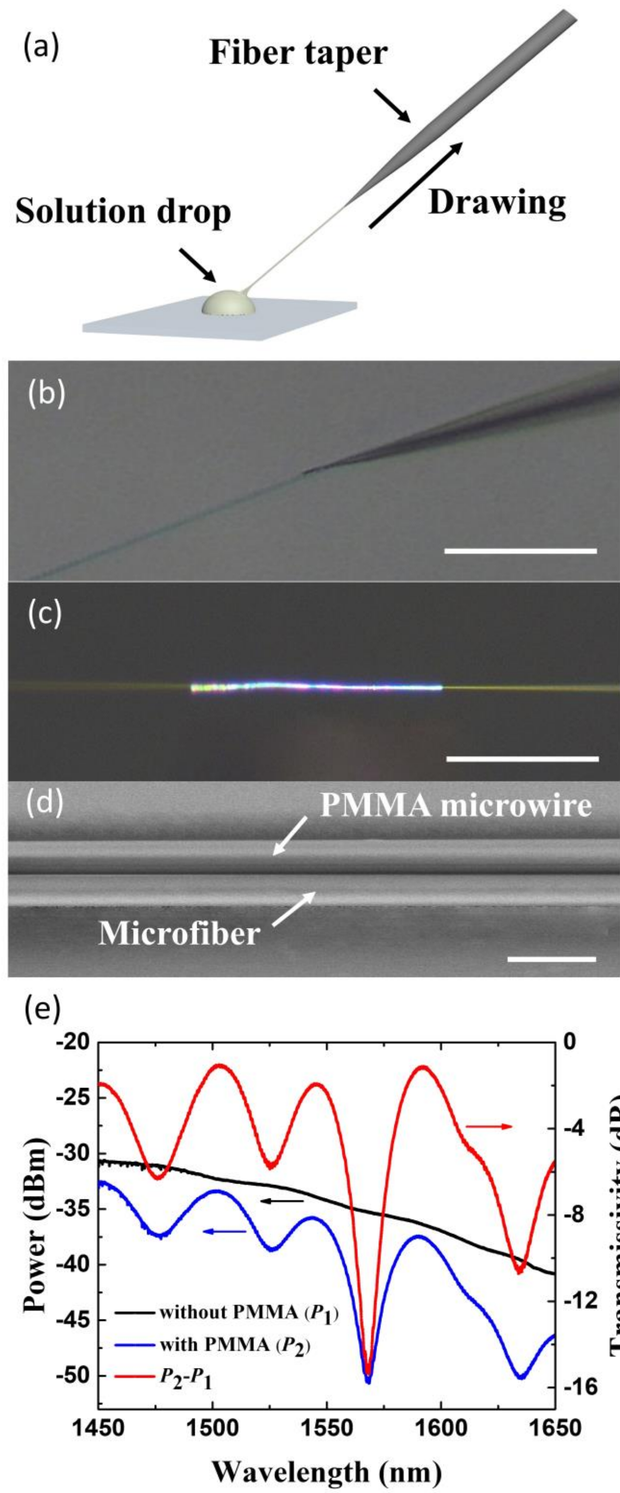

Fig. 2. (a) Fabrication of a PMMA microwire; (b) PMMA microwire picked up using a fiber probe; (c) micrograph, (d) SEM, and (e) output spectrum of the hybrid microfiber coupler. Scale bar: (b) (c) 100 μm, (d) 2 μm

we measured the output spectrum of the sample without and with the PMMA microwire, respectively. The black and blue curves represent the transmission powers of the sole silica microfiber (P_1) and the silica microfiber – PMMA microwire hybrid coupler (P_2). Therefore, the red curve ($P_2 - P_1$) indicates the influence of adding the PMMA microwire. It can be observed from Fig. 2(e) that the loss caused by the added

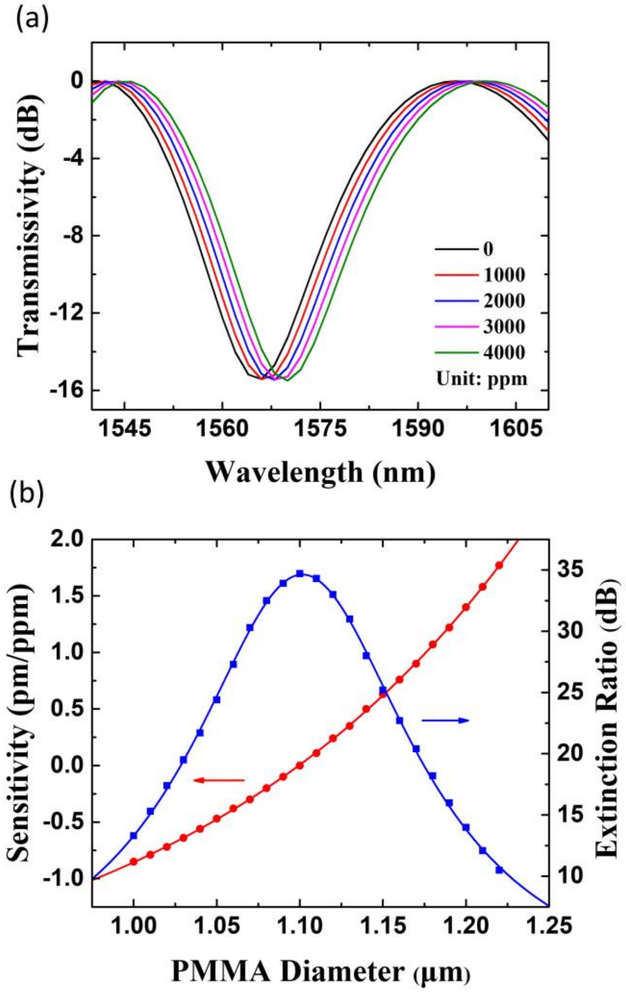

Fig. 3. Simulations of (a) output spectra with different ethanol concentrations, (b) relationship between the diameter of the PMMA microwire and sensitivity (red line) or extinction ratio (blue line)

microwire was approximately 1.5 dB and the extinction ratio, which indicates the ratio of the maximum and minimum transmission intensities, was approximately 15 dB. The dips in the transmission spectrum would be sharper and denser with a longer microwire, in which case could improve the resolution to some extent.

III. Theoretical Simulation

The hybrid microfiber coupler can be illustrated as two touching cylindrical waveguides with different materials and refractive indices. The coupling coefficients of silica microfiber and PMMA microwire are given by Eq. (1) [26].

$$\begin{aligned} \kappa_1 &= \frac{2\pi}{\lambda} n \int_S \left[\Delta n_1(x,y)\right] \cdot e_1(x,y) \cdot e_2(x,y) dS \\ \kappa_2 &= \frac{2\pi}{\lambda} n \int_S \left[\Delta n_2(x,y)\right] \cdot e_1(x,y) \cdot e_2(x,y) dS \end{aligned} , \quad (1)$$

where n refers to the refractive index of air; Δn_1 and Δn_2 represent the relative differences between the refractive indices of silica or PMMA and air, respectively; e_1 and e_2 denote the normalized electric field distributions of the microfiber and PMMA microwire, respectively. The normalized optical power flow along the z-direction of the microfiber is given by Eq. (2) [26].

$$P(z) = [\cos(\sigma z) - \frac{i\delta}{\sigma}\sin(\sigma z)]^2 , \quad (2)$$

where $\sigma = \sqrt{\kappa_1\kappa_2 + \delta^2}$ and δ is defined as the mismatch between the two waveguides, which equals half of the difference between the propagating constants β_1 and β_2 of the two waveguides. The propagating constant is defined as $\beta_j = \frac{2\pi}{\lambda} n_{eff}$ (j = 1, 2), where n_{eff} is the effective refractive index and is related to the diameter of the waveguide.

The propagation property of the hybrid microfiber coupler and its wavelength shift with the change in concentration of ethanol gas were simulated using finite element analysis before the experiments. We chose to detect ethanol gas, as ethanol was common in the laboratory and its vapor in the environment might deteriorate the performance of some delicate instruments. We used Comsol Multiphysics to calculate the electric field distributions of the two waveguides and utilized these values in MATLAB to calculate κ and $P(z)$.

When ethanol gas is introduced, the ambient refractive index increases. PMMA can absorb the gas and thus, its refractive index becomes smaller. In ethanol solution, swelling resulted from softening and plasticization of PMMA is obvious [27]. In ethanol gas with low concentration, the effect is weak. According to the previous results [28, 29], the refractive indices of PMMA microwire and surroundings change by less than 10^{-6}/ppm and ~10^{-9}/ppm, respectively, and the radius of the PMMA microwire increases by less than 10^{-6}/ppm. Therefore, when the concentration of ethanol gas is increased, transmission spectra are obtained as illustrated in Fig. 3(a). The initial diameters of the silica microfiber and PMMA microwire were set as 1.2 μm. We can foresee that the hybrid coupler is more sensitive to ethanol gas than pure silica microfiber as the latter only responses to the environment refractive index.

Moreover, as expressed in Eq. (2), the transmission is also related to the waveguide mismatch δ, and hence, we consider that the sensitivity is affected by the diameters of the waveguides. In order to achieve the optimal sensitivity of the hybrid coupler, we calculated the dependence of concentration sensitivity on the diameter of the PMMA microwire of wavelength approximately 1550 nm. The diameter of the silica microfiber was set as 1.2 μm and the diameter of the PMMA microwire was varied from 1.0 μm to 1.2 μm. As illustrated in Fig. 3(b), the red and blue curves represent the variation tendency of sensitivity and extinction ratio, respectively, with the diameter of the PMMA microwire. On the one hand, the extinction ratio achieves the maximum but the sensitivity of the hybrid coupler declines to zero at the critical diameter of approximately 1.1 μm, where $\delta = 0$ and $\Delta\kappa_1\kappa_2$ compensates for $\Delta\delta^2$ with the change of the ethanol gas concentration. When the diameter of the PMMA microwire is smaller or larger than 1.1 μm, the spectrum has a blueshift or redshift, respectively. On the other hand, although the sensitivity improves when the diameter of the PMMA microwire deviates from the critical

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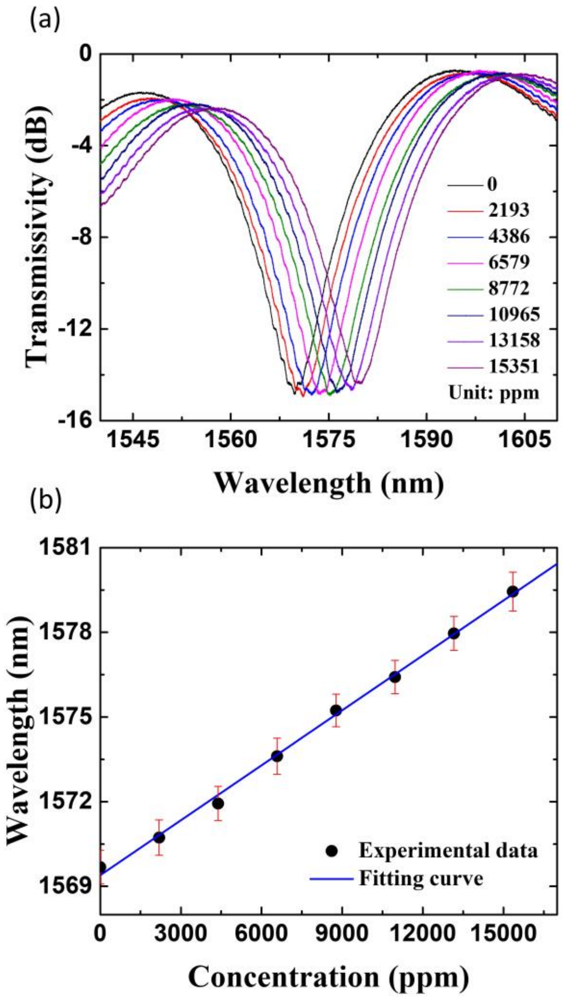

Fig. 4. (a) Transmission spectra with different concentrations of ethanol vapor. (b) Resonant wavelength shift as a function of the vapor concentration. The sensitivity was approximately 0.65 pm/ppm.

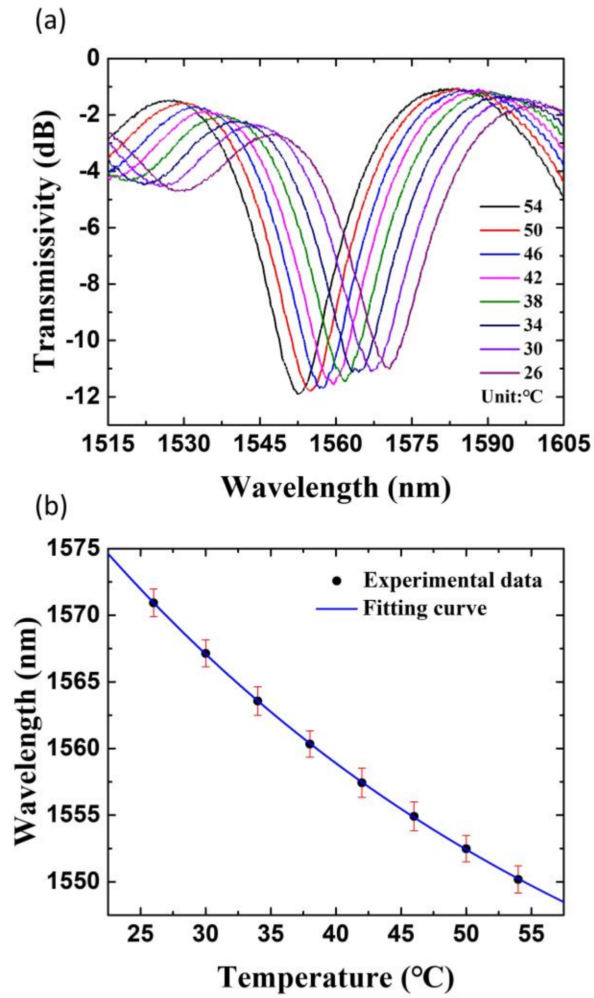

Fig. 5. Resonant wavelength shift as a function of temperature. The sensitivity at room temperature was approximately −942 pm/°C.

diameter, the extinction ratio decreases. Herein, we consider the optimal diameter of the PMMA microwire to be approximately 1.2 μm, because both sensitivity and extinction ratio are acceptable at this diameter.

IV. Experiments and Discussions

In order to investigate the response to different concentrations of ethanol vapor, the sample was enclosed in a glass tube, which served as the gas fluidic chamber with two rubber plugs sealing both sides, as illustrated in Fig. 1(b). The inner diameter and height of the tube were 3.5 cm and 20 cm, respectively.

The hybrid coupler sensor was also sensitive to water vapor and other gases that could be absorbed by PMMA. It was well packaged, ensuring that the wavelength shifted only with the input of ethanol gas during the experiment. However, in practice, crosstalk owing to humidity and other impurities should be considered. Ethanol microfluids were injected into the glass tube using a syringe pump (Shenchen, SPLab01). Every time 1 μL of ethanol was injected and the liquid was being volatilized, we discovered an apparent redshift of the resonant wavelength, as demonstrated in Fig. 4(a). We added ethanol droplets several times and the resulting ethanol gas concentrations were calculated based on the concentration of ethanol in the solution and the volume of the chamber. Figure 4(b) shows the relationship between the resonant wavelength and concentration of ethanol molecules. It can be observed that, with the increase in the concentration of ethanol, the resonant wavelength linearly increased. The wavelength shift response caused by the change in the concentration of ethanol was approximately 0.65 pm/ppm. As the resolution of OSA was 20 pm, the detection limit of the gas sensor could be as low as 30 ppm. This sensitivity is similar to that of previously reported fiber-device-based [29-33] and semiconductor electrical [34, 35] gas sensors.

There are several possible reasons for the discrepancy between the theoretical simulation and experimental results. First, the deviation in diameter and uniformity of the silica microfiber and PMMA microwire used in the experiment might have caused a small discrepancy from the ideal theoretical model. Second, as shown in Fig. 2(c), the PMMA microwire adhered imperfectly to the microfiber. It might have contained distortion, tension, and interspace, resulting in non-uniformity in the two-waveguide coupling. Third, temperature variation

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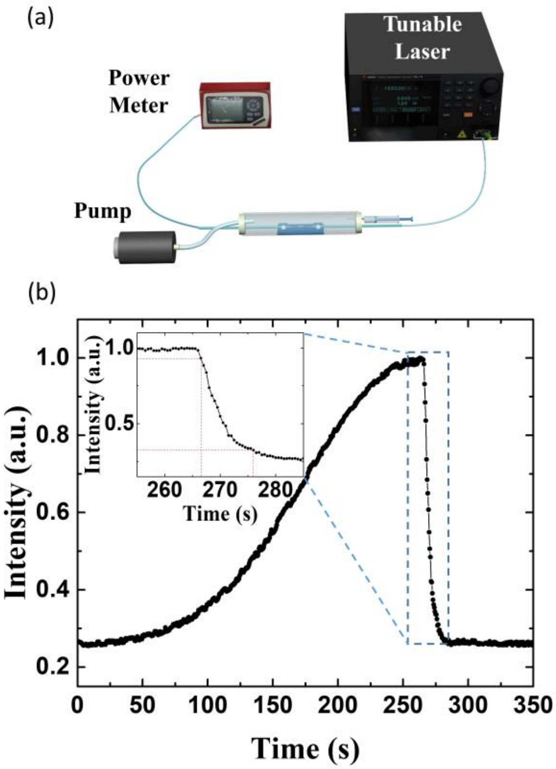

Fig. 6. (a) Experimental setup to measure the dynamic response. (b) Time response of the coupler. Inset shows that the recovery time was 9.4 s.

was also a non-negligible factor as the volatilization could reduce the temperature of the environment. Thus, we measured the coupler response to temperature over the range of room temperature to approximately 55 °C. As illustrated in Fig. 5, the sensitivity at room temperature was approximately −942 pm/ °C. Under these circumstances, the temperature must be sufficiently stable during the experiment.

Furthermore, we measured the dynamic response to ethanol vapor. The setup of the experiment is illustrated in Fig. 6(a). Two pigtails of the coupler were connected to a tunable semiconductor laser (Santec, TSL-710) and a power meter (Thorlabs, PM100D), respectively. The wavelength of the probe light source was chosen at the falling edge of the output spectrum. The power meter was utilized to record the output power in real time. Further, a conduit was inserted into the chamber using a rubber plug. The other end of the conduit was connected to a mechanical air pump, which could extract the vapor from the chamber in a short time. We used a syringe to inject a tiny drop of ethanol (approximately 10 μL) into the chamber. When the liquid was evaporating, the output spectrum redshifted gradually, and thus, the power at the chosen wavelength increased, as demonstrated in Fig. 6(b). It can be observed from the figure that the rising curve is not very smooth, which is probably due to the airflows generated from evaporation. After the output power achieved the maximum and became stable for some time, the pump was turned on and the power decreased to the previous value after a few seconds, indicating complete removal of vapor molecules from the PMMA microwire and the atmosphere. Benefit from the small size of the sensor, its response time was approximately 9.4 s, which is better than that of standard-size fiber sensors [29, 33]. The order of magnitude is close to that of photonic crystal fiber (PCF), while the size of PCF is much bigger [32]. The hysteresis feature of the sensor mostly depends on the velocity of airflows. As the power of the mechanical pump used in the experiment was not very large, it limited the sensor performance in the measurement of the dynamic response. We believe that the actual response time would be faster than 9.4 s.

V. Conclusions

In conclusion, we developed a versatile gas sensor based on a hybrid PMMA–silica microfiber coupler, which is easy to fabricate and has good sensitivity. The device was constructed using a silica microfiber with a PMMA microwire adhering to the side of its waist region. The resonant wavelength was proportional to the analyte concentrations, indicating that this hybrid coupler sensor can be effective for the detection of gas concentration. The sensitivity of the sensor to ethanol vapor was 0.65 pm/ppm. We also analyzed the response time, which indicated that this sensor has potential for applications in dynamic measurement.

Future work will focus on optimizing the performance of the hybrid coupler gas sensor by increasing the coupling coefficients of the two waveguides to improve the spectral resolution of the sensor. For practical applications, it is necessary to well package and protect the coupler [36, 37]. We can insert the coupler into a thin glass capillary with a diameter of ~200 μm, and both sides are sealed with UV adhesive. Several microchannels can be drilled along the capillary by use of a femtosecond laser. Furthermore, it is still a challenge for materials such as polymers to respond to specific gases. Thus, extensive studies should be conducted to improve the selectivity of the sensor.

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