## Highly sensitive optical hydrogen sensor using circular Pd-coated singlemode tapered fibre

## J. Villatoro, A. Díez, J.L. Cruz and M.V. Andrés

A novel optical hydrogen sensor, based on the absorption change of the evanescent fields in a circular Pd-coated singlemode tapered fibre is presented. The proposed sensor is polarisation independent and its sensitivity is adjustable by means of the taper diameter, interaction length, and/or light wavelength. A simple light transmission measurement setup is used to test the sensor. The sensor is suitable for the detection of low hydrogen concentrations with high sensitivity and fast time response. Transmission changes as high as 60% are demonstrated.

*Introduction:* In the past few decades, hydrogen has attracted much attention in the scientific community owing to its superior properties and characteristics as a carrier energy over conventional fuels. However, liquid or gaseous hydrogen is very volatile, extremely flammable, and highly explosive, e.g. a gaseous hydrogen leakage in air greater than 4% at room temperature and normal pressure leads to an explosive atmosphere which is easily ignitable. Therefore, the development of sensors for detecting hydrogen concentrations below such explosive limit is very important. To date, several optical hydrogen sensors based on different working principles have been reported [1 - 6]. Optical sensors seem to be the most appropriate sensors in dangerous atmospheres owing to their lack of sparking possibilities and high sensitivity.

In this Letter, we present a new optical hydrogen sensor based on the absorption change of the evanescent fields in a Pd-coated singlemode tapered fibre. This sensor is polarisation independent and its sensitivity can be adjusted by means of the taper diameter and the interaction length, which can be easily tailored during the taper fabrication process [7], and by the light wavelength. Hydrogen concentrations below the lower explosive limit were detected with high sensitivity and fast time response.



Fig. 1 Schematic diagram of sensor structure and experimental setup

*a* Schematic diagram of sensor structure.  $\phi$  is taper diameter and  $L_{in}$  is interaction length *b* Experimental setup

SMF: singlemode fibre

*Basis:* In a singlemode tapered fibre, the reduction of the core and cladding diameters causes the evanescent fields to spread out across the cladding and to reach the outer air-cladding boundary (see Fig. 1*a*). When the taper waist is coated with a Pd layer, the propagation constant of the fundamental mode is modified owing to the interaction with the layer. Since the palladium refractive index is complex, the fundamental mode of the structure exhibits an attenuation coefficient  $\gamma$  different from zero.

The sensor behaviour under the presence of hydrogen is found by taking into account the well-known properties of a palladium thin film when it is exposed to hydrogen [8]. Palladium has the ability to form hydride, the optical properties of which are different than those of an H<sub>2</sub>-free Pd film. Formation of hydride leads to a decrease in both the real and imaginary parts of the palladium complex permittivity. Consequently, the attenuation coefficient of the fundamental mode of the structure decreases, therefore the transmission of the device increases. The transmitted power, when the sensor is exposed to hydrogen, may be expressed as  $I = I_0 \exp[2\Delta\gamma L_{in}]$ , where  $\Delta\gamma$  is the change of the attenuation coefficient,  $L_{in}$  the interaction length, and  $I_0$  the transmitted power when no hydrogen is present. A standard boundary-value method was used to calculate  $\Delta\gamma$  for various hydrogen concentrations. As expected, the theoretical results indicate that the relative transmission of the sensor,  $I/I_0$ , increases with increasing hydrogen concentration. Furthermore, for a given hydrogen concentration  $I/I_0$  increases with wavelength and with the reduction of the taper diameter.



Fig. 2 Transmission spectra for 2%  $H_2$  concentration with TM-, TE- and unpolarised light

Sensor parameters:  $\phi = 25 \,\mu\text{m}$ ,  $L_{in} = 15 \,\text{mm}$ , and  $12 \,\text{nm}$  Pd layer ...... TM-polarised light

– – TE-polarised light



Fig. 3 Relative transmission against time for different hydrogen concentrations

Sensor parameters:  $\phi = 20 \,\mu\text{m}$ ,  $L_{in} = 15 \,\text{mm}$ , and  $12 \,\text{nm}$  Pd layer

*Experimental procedure and results:* A schematic representation of the sensor structure is shown in Fig. 1*a*. A standard telecommunications fibre was tapered adiabatically to a uniform diameter over a length of several millimetres. The introduction of the taper was performed by using the travelling-burning technique [7]. The losses of the tapered fibres were typically < 0.1 dB. Three palladium depositions, each 12 nm thick, were evaporated in high vacuum onto the taper uniform waist, rotating the fibre 120° between two consecutive depositions. This procedure gives quasi-circular coatings the thickness of which is close to uniformity and makes the device polarisation insensitive [9].

The sensor was tested using the experimental setup shown in Fig. 1*b*. All the measurements were carried out under normal conditions. A PVC tube was used as a gas cell wherein a homogeneous mixture of hydrogen and nitrogen (used as a carrier gas) was flowing. Both hydrogen and nitrogen flow rates were individually controlled by using high-precision accucal flow meters. Two kinds of measurements were carried out. First, the power transmission spectra were recorded for a fixed hydrogen concentration with TE-, TM- and unpolarised light. Secondly, the transmitted power was measured for different hydrogen concentrations using an LED. The results are shown in Figs. 2 and 3, respectively. From Fig. 2 it is clear that similar results are obtained no matter the polarisation of the light, which confirms the light polarisation insensitivity of our device. As indicated by the theoretical calculations, the sensitivity of the sensor increases with wavelength. It is worth noting that the biggest

transmission changes occur at wavelengths around  $1.55\,\mu m$ , at which inexpensive semiconductor laser diodes or LEDs are widely available.



Fig. 4 Calibration curve of the sensor described in Fig. 3
experimental data

– lower explosive limit

Results shown in Fig. 3 were obtained with an LED, peak wavelength of 1600 nm and  $3\,\mu$ W of optical power, and a photodetector. Note that the sensor absorbs and desorbs hydrogen very rapidly. When the hydrogen flow is brought back to zero, the relative transmission reaches the baseline, which reflects the reversibility of the sensor. Fig. 4 shows the calibration curve of the sensor described in Fig. 3. A maximum relative transmission increment of nearly 60% is obtained for a 10% hydrogen concentration. Note the dramatic changes of the relative transmission for hydrogen concentrations below the lower explosive limit.

*Conclusions:* A novel optical hydrogen sensor using a circular Pdcoated singlemode tapered fibre is presented. The sensor is based on the absorption change of the evanescent fields in the palladium-coated tapered waist. The proposed sensor is polarisation independent and its sensitivity can be adjusted by the interaction length, taper waist diameter, and the light wavelength. It is shown that with a low-power LED and a single photodetector it is possible to detect hydrogen concentrations below the lower explosive limit with very high sensitivity and fast time response. A further study of the proposed sensor will be reported at a future date.

*Acknowledgments:* J. Villatoro is grateful to Conacyt (Mexico) for a post-doctoral fellowship. The authors acknowledge the financial support of the Comisión Interministerial de Ciencia y Tecnologia (Grant 1FD97-0684).

11 June 2001

© IEE 2001 Electronics Letters Online No: 20010716 DOI: 10.1049/el:20010716

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## References

- 1 BUTLER, M.A., and GINLEY, D.S.: 'Hydrogen sensing with palladium-coated optical fibers', J. Appl. Phys., 1988, 64, pp. 3706–3712
- 2 CHADWICK, B., TANN, J., BRUNGS, M., and GAL, M.: 'A hydrogen sensor based on the optical generation of surface plasmons in a palladium alloy', *Sens. Actuators B*, 1994, **17**, pp. 215–220
- 3 BUTLER, M.A.: 'Micromirror optical-fiber hydrogen sensor', *Sens. Actuators B*, 1994, **22**, pp. 155–163
- 4 TABIB-AZAR, M., SUTAPUN, B., PETRICK, R., and KAZEMI, A.: 'Highly sensitive hydrogen sensors using palladium coated fiber optics with exposed cores and evanescent field interactions', *Sens. Actuators B*, 1999, 56, pp. 158– 163
- 5 SEKIMOTO, S., NAKAGAWA, H., OKAZAKI, S., FUKUDA, K., ASAKURA, S., SHIGEMORI, T., and TAKAHASHI, S.: 'A fiber-optic evanescent-wave hydrogen gas sensor using palladium-supported tungsten oxide', *Sens. Actuators B*, 2000, **66**, pp. 142–145
- 6 TOBISKA, P., HUGON, O., TROUILLET, A., and GAGNAIRE, H.: 'An integrated optic hydrogen sensor based on SPR on palladium', *Sens. Actuators B*, 2001, **74**, pp. 168–172
- 7 KENNY, R.P., BIRKS, T.A., and OAKLEY, K.P.: 'Control of optical fiber taper shape', *Electron. Lett.*, 1991, **27**, pp. 1654–1656
- 8 WYRZYKOWSKI, K., RODZIK, A., and BARANOWSKI, B.: 'Optical transmission and reflection of PdHx thin films', J. Phys., Condens. Matter, 1989, 1, pp. 2269–2277
- 9 DIEZ, A., ANDRES, M.V., and CRUZ, J.L.: 'Hybrid surface plasma modes in circular metal-coated tapered fibres', *J. Opt. Soc. Am. A*, 1999, **16**, pp. 2978–2982