An optical fiber methane gas sensing film sensor based on core diameter mismatch

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An optical fiber evanescent wave methane gas sensor based on core diameter mismatch is reported. The sensor consists of a multimode fiber in which a short section of standard single-mode fiber, coated with the inclusion of cryptophane molecules E in a transparent polysiloxane film, is inserted. The sensing principle is analyzed by optical waveguide theory. For different sensing film thicknesses and interaction lengths, the sensor signal is investigated within the methane concentration range of 0−14.5% (v/v). It is shown that the sensor signal with the thickness of 5 μm and the interaction length of 3 mm strengthens linearly with the increasing concentration of methane, with a slope of 0.0186. The best detection limit of the sensor for methane is 2.2% (v/v) with a response time of 90 s. This sensor is suitable for the detection of methane concentration below the critical value of 5%.

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Gas accident is always the main threat to safe production in coal mine, which severely restricts the development of national economics. Thus, it is necessary to prevent and control gas accident. So far, monitoring the concentration of methane gas, which is the most important component in coal mine, is an effective approach.

Recently, several techniques have been developed for methane detection including catalytic combustion[1], semiconductor detectors[2], electrochemistry[3], optical methods[4,5], etc. Among the optical methods, optical fiber sensors based on evanescent wave are especially suitable for detecting methane gas in coal mine, because the sensors possess the advantages of safe operation in hazardous environments, immunity to electromagnetic field interference, remote monitoring, distributed measurement, small size, and light weight[6].

A tapered optical fiber evanescent wave methane sensor of a small diameter ranging from 1.8 to 7.0 μm has been investigated[7], whose minimum detectable concentration of methane is less than the 5% lowest explosive limit. A D-fiber evanescent wave methane sensor was proposed[8], with a resolution in the region of 1000 ppm methane. Wu *et al.* reported an optical fiber sensor based on mode-filtered light for methane gas[9], for which the detection limit is 0.06% (v/v) with cryptophane A, and the response time is about 5 min. However, these methods need special equipment to fabricate the sensor, and have the disadvantages of high cost in fabrication and difficulty in operation. The D-fiber sensor has high background signal levels from interference effects, system degradation through surface contamination, low sensitivity and selectivity[10]. To improve the selectivity of the sensor, an optical fiber evanescent wave methane gas sensor based on polymer-clad silica (PCS) fibers was proposed[11], which was covered with the inclusion of cryptophane E in a transparent polysiloxane film. The sensor shows a high selectivity for the complexation process, and its detection limit for methane is 6% (v/v) with cryptophane E. However, the PCS fiber is expensive, high loss in transmission, and cannot match along with conventional step or graded-index optical fiber. To overcome these shortages, a new optical fiber evanescent wave methane sensor based on core diameter mismatch was developed with refractive index change of sensing film[12].

Figure 1 presents the schematic diagram of optical fiber methane gas sensor based on core diameter mismatch. A piece of single-mode fiber (SMF) of length $L_{\rm SM}$ was longitudinally sandwiched between two multi-mode fibers (MMFs). The core diameters of SMF (SMF-28, Corning) and MMFs (G.651.1, YOFC) were 9 and 62.5 μm, respectively. The outer diameters of all fibers were 125 μm. To fabricate the structure, one only requires cleaving and fusion splicing the fibers, which can be easily carried out with widespread fiber optical equipments. To realize methane sensing, the section of SMF was coated with the inclusion of cryptophane E in a transparent polysiloxane film. Several samples with different interaction lengths $L_{\rm SM}$ ranging from 2 to 4 mm were fabricated for methane sensing experiments.

According to geometrical optics, the incident angle θ_1 can be related to the oblique angle α_0. At the interface of air/MMF core, $n_{\rm a}\sin\alpha_0=n_3\sin\beta_1$, where $n_{\rm a}$ is the refractive index of air, and n_3 is the refractive index of the MMF core.

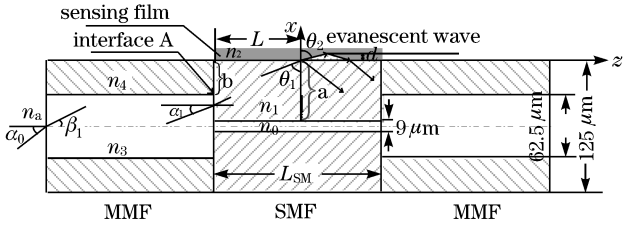

Fig. 1. Schematic diagram of optical fiber methane gas sensor based on core diameter mismatch.

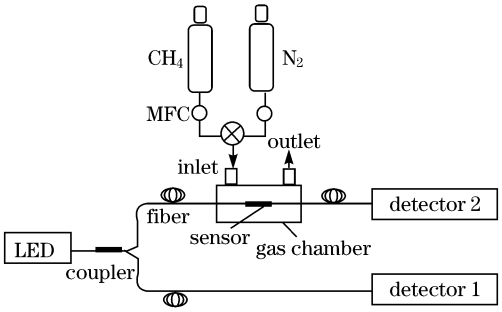

Fig. 2. Experimental setup. LED: light emitting diode.

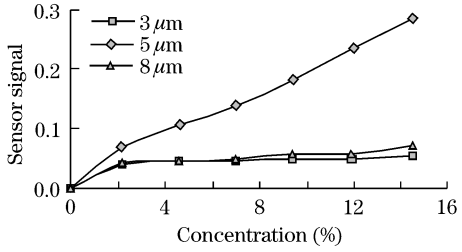

Fig. 3. Sensor signal versus methance gas concentration for different thicknesses of sensing film.

When the light transmitted through the interface A with an oblique angle α_1 to refract into the SMF cladding and transmit to the interface between the SMF cladding and the sensing film, where the incident angle θ_1 changes from $\arctan(L/a)$ to $\arctan(L/b)$. θ_2 is the refraction angle in the medium with the refractive index of n_2, θ_2=arcsin$[(n_1/n_2)\cdot\sin\theta_1]$. As the light transmits along the multimode optical fiber, the critical angle θ_c^{MM}=arcsin(n_4/n_3).

At the interface of SMF cladding/sensing film, the electromagnetic wave is not influenced by the transmitted light along y, z orientation, but the transmitted wave's amplitude decreases very rapidly with the depth (x) orientation of penetration, and its attenuation degree shows a close correlation with the change of refractive index of the sensing film. It can be explained by utilizing the effective penetration depth on the basis of[13]

$$d = \frac{\lambda_1}{2\pi\sqrt{\sin^2\theta_1 - \left(\frac{n_2}{n_1}\right)^2}}, \quad (1)$$

where λ_1 is the wavelength in the SMF cladding, n_1 is the refractive index of the SMF cladding, n_2 is the refractive index of polymer sensing film, d is the evanescent wave penetration depth, and θ_1 is the incident angle of the ray with respect to the normal to the cladding/sensing film interface in the sensing region.

When the sensing film is exposed to methane gas, its refractive index n_2 will drop with the increase of methane gas concentration. According to Eq. (1), the evanescent wave penetration depth d varies with the change of refractive index n_2, further affecting the output light power of the sensor. When the penetration depth d is larger, light transmission loss is higher, and the corresponding output light power of the sensor is weaker. When d is smaller, the output light power is stronger. Therefore, only by observing the output light power of the sensor, the concentration of methane gas can be obtained.

The experimental setup used to test the sensors consisting of two independent parts is shown in Fig. 2. One part was the gas flow control system, which included a gas chamber constructed with a piece of stainless steel tube, an inlet and an outlet that allow the gas to flow in and out. Two mass flow controllers (MFCs) that were individually controlled from an electronic unit were employed to precisely control the flow of methane and nitrogen gas. The other part was the optical system, which included a low-power light emitting diode (LED), centered at 1310 nm with 30-nm spectral width, a 3-dB coupler, graded-index MMF with 62.5-μm core diameter, and two detectors (AQ2140 optical multimeter, Ando). By a high-precision fiber cleaver (FC-6S, Sumitomo Electric Ltd.) and a fusion splicer machine (TYPE-39, Sumitomo Electric Ltd.), the samples with interaction length L_{SM} of 2−4 mm were spliced to one arm of the fiber coupler, respectively. And the other arm of the coupler was spliced to several meters of un-coated graded-index MMF as a reference to compensate possible fluctuations of the LED.

Using the "dip-coating" technique at a withdrawal speed of 12 cm/min, the polymer sensing film was deposited on the section of SMF. A homogeneous surface coating was observed by optical microscopy. Several deposition steps were performed, and the film thickness was from 3 to 8 μm and optimized to obtain the lower detection limit. The ratio of output light power transmitted by the sensor (P_T) to the power transmitted by the reference arm (P_R) is used to calculate the sensor signal $S = (P_T/P_R)\times 100$, where $P_T = \Delta P_{CH_4} = P_{CH_4} - P_{N_2}$, P_{CH_4} and P_{N_2} are output light powers of the sensor obtained at various concentrations of methane (CH_4) and pure nitrogen (N_2), respectively.

Figure 3 presents the response of the sensor with different sensing film thicknesses to various concentrations of methane gas. As is shown, the sensor signal strengthens with the increase of methane concentration basically. For the film thicknesses of about 3 and 8 μm, the sensor signal slowly rises to a stable value when methane gases of different concentrations are introduced to the sensor. Compared with the cases of 3 and 8 μm, the sensor with thickness of about 5 μm shows good response characteristic and high sensitivity for methane gas. So, the sensor with film thickness of 5 μm is used for further measurements.

At the same time, the interaction length also influences the response characteristic of the sensor, as shown in Fig. 4. For each interaction length, the sensor shows similar response trend to methane. When the length increases

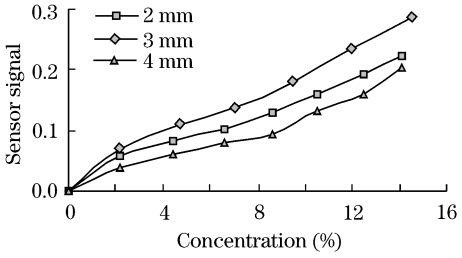

Fig. 4. Sensor signal versus methane gas concentration for different lengths of the sensing region.

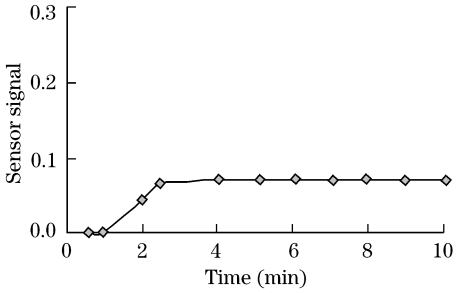

Fig. 5. Response curve of the sensor to methane gas.

from 2 to 4 mm, the sensitivity (the ratio of sensor signal transmitted per percent of methane) is 0.0186 for 3 mm, 0.0148 for 2 mm, and 0.0133 for 4 mm. Obviously, the sensor with interaction length of 3 mm shows the highest sensitivity in the methane concentration range of 0−14.5% (v/v), with a slope of 0.0186 and a correlation coefficient of 0.9903, which agrees with the theoretical analysis result[12,14]. The best detection limit of the sensor for methane is 2.2% (v/v) with cryptophane E.

Figure 5 further presents the response curve of the sensor for methane gas. As is shown, the sensor signal is almost zero at pure nitrogen. When the sensor is exposed to 2.2% (v/v) methane gas, the sensor signal increases rapidly and reaches a stable value with the response time of 90 s, superior to that of Ref. [11].

In conclusion, an optical fiber methane gas sensing film sensor based on core diameter mismatch is developed by coating a polymer film including cryptophane molecules E in the sensing region. For the film thickness of 5 μm and interaction length of 3 mm, the detection limit of the sensor is 2.2% (v/v), with the response time of 90 s. This sensor is sensitive, simple in structure, low cost in fabrication, and easy in operation.

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