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**Four-parameter gas optical fiber sensor based on surface plasmon
resonance (SPR) for simultaneous detection of methane, relative
humidity, hydrogen, and hydrogen sulfide**

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Abstract

A 4-parameter gas sensor that can simultaneously detect methane, relative humidity, hydrogen, and hydrogen sulfide is designed and analyzed. It uses a 4D-type coreless optical fiber as the substrate, gold as the plasmonic medium, and cryptophane-A/ultraviolet-cured fluorosiloxane complex (Crypt-A/UVCFS), polyvinyl alcohol (PVA), Pd-WO₃, and zinc oxide (ZnO) as the sensing materials. Numerical analysis shows that in the ranges of methane concentrations of 0%-2%, relative humidity of 30%-70%, hydrogen concentration of 0%-2%, and hydrogen sulfide concentration of 0-80 ppm, the average methane sensitivity, relative humidity sensitivity, hydrogen sensitivity, and hydrogen sulfide sensitivity are -76 nm/%, -2.2 nm/%RH, -153 nm/%, and 15 nm/ppm, respectively. This sensor achieves simultaneous and integrated detection of four parameters without spatiotemporal deviations and has high practical potential in environmental sensing and related applications.

1. Introduction

Methane, water vapor, hydrogen, and hydrogen sulfide, the main gases encountered in oil and gas exploitation, can easily cause unpredictable losses, and therefore, real-time monitoring of them is very important [1]. Compared with traditional electrical component sensors, optical sensors not only avoid the risk of explosion caused by electromagnetic characteristics, but also have higher sensitivity and durability [2], [3].

Optical fibers used in sensors are mainly divided into the interferometric type and the surface plasmon resonance (SPR) type. The interferometric type generally has technical bottlenecks such as a narrow monitoring range and low integration, making it difficult to adapt to complex environments, while the SPR type overcomes the above limitations, delivers comprehensive sensing performance, and has become a research hotspot [4], [5]. For example, Chen et al. have detected hydrogen sulfide in the concentration range of 0-100 ppm with a maximum sensitivity of 21.76 pm/ppm by coating the surface of the optical fiber with titanium dioxide nanoparticles and multi-walled carbon nanotubes [6]. Zhao et al. have detected hydrogen in the range of 0% to 1% with an average sensitivity of 1.09 nm/% by coating a PdWO_3 film on the photonic crystal fiber [7]. Zheng et al. have coated a PDMS/Pt- WO_3 composite film onto a single-mode fiber to achieve a maximum sensitivity of -52.12 nm/% over the hydrogen concentration range of 0%- 1.8% [8]. Liu et al. have deposited a nanoscale polysiloxane film on silver to achieve an average sensitivity of 6.643 nm/% over the methane concentration range of 0% - 3.5% [9]. However, the sensitivity is relatively low. Xu et al. have coated a graphene-molybdenum disulfide film on the optical fiber to improve

the average sensitivity to 63.4 nm/% in the hydrogen range of 0.5 - 3.5% [10]. Peng et al. have deposited a polyvinyl alcohol (PVA) film on a tapered optical fiber, which shows a humidity sensitivity of -1.5 nm/% within the humidity range of 46% - 93% [11].

Although traditional single-parameter optical fiber SPR sensors have high sensitivity, they may not work well in the field due to the complex environment and strong coupling as a result of the sensing principle of SPR sensors. Multiple gases can affect the sensing properties and produce inaccurate results. By integrating different types of sensing materials with specific recognition capabilities into the same segment of the optical fiber, matrix demodulation can be performed to sense different media. Moreover, the higher the integration level, the higher the accuracy. For instance, Liu et al. have designed a tellurite photonic crystal fiber dual-parameter gas sensor for methane and hydrogen with maximum sensitivities of 4.03 nm/% and -14.19 nm/% for methane and hydrogen, respectively [12].

In this work, in order to improve the level of integration, we use a coreless fiber as the basis, fabricate four planes along the cross-section direction by material reduction, plate them with gold, and coat sensing materials with low crosstalk and high specific recognition capability: cryptophane-A/ultraviolet-cured fluorosiloxane complex (Crypt-A/UVCFS), polyvinyl alcohol (PVA), Pd-WO₃ and zinc oxide (ZnO) to accomplish the simultaneous detection of methane, relative humidity, hydrogen and hydrogen sulfide. Numerical analysis shows that in the ranges of methane concentration of 0%-2%, relative humidity of 30%-70%, hydrogen concentration of 0%-2%, and hydrogen sulfide concentration of 0-80 ppm, the average methane sensitivity is -76

nm/%, the average relative humidity sensitivity is -2.2 nm/%RH, the average hydrogen sensitivity is -153 nm/%, and the average hydrogen sulfide sensitivity is 15 nm/ppm. This sensor, which solves the crosstalk problem plaguing complex gas sensing and accomplishes simultaneous multi-component detection at the same spatial point, has large practical potential.

2. Model and Theory

2.1. Model

Fig. 1(a) depicts the structure of the sensor with the following structural parameters: radius of the coreless fiber $r = 62.5$ μm , side-polished depth $l = 0.13r$, thickness of the sensitive material $D = 0.05r$, and thickness of the gold film $h = 40$ nm. The thickness of the ZnS film is x , and the thickness of the ZnO film is $D - x$. The modal characteristics of the sensor are analyzed by the finite element method, and the infinite space environment is simulated using a perfectly matched layer (PML).

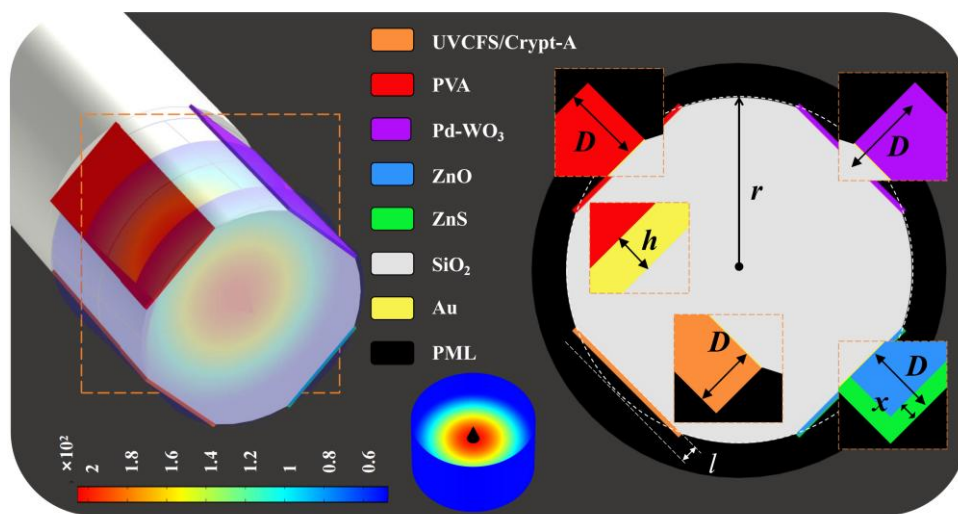


Fig. 1. Cross-sectional structure of the sensor.

The preparation process of the sensor is illustrated in Fig. 2. A femtosecond laser is used to side-polish the four sides of the coreless optical fiber [13], and magnetron sputtering is performed to deposit a gold film on each of the four planes [14]. The sol-gel method (SGM) is used to coat the four planes with uniformly textured UVCFS/Crypt-A, PVA, Pd-WO₃, and ZnO thin films, respectively [15], [16].

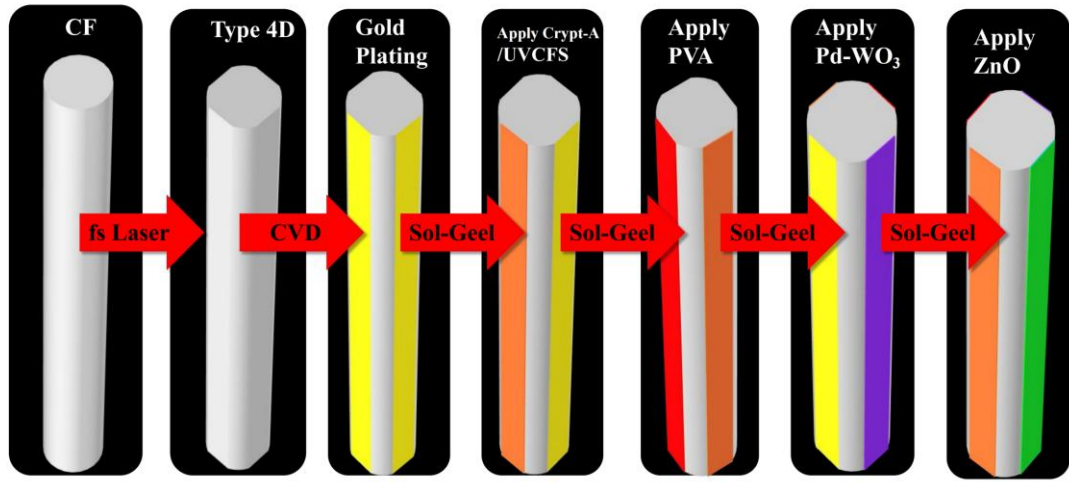


Fig. 2. Preparation process of the sensor.

2.2. Theory

The bulk material of the coreless optical fiber is fused silica, and its refractive index (n) is expressed by the Sellmeier equation:

$$n^2(\lambda, T) = 1.31552 + A_1 T + \frac{(B_1 + B_2 T) \lambda^2}{\lambda^2 - (B_3 + B_4 T)} + \frac{(C_1 + C_2 T) \lambda^2}{\lambda^2 - C_3}, \quad (1)$$

where λ is the wavelength of the incident light, T is the ambient temperature, $A_1 = 6.90754 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$, $B_1 = 0.788404$, $B_2 = 2.35835 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$, $B_3 = 0.0110199 \text{ } \mu\text{m}^2$, $B_4 =$

$0.584758 \times 10^{-6} \text{ } ^\circ\text{C}^{-1} \cdot \mu\text{m}^2$, $C_1 = 0.91316$, and $C_2 = 0.548368 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$, $C_3 = 100 \mu\text{m}^2$. The dielectric constant of gold is derived by the Drude-Lorentz model[17]:

$$\varepsilon_{\text{Au}}(\omega) = \varepsilon_1 + i\varepsilon_2 = \varepsilon_\infty - \frac{\omega_p^2}{\omega(\omega + i\omega_c)}, \quad (2)$$

where ε_1 and ε_2 are the real and imaginary parts of the metal dielectric constant, respectively, $\varepsilon_\infty = 9.48$ is the dielectric constant of gold at high frequencies, ω is the angular frequency of the incident light, $\omega_p = 1.36 \times 10^{16} \text{ rad/s}$ represents the angular frequency of the plasma, and $\omega_c = 1.45 \times 10^{14} \text{ rad/s}$ represents the damping frequency.

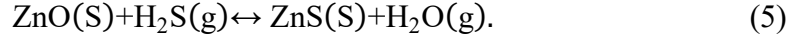
The UVCFS/Crypt-A thin film is a porous material that can achieve rapid physical adsorption of methane. In the concentration range of 0%-2%, the refractive index varies linearly with the methane concentration:

$$n_{\text{UVCFS/Crypt-A}} = 1.4478 - 0.0038c(\text{CH}_4). \quad (3)$$

The relationship between the refractive index of PVA and relative humidity in the range of 10%RH to 90%RH is described by the following equation:

$$n_{\text{PVA}} = -3 \times 10^{-6} RH^2 - 10^{-4} RH + 1.4738. \quad (4)$$

The zinc oxide thin film exhibits high sensitivity to hydrogen sulfide. At room temperature, the following reversible reaction occurs between H_2S and ZnO :



There is a large difference between the refractive indexes of hydrogen sulfide and zinc oxide, as shown below [18]:

$$n_{\text{ZnO}}^2=2.81418+\frac{0.87968\lambda^2}{\lambda^2-0.3042^2}-0.00711\lambda^2, \quad (6)$$

$$n_{\text{ZnS}}^2=8.393+\frac{0.14383}{\lambda^2-0.2421^2}+\frac{4430.99}{\lambda^2-36.71^2}. \quad (7)$$

At room temperature, the reaction between hydrogen sulfide and zinc oxide depends on the degree of diffusion. As shown in Fig. 3(a), in the concentration range of 0-100 ppm, the relationship between the reaction degree and hydrogen concentration is shown in the following [19]:

$$\frac{x}{D}=1-0.75c(\text{H}_2). \quad (8)$$

As shown in Fig. 3(a), as the concentration of hydrogen sulfide increases, x/D also increases, which in turn changes n near the gold film, eventually causing the resonance wavelength (RW) to shift[20].

The Pd-WO₃ thin film exhibits extremely high detection selectivity for hydrogen. As shown in Fig. 3(b), H₂ dissociates and adsorbs as hydrogen atoms on the Pd surface, and H⁺ and e⁻ are embedded together into the lattice of WO₃. When the concentration of hydrogen sulfide decreases, the diffusion degree decreases, allowing oxygen to enter,

and H_XWO_3 will be oxidized and converted back to WO_3 . In the concentration range of 0%-2%, the relationship between the refractive index of the hydrogen-sensitive thin film and the hydrogen concentration is shown below [21]:

$$n_{Pd-WO_3} = 1.995 - 0.00067 c(H_2). \quad (9)$$

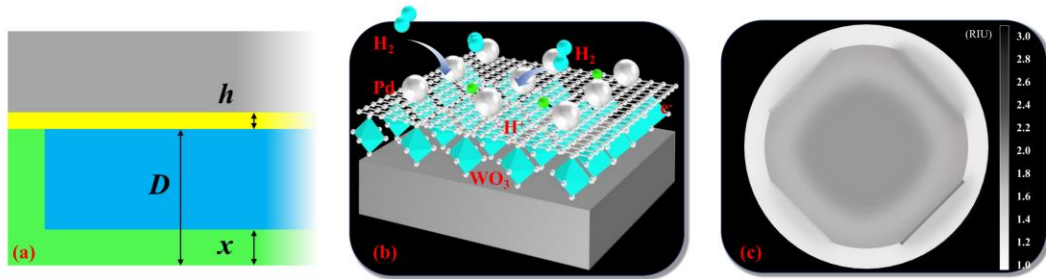


Fig. 3. (a) Structure of the hydrogen-sensitive film; (b) Hydrogen sensing mechanism on the Pd- WO_3 surface; (c) Refractive index distribution of the sensor cross-section.

Although the refractive index of the sensing film is greater than that of the optical fiber, the film thickness is small, so the ambient gas environment reduces the refractive index distribution outside the gold film. As shown in Fig. 3(c), the sensor still meets the total reflection condition and can confine light within the fiber core, as shown in Fig. 4. When the fundamental mode of the optical fiber matches the phase of the surface plasmon polariton (SPP) mode near the metal film, resonance coupling occurs, and a resonance peak appears in the loss spectrum of the sensor. When the gas concentration changes, n of the sensing material changes, in turn shifting the SPR resonance peak. By calculating the wavelength shift, an accurate measurement of the target parameter can be achieved. The calculation formula of light wave loss is as follows [22].

$$\alpha_{\text{loss}} = 8.686 \times \frac{2\pi}{\lambda} \text{Im}(n_{\text{eff}}) \times 10^7 \text{ (dB/cm)}. \quad (10)$$

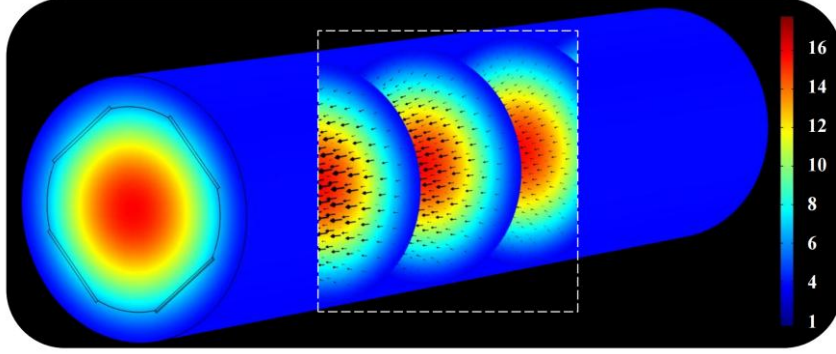


Fig. 4. 3D electric field distributions of y-pol FM.

The wavelength sensitivity is calculated by formula (13). Substituting the sensitivities of the four planes into the demodulation matrix (14), the accurate calculation of the contents of the four gases can be achieved [23].

$$S_i(X) = \frac{\Delta\lambda_{\text{peak}i}}{\Delta X} \quad \text{and} \quad (11)$$

$$\begin{bmatrix} \Delta RW_1 \\ \Delta RW_2 \\ \Delta RW_3 \\ \Delta RW_4 \end{bmatrix} = \begin{bmatrix} S_1(\text{CH}_4) & S_1(RH) & S_1(\text{H}_2) & S_1(\text{H}_2\text{S}) \\ S_2(\text{CH}_4) & S_2(RH) & S_2(\text{H}_2) & S_2(\text{H}_2\text{S}) \\ S_3(\text{CH}_4) & S_3(RH) & S_3(\text{H}_2) & S_3(\text{H}_2\text{S}) \\ S_4(\text{CH}_4) & S_4(RH) & S_4(\text{H}_2) & S_4(\text{H}_2\text{S}) \end{bmatrix} \begin{bmatrix} \Delta c(\text{CH}_4) \\ \Delta RH \\ \Delta c(\text{H}_2) \\ \Delta c(\text{H}_2\text{S}) \end{bmatrix}, \quad (12)$$

where $S_i(X)$ represents the sensitivity of the i^{th} ($i = 1, 2, 3, 4$) loss peak with respect to X (X representing $c(\text{CH}_4)$, $c(\text{H}_2)$, or $c(\text{H}_2\text{S})$ respectively) and ΔRW_i denotes the offset of the i^{th} loss peak. The sharpness of the loss peak, an important metric to evaluate the optimization degree of the sensor, is calculated as follows [24]:

$$SHP = \frac{Loss}{FWHM} \text{ (dB}/\mu\text{m}^2\text{)}, \quad (13)$$

where SHP is the sharpness of the loss peak and $FWHM$ represents the full-width at

half-maximum of the loss peak. Since the y-polarized fundamental mode (y-pol FM) in the fiber core can better couple with the SPP in the gold film, the y-pol FM is analyzed to derive the sensitivity.

Fig. 5(a) shows the spectral characteristics of the sensor when $c(\text{CH}_4) = 0.5\%$, $RH = 40\%$, $c(\text{H}_2) = 0.5\%$, and $c(\text{H}_2\text{S}) = 60$ ppm, showing the real part of the effective refractive index of the SPP mode ($\text{Re}(n_{\text{eff}}^{\text{SPP } i})$, $i=1,2,3,4$), the real part of the effective refractive index of the y-pol FM ($\text{Re}(n_{\text{eff}}^{\text{y-pol FM}})$), and the variation of loss with wavelength. The curves of $\text{Re}(n_{\text{eff}}^{\text{y-pol FM}})$ and $\text{Re}(n_{\text{eff}}^{\text{SPP } i})$ intersect at the four loss peaks, indicating that the phase matching condition is satisfied at these four resonant wavelengths (RW_i , $i = 1, 2, 3$). Fig.5(b) shows the electric field patterns of the y-pol FM and the SPP mode at the RW , and obvious coupling between the two is observed [25].

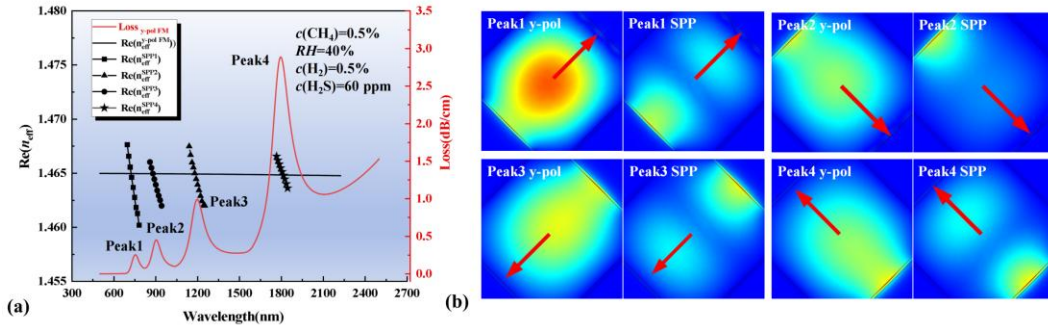


Fig. 5. (a) Loss spectra of the y-pol FM and $\text{Re}(n_{\text{eff}})$ for y-pol FM and SPP and (b) Electric field distributions of y-pol FM and SPP.

3. Structure Optimization

To improve the sensing properties, the total sharpness of resonance peaks (S_{SUM}), total spacing (T_{SP}), and total loss (S_{loss}) are evaluated, and the structural parameters of

the sensor (l , D , and h) are optimized. Specifically, $S_{\text{SUM}} = \sum_{i=1}^3 S_{\text{HP}i}$, where $S_{\text{HP}i}$ is the sharpness of the i^{th} resonance peak; $TSP = RW4 - RW1$; $S_{\text{loss}} = \sum_{i=1}^4 S_{\text{loss}i}$. Larger S_{SUM} and T_{SP} and smaller S_{loss} translate into higher resolution, a wider detection range, and higher transmission efficiency, respectively [26].

3.1 Side-Throw Depth Optimization

As shown in Fig. 6, the optimization range of the side-polishing depth l is $0.1r$ - $0.25r$ μm . When $l < 0.1r$, the loss spectrum no longer exhibits 4 distinct peaks. When $l > 0.25r$, the 4 side-polished planes intersect. Fig. 6(a) shows that as l increases, the loss spectrum does not shift significantly, but the loss increases. This is because l does not affect the sector-shaped distribution of the cross-sectional refractive index, but instead reduces the coupling distance and increases the loss. Figs. 6(b) and (c) also show that when $l = 0.16r$, the loss peaks have relatively high sharpness, spacing, and low loss. Therefore, $l = 0.16r$ is selected as the optimal value.

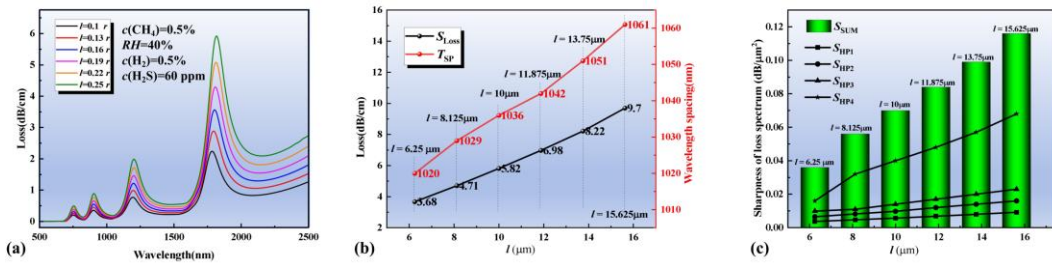


Fig. 6. (a) Loss spectra of y-pol FM, (b) S_{Loss} and S_{SUM} , and (c) T_{SP} of the three loss peaks for l between $0.1r$ and $0.25r$.

3.2 Optimization of Film Thickness

As shown in Fig.7, the optimization range of the gold film thickness h is 30-60 nm.

When $h < 25$ nm, uniform deposition is difficult. When $h > 50$ nm, the loss spectrum no longer has an obvious loss peak. Fig.7(a) shows that as h increases, the loss spectrum redshifts, because a thicker gold film reduces n_{eff} of the SPP mode, in turn decreasing n_{eff} of the matched y-pol FM. According to $n = c/v$ and $\lambda = v/f$, it can be concluded that λ increases. When $h = 35$ nm, the sharpness of the loss peak is the highest, and the loss is relatively low, so $h = 35$ nm is selected.

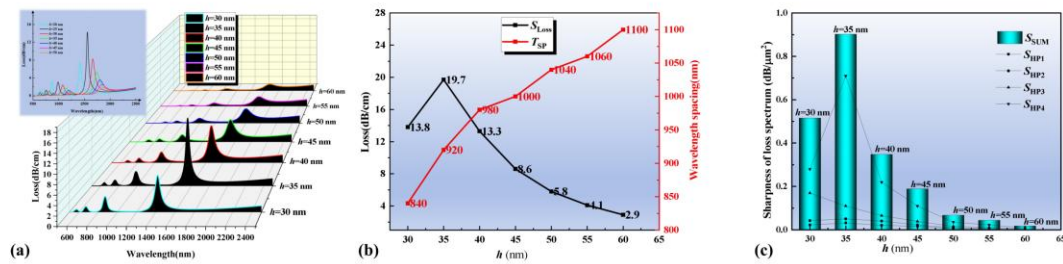


Fig. 7. (a) Loss spectra of y-pol FM, (d) S_{Loss} and S_{SUM} , and (c) T_{SP} of the three loss peaks for h between 30 nm and 60 nm.

3.3 Optimization of Sensing Material Thickness

As shown in Fig. 8, the optimization range of the sensing film thickness D is $0.03r - 0.13r$. When $D < 0.03r$, the loss peak is not obvious. When $D > 0.13r$, the sensing layer is prone to peeling off. Fig. 9(a) shows that as D increases, the peak spacing increases and stabilizes, while the loss and sharpness show a fluctuating change of increase-decrease-increase. In order that the peak spacing and sharpness are maintained at relatively high values, when D is $0.11r$ and the loss is low, $D = 0.11r$ is selected.

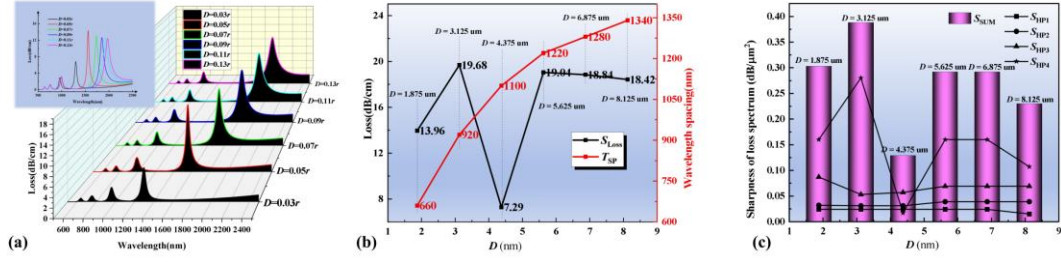


Fig. 8. (a) Loss spectra of y-pol FM, (d) S_{LOSS} and S_{SUM} , and (c) T_{SP} of the three loss peaks for D between $0.03r$ and $0.13r$.

4. Results

The characteristics of the sensor can be determined using the experimental platform shown in Fig. 9. Light from a supercontinuum light source is transmitted to the coupler through a single-mode fiber. One end of the coupler is connected to the sensor, and the other end is connected to the spectrometer. After the optical signal is converted into an electrical signal, it is transmitted to the computer for analysis, and the sensor is placed in the control box.

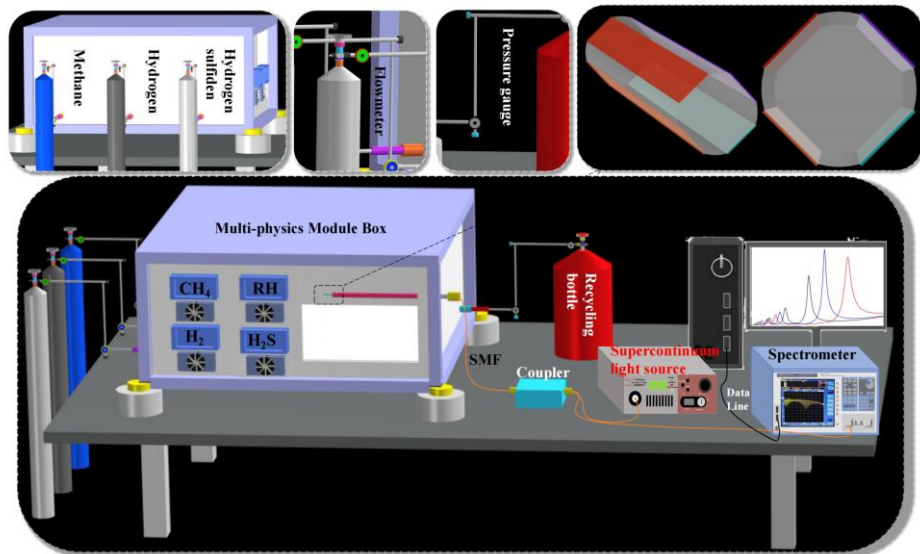


Fig. 9. Experimental platform.

Fig.10 shows the sensitivity of the optimized sensor. As shown in Fig.10(a), when the methane concentration increases from 0% to 2%, Peak1 blueshifts. Linear fitting shows that the average methane sensitivities of each peak are $-76 \text{ nm/\%}$, $-3.4 \text{ nm/\%}$, $-3.8 \text{ nm/\%}$, and $-5.6 \text{ nm/\%}$, respectively. As shown in Fig. 10(b), when the relative humidity increases from 30% to 70%, Peak2 blueshifts as well, and linear fitting reveals average relative humidity sensitivities of $0.11 \text{ nm/\%RH}$, $-2.2 \text{ nm/\%RH}$, $0.21 \text{ nm/\%RH}$, and $-0.18 \text{ nm/\%RH}$, respectively. As shown in Fig.10(c), when the hydrogen concentration changes uniformly within the range of 0%-2%, Peak3 blueshifts, and the average hydrogen sensitivities are $2.1 \text{ nm/\%}$, $4.2 \text{ nm/\%}$, $-153 \text{ nm/\%}$, and $-4.6 \text{ nm/\%}$, respectively. Fig.10(d) shows that when the hydrogen sulfide concentration increases from 0 to 80 ppm, Peak4 redshifts, and the average hydrogen sulfide sensitivities are determined to be 0.025 nm/ppm , 0.051 nm/ppm , 0.061 nm/ppm , and 15 nm/ppm , respectively. Substituting the above sensitivity coefficients into matrix (12) and performing an inverse operation, the variations of methane, relative humidity, hydrogen, and hydrogen sulfide can be demodulated.

$$\begin{bmatrix} \Delta c(\text{CH}_4) \\ \Delta RH \\ \Delta c(\text{H}_2) \\ \Delta c(\text{H}_2\text{S}) \end{bmatrix} = \begin{bmatrix} -76 \text{ nm/\%} & 0.11 \text{ nm/\%RH} & 2.1 \text{ nm/\%} & 0.025 \text{ nm/ppm} \\ -3.4 \text{ nm/\%} & -2.2 \text{ nm/\%RH} & 4.2 \text{ nm/\%} & 0.051 \text{ nm/ppm} \\ -3.8 \text{ nm/\%} & 0.21 \text{ nm/\%RH} & -153 \text{ nm/\%} & 0.061 \text{ nm/ppm} \\ -5.6 \text{ nm/\%} & -0.18 \text{ nm/\%RH} & -4.6 \text{ nm/\%} & 15 \text{ nm/ppm} \end{bmatrix}^{-1} \begin{bmatrix} \Delta RW_1 \\ \Delta RW_2 \\ \Delta RW_3 \\ \Delta RW_4 \end{bmatrix}$$

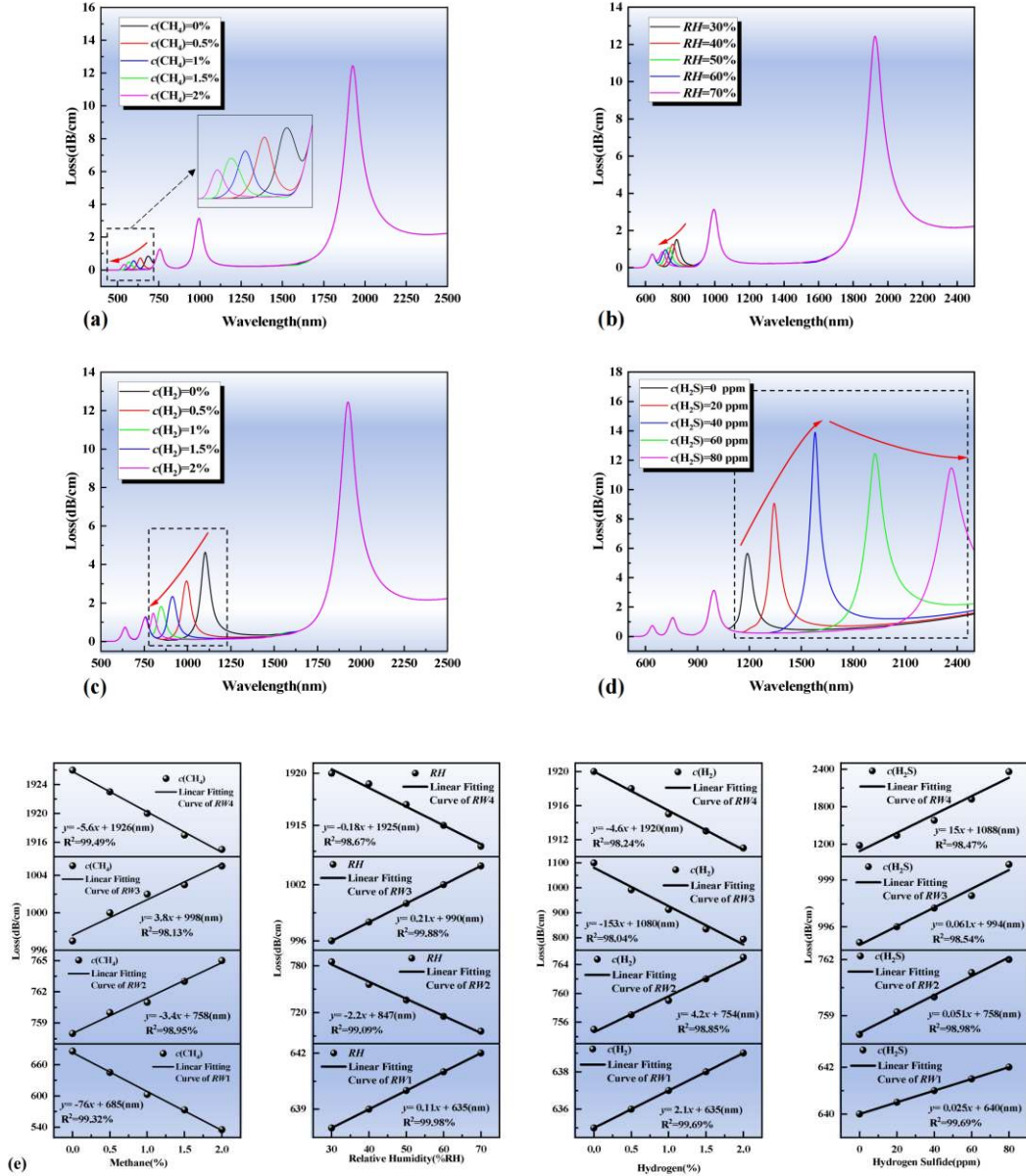


Fig. 10. (a)-(d) Loss spectra of the sensor and linear fitting curves of RW when the concentration of a single gas changes; (e) Linear fitting curve of RW.

As shown in Fig.11, the changes in the loss spectrum are simulated when the four quantities increase simultaneously. The four loss peaks shift at the same time, verifying that the sensor can function in a complex gas environment for simultaneous detection of multiple gases.

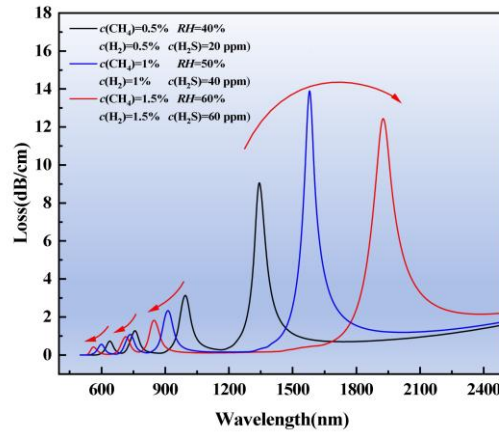


Fig. 11. Changes in the sensor loss spectrum when the methane concentration, relative humidity, hydrogen concentration, and hydrogen sulfide concentration change simultaneously.

5. Discussion

Table 1 compares the sensing characteristics of different SPR gas sensors. It is noted that our sensor is capable of detecting 4 parameters simultaneously while delivering high sensitivity.

Table1. Comparison of the sensing properties of different optical fiber SPR gas sensors.

Refs	CH ₄	RH	H ₂	H ₂ S
[9]	0%-3.5%	/	/	/
	6.6nm/%			
[11]	/	46% - 93%	/	/
		1.542 nm/%RH		
[27]	/	/	/	0-100ppm
				13nm/ppm

[12]	0%-2%	/	/	0%-2%
	4.03 nm/%			-14.19 nm/%RH
[This	0%-2%	30%RH-70%RH	0%-2%	0-80ppm
Work]	-76 nm/%	-2.2 nm/%RH	-153 nm/%	15 nm/ppm

During manufacturing, there will be inevitable deviations in the structural parameters. The sensitivity fluctuations are analyzed when the structural parameters l , h , and D are within the manufacturing error range of $\pm 3\%$. The average sensitivity fluctuation for all gases is less than 7% and in an acceptable range, leading to a high tolerance of manufacturing errors.

Table 2. Effects of manufacturing tolerance on sensitivity.

Param.	Tol.	$S_1(\text{CH}_4)$	$S_2(\text{RH})$	$S_3(\text{H}_2)$	$S_4(\text{H}_2\text{S})$
D	-3%	-75 nm/%	-2.1 nm/%RH	-150nm/%	-15 nm/%
	0	-76 nm/%	-2.2 nm/%RH	-153nm/%	-15 nm/%
	+3%	-78 nm/%	-2.3 nm/%RH	-155nm/%	-15 nm/%
h	-3%	-78 nm/%	-2.4 nm/%RH	-156nm/%	-16 nm/%
	0	-76 nm/%	-2.2 nm/%RH	-153nm/%	-15 nm/%
	+3%	-74 nm/%	-2.1 nm/%RH	-151nm/%	-15 nm/%
l	-3%	-77 nm/%	-2.3 nm/%RH	-153nm/%	-15 nm/%
	0	-76 nm/%	-2.2 nm/%RH	-153nm/%	-15 nm/%

+3%	-75 nm/%	-2.1 nm/%RH	-153nm/%	-14 nm/%
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The preparation process of the four-parameter optical fiber gas sensor shown in Fig.2 is quite mature. The sensor uses gold as the plasmonic material, which has excellent oxidation resistance. Meanwhile, the four sensitive materials are reversible in both physical adsorption and chemical reactions, making the sensor highly reusable.

5. Conclusion

A four-parameter optical fiber gas sensor that can simultaneously detect methane, relative humidity, hydrogen, and hydrogen sulfide is designed and analyzed. Numerical analysis shows that in the ranges of methane concentration of 0%-2%, relative humidity of 30%-70%, hydrogen concentration of 0%-2%, and hydrogen sulfide concentration of 0-80 ppm, the average methane sensitivity, humidity sensitivity, hydrogen sensitivity, and hydrogen sulfide sensitivity are -76 nm/%, -2.2 nm/%, -153 nm/%, and -15 nm/ppm, respectively. In addition, the sensor has excellent stability, showing an average sensitivity fluctuation of less than 7%, which is within the typical manufacturing tolerance of $\pm 3\%$. The ability to detect these four parameters at the same time has seldom been achieved, and this sensor is a technological breakthrough with high practical potential.

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