Distributed and discrete hydrogen monitoring through optical fiber sensors based on optical frequency domain reflectometry

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Abstract

The potential of discrete and distributed fiber-based sensors exploiting the Rayleigh scattering signature of doped amorphous silica is investigated for the real time monitoring of molecular hydrogen (H₂) detection. We showed that the impact of the refractive index changes induced by the H₂ diffusion into the silica host matrix can be used to detect and quantify this gas presence through two approaches: first via the related fiber length variation and second through the observed spectral shift. Comparing the obtained results with H₂ diffusion calculations, we can estimate the sensor sensitivity thresholds to be \( \sim 10^{16} \text{ molecule cm}^{-3} \) for the distributed measurements (spatial resolution better than 1 mm) and below \( \sim 10^{19} \text{ molecule cm}^{-3} \) for the discrete-one. The presented architecture of the sensor is well adapted to the monitoring of slowly evolving H₂ concentrations such as the ones expected in nuclear waste repositories as the time response of the sensor remains limited by the diffusion of the gas within the optical fiber. These threshold values and time responses can be easily improved by optimizing the length, the composition and/or the geometry of the sensing fiber.

1. Introduction

Hydrogen is a key element in various and numerous applicative domains: fault diagnosis, aerospace engineering, petroleum exploration, chemical processing… Indeed, this gas plays an important role as an energy carrier as well as chemical reactant. So over the last decades many studies were conducted and devoted to both the detection of hydrogen presence and to the quantification of its concentration. These research activities were motivated by several specific considerations such as: non pollution, sustainability, abundant availability… This huge interest is also driven by other intrinsic properties: high diffusion coefficient and buoyancy, minimum ignition energy as combustion characteristic with a high heat of burning process, wide inflammable range (4%–75%), high burning velocity, detonation sensitivity.

In fact, several technologies regarding sensors for H₂ detection were already reported describing a large panel of measurement methods. Recent review papers [1, 2] reported the different hydrogen detection technologies, highlighting their performances and the main challenges concerning their optimization in terms of accuracy, sensitivity and highly selective responses.

The most commonly used transducers are based on Pd or WO₃ thin films. In these cases, the typical probe structures were associated to the intrinsic physicochemical characteristics of the sensing thin film that change according to the H₂ concentration, so the transmitted probe optical signal will also carry the corresponding signature through intensity evolutions [3–10] or wavelength shifts [11–20]. In addition, many other studies targeted the optical response modifications based on interferometers [21–26] or surface plasmon resonance [27–33].

However, in most cases involving the optical fibers these components are mainly used as passive wave guides for the signal response transport, while the H₂ presence is revealed by an additional and external transducer, such as a thin film [34]. So, all the above mentioned sensors lead to a discrete and localized H₂ concentration...
measurement. Although they can be multiplexed in the environment of interest, a complete mapping of the H$_2$ concentration and distribution cannot easily be performed with these sensor technologies and architectures.

In this study, we focused on an innovative scheme based on a distributed technique, using one unique optical fiber, which allows us to provide a H$_2$ mapping of an entire region. The motivation is pushed up by the increasing demand for such sensors in harsh environments mixing high irradiation doses and temperature constraints such as those encountered in nuclear power plants and radioactive disposal facilities [35, 36].

It is well known that H$_2$ diffuses in amorphous SiO$_2$ matrix at room temperature and above. In a simplified situation where there is no dissociation of the hydrogen molecules and keeping the cylindrical symmetry of the fiber (assuming that the fiber is long relative to its diameter), the diffusion equation is reduced to [37]:

$$\frac{\partial C(r, t)}{\partial t} = D \left( \frac{\partial^2 C(r, t)}{\partial r^2} + \frac{1}{r} \frac{\partial C(r, t)}{\partial r} \right),$$

where $C(r, t)$ is the time and spatial dependent concentration, $r$ is the radial coordinate along the fiber transverse cross-section and $D = D_0 e^{-\frac{E_a}{k_B T}}$ is the diffusivity of H$_2$ in SiO$_2$ [37] with $E_a$ the activation energy of H$_2$, $k_B$ the Boltzmann constant and $T$ the temperature.

Moreover, Swart et al [37] have demonstrated that H$_2$ diffusion induces a variation of the refractive index ($n$) in the doped amorphous silica of the optical fiber cores. Since, considering that optical fiber sensor (OFS) scattering-based techniques uses the $n$ changes in order to determine the perturbation (usually temperature and/or strain) applied to the optical fiber (OF) [38], they can be employed for the detection of H$_2$ concentration too, as it was discussed in [35] for Brillouin-based sensors. In particular Optical Frequency Domain Reflectometry (OFDR) technique is suitable to this objective as its spatial resolution can be within the order of 40 $\mu$m over a probed optical length of 70 m. Indeed, its measurement principle is based on swept-wavelength interferometry [39]. The tested optical fiber is along one arm of a Mach–Zender interferometer, so the light of a tunable laser source is split and then recombined by two 3 dB couplers. The detector is placed at the output of the second coupler to acquire the fiber backscattering amplitude. Next a Fourier transform is performed to obtain the Rayleigh signature as a function of the fiber length [39, 40] which depends on the effective refractive index $n_{\text{eff}}$ of the OF by:

$$L = \frac{ct}{2n_{\text{eff}}},$$

It is then possible to monitor the small length changes, $\Delta L$, induced by the diffusion of the H$_2$ in and out of the fiber. In (2) $c$ is the speed of light and $\tau$ is time needed to cover the distance 2$L$ (in forward and backward directions).

Moreover, OFDR allows performing very precise distributed measurements along the tested fiber length through the cross-correlation of a reference trace, for which the environmental conditions are known, and successive perturbed traces [39, 40]. The result of this cross-correlation analysis is the spectral shift, $\Delta \nu$, which can be used to follow the applied perturbation and its distribution. As regards the refractive index changes, the empirical $\Delta \nu$ dependence is known [41] as:

$$\frac{\Delta \nu}{\nu_C} = \frac{\Delta n}{n_{\text{eff}}}$$

with $\nu_C$ the central frequency of the OFDR laser. Finally, exploiting this spectral shift, distributed measurements of H$_2$ along the fiber propagation axis appears feasible.

This study deals with the evaluation of these two measurement approaches for real time sensing of H$_2$ detection with the particular aim to demonstrate the potential of the unique very spatially-resolved OFDR-based systems. The obtained results are compared with measurements of H$_2$-induced excess attenuation in the infrared domain and with H$_2$ diffusion calculations.

2. Materials and methods

To perform the study, a germanosilicate commercial SMF28 fiber from CORNING was coiled in 5 cm diameter loops and was treated in H$_2$ atmosphere. As reported in figure 1, the H$_2$-loading phase was performed at controlled room temperature (accuracy of 1 °C) in a 250 ml high-pressure vessel, PARR 4651. The vessel was filled with high-purity technical H$_2$ gas up to a maximum pressure of 150 bars, which is controlled thanks to a pressure sensor inside the vessel. The H$_2$-loading duration lasted 14 d and it was followed by an out-diffusing period of the same duration. So these two cycles were monitored during 28 d. The treatment conditions were chosen on the basis of the cylindrical geometry of the samples and the diffusion coefficient of H$_2$ in silica [42].

During the entire experiment, the OFDR (OBR4600 from Luna Technologies) traces, see top of figure 1(a), were continuously recorded (every 5 min, acquisition time of ~1 min) in order to investigate the $n$ induced
changes in terms of both the OF $\Delta L$ (local measurement) and $\Delta \nu$ (distributed measurements, along the 13.5 m sample length). In figure 1 (a) the optical path of the sample is highlighted: the OF from 0 m to $\sim$5.5 m served as connection from the OBR equipment to the fiber inside the vessel which starts at $\sim$5.5 m (beginning of the red rectangle in figure 1(a)). As one can notice, there is no change in the scattering amplitudes thus meaning that the connection between the pigtail and the sensing fiber did not induce additional losses along the optical path.

Moreover and in order to perform well defined distributed traces all along the sample length in the vessel, few tenths of centimeters ($\sim$50 cm) at the OF end were kept out the vessel. After the end of the fiber, at $\sim$19.5 m, the amplitude of the reflected spectra decreases to the noise level (from 19.5 to $\sim$22 m in figure 1(a)). An example of the distributed measurement is reported in bottom of figure 1(a). It is possible to recognize, from this trace, the difference in the spectral shift between the coiled fiber inside the vessel (from $\sim$5.5 to $\sim$19 m) and the part of the fiber that was outside the vessel, i.e. a segment of the connection between the fiber and the OBR and the end of the OF sensing fiber.

In this study, the hydrogen induced absorption (HIA) was also monitored during the same two sequences of $\text{H}_2$ loading and out diffusing phases. However, as these measurements cannot be conducted simultaneously with OFDR ones, they were successively performed just one after the other. Therefore, we kept exactly the same experimental conditions ($\text{H}_2$ loading pressure, temperature, duration...) for both the OFDR and HIA runs. As reported in figure 1(b), the transmitted signal of the white light source (WLS), passing through the optical fiber, is recorded thanks to the near infrared (NIR) spectrum analyzer (nominal wavelength range from 800 nm to 2 $\mu$m). It is indeed well-known that $\text{H}_2$ diffusion in silica induces high losses due to the $\text{H}_2$ overtone absorption band at 1240 nm [43]. This HIA signature was monitored during the $\text{H}_2$-loading and outgassing sequences, serving as reference measurement for the calculations of the gas concentration evolution during the entire experiment.

3. Results and discussion

Figure 2(a) reports the Rayleigh scattering pattern for the tested fiber before and at the end of the $\text{H}_2$ loading periods. The scattering amplitude signature along the $\text{H}_2$ loaded fiber remains almost unchanged when the $\text{H}_2$
concentration varies from 0% to 95%. This result is very important since it shows that the HIA is not a limiting factor for OFDR operating around 1550 nm as these excess losses do not reduce significantly the Rayleigh scattering amplitude all along the fiber length. Moreover, from the zoom of the traces at the fiber end, we clearly distinguish a change of the fiber length (shown in figure 2(b)) when the H₂ diffuses, first quickly through the acrylate coating, then into the OF thus confirming that the n changes due to the gas loading can be monitored in these experimental conditions. The ΔL induced by the H₂ was, at the end of the loading, 1.2 cm long corresponding to a n variation of ∼10⁻³ in good agreement with literature where a refractive index change of 0.9 × 10⁻³ was reported in the case of a treatment at 45°C and 105 bars [37].

Intermediate distributed measurements along the OF are reported in figure 3. We note that Δν absolute value increases when H₂ concentration increases as the refractive index indeed increases in this loading regime. It is worth noticing also that the spectral shift remains constant along the 13.5 m length of tested OF proving a uniform gas loading along the tested sample during the whole experiment.

Results of figures 2 and 3 are very promising since they demonstrated that OFDR based systems, thanks to their high spatial resolution, can be powerful tools for point and distributed real time H₂ monitoring. The evolutions of these results are reported in figure 4 during both H₂-loading and outgassing phases. Their responses in terms of ΔL (figure 4(a)) and Δν (figure 4(b)) confirm the potential and the ability of these sensors to follow the hydrogen level in a large concentration range scale. Figure 4 also displays diffusion calculation results obtained thanks to equation (1). In order to achieve calculations that are as much representative as possible with our test conditions, HIA measurements were used to determine the diffusion constant, D, of equation (1). For the two H₂ phases (loading and out diffusing) the obtained D values are: 

\[ D_{in} = (2.7 ± 0.3) \times 10^{-11} \text{ cm}^2 \text{ s}^{-1} \quad D_{out} = (2.0 ± 0.3) \times 10^{-11} \text{ cm}^2 \text{ s}^{-1} \]

in good agreement with literature [37]. The different values of D for loading and outgassing sequences are substantially due to changes of temperature and
pressure during these two phases. Moreover, from figure 4 it can be seen in the case of H2 loading both ΔL and Δν follow pretty well the diffusion and outgassing calculations.

However, we can point out some differences with a main one at the beginning of the outgassing phase. The concentration mismatch between 1.1 × 10^6 s and 1.3 × 10^6 s is mostly due to the fact that the successive OFDR and HIA measurements were not performed simultaneously as explained in section 2.

In order to evaluate and highlight the performances of OFDR sensors for the H2 detection, we reported, in figure 5, the evolution of the H2-concentration as a function of ΔL and Δν during both the loading (figure 5(a)) and the outgassing (figure 5(b)) phases. It is interesting to notice that when C_{H2} > 2 × 10^{19} n_{molecule} cm^{-3} (0.3%) we observe a linear dependence between ΔL and molecular hydrogen concentration. This tendency is preserved until concentration levels of 6 × 10^{21} n_{molecule} cm^{-3}, i.e. 95%. Moreover, this linear dependence is verified in both loading and outgassing phases. Another important result pointed out from figure 5(a) is that distributed measurements are more sensitive with respect to the ΔL evaluation (using 13.5 m long fiber). Indeed, it can be clearly seen from figure 5(a) that appreciable variations of Δν are observed for H2 concentration within the order of 10^{18} n_{molecule} cm^{-3} (0.002%), whereas length variations are detected for concentrations higher than ∼4 × 10^{17} n_{molecule} cm^{-3} (0.006%).

It is worth noticing that, although distributed measurements are more sensitive than local ones, their detection limit cannot be easily improved by changing the experimental configuration. In other words, it is possible to greatly enhance the point measurements sensitivity rather than the distributed ones. Indeed, since this measure is based on the length variation detection, more fiber sensing length is used more easily (and quickly) H2 is detected as far as a sensitive and high spatial resolution system is used.

![Figure 4. Real time H2 monitoring of hydrogen presence and concentrations during the loading as well as the out-diffusing phases. The results are reported in terms of (a) ΔL and (b) −Δν/ν measurements. Dotted red line shows the diffusion calculation results obtained from equation (1).](image)

![Figure 5. H2 concentration evolution as a function of ΔL (blue points) and −Δν/ν (red point) during (a) loading and (b) outgassing phases.](image)
Although the results reported in this paper have demonstrated that the real-monitoring of H₂ using OFDR based sensors is efficient, they have also highlighted some possible progresses for this kind of sensor as the OFDR is coupled with a commercial SMF28 optical fiber. It has been showed that the reaction time of the hydrogen detection is relatively long when the H₂ concentration level is low. In order to improve the reactivity of OFDR sensors it could be useful to use as sensitive element different optical fibers with adapted geometry (reduced cladding diameter and/or hole-assisted optical fibers) or using photonic crystal fibers with large holes allowing a quicker diffusion of molecular hydrogen into the light propagation zone. Moreover, OFDR sensors are sensitive to environmental changes such as temperature variation or evolving strain. Even if it is possible to design proper sensor architecture in order to eliminate the strain contribution from the spectral shift, temperature variation influence is more complex to control. As already suggested in [35], the use of hermetic-coated optical fibers (such as carbon coating) can be useful for the monitoring of the only temperature parameter in order to decouple the hydrogen induced changes from temperature variation. The use of palladium-based optical fibers as sensing element for OFDR could be also explored improving the H₂ detection with low impact on the optical losses along the fiber path and giving the possibility to perform distributed measurement with high hydrogen sensitivity.

4. Conclusions

This work demonstrates the potential of OFDR-based sensing for the real time monitoring of molecular hydrogen detection (localization and concentration quantification) with Telecom-grade optical fibers. The feasibility of two measurement approaches has been successfully demonstrated. The first one is based on the detection of fiber sensing length variation due to the refractive index changes caused by the H₂ diffusion. The second exploits the observed spectral shift in presence of H₂ gas. Thanks to additional HIA measurements, it was possible to extract the diffusion constants of H₂ during loading and outgassing phases of our experiments, allowing calculating the H₂ diffusion in the studied optical fiber. The comparison between OFDR results and the calculations highlighted a linear correlation between both ΔL and Δν and the gas concentration when \( C_{H_2} > 2 \times 10^{19} \text{n mole} \text{cm}^{-3} \). The evaluation of the sensitivity limits of these sensors has shown that distributed measurements with spatial resolution below 1 mm are possible for concentrations above \( \sim 10^{16} \text{n mole} \text{cm}^{-3} \). These promising results allow designating the OFDR based sensors as very good candidates for H₂ monitoring, as their temperature dependence can be easily overcome by coupling the measurements on the sensitive fiber with additional ones on a fiber with a carbon-coated fiber preventing the hydrogen presence in its core.

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