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Advancing Hydrogen Sensing for Sustainable Aviation: A Metal Hydride Coated TFBG Optical Fibre Hydrogen Sensor

Kasun P. DISSANAYAKE*[1], H. Sandra DEWI[1], Herman SCHREUDERS[2], Lars J. BANNENBERG[2], Roger M. GROVES[1]
[1] Department of Aerospace Structures and Materials, Delft University of Technology, The Netherlands
[2]Faculty of Applied Sciences, Delft University of Technology, The Netherlands
*k.p.w.dissanayake@tudelft.nl

Abstract. Hydrogen, which serves as a major driver of sustainable aviation, requires precise sensing methods. This study focuses on the development of a metal hydride-coated tilted fibre Bragg grating (TFBG) based sensor for hydrogen detection, with tantalum as a novel sensing material for fibre optic hydrogen sensing. Magnetron sputtering has been employed to deposit nanometer-scale metal films onto the optical fibre surface of the TFBG structure. In this proof of concept work, changes in both amplitude and the centre wavelength of cladding resonances of the TFBG transmission spectrum were observed in the hydrogen concentration range from 0.01% to 4% at room temperature.

Keywords: fibre optic sensors, tilted fibre Bragg grating, hydrogen sensors, metal hydrides

1. Introduction

The aviation industry is undergoing a transformative shift towards sustainability. Accentuated by high gravimetric energy density and clean exhaust resulting from combustion, hydrogen has emerged as a prominent green fuel to combat the significant carbon dioxide emissions associated with conventional aviation fuels [1]. However, as hydrogen and air mixtures can be explosive, harnessing the potential of hydrogen as an aviation fuel requires advanced sensing technologies capable of accurate monitoring of hydrogen concentrations in harsh operational environments. Two critical challenges are monitoring hydrogen concentrations near high-temperature engines and around high-pressure, cryogenic hydrogen storage tanks for leak detection [2].

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Fibre optic sensors offer unique advantages in these demanding conditions due to their robustness under harsh conditions, small in size, immunity to electromagnetic interference, spark-free operation, and remote sensing capabilities [3]. Among the various configurations of fibre optic sensors used for hydrogen detection, such as micro-mirror [4] and surface plasmon resonance (SPR) based sensors [5], those based on tilted fibre Bragg gratings (TFBGs) have emerged as top contenders [6,7] due to their increased sensitivity to changes in external refractive index (RI) [8] and potential multi-point operability. An optical hydrogen sensor can be developed by depositing a suitable metal hydride on the surface of a TFBG. The metal hydride gradually absorbs hydrogen in the surrounding environment, leading to gradual alterations in its optical properties, which can then be detected and observed by monitoring the TFBG's transmission spectrum.

Metal hydrides are prominent sensing materials studied for hydrogen sensing, especially palladium (Pd). While previous literature has explored Pd coated fibre optic sensors [3,9] for hydrogen detection, issues related to limited sensing range, delamination, and hysteresis in response have been observed [10], calling for innovative solutions. Recently, tantalum (Ta) has been introduced as a potential candidate material to address the limitations observed with Pd [11,12].

In this proof-of-concept work, a novel fibre optic hydrogen sensor has been developed by combining Ta as the sensing material with a TFBG as the optical transducer, leveraging the benefits of both components. The following sections discuss the sensing principle, the experimental setup used, and the sensor response to varying hydrogen environments in detail. The developed Ta-based TFBG was exposed to gas flows equivalent to hydrogen concentrations ranging from 0.01% to 4% at room temperature (23°C) to evaluate its performance. This concentration range was selected based on the lower limit of hydrogen concentrations (4%), which is within the range susceptible to explosive mixtures with atmospheric oxygen [13]. These preliminary results underscore the significant potential of our approach in developing highly sensitive and highly selective fibre optic hydrogen sensors tailored specifically for hydrogen-powered aviation applications.

2. Sensing Principle

The fabrication process of the sensor involved applying nanometres thick metal thin films on a TFBG fibre surface using a magnetron sputtering technique. The metal layer stack deposited on the surface of the TFBG consists of two layers: a 50 nm Ta thin film layer acting as the primary sensing element and a 15 nm $Pd_{0.6}Au_{0.4}$ capping layer. The capping layer serves a dual purpose: protecting the Ta layer from oxidation and catalysing the hydrogen dissociation process which involves separating hydrogen molecules into individual atoms [14]. As outlined in [15], the sensing metal layer stack can reversibly absorb hydrogen, leading to the formation of metal hydrides and altering its optical properties, notably the refractive index (n) and the extinction coefficient, with exposure to even minute hydrogen concentrations. Then the TFBG optical transducer converts these changes into an optical signal by changing the characteristics (amplitude and wavelengths) of the transmission spectrum of the Ta deposited TFBG. The degree of these changes depends on the complex RI of the metal hydride sensing layer, which is influenced by the concentration of hydrogen in the surrounding environment. Consequently, the characteristics of the TFBG transmission spectrum can be utilized to monitor and quantify the hydrogen concentration in the surrounding environment. This serves as the sensing principle of the TFBG hydrogen sensor developed in this work.

The centre wavelength of the Bragg peak of the TFBG can be defined as [16];

$$\lambda_{Bragg} = [n_{eff}^{core}(\lambda) + n_{eff}^{core}(\lambda)]\Lambda_g/\cos\theta \qquad (1)$$

The centre wavelength of the i^{th} cladding resonance of the TFBG transmission spectrum can be defined by [17];

$$\lambda_{clad,i} = \left[n_{eff}^{core}(\lambda) + n_{eff,clad}^{i}(\lambda)\right]\Lambda_g/\cos\theta \qquad (2)$$

Reflectivity (R) or the amplitude of individual cladding mode resonances can be defined by [16];

$$R = \tanh^2(kL) \qquad (3)$$

where:

$n_{eff}^{core}(\lambda)$ – effective RI of the core mode
$n_{eff,clad}^{i}(\lambda)$- effective RI of the i^{th} order cladding mode
λ- wavelength
L- length of the TFBG
k- coupling coefficient between i^{th} order cladding mode and the core mode
Λ_g– distance between two tilted grating planes
θ – tilt angle of the TFBG

In equations (2) and (3), $n_{eff,clad}^{i}(\lambda)$ and k are functions of the RI of the deposited hydrogen sensing metal layer. This RI changes based on the surrounding hydrogen concentration, leading to changes in $\lambda_{clad,i}$ and R. Therefore, the characteristics of the TFBG transmission spectrum can be utilised to monitor and quantify the hydrogen concentration in the surrounding environment.

3. Sensor Fabrication

After cleaning the fibre optic surface with acetone, a stack of metal thin film layers was deposited on the TFBG surface using magnetron sputtering (AJA Int. with base pressure of 10^{-6} Pa). The optical fibre containing the TFBG was mounted on a plate and rotated during the deposition under 0.3 Pa of argon (Ar) to ensure a uniform coating. The sputtering parameters and conditions matched those detailed in our previous work [11]. Deposition thickness was controlled by controlling the deposition time.

Fig. 1. shows a microscopic image of the deposited metal thin film alongside the uncoated regions of the optical fibre surface containing the TFBG. The optical microscope image revealed a uniform and homogeneous deposition. The TFBGs used in this work were fabricated by B-Sens BV using a phase mask technique on a single mode fibre with a core RI of 1.45 and a cladding RI of 1.445. A 6 degree tilt angle was chosen to achieve the maximum cladding mode coupling to the core mode in the wavelength region of 1510 nm to 1590 nm, defined by the operating wavelength region of the NI PXIe-4844 FBG interrogator from National Instruments. It has a dynamic range of 40 dB and a wavelength resolution of 4 pm. A 10 mm length of TFBG was used.

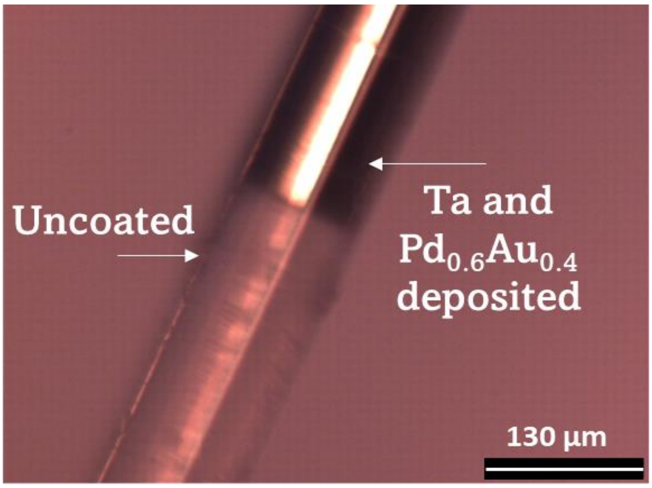

Fig. 1. Optical microscopic image of the sensing material (metal thin film stack) deposited TFBG surface

Fig. 2. shows the transmission spectrum of the TFBG before and after the deposition of the metal sensing layer. As expected, a reduction of the amplitude of the cladding peaks were observed due to the higher RI of the metal thin films compared with the cladding RI [18]. Cladding guided modes begin to couple more with the high-indexed metal thin films, reducing their amplitude in the transmission spectrum. Another significant observation is the observed split behaviour of cladding resonances compared to the uncoated transmission spectrum. This behaviour can be attributed to the lifting of mode degeneracy of linearly polarized (LP) modes and the appearance of their vector modes as closely split resonances under interrogation with unpolarized light [16].

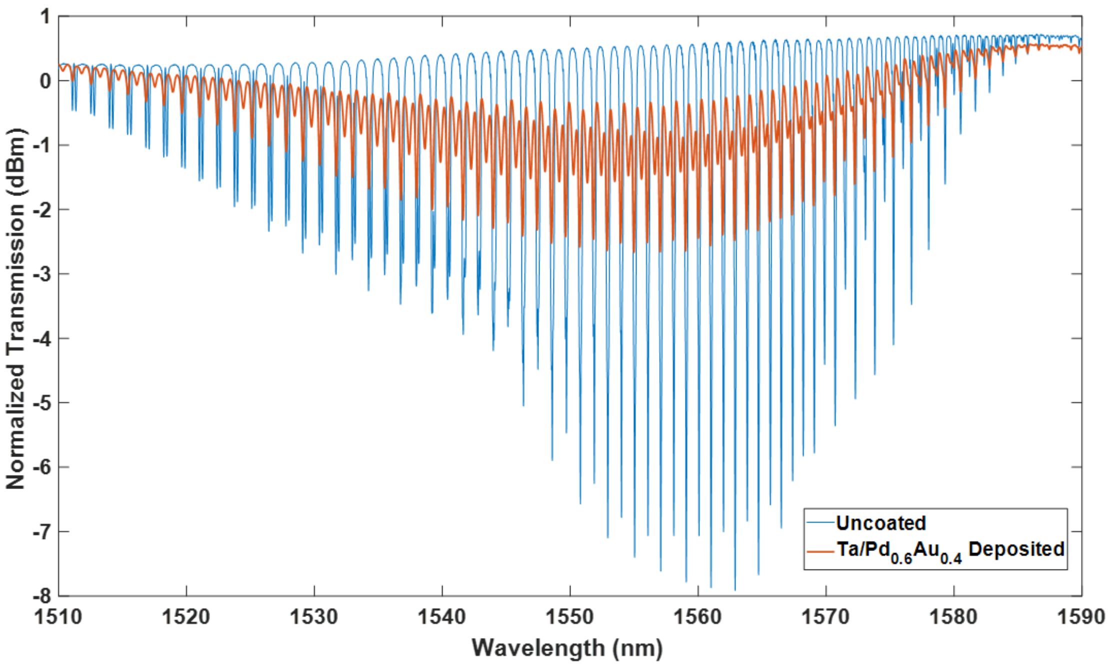

Fig. 2. Transmission spectrum of the bare and metal thin film deposited TFBG

4. Experimental Setup

Fig. 3. illustrates the experimental setup employed to test the performance of the Ta based TFBG fibre optic hydrogen sensor under various hydrogen concentrations. A leak-free gas chamber was connected via three Mass Flow Controllers (MFC) to three cylinders of gas containing 100% Ar, 4% H_2 in Ar, and 0.1% H_2 in Ar. MFCs were used to mix and control the flow rates of gas from the cylinders to achieve the desired hydrogen concentrations in the chamber. Total flow to the chamber was controlled at 200 sccm and the hydrogen concentration inside the chamber was calculated based on the ratio of flows from the three gas cylinders. The sensing material-deposited TFBG was mounted inside the chamber to ensure that the Ta-deposited area did not touch the chamber walls. It was then connected to the NI PXIe-4844 interrogator via a fibre optic feedthrough from SQS Vláknová Optika A.S. The transmission spectrum of the TFBG sensor was continuously monitored and recorded at room temperature over a series of hydrogen concentrations of 0.01%, 0.05%, 0.1%, 1%, 2%, and 4% to investigate the performance of the developed Ta metal hydride based TFBG hydrogen sensor.

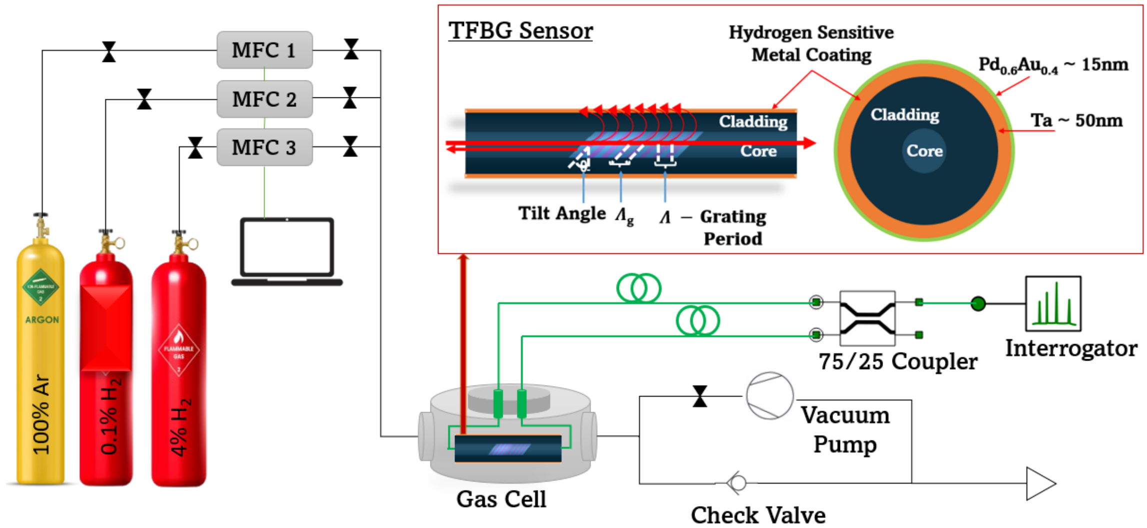

Fig. 3. Experimental setup and the schematic of the Ta-based TFBG hydrogen sensor

5. Results and Discussion (Response to hydrogen)

Fig. 4 depicts the recorded transmission spectra of the Ta-based TFBG sensor under varying hydrogen concentrations. Continuous perturbations of both amplitude and the centre wavelength of the cladding mode resonances were observed in the transmission spectra starting from exposure to 0.01% of hydrogen concentration. Normalized and zoomed areas of two of the most prominent cladding resonances, which have been used to quantify the amount of changes observed, are presented in Fig. 5. As can be seen from Fig. 5(a), the amplitude of the split cladding resonances were observed to decrease with increasing hydrogen concentrations. As shown in Fig. 5(b), a red shift of the centre wavelength of the cladding resonances were observed with increasing hydrogen concentrations. These changes are highlighted in Fig. 6(a). and Fig. 6(b).

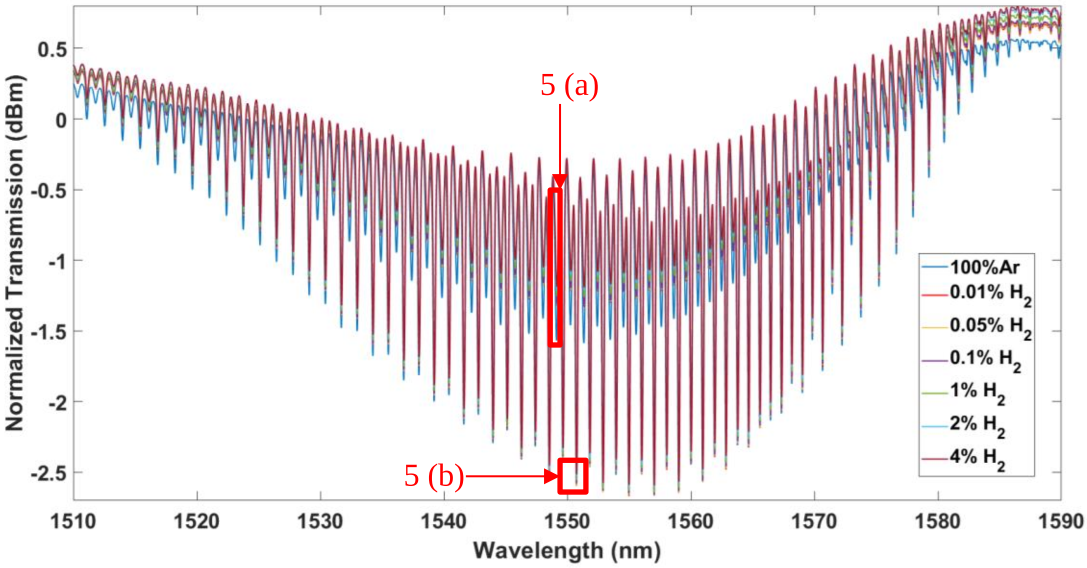

Fig. 4. Transmission spectra of Ta-based TFBG sensor under different hydrogen environments

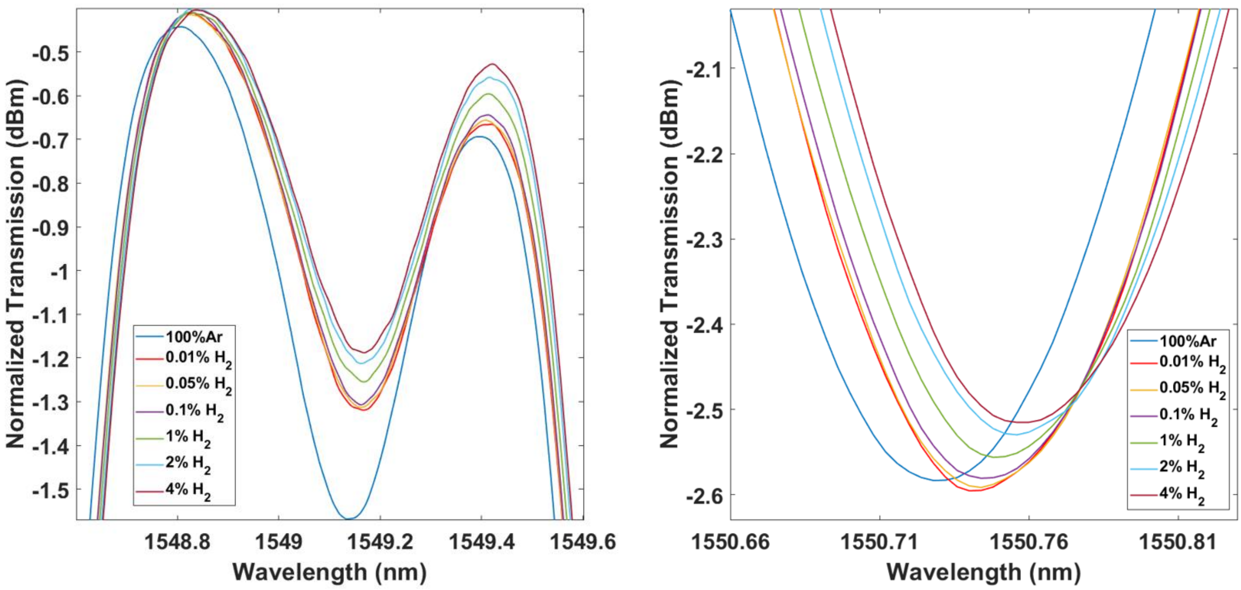

Fig. 5 (a) Amplitude **(b)** wavelength response of the cladding resonances under different hydrogen environments

A similar non-linear response can be observed for both amplitude and centre wavelength characteristics with exposure to increasing hydrogen concentrations from 0.01% to 4%. Two clear hydrogen concentration regions (0.01% - 2% and 2% - 4%) can be seen with different sensitivities in response. This sensitivity difference at different hydrogen concentration regions shows that the changes in the optical properties of the Ta metal-hydride layer is not linear, a behaviour that was also reported in [11]. In the hydrogen concentration range from 0.1% to 2%, we observed an amplitude-changing response of 0.05 dB/%H_2 and a wavelength shift of 5.8 pm/%H_2. However, we observed a reduction in these responses in the higher concentration range, from 2% to 4%. These initial results suggest that the developed Ta-based TFBG hydrogen sensor could potentially be utilised for monitoring lower concentrations of hydrogen below 4% with high sensitivity, indicating its potential applications in industries such as hydrogen powered aviation.

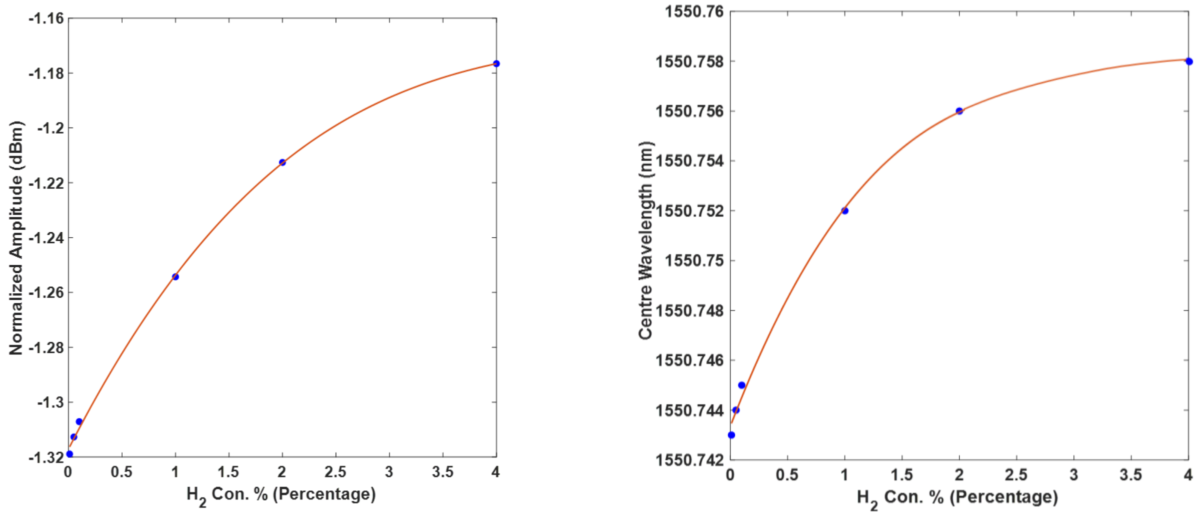

Fig. 6 (a) Amplitude **(b)** wavelength response with varying hydrogen concentrations (The red line serves as a guide to the trend)

6. Conclusion

A Ta metal-hydride-coated TFBG sensor has been developed and tested under various hydrogen concentrations up to 4% to evaluate its performance in this proof-of-concept work. The lowest hydrogen concentration measured was 0.01%, achieving an amplitude sensitivity of 0.05 dB/%H_2 and a wavelength response of 5.8 pm/%H_2 over the 0.01% to 4% H_2 concentration range at room temperature. These initial results suggest that this novel design using Ta and TFBG-based optical hydrogen sensors has great potential for use in aviation for detecting low hydrogen concentrations. Future work includes expanding the operating temperature range to low (-60°C) and high (>100°C) temperatures.

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