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Effect of hydrogen gas on FBG-based optical fiber sensors for downhole pressure and temperature monitoring

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Abstract: The influence of hydrogen gas on Fiber Bragg Grating (FBG)-based optical fiber sensors has been validated experimentally. More in particular, the focus was on FBGs written in the so-called Butterfly Micro Structured Fiber that targets simultaneous pressure and temperature monitoring with a minimum in cross-sensitivity to be used in, for example, downhole applications for the oil and gas market. The hydrogen-induced pressure and temperature errors from this type of sensor have been quantified as a function of the partial hydrogen pressure. The induced errors can be related to the diffusion of the hydrogen into the microstructure and to refractive index changes due to the presence of the hydrogen in the micro holes and penetration of it into the fiberglass. Furthermore, we have also shown that the hydrogen-induced errors scale with the partial hydrogen pressure.

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1. Introduction

Fiber optic sensing has demonstrated its benefits for sensing of different critical parameters in downhole applications [1–5]. With respect to downhole temperature monitoring, Distributed Temperature Sensing (DTS) is a well-known intensity-based detection method within this industry [3,6,7]. DTS allows picturing the well temperature as a function of depth down to several kilometers with meter sized resolution. The technique relies on the encoding of temperature information contained in the Raman back scattered signal (Stokes and Anti-Stokes components). Another method relies on the use of Fiber Bragg Gratings (FBG) [4,8,9]. This technology yields quasi-distributed sensing: FBGs with different Bragg wavelengths can be inscribed along the same sensing fiber but the number of FBGs that can be multiplexed is rather limited when compared to DTS. Each Bragg resonance is intrinsically sensitive to temperature and strain and thus by eliminating strain effects, they can be equally employed to capture temperature profiles down an oil well.

A common characteristic for downhole environments is the presence of hydrogen [10]. Intensity-based sensing systems, like DTS, are affected by hydrogen as it diffuses into the optical fiber and thereby induces photo-darkening which increases the optical attenuation in the sensing fiber [5,11]. Researchers have proposed different mitigation techniques, such as reducing the penetration rate of hydrogen by means of a hermetic fiber coating [12] or by using self-calibrating methods involving either dual wavelength scanning or exploiting two sensing fibers [13–15]. For FBG wavelength-based sensing systems, the attenuation itself is
less of an issue but the presence of hydrogen results in an additional Bragg wavelength shift [16]. This drift of the Bragg wavelength stems from the change of the refractive index of the silica and the formation of Ge-OH or Si-OH depending on the hydrogen pressure level and on the temperature [17–22].

More recently, FBG-based sensors fabricated in Micro-Structured optical fiber (MS-FBG) have also been considered for sensing critical parameters in downhole applications [23,24]. The effect of the presence of hydrogen on such sensors has also been studied [25,26] to result in an additional wavelength shift due to the diffusion of hydrogen in the micro structure [25,27,28]. The induced wavelength shift was found to be nearly 1 nm after exposure to 200 bar of hydrogen pressure at 80 °C for a week, which corresponds to a fictitious temperature shift of ~100 °C, considering a typical temperature sensitivity of around 10 pm/°C.

Recently, we have also reported on the usage of MS-FBGs for downhole applications. More particularly, the usage of FBGs written in highly-birefringent Butterfly micro-structured fibers for simultaneous measurement of pressure and temperature [29–31]. The behavior of such specialty sensors when exposed to a hydrogen-rich environment was studied and will be reported in this document. We specifically evaluate the performance of temperature and pressure monitoring with the proposed MS-FBG sensor together with other conventional FBG based sensors. The modification of the refractive index in the fiber glass due to hydrogen diffusion attributed to the main cause on the apparent temperature shift. For pressure sensing, the disappearance of pressure difference due to hydrogen diffusion appears to be the primary reason for the apparent pressure error. In section 2, we introduce the autoclave facility used to load the sensors with hydrogen. Section 3 covers the experimental data and the analysis of the induced errors for each sensor when being loaded with pure hydrogen. In section 4, the hydrogen induced effects have been studied when using a gas mixture with partial hydrogen pressure. Based on the experiments which were discussed in each section, the influence of hydrogen on MS-FBG sensor is estimated. More importantly, the induced error can be quantified with respect to partial hydrogen pressure and working temperature in the environment. With that, the product adaptability of the MS-FBG sensor in downhole applications, especially to the influence of the hydrogen gas can be evaluated.

2. Experimental set-up

2.1 Gas loading facility

![Fig. 1. Autoclave facility for simulating a hydrogen-rich environment.](image)

To create a hydrogen-rich environment, we use a hydrogen loading autoclave which is commonly used in view of increasing the photosensitivity of (standard) fibers prior to grating inscription [32,33]. The autoclave consists of a stainless steel cylinder with an inner cavity of
20 cm in height and 13 cm diameter. It is equipped with four epoxy-based fiber feed-throughs with FC-connectors, which allow for online monitoring of samples during gas loading. The temperature of the autoclave can be controlled by an external oven. The epoxy-based fiber feed-throughs limit the maximum operating pressure and temperature to 80 bar and 80 °C. The temperature and pressure in the autoclave are continuously monitored with reference gauges. Figure 1 shows a schematic arrangement of the autoclave set-up.

2.2 FBG-based optical fiber sensors

We experimented with three types of FBG-based sensors. The first is an FBG in a Polyimide coated pure silica fiber written with a femtosecond (fs) laser by means of a phase mask. This sample will further be referred to as the Femto Second Grating or ‘FSG’. The second is a Draw Tower Grating or ‘DTG’ with Ormocer coating written with a UV laser using a Talbot interferometer. The sample was provided by FBGS International. The third is an FBG in the Butterfly Micro-Structured fiber (MS-FBG) written again with a fs-laser and phase mask. A typical reflection spectrum of the Butterfly MS-FBG sensor features two Bragg resonances due to the birefringent nature of this fiber. One peak corresponds to light polarized along the fast axis, whilst the other to light polarized along the slow axis. More information about the typical reflection spectrum of the Butterfly MS-FBG sensor and the cross section image of the Butterfly MSF can be found in [29–31]. The unique ability of this sensor is that pressure changes appear as changes in the peak separation (i.e. the wavelength difference between the fast and slow axes) while temperature can be monitored by tracking the change of the individual Bragg wavelengths. Since temperature appears as common mode for both Bragg wavelengths, it becomes cancelled in the peak separation and in this way, most of the pressure-temperature cross-sensitivity can be eliminated [29–31]. We used two such MS-FBG sensors. The first has its fiber end sealed by collapsing the micro-holes with an electrical arc of a fusion splicer in order to be sensitive to environmental pressure. Since this sealing process is done under atmospheric pressure, sealed MS-FBG sensors will sense the differential pressure i.e. the difference in pressure with respect to the atmospheric pressure that is present in the micro-holes. The other sample has its fiber end untreated and so with open micro-holes. This sample is not sensitive to pressure changes because the pressure inside the micro holes will always be the same as the pressure outside. The Bragg resonances of each FBG sensor are tracked using a Micron Optics SM125-500 interrogator combined with an HP 11896A polarization scrambler. All sensors are spliced to a FC/APC pigtail with a lead-in cable spooled on a metal rim. The sensing region of the MS-FBG sensors was kept straight by taping it before and after the FBG to the inner wall of the autoclave.

3. Experiments with sensors in a single gas environment

3.1 Nitrogen loading

The aim of this first experiment was primarily to verify whether both the autoclave and the sealed MS-FBG sensor are properly sealed. We examine this by loading nitrogen gas into the autoclave since this is not explosive. The experiment was carried out in the following stages: (1) pressurization of the autoclave with nitrogen from atmospheric pressure to 20 bar and then to 80 bar sequentially, (2) heating up the autoclave to a temperature of 80 °C with a holding time of a few hours, (3) leave the autoclave to cool down to room temperature (RT) and (4) release the pressure in the autoclave down to atmospheric pressure. Notice that the heating step also causes the pressure to increase since the autoclave should be regarded as a closed volume.

Figure 2(a) shows the evolution of the Bragg resonances of the open MS-FBG sensor during nitrogen loading, together with the reference pressure and temperature readings. The recorded wavelength shifts for both the slow and fast axes have been normalized for clarity. The figure also indicates the different stages (1) to (4) explained above. A few transients in
the wavelength shifts for both Bragg resonances can be observed during stage (1), which actually correspond to the moments when the pressure is increased in the autoclave. The sudden change in pressure in the closed volume of the autoclave induces a sudden change in temperature, which quickly dissipates again over time. Since the individual wavelengths are sensitive to temperature changes, this explains the transients. Apart from that, both Bragg resonances shift first to longer (shorter) wavelength as the nitrogen pressure increases (decreases) during the warming up (cooling down) period and we can see a good correspondence between the wavelength shift and the pressure reading. However, the magnitude of the shift for the fast axis Bragg resonance is larger than that for the slow axis.

The wavelength correspondence to pressure and the change in peak separation can both be related to the change in refractive index that occurs due to the presence of the pressurized nitrogen gas in the micro holes. As the pressure increases, the refractive index will change accordingly. This also causes the Bragg wavelength to increase since the light from both modes overlaps partly with the air holes. However, the light guided along the fast-axis has more overlap with the micro-holes and therefore it experiences a larger change in effective refractive index (and wavelength) compared to the slow-axis and hence there is a net change in peak separation: a pressure increase corresponds to a drop in peak separation and vice versa, see Fig. 3. The peak separation decreases with 42 pm with a pressure increase of 80 bar and returns to its initial value when the autoclave pressure has been released. As stated before, this initial loading test with nitrogen was primarily intended as a sealing check of the autoclave and sealed sensor. But it also nicely illustrates the effect of the gas in the micro-holes on the refractive index and wavelengths. This effect will also be present during the hydrogen loading tests, see next section.

Similarly, Fig. 2(b) shows the evolution of the Bragg resonances of the sealed MS-FBG sensor. Both Bragg resonances respond to pressure in the way it is understood for sealed samples of this fiber type [29,30]: the slow axis Bragg resonance features a positive and larger pressure sensitivity, whilst the fast axis Bragg resonance has a negative and lower pressure sensitivity during stages (1) and (2). The sealed MS-FBG sensor operates as an actual pressure sensor as expected. The sensing functionality of the sealed MS-FBG becomes obvious when considering the peak separation as shown in Fig. 3, which follows the reference pressure reading. The influence of temperature changes on the pressure measurements is negligible since both Bragg resonances experience the temperature variation as a common mode effect that is cancelled out by taking the difference. We obtain a pressure sensitivity of the sealed MS-FBG sensor of 2.8 pm/bar (a total peak separation change of 229 pm over a pressure difference of 81.7 bar). This proves that the sealed sensor is indeed properly sealed.

The FSG and DTG samples behave similarly to standard FBG based sensors during pressure cycling. At stage (1), a typical pressure sensitivity of around −0.2 pm/bar was observed. Afterward, the wavelength shifts are mainly dominated by the change in temperature. The net wavelength changes are around +0.746 nm and +0.651 nm for the DTG and FSG sensor respectively.

The findings of the nitrogen loading tests can be summarized as follows. The sealed MS-FBG acts as a pressure sensor as the change of hydrostatic pressure can be encoded into the change of peak separation and a positive pressure sensitivity in peak separation was observed. On the other hand, the open MS-FBG sensor is acting as a refractive index sensor since the pressure changes will predominantly cause the refractive index of the nitrogen in the micro holes to change and this in turns induces wavelength shifts in the light guided in the fast and slow axes. Due to the difference in overlap, the effect is larger in the fast axis and this causes a net negative change in peak separation.
3.2 Hydrogen loading

After the initial verification step with nitrogen gas, we also evaluated the influence of hydrogen loading on the different FBG sensors. Hydrogen is expected to behave differently since it is known to diffuse into solids like e.g. silica. The hydrogen loading tests were conducted as follows: (1) loading with hydrogen gas up to 80 bar at room temperature while monitoring the pressure stability using the reference pressure gauge, (2) heating of the autoclave up to 80 °C and wait until the sensor readings have completely stabilized (diffusing in step), (3) cooling down to room temperature, (4) release of the hydrogen gas and (5) heating of the autoclave back to 80 °C and wait again until all sensor readings have completely stabilized (diffusing out step).

3.2.1 Diffusing in

Figure 4 shows the evolution of the individual Bragg resonances and of the peak separation for the open MS-FBG sensor during stages (1) to (4) as described above. It can be seen that both Bragg resonances shift to longer wavelengths when the pressure increases from atmospheric pressure to 80 bar hydrogen gas at room temperature. However, it can be seen
that the wavelength shifts are not instantaneous but it takes a certain amount of time for the wavelengths to stabilize. This can be linked to the diffusion of the hydrogen gas into the micro-holes, which is not instantaneous because the holes are initially filled with air. We observe again that the shift of the fast axis Bragg resonance is larger than that of slow axis, resulting in a net decrease of the peak separation, as shown in Fig. 4(b). We cannot observe any sign of leakage during the first 24 hours at room temperature during stage (1). Therefore, it is safe to increase the temperature up to 80 °C, stage (2). It results in a pressure increase of 20 bar in the closed autoclave volume. The peak separation then increases from −21 pm to −13 pm. This change in peak separation is opposite to what is expected in view of a pressure increase in the micro holes since this should result into a further decrease. Therefore, another effect should be present that is more important than the refractive index increase in the micro holes. The most plausible explanation is that this is the result from the hydrogen penetrating the fiber glass at the level of the core during the initial heating. Hydrogen entering the fiber core will also increase the refractive index but its effect will be opposite on the peak separation since the light from the slow axis now has the most overlap with the glass core.

Note that the long waiting time of almost 150 hours at 80°C and 100 bar in stage (2) is mainly required for diffusion in the sealed MS-FBG sensor. The diffusion in the open MS-FBG sensor is of the order of tens of minutes, in line with the time constant reported in [28]. After cooling down (stage 3) and release of the hydrogen gas pressure (stage 4), the hydrogen gas present in the glass and the micro-holes diffuses out again and the peak separation of the open MS-FBG sensor returns to its initial value.

Similarly, Fig. 5 shows the evolution of the individual Bragg resonances and of the peak separation for the sealed MS-FBG sensor during stages (1) to (4). Initially, the sealed MS-FBG reacts to the 80 bar of hydrogen gas with both Bragg resonances moving away from each other, resulting in an increase of 0.21 nm in peak separation. Note that the peak separation slightly decreases along stage (1), while the reference pressure reading remains constant. This indicates that the hydrogen is already starting to diffuse into the air holes of the MSF. The diffusion rate is low because we are still at room temperature. Increasing the temperature to 80° yields an additional increase in peak separation of 51 pm, which matches with the additional pressure increase of 20 bar at the beginning of stage (2). The higher temperature clearly also speeds up the diffusion rate and it leads to a quick reduction of the peak separation almost down to the initial value at atmospheric pressure. Since the hydrogen diffuses through the glass, the differential pressure will eventually completely vanish when a sealed MS-FBG sensor is placed in a hydrogen rich environment for a sufficiently long time.
On the other hand, the individual Bragg resonances of the sealed MS-FBG sensor both shift to longer wavelengths when the hydrogen gradually diffuses into the microstructure, and this behavior is similar to that of the open MS-FBG sensor. The wavelength shift for the fast axis resonance is larger than that of the slow axis. Both curves eventually join almost completely at the end of stage (2) as shown in Fig. 5(a). After almost 150 hours, the peak separation of the sealed MS-FBG sensor reaches a value of −12 pm. The microstructure of the sealed MS-FBG sensor is now filled with hydrogen at 100 bar and 80 °C and hence becomes identical to the open MS-FBG. Both MS-FBG sensors (open and sealed) eventually feature an identical behavior in the same hydrogen rich environment.

As we release the pressure in stage (3), the sealed MS-FBG sensor experiences a negative differential pressure since now the 80 bar of hydrogen is trapped inside the microstructure and it largely exceeds the pressure at the outside. This is reflected in the peak separation being negative (from +0.21 nm initial separation to −0.21 nm after external pressure release), see Fig. 5(b). During stage (4), the peak separation gradually increases again as the hydrogen starts to diffuse out of the microstructure at room temperature.

![Figure 5](image)

**Fig. 5.** Change of (a) normalized Bragg resonances and (b) peak separation of the sealed MS-FBG sensor during the hydrogen loading experiment corresponding to stages (1) to (4).

Figure 6 shows the change in Bragg wavelength for the FSG and DTG sensors during stages (1) to (4) of the hydrogen loading experiment. Both sensors simply react to hydrogen pressure with a minor and negative pressure sensitivity during stage (1). They also feature a slight increase of 10-20 pm during the first 24 hours as the hydrogen diffusion rate is low at room temperature and because the sensor gratings are protected by the coating. At the beginning of stage (2), Bragg resonances move to longer wavelength as both the temperature and the hydrogen diffusion rate increase. After around 20 – 24 hours in stage (2), the wavelength shift stabilizes at +1.248 nm and +1.308 nm for the FSG and DTG, respectively. Note that the wavelength shift stems from both the temperature difference and the hydrogen loading. We will estimate the error in temperature and in pressure induced by hydrogen diffusion in section 3.3. Similar to the open MS-FBG sensor, the 150 hour waiting time is not necessary neither for the FSG nor for the DTG sensor. After cooling down and release of the hydrogen pressure, the remaining hydrogen induced wavelength shift is +0.567 nm and +0.594 nm at room temperature for the FSG and DTG sensors, respectively. These wavelength shifts are coming purely from the hydrogen diffusin into the silica.
3.2.2 Diffusing Out

In stage (5), the autoclave temperature was increased to 80 °C to speed up the out-diffusion process. Figure 7(a) shows the Bragg wavelength evolutions for the open MS-FBG sensor. The hydrogen contained in the air holes has already diffused out of the microstructure as soon as the autoclave pressure was released (stage 4). Therefore, this out-diffusion stage is not necessary for the open MS-FBG sensor: both Bragg resonances simply respond to the temperature change, as shown in Fig. 7(b). The peak separation remains stable, indicating as expected that both Bragg resonances experience the temperature change as a common mode effect.

Similarly, Fig. 8 shows the wavelength evolutions for the sealed MS-FBG sensor during the out-diffusion step. Both the Bragg wavelengths and the peak separation evolve in a complementary way to the results reported in Fig. 5. The trapped hydrogen gas now diffuses out of the microstructure leading to a decrease of the trapped hydrogen pressure and resulting in a decrease of the individual Bragg resonances and an increase of the peak separation. After almost 150 hours, the peak separation reaches zero, indicating that the internal pressure of the sealed MS-FBG sensor has been restored to atmospheric pressure.
Figure 9 shows the change of the Bragg resonances for the FSG and DTG sensors during the out-diffusion. The hydrogen induced wavelength shifts gradually vanish as the hydrogen diffuses out of the fiber.

Fig. 8. Change of (a) Bragg resonances and (b) peak separation of the sealed MS-FBG sensor during the hydrogen out-diffusion.

Fig. 9. Change of Bragg resonances for (a) FSG in pure silica fiber and (b) DTG during hydrogen out-diffusion.

3.3 Hydrogen-induced measurement error

For the sealed MS-FBG sensors, the change of the Bragg resonances can be described by a linear calibration model. It involves the contribution of the pressure change ($\Delta p$) multiplied by the pressure sensitivity ($a$ and $c$) and that of the temperature change ($\Delta T$) multiplied by the temperature sensitivity ($b$ and $d$), as given by Eq. (1) [31].

$$
\begin{bmatrix}
  a & b \\
  c & d
\end{bmatrix}
\begin{bmatrix}
  \Delta p \\
  \Delta T
\end{bmatrix}
= \begin{bmatrix}
  \lambda_{B1} - \lambda_{B1,0} \\
  \lambda_{B2} - \lambda_{B2,0}
\end{bmatrix}
$$

where $\lambda_{B1,0}$ and $\lambda_{B2,0}$ indicate the fast and slow axis Bragg resonances at a reference temperature and pressure. The sensitivity coefficients could be obtained from dedicated pressure and temperature calibrations. They were found to be $a = -0.1$ pm/bar, $b = 9.4705$ pm/°C, $c = 2.725$ pm/bar and $d = 9.4801$ pm/°C. Since the coefficients are not identical, the equation can be inverted and doing so yields the calibration formulas to convert the
wavelengths into pressure and temperature data, see Eq. (2). These equations will be used to calculate the pressure and temperature errors by comparing the calculated values with the reference measurements.

\[
\begin{align*}
\Delta p &= \frac{1}{ad-bc} \left[ d(\lambda_{b1} - \lambda_{b1,0}) - b(\lambda_{b2} - \lambda_{b2,0}) \right] \\
\Delta T &= \frac{1}{ad-bc} \left[ a(\lambda_{b2} - \lambda_{b2,0}) - c(\lambda_{b1} - \lambda_{b1,0}) \right]
\end{align*}
\]

For the open MS-FBG sensor, the above equations and coefficients are still used to calculate the corresponding temperature and pressure. The wavelength changes of the open MS-FBG sensor are mainly originating from the refractive index modifications when hydrogen is present in the air holes and in the fiber glass. The open MS-FBG sensor can quickly reveal the response of both Bragg wavelengths when hydrogen diffuses through the open channels. Doing so yields the data presented in Fig. 10(a). The temperature error for the open MS-FBG sensor at 80 °C and 100 bar of hydrogen is around 74 °C, which comes purely from the refractive index changes from the hydrogen in the MS-FBG. Similarly, the refractive index induced drop in peak separation leads to an apparent pressure drop of \(-4.6\) bar, causing the overall pressure error to increase from 100 to 104.6 bar.

For the sealed MS-FBG sensor, the influence on temperature monitoring is rather limited in the beginning when the hydrogen diffusion rate is low, as shown in Fig. 10(b). The temperature difference however starts to grow when the hydrogen starts to diffuse into the air holes and through the glass, yielding eventually to a total induced temperature error of around 75 °C. We thus find good agreement between the two MS-FBG sensors at the end of the hydrogen diffusing in step. In terms of pressure monitoring, the pressure reading in the sealed MS-FBG sensor gradually drops to zero as the hydrogen diffuses into the air holes. One could expect that the pressure difference becomes zero from the moment when the hydrogen pressure in the air holes is the same as in the autoclave. But due to the difference in effective refractive index induced by the hydrogen in the air holes and glass, the calculated pressure for the sealed MS-FBG sensor will be approximately 4 bar lower. Again, good agreement between open and sealed MS-FBG sensors could be found.

When the hydrogen is removed from the autoclave, the sealed sample initially gives a similar temperature difference as just before the hydrogen removal. The pressure error becomes negative because the pressure of the trapped hydrogen is larger than the pressure in the autoclave and hence the net pressure difference is negative. During the out-diffusion, both the temperature as well as the pressure error gradually restore to zero.

For the conventional FBG sensors, the response of the temperature changes can be approximated to the response of the wavelength variations with a linear relationship. The used linear coefficients are 10.25 pm/°C and 11.6 pm/°C for the FSG and for the DTG sensor, respectively. Figure 11 shows the calculated temperature during the hydrogen loading test for the FSG and DTG. Due to the diffusion of hydrogen, the wavelength shifts will be translated into apparent temperature shifts. Eventually, the temperature differences are around 58 °C and 49 °C for the FSG and DTG sensor, respectively. These apparent temperature shifts will vanish again once all hydrogen has diffused out from the fiber.

In these initial experiments, the pressure and temperature errors have been quantified under a pure hydrogen gas environment. In the next step, we will study the influence on the same set of FBG sensors in case of hydrogen-nitrogen gas mixtures.
4. Experiments with sensors in a gas mixture

For gas mixtures, Dalton’s law of partial pressures states that the overall pressure equals the sum of the individual partial pressures of each gas [34]. Hence, as far as FBG based sensors are concerned, hydrogen induced measurement errors should only result from partial hydrogen pressure and should not be related to the overall gas pressure. To verify this, we experimented with sensors exposed to a nitrogen-hydrogen gas mixture. We used again two MS-FBG sensors, one open and one sealed. As for the conventional FBG sensors, we only used the FSG since the behavior of the DTG was found to be very similar.

4.1 Nitrogen-hydrogen loading

We follow a procedure that is similar as described in the preceding sections, but now using two gases. (1) first we load 20 bar of hydrogen at room temperature, (2) we heat the autoclave to 80 °C and wait until the diffusion is complete, (3) we let the autoclave cool down to room temperature and then we increase the total pressure up to 80 bar by adding nitrogen, (4) we heat the autoclave again to 80 °C and wait sufficiently long, (5) we let the autoclave to cool down to room temperature and then we release the pressure, (6) we heat the autoclave up to 80 °C again and we wait for the out-diffusion to complete and (7) finally we let the autoclave

![Fig. 1.](image1.png)

**Fig. 10.** Calculated temperature and pressure for the (a) open and (b) sealed MS-FBG sensor during the hydrogen loading tests.

![Fig. 2.](image2.png)

**Fig. 11.** Calculated temperature and pressure from the (a) FSG and (b) DTG sensor during the hydrogen loading tests.
to cool down to room temperature again. We will immediately look to the induced temperature and pressure errors like it was done in section 3.3.

4.2 Nitrogen-hydrogen induced measurement error

4.2.1 Diffusing in

The results presented in this section correspond to the aforementioned stages (1) - (4). The sealing condition of the autoclave was checked in the first 84 hours before starting the loading cycle and is not presented in the plots below. Figure 12 shows the calculated pressure and temperatures for the open MS-FBG sample. Due to the presence of hydrogen in the air holes and the diffusion of hydrogen into the fiber glass, the temperature error is 20 °C and the pressure error is 27.3 bar (26 bar actual pressure of hydrogen at 80 °C and 1.4 bar extra due to the induced refractive index changes). The gap between stages (3) and (4) is due to a scheduled power maintenance. Nevertheless, the loaded hydrogen gas is still properly preserved in the autoclave after the maintenance. Next, an additional 60 bar of nitrogen is loaded into the autoclave, resulting into a temperature error increase from 20 °C to 48 °C and a pressure difference increase from −1.4 bar to −11 bar due to the change of the effective refractive index in the air holes. These errors stay constant for the next 144 hours before the release of the pressurized gases from the open MS-FBG sensor.

![Fig. 12. Calculated temperature and pressure as a comparison to the references for the open MS-FBG sensor during the different loading steps with the hydrogen-nitrogen gas mixture.](image)

The results for the sealed MS-FBG sensor are presented in Fig. 13. The temperature and pressure errors are gradually increasing as hydrogen is diffusing into the air holes during stage (2). From the open sample, we would expect the final apparent pressure reading to be −1.4 bar but at the end of stage (2) it has reached only around −0.9 bar. This indicates that the diffusion is not yet fully completed. During stage (3), 60 bar of nitrogen is injected into the autoclave to create the mixture. The sealed MS-FBG sensor already has 20 bar of trapped hydrogen and therefore sees only around 59 bar (80 −20 − 0.9 bar). During stage (4), the temperature is increased to 80 °C. Initially, we can still see some remaining diffusion, which is the continuation from the previous step. It gradually levels off to a constant reading, which is approximately 30 bar below the reference pressure. This is slightly lower than the expected total of 27.3 bar coming from the open sensor but this difference is most likely originating from the sensitivity coefficients \((a, b, c \text{ and } d)\), that are slightly different for a sensor filled with 20 bar of hydrogen compared to a similar sensor filled with 1 bar of air.
Fig. 13. Calculated temperature and pressure as a comparison to the references for the sealed MS-FBG sensor during the different loading steps with the hydrogen-nitrogen gas.

So to summarize: in an environment with a partial hydrogen pressure of 100 bar at 80 °C, the induced temperature and pressure error can reach 104 bar and 75 °C, respectively, as shown in Fig. 10(b). And in case the partial hydrogen pressure is reduced to 26 bar at 80 °C, the induced temperature and pressure errors also reduce to 27.3 bar and 20 °C, respectively. Therefore, the hydrogen-induced error in temperature and pressure sensing is only related to the partial hydrogen pressure and the errors scale almost linearly with the partial pressure.

The apparent temperature shift for the FSG sensor in the gas mixture test is around 25 °C during stage (2) and around 20 °C during stage (4), respectively. This reduction in apparent temperature with 5°C comes primarily from the pressure sensitivity of the FSG in combination with the applied pressure (80 bar during stage 4 instead of 20 bar in stage 2). Therefore, also here no extra temperature error could be observed by adding the nitrogen.

4.2.2 Diffusing out

This section reports the results of stages (5) - (7). For the open MS-FBG sensor, the calculated temperature and pressure return to their normal status when the gas mixture has diffused out of the open air holes. The out-diffusion process for the FSG sensor is of the order of tens of minutes. For the sealed MS-FBG, the trapped hydrogen in the air holes starts to diffuse out to the autoclave after the gas mixture in the autoclave has been released. A negative pressure difference around the core region is felt by the sealed MS-FBG sensor when the gas mixture has been released. The amount of negative pressure also corresponds to the pressure difference for the open MS-FBG when its air holes are filled with 26 bar of hydrogen at 80 °C. In the end, both calculated readings match with the reference gauges again.

5. Summary and conclusion

In this document, the effect of hydrogen on different FBG based optical fiber sensors was validated experimentally. The focus was primarily on FBG sensors used for downhole temperature and pressure monitoring, where hydrogen is known to be present. Of particular interest were the femtosecond written FBG sensors in the Butterfly Micro-Structured fiber (MS-FBG). Both open as well as sealed samples of this MS-FBGs were exposed first to pure hydrogen and later on also to a hydrogen-nitrogen mixture. In both cases, the samples were kept in an autoclave where both pressure and temperature could be controlled. For the evaluation, the measured pressure and temperature from the FBG-based sensors was compared with the readings from the reference sensors so that the measurement errors coming
from the hydrogen could be quantified. From the pure hydrogen loading tests, it was found that the responses from the sealed samples evolved towards those of the open samples and eventually, their response was found to be identical. This indicated that the hydrogen diffuses through the glass into the microstructure for the sealed samples. Because the diffusion time is different for the closed compared to the open samples, we could clearly separate the two main mechanisms behind the hydrogen induced errors: (1) the first being the disappearance of the (partial) hydrogen pressure from the pressure reading due to the diffusion of the hydrogen through the glass into the microstructure and (2) the second being a change of the individual Bragg wavelengths and also of the peak separation due to a change in the refractive index originating from the hydrogen entering the microstructure and penetrating the fiber glass. The changes in the individual wavelengths are causing apparent temperature shifts whereas a change in the peak separation causes an apparent pressure shift.

The tests with the hydrogen-nitrogen mixture was done to confirm that the driving parameter behind this process is the partial hydrogen pressure. This could be confirmed and it indicates basically that the size of the hydrogen induced errors scale almost linearly with the partial hydrogen pressure. Although the pressure and temperature errors shown in this work may seem relatively large, it should be kept in mind that these are for relatively large partial hydrogen pressures (80 bar and 20 bar). In practice however, the hydrogen level in a downhole environment is typically a few order of magnitude smaller. Therefore, the hydrogen induced errors will scale accordingly and therefore can be expected to be much smaller. The adaptability of the MS-FBG sensor for temperature and pressure monitoring in downhole applications is feasible if the hydrogen level is of the order of the present work. Some mitigation techniques would be required for MS-FBG sensor if the hydrogen induced error is above the required measurement accuracy.

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References