

Temperature-Compensated Hydrogen Sensing with In-Fiber Dual-FBG Fabry-Pérot Structures and Palladium-Based Thin Films

Anja Braunmüller¹, Fabian Buchfellner¹, Markus Becherer², Johannes Roths¹

¹*Photonics Lab, Munich University of Applied Sciences, Munich, 80335, Germany*

²*School of Computation, Information and Technology (CIT), Technical University of Munich, Munich, 80333, Germany*

anja.braunmueller@hm.edu (Anja Braunmüller), fbuchfel@hm.edu (Fabian Buchfellner), markus.becherer@tum.de (Markus Becherer), roths@hm.edu (Johannes Roths)

Abstract

Hydrogen sensors based on in-fiber dual-FBG Fabry-Pérot (FP) structures present an advanced sensor platform with the ability to achieve a highly precise detection of hydrogen and temperature. By selectively covering the cavity formed between two UV imprinted Bragg reflectors with a Pd80:Au20 nanofilm provides the sensor with the potential for simultaneous measurements of temperature and hydrogen concentration in a single optical fiber and sensor element. For this purpose, current efforts aim for decoupling the sensor response in regard of temperature and hydrogen-induced strain by applying a fully zero-point referenced sensor calibration scheme. This work addresses the fabrication of such a dual-FBG FP sensor under consideration of suitable design parameters and its subsequent calibration. For the latter, the determination and compensation of the intrinsic temperature response of the sensor will be demonstrated on the example of measurements of H₂ concentrations between 100 and 20,000 ppm at temperatures of 30°C and 50°C. This compensation substantially reduces the cross-sensitivity of the sensor regarding temperature- and hydrogen-induced wavelength shifts. Above all, however, the results obtained with this sensor configuration show hydrogen measurements free of hysteresis with the capability to extend for multi-point sensor probes by wavelength division multiplexing.

Keywords: Hydrogen sensing, optical fiber sensing, temperature compensation, palladium-based metal-hydride, fiber Bragg grating, Fabry-Pérot

Introduction

In recent years, hydrogen (H₂) and hence the research into suitable sensor technologies has significantly gained importance [1-3]. Considering the high flammability of hydrogen in particular [4], it is crucial that sensors are capable of reliably detecting the present H₂ concentration at high precision. Such sensors are required not only for detecting leakages, but for example also the hydrogen concentration in inert gases [5,6]. For industrial applications, however, they must comply with ISO 26142 [7], which specifies the technical requirements summarized in Tab. 1. Fiber-based sensor systems have proven to be particularly promising in this context, since these offer a number of advantages, including the possibility of multiplexing, immunity to electromagnetic interference and their usability in explosive environments due to galvanic insulation. This makes fiber Bragg gratings (FBGs) an especially powerful sensor technology, which is usually applied to enable the detection of parameters such as temperature and strain [8,9]. The use of functionalizing coatings makes

it possible to extend their range of applications for measuring further parameters such as the concentration of gaseous H₂ through Palladium (Pd) based thin films [10-13].

Tab 1: Requirements for H₂ sensors according to ISO 26142 [7]

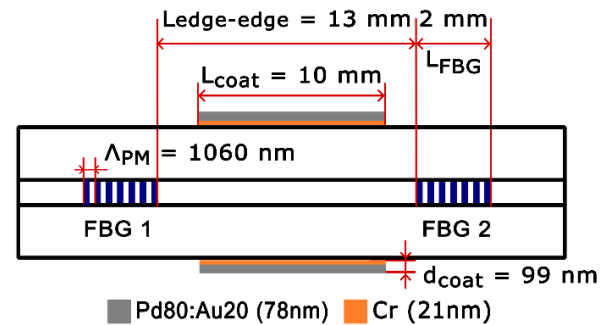
Parameter	Specification
Measurement Range	Up to 4 vol-%
Detection Limit	100 ppm
Accuracy	±50% (≤ 500 ppm) ±25% (≥ 500 ppm)
Repeatability	±10%
Time of Response	≤ 30 s
Time of Recovery	≤ 60 s
Temperature	- 20 °C to 50 °C
Pressure	80 kPa to 108 kPa
Humidity	20 % to 80 %

In 1991, Butler [14] first presented a hydrogen sensor based on a Pd-coated, fiber-optical Mach-Zehnder interferometer. Sutapun et al. [10] later transferred this concept to fiber Bragg gratings by coating a single FBG entirely with a thin layer of pure palladium. Therefore, the mechanical strain arising from the H₂ absorption in the Pd film created a measurable shift of the Bragg wavelength and hence a suitable sensor signal. Ever since, significant progress has been made in optimizing this sensor concept, for example in terms of sensitivity, response time, durability and fabrication feasibility [15].

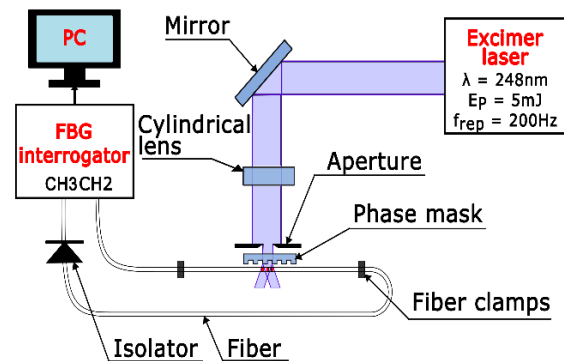
The in-fiber hydrogen sensor presented in this work offers a further improvement by employing an interferometric approach based on a Fabry-Pérot etalon. Applying Pd-coated dual-FBG Fabry-Pérot cavities, allows the suggested sensor platform to provide an increased sensor response and hydrogen measurements at higher resolution, while keeping the spectral selectivity provided by the FBGs. Apart from its potential regarding wavelength multiplexing, the key advantage of this arrangement lies in the possibility of dual parameter sensing [16,17]. An essential step to achieve this is the compensation of the intrinsic temperature sensitivity of the sensor as part of an appropriate sensor calibration, which is addressed in this work.

Sensor Fabrication

The sensor architecture of the fiber FP structures presented in this report is schematically shown in Fig. 1a including the manufacturing parameters. The sensor consists of two FBGs, which are created by photo imprinting a refractive index modulation into the core of a Germanium doped fused silica fiber according to the procedure described by Hill et al. [18]. Therefore, a single mode fiber with a diameter of 80 μm is positioned within the horizontal focal line of a KrF laser (248 nm) created with a cylindrical lens as shown in Fig. 1b. In addition, an adjustable aperture slit for setting the length of the inscribed FBG and a phase mask are placed in the beam path between cylindrical lens and photosensitive fiber. In this case, a phase mask with a pitch of $\Lambda_{\text{PM}} = 1060 \text{ nm}$ was used. The mask is the central component of the FBG fabrication setup. It creates a spatially modulated intensity pattern in the region, where the partial beams of the +1st and -1st diffraction order overlap. This leads to locations of high and low UV radiation, where the corresponding increase of the refractive index (RI) leads to the desired periodic RI modulation in the fiber's core.



(a) Schematic of the dual-FBG Fabry-Pérot based H₂ sensor



(b) Experimental setup for the FBG inscription with UV exposure through a phase mask

Fig 1: Fabrication of a dual-FBG Fabry-Pérot based H₂ sensor

After the inscription of the first FBG, a second grating is created at an edge-to-edge distance of 13 mm by applying identical parameters. Both gratings act as wavelength selective mirrors forming a cavity according to an in-fiber FP etalon. The reflection spectrum of the created etalon measured with an optical 4-channel I4G FBG interrogator (Optics 11, Netherlands) is shown in Fig. 2. The resulting signal consists of a high frequency signal with multiple narrow spikes (yellow) enclosed by a wider envelope (red), whose shape is defined by the line-shape of the two FBGs. Whereas the shift of the cavity-induced high frequency signal is tracked by the shift of the most central minimum, the so-called fringe, the FBG-induced envelope is represented by the vertex of a parabolic fit through the peaks of the FP spikes.

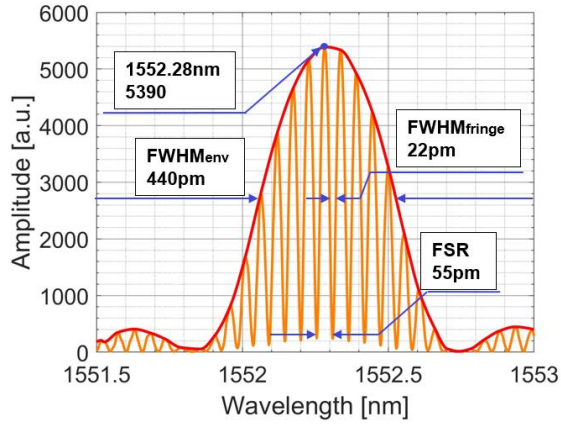


Fig 2: Spectrum of the FBG-induced FP etalon with envelope and comb-like modulation

Parameters such as the fiber diameter, FBG reflectivity and cavity length were intentionally selected to increase the sensor's strain sensitivity and the precision of both fringe and vertex signal. Accordingly, the cavity length must suffice to obtain enough support points for the vertex fit, while a larger FBG length results in a higher reflectivity, a narrower linewidth of the reflection spectrum and a higher precision of the fringe signal due to steeper transitions between maxima and minima. Further details to the fabrication of this sensor can also be found in [12].

In order to use the introduced sensor for the detection of hydrogen the optical cavity of the fabricated FP etalon is modified by partially coating its cavity with a Pd₈₀:Au₂₀ thin film via magnetron sputtering, while the FBGs remain unmodified (see Fig. 1a). Since Pd alone shows a tendency to peel off due to its poor adhesion towards optical fibers [10], the actual coating includes an intermediate chromium (Cr) layer before the gas-sensitive Pd alloy is deposited by accessing the Pd and Au targets simultaneously with different sputter powers. Due to the pronounced hysteresis behavior of Pd at its miscibility gap, gold is added to stabilize the phase transition of PdH_x by expanding the host lattice and increasing the H₂ solubility of the metal alloy [19]. For monitoring purposes, a fragment of a silicon (Si) wafer was coated in the same run as the fiber sensor. This enables the determination of the exact coating composition after the sputter process. The result of the performed energy-dispersive X-ray (EDX) measurement is shown in Tab. 2. From the scanning electron microscopy (SEM) image in Fig. 3, a thickness of approximately 21 nm is deduced for the Cr layer, while the Pd film measures roughly 78 nm.

Tab 2: Elemental composition of the H₂-sensitive coating determined by EDX

Element	Atom-%
Cr	3.05
Pd	78.99
Au	17.96

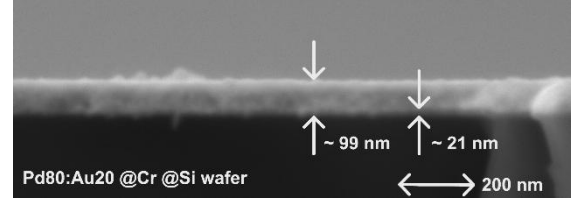


Fig 3: SEM image of the coating deposited via magnetron sputtering on a fraction of an Si wafer

Sensor Principle

Palladium (Pd) is known for its high H₂ solubility. In case of exposure, H₂ dissociates and diffuses into the coating under the formation of the metal hydride PdH_x. The hydrogen load leads to a volumetric expansion of the Pd lattice [20,21] and hence transfers a strain on the fiber along the FP cavity, supported by the reduced fiber diameter of only 80 μm compared to the 125 μm of a standard SM fiber [22]. The provoked strain again results in an increase of the optical path within the cavity and therefore in a measurable wavelength shift of the comb-like interference fringes. While the coated cavity reacts to both - a change in hydrogen concentration and temperature - with a shift in fringe wavelength, the FBGs remain uncoated. This means that a change in the spectral position of the envelope's vertex can be clearly attributed to a change in temperature, enabling compensation for temperature-related wavelength changes within a single spectrum. The compensated wavelength shift $\delta\lambda$ can be calculated via

$$\begin{aligned}
 \delta\lambda(c_{H_2}, \vartheta) &= \Delta\lambda_f - \Delta\lambda_{env} \quad (1) \\
 &= \lambda_f(c_{H_2}, \vartheta) - \lambda_f(c_{H_2} = 0 \text{ ppm}, \vartheta = 0^\circ\text{C}) \\
 &\quad - [\lambda_{env}(\vartheta) - \lambda_{env}(\vartheta = 0^\circ\text{C})] \\
 &= \lambda_f(c_{H_2}, \vartheta) - \lambda_{env}(\vartheta) - \delta\lambda_0
 \end{aligned}$$

with

$$\begin{aligned}
 \delta\lambda_0 &= \lambda_f(c_{H_2} = 0 \text{ ppm}, \vartheta = 0^\circ\text{C}) \\
 &\quad - \lambda_{env}(\vartheta = 0^\circ\text{C}) \quad (2)
 \end{aligned}$$

where $\Delta\lambda_{env}$ denotes the change in vertex wavelength, while $\Delta\lambda_f$ indicates the shift in fringe wavelength and $\delta\lambda_0$ denotes their joint zero reference, which is obtained as part of the subsequent temperature compensation procedure, described in detail in [12].

Temperature compensated H2 measurements

In order to determine the sensor's H2 sensitivity, it was integrated in the setup shown in Fig. 4.

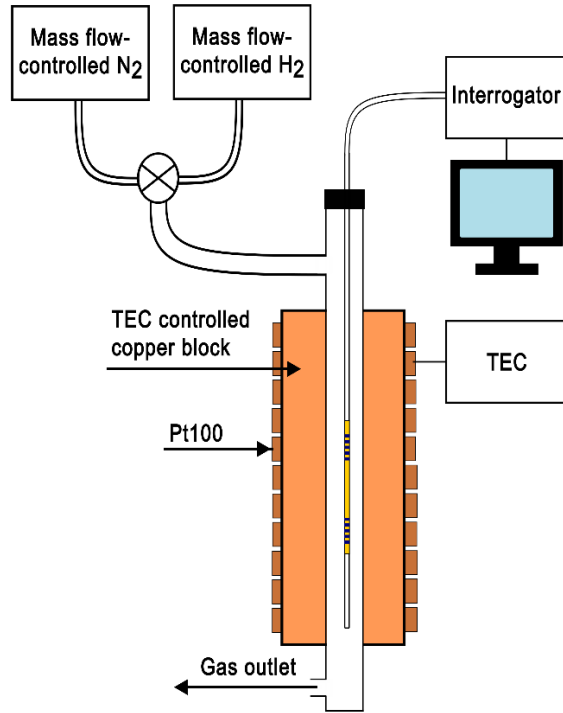


Fig 4: Schematic of the H2 calibration system

The calibration system consists of a glue-sealed tube holding the fiber sensor. This tube is temperature-controlled with an array of 12 Peltier elements and is connected to a mass flow-controlled gas supply making it possible to set a specific concentration of gaseous H2 surrounding the sensor by controlling the ratio between H2 and N2 flow. Within the scope of these measurements, the sensor was exposed to H2 concentrations in the range of 100 ppm to 20,000 ppm at temperatures of 30°C and 50°C. These measurements were performed at cycles of increasing and decreasing H2 concentration to assess the hysteresis behavior of the sensor. Fig. 5 shows the signals arising for both, the envelope vertex (violet) and fringe (yellow). As expected, the fringe signal shows a distinct modulation due to the change in H2 concentration, while the vertex signal remains almost constant during an isothermal hydrogen cycle.

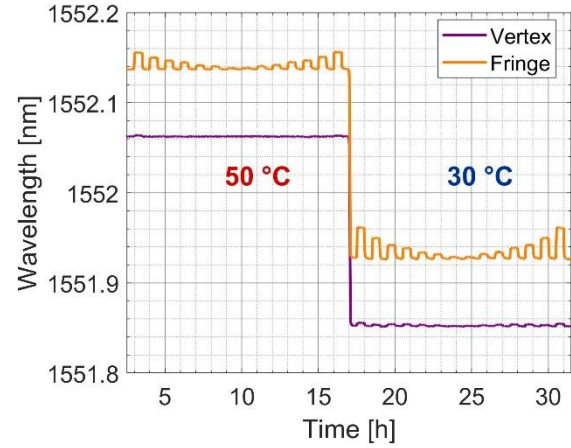


Fig 5: Fringe and vertex signal recorded at 30°C and 50°C for H2 concentrations between 100 and 20,000 ppm

The first step to a successful temperature compensation requires the determination of the reference wavelengths $\lambda_f(cH_2 = 0\text{ppm}, \vartheta = 0^\circ\text{C})$ and $\lambda_{env}(\vartheta = 0^\circ\text{C})$. Since the temperature response of FBG based sensors follows a quadratic function [23], these references emerge from the y-axis intercept of a second order polynomial fitted to the measurement signals obtained for fringe and vertex. The fit function

$$\lambda_{q, fit}(cH_2, \vartheta) = \alpha_1 \vartheta^2 + \alpha_2 \vartheta + \alpha_3 \quad (3)$$

(q = 'f' or 'env') yields the fit parameters denoted in Tab. 3 with α_3 corresponding to the zero-reference of the according signal. Using these parameters, $\delta\lambda_0$ can be calculated.

Tab 3: Fit coefficients resulting for the intrinsic temperature response of fringe and vertex signal of the sensor

λ_i	α_1 [pm/°C ²]	α_2 [pm/°C]	α_3 [nm]
$\lambda_f(cH_2, \vartheta)$	-0.098	15.998	1551.582
$\lambda_{env}(\vartheta)$	0.017	9.150	1551.562

Based on measurements of the vertex $\lambda_{env}(\vartheta)$ and fringe wavelengths $\lambda_f(cH_2, \vartheta)$, the difference results in the temperature-compensated wavelength shift $\delta\lambda$, because fringe and vertex wavelength show equal temperature dependencies. This is evident from the shared baseline for cycles at different temperatures in Fig. 6. The sensor clearly resolves H2 concentrations to a level as low as 100 ppm at both temperatures.

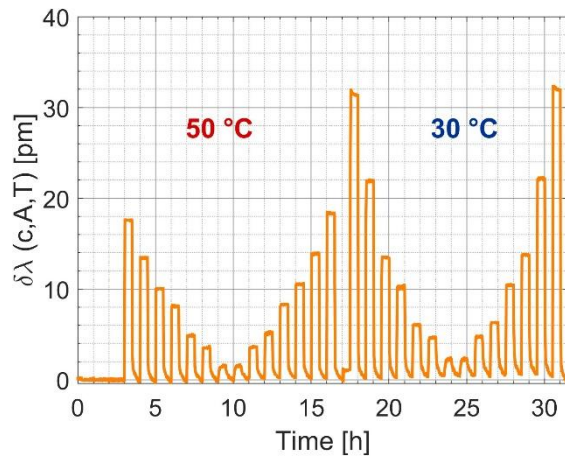


Fig 6: Time-resolved zero-referenced signal $\delta\lambda$ for H_2 concentrations ranging from 100 to 20,000 ppm at 30°C and 50°C

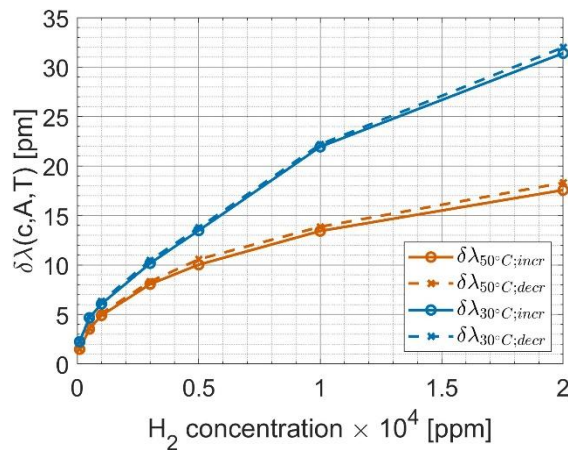


Fig 7: Temperature-compensated sensor response $\delta\lambda$ as a function of the H_2 concentration in the surrounding gas atmosphere in case of increasing and decreasing hydrogen load at 30°C and 50°C

The curves in Fig. 7 show the characteristic hydrogen curves at constant temperatures with a continuous, strongly nonlinear increase of $\delta\lambda(c_{H_2}, \vartheta)$ towards higher H_2 concentrations representing a particularly high sensitivity of the sensor at small concentrations. At the same time, the increasing and decreasing H_2 loadings are consistent, indicating a reversible, hysteresis-free sensor response. Even after the compensation of intrinsic temperature effects, a remaining temperature dependence was observed, being a result of the temperature dependent solubility of H_2 in the Pd thin film. This indicates the need of a temperature-dependent calibration procedure. Although the overall signal is highly nonlinear, Eq. (4) gives an adequate description of the sensor behavior with respect to H_2 concentration and temperature, given by

$$\delta\lambda(c_{H_2}, \vartheta) = \beta_1(\vartheta)\sqrt{c_{H_2}} + \beta_2(\vartheta)c_{H_2} + \beta_3(\vartheta)c_{H_2}^2 \quad (4)$$

The fit parameters β_i corresponding to the characteristic curves for the considered temperatures of 30°C and 50°C shown in Fig. 7 are summarized in Tab. 4.

Tab 4: Fit coefficients resulting for the H_2 dependent behavior of the hydrogen induced wavelength shift $\delta\lambda$

ϑ [°C]	β_1 [pm/ $\sqrt{\text{ppm}}$]	$\beta_2 \times 10^{-4}$ [pm/ppm]	$\beta_3 \times 10^{-9}$ [pm/ppm ²]
30	0.178	3.913	-2.366
50	0.170	3.254	1.876

While a straight forward compensation of the temperature dependent H_2 solubility is not possible, Buchfellner et al. [12] presented an algorithm based on iterative local linearization that allows a full calibration of the sensor under consideration of the sensitivities of fringe and vertex determined during the described compensation process.

Conclusion

The presented dual-FBG Fabry-Pérot sensor offers a highly precise detection of small H_2 concentrations. This sensor shows a reversible sensor response providing one of the highest sensitivities yet reported for unmodified, intact optical fibers (no etching or tapering required). Its suitability for multiplexing makes this sensor concept especially appealing. Additionally, it allows the compensation for internal temperature effects. Future efforts aim to characterize this sensor for broader H_2 and temperature ranges in various background compositions and for multi-point, multi-parameter sensor probes.

Funding

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