SENSORDEVICES 2025
DECOR Post-doctoral project

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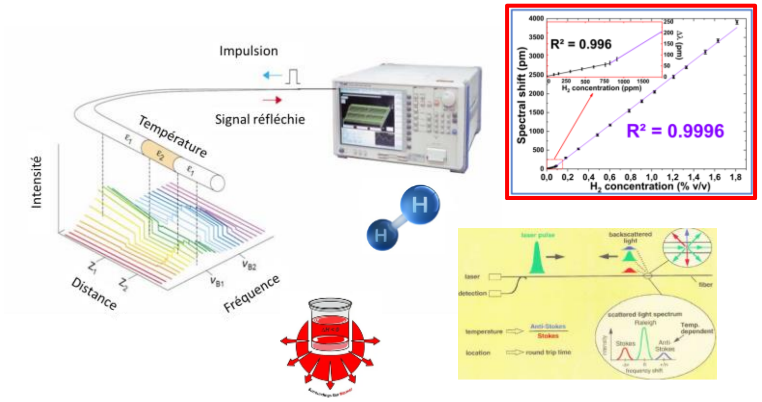

Fast, sensitive and distributed optical fiber detection of hydrogen gas

Post-doctoral fellow: TOUCHET Kévin, Doctor of Physic, Chemistry and Analytic

Starting: 06/03/2023

Founding: PTC-ID – project PROH2

IARIA

Team members:

LADACI Ayoub, *CHEYMOL Guy, BOURGES Firmin :* CEA/**DES**/ISAS/DRMP/SRMA/LISL
ZEKRI Elisabeth : *CEA/**DES**/ISAS/DRMP/SPC/LC2R*
DENIAU Guy : *CEA/**DRF**/IRAMIS/NIMBE/LICSEN*
LAFFONT Guillaume, MAGNE Sylvain, MAURIN Laurent: *CEA/**DRT**/List/SMCD/LSPM*

1. Context

SAFETY of gases in industrial environments!

<u>Fields of application</u>:

-Energetic source/vector: CH_4 and H_2

-Chemical synthesis:

CO : metal production

H_2 : NH_3 production, MetOH, refining

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Dangers and risks:

-Inflammable and explosive: CH_4, $\mathbf{H_2}$, CO

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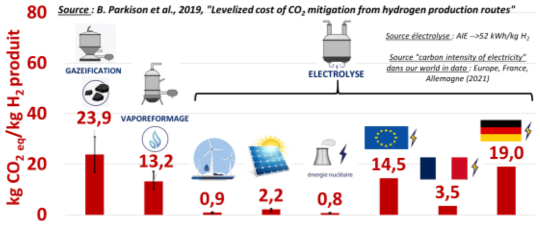
Ecological transition/low carbon energies:

-H_2



Combustion heat	
Hydrogen	**141 766 kJ/kg**
Methane	55 505 kJ/kg
Oil	43 070 kJ/kg

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Hydrogen: Dangers and risks:

1. *D. A. Crowl et al., 2007, « The hazards and risks of hydrogen »*
2. *K. Surla, 2020, TI, « Hydrogène »*

Danger factors	Hydrogen	Methane
Flammability limit (air) [1]	**4.0 à 75 % v/v** [1.2] (détonation: 13 à 65 % v/v) [2]	**5.3 à 15 % v/v**
Deflagration index [1]	**550 bar*m/s**	**55 bar*m/s**
Diffusion coefficient (air) [2]	**0.61 cm²/s**	**0.16 cm²/s**

Hydrogen uses, storage and transportation (15% actually)

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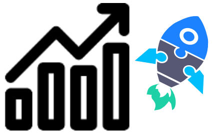

Major element of risk reduction: → **Early leak detection**

H_2 detectors already exist[3,4,5] :

-Catharometer

-Amperometer

-Semi-conductor resistance (MOx)

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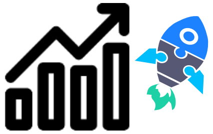

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Texas City refinery incident (USA, 2005)
The explosion killed 15 people and injured more than 170 (hydrocarbon gas leak)

Fossile gas explosion in Ghislenghien (Belgium, 2004)
Death of 24 people and serious injuries for more than 120 others

Hydrogen explosion in solar panel manufacturing plant – Heyuan (China, 2015)

Hydrogen explosion - Oslo (Norway, 2019)
The event led to the temporary suspension of the hydrogen station network in Norway

Hydrogen explosion - Fukushima (Japan)

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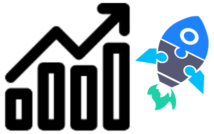

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Ponctual measurement systems

→ Development of **real-time, in-situ and distributed** fiber optic hydrogen leak detector

Realization of distributed hybrid fiber optic sensors!

2. Initial objective of the project

Objective

1) Detection at ambient T et P of 2% v/v H_2 (4 % v/v max limit)
2) Coating fixed along the length of the fiber,
3) Passive reconditioning for multiple measurements (without any intervention and fast)
4) Stable over time, in humidity (selectivity +)

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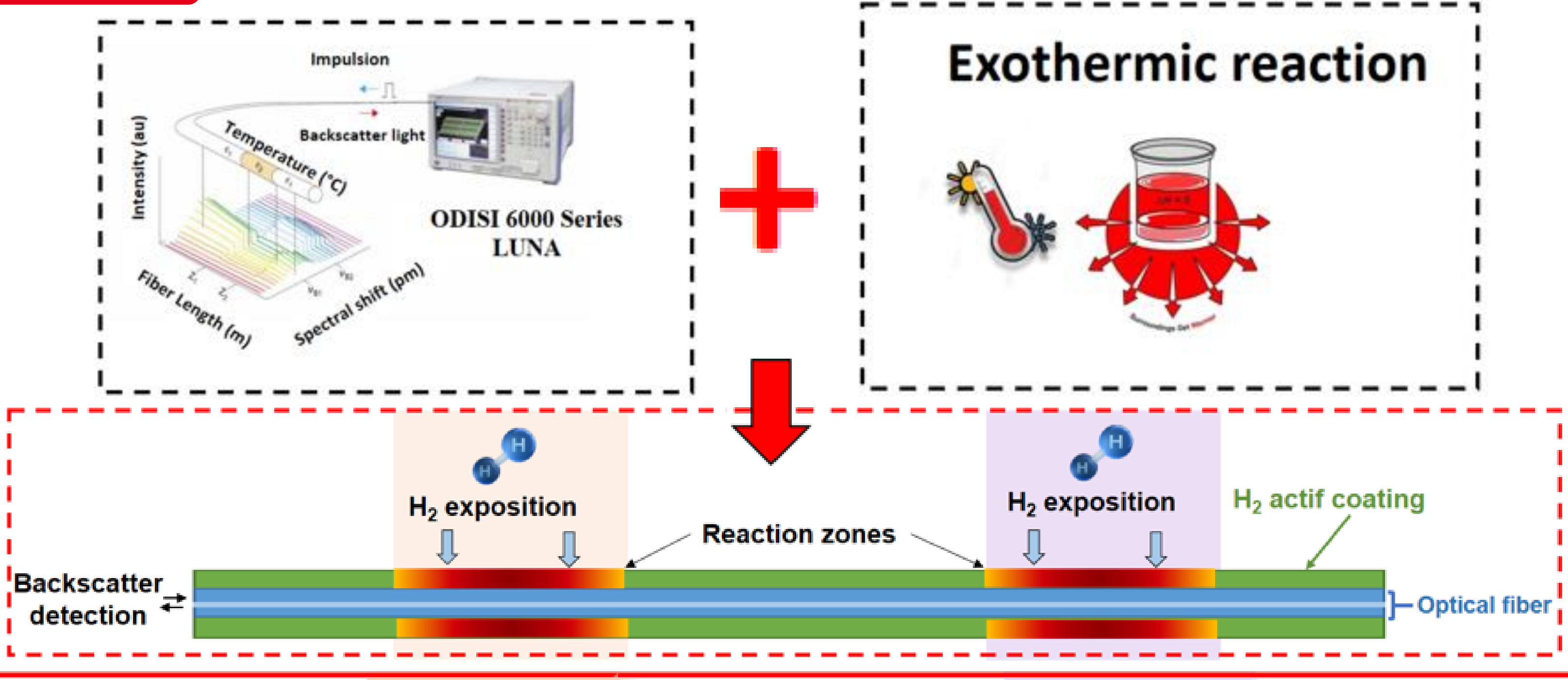
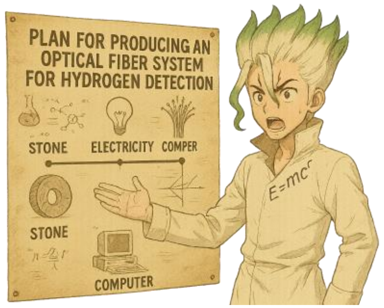
Principle

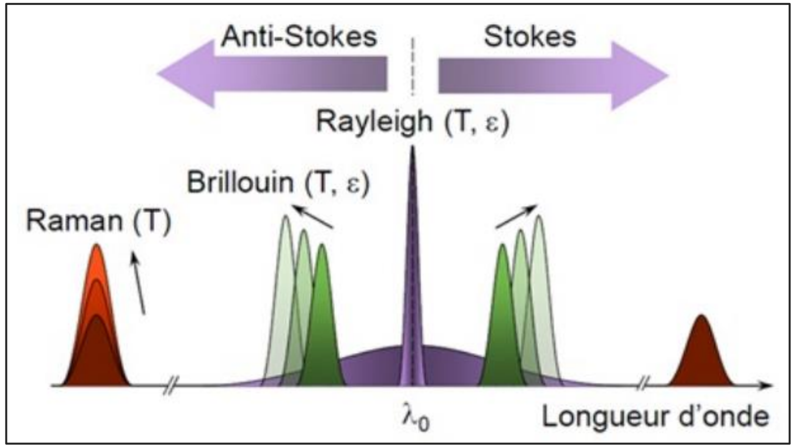




3.1. Optical fiber detector:

Rayleigh/Brillouin/Raman backscattering

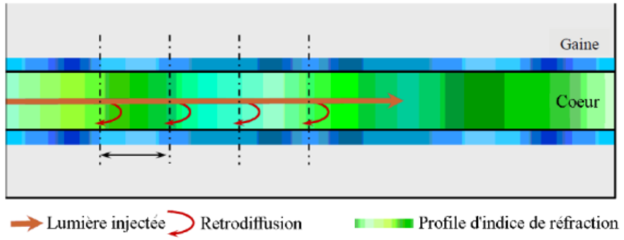
In OF: Elastic (Rayleigh) and inelastic (Raman, Brillouin) diffusion phenomena



I. Planes, 2019, PhD, « Performances des capteurs à fibre optique basés sur les diffusions Brillouin, Rayleigh et Raman en environnement sévère »

Raman = f(T) ; Brillouin et Rayleigh=f(T, ε)

Rayleigh backscattering case[1]**:**



A telecom OF = few defaults

Rayleigh diffusion → local fluctuations of refraction index

(non-periodic but cst).

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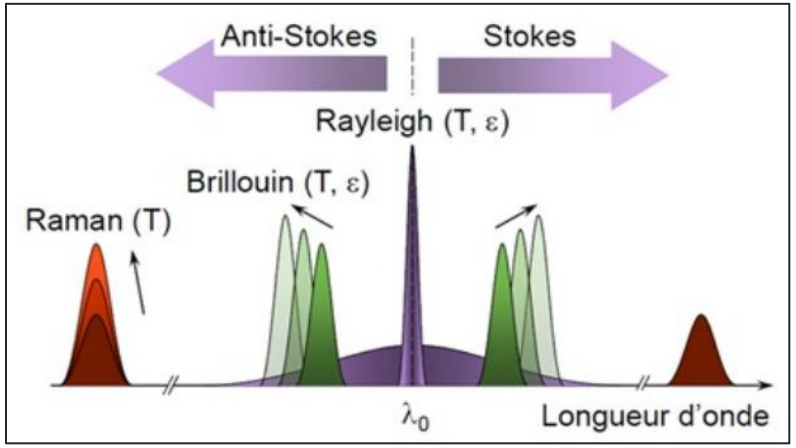

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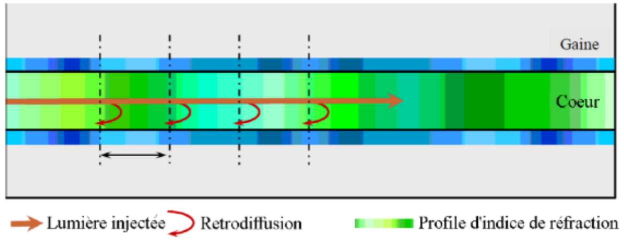

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D. Holler, et al. Optical Fiber Technology 55 (2020) 102160

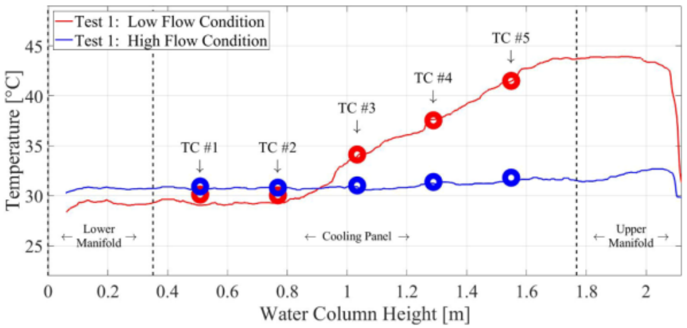

Table 3. The main characteristics of DOFSs.

Scattered signal	Technique	Spatial resolution	Sensing range	Temperature accuracy	Strain accuracy
Rayleigh	OTDR	1 m	~100 km	15 °C	
	OFDR	From 1 cm to 10 μm	From 2 km to 10 m	0.1 °C	1 με
Brillouin	BOTDR	1 m	~20–50 km	10 °C	60 με
	BOTDA	<50 cm	~100 km	1 °C	20 με
Raman	OTDR	<50 cm	~100 km	1 °C	—

S. Girard et al., 2018, « Recent advances in radiation-hardened fiber-based technologies for space applications », p.15/49

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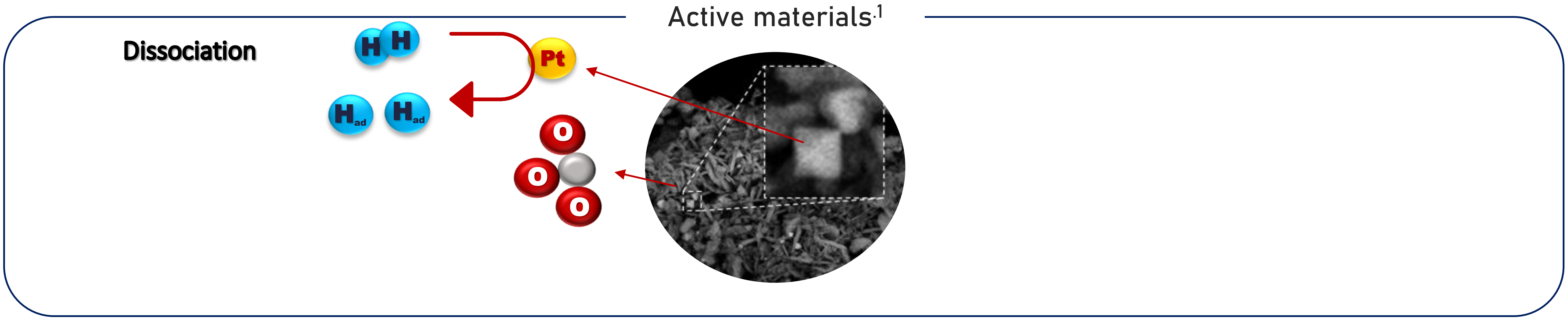
3.2. Active hydrogen coating:

How is it working ?



[1] K. Shun et al., Chem. Sci. 2022

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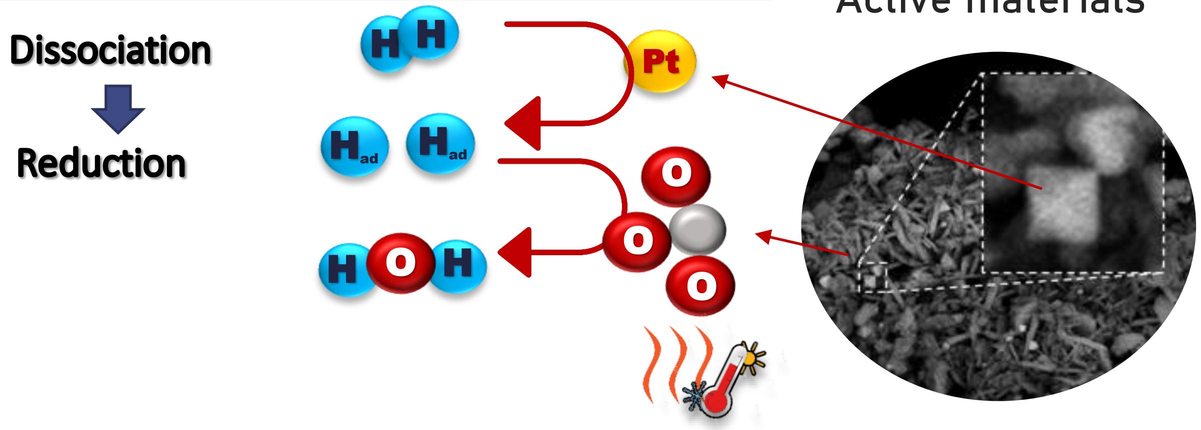

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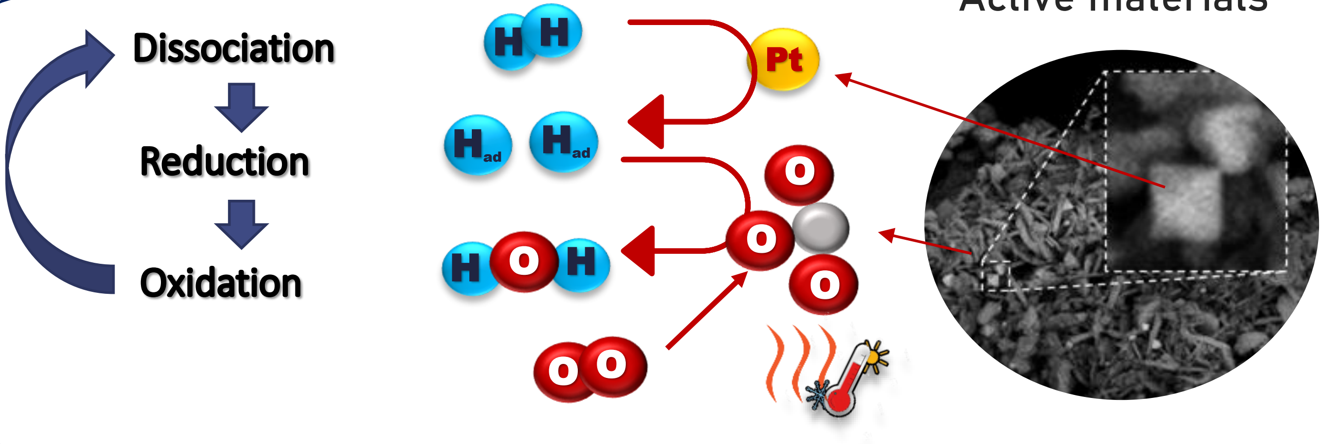

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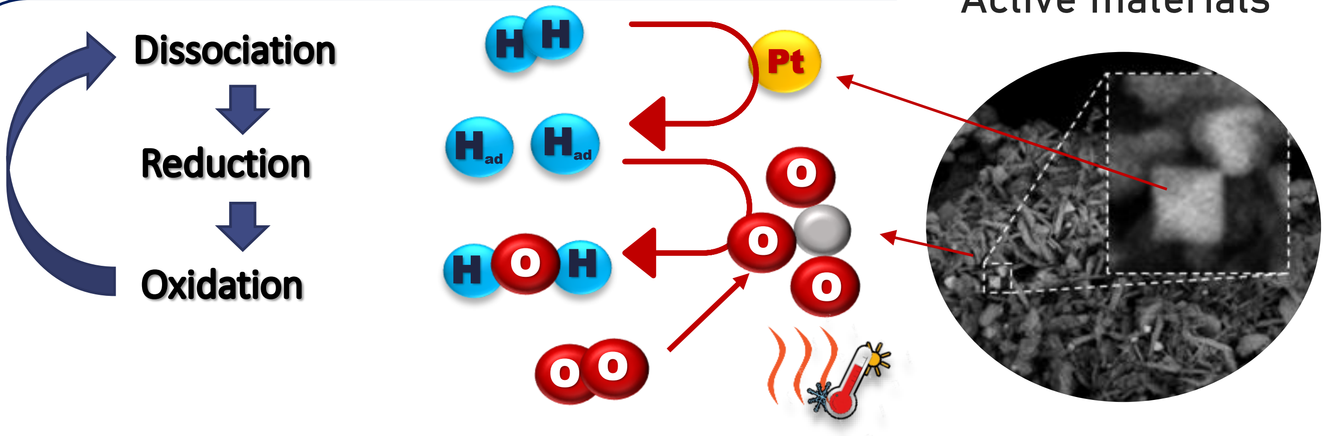

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Physical/Structural optimization

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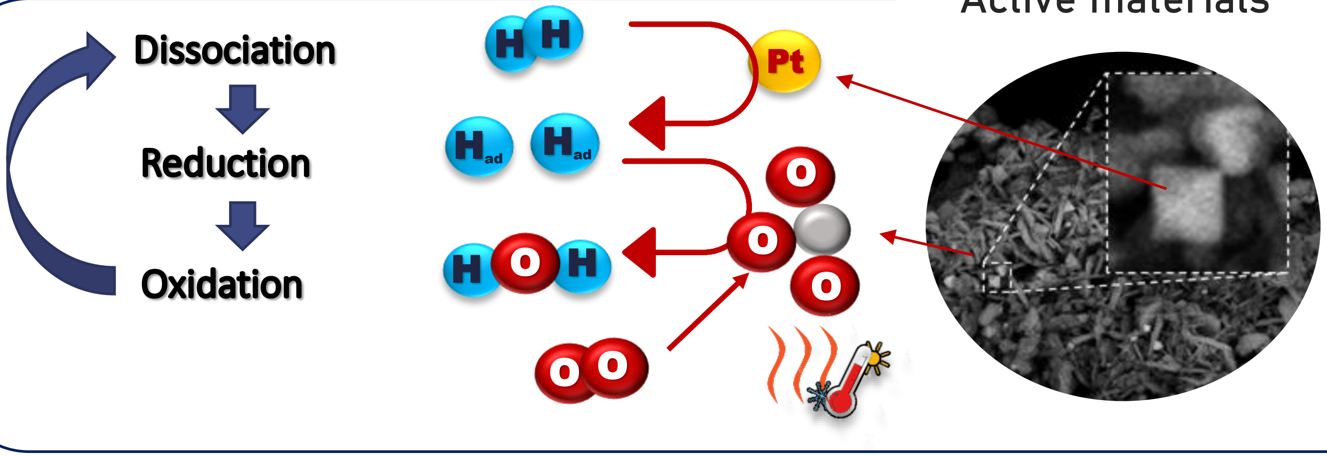

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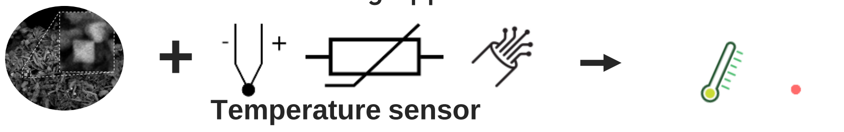
Sensing applications



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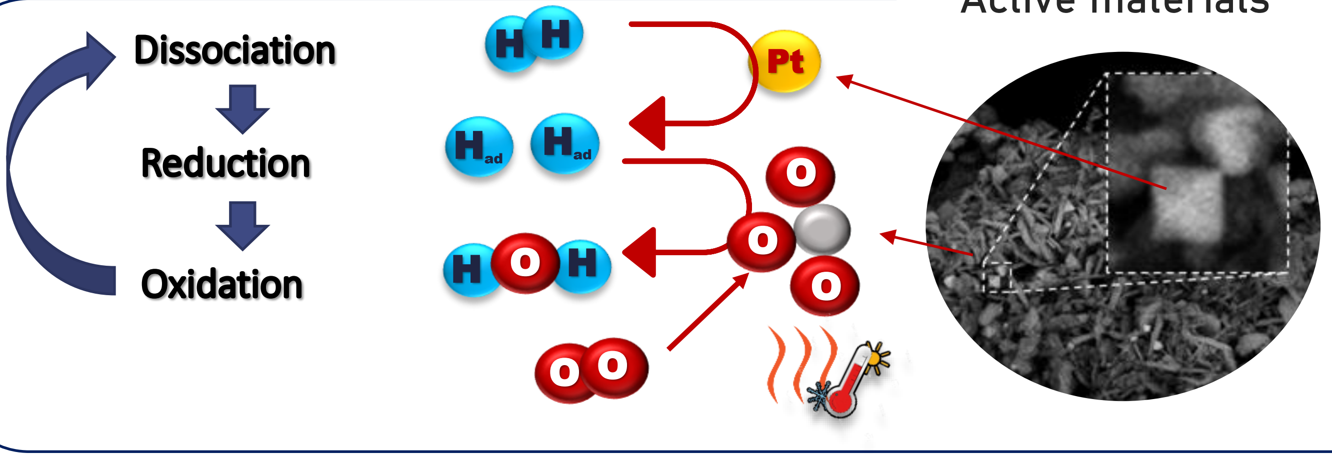

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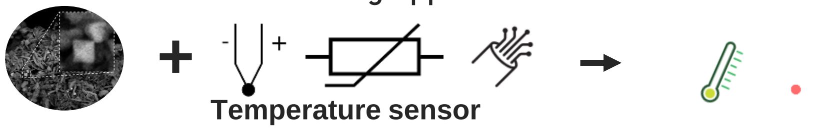
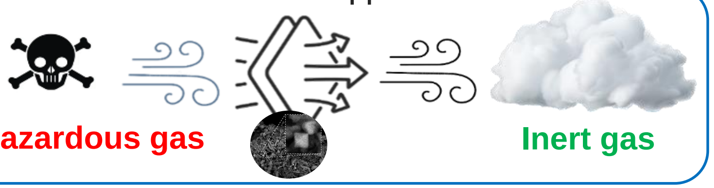


Temperature sensor

Neutralization applications



Hazardous gas

Inert gas

[1] K. Shun et al., *Chem. Sci.* 2022

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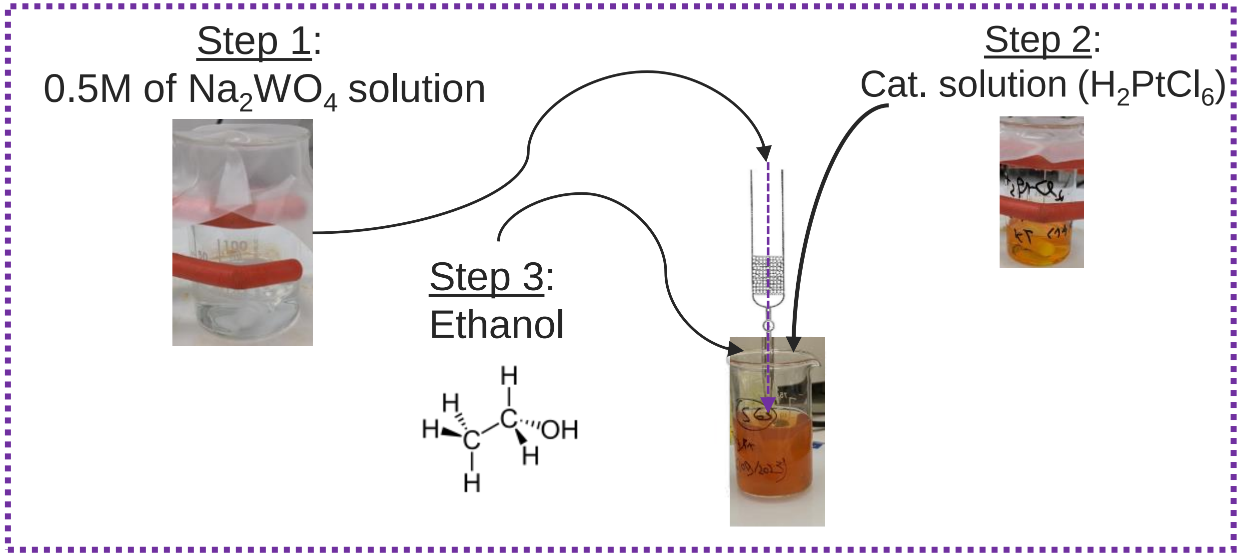
(2) Metal oxides coating: cat./WO_3

General Sol-Gel process protocol: Cat./WO_3 [1, 2]:



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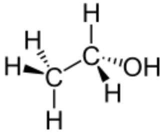
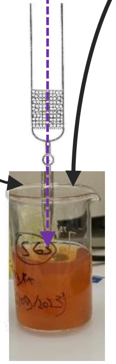
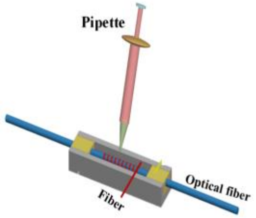




Step 4'

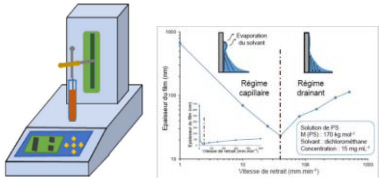
Step 4 : Deposition

Mold



Or:

Dip-coating



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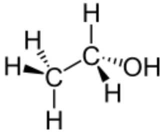

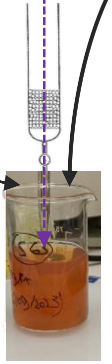

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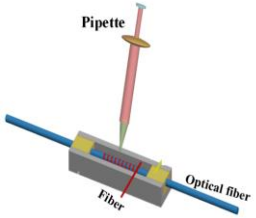

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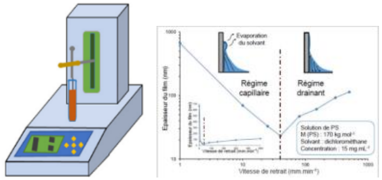
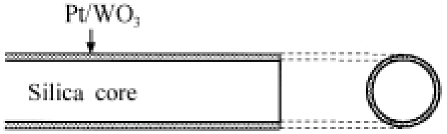


Step 5:
Drying at room temperature

Amorphous oxide



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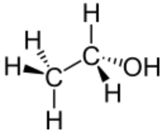

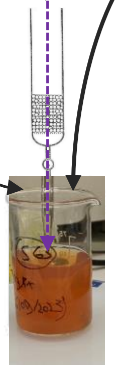

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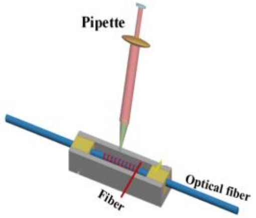

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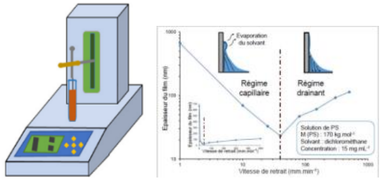

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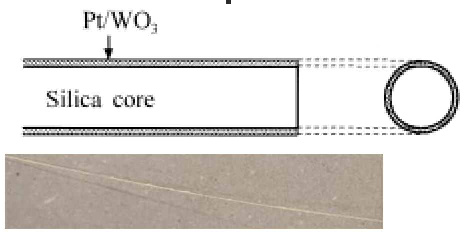

Step 6'

Step 6: Calcination

Crystalline oxide

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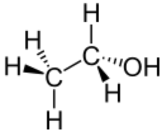

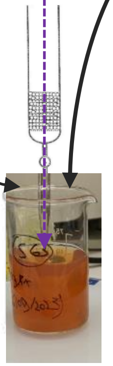

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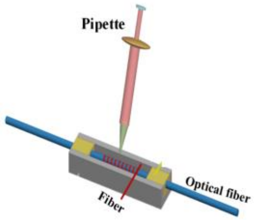

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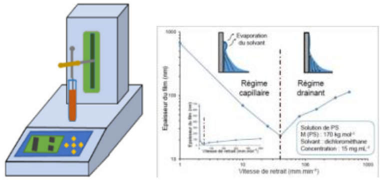

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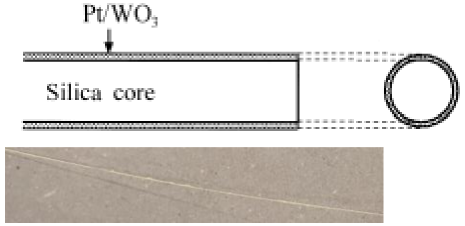

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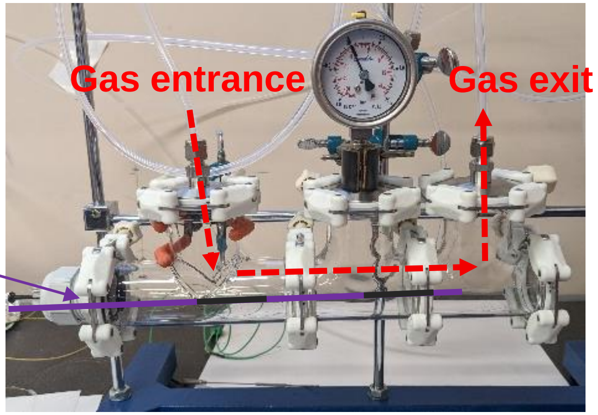
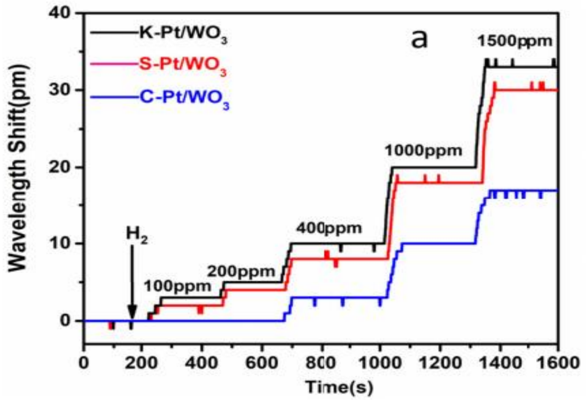
Hydrogen detection test:

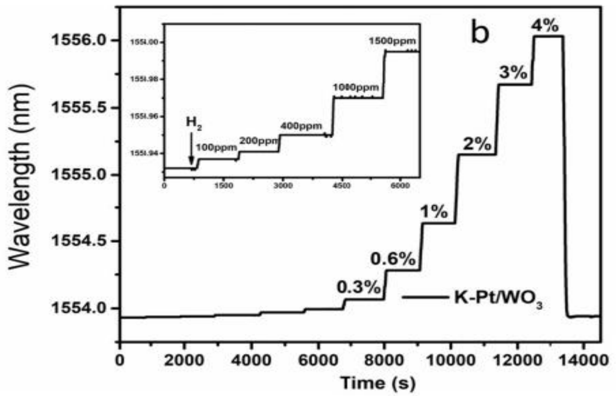
Fiber with coating



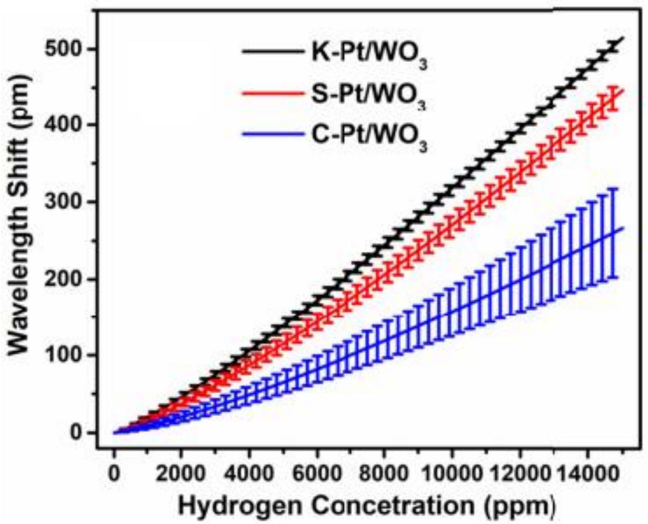
[1] S. Okazaki et al.,, 2003, Sens. Actuators B Chem., « Sensing characteristics of an optical fiber sensor for hydrogen leak »
[2] S. Sekimoto et al.,, 2000, Sens. Actuators B Chem., « A fiber-optic evanescent-wave hydrogen gas sensor using palladium-supported tungsten oxide »

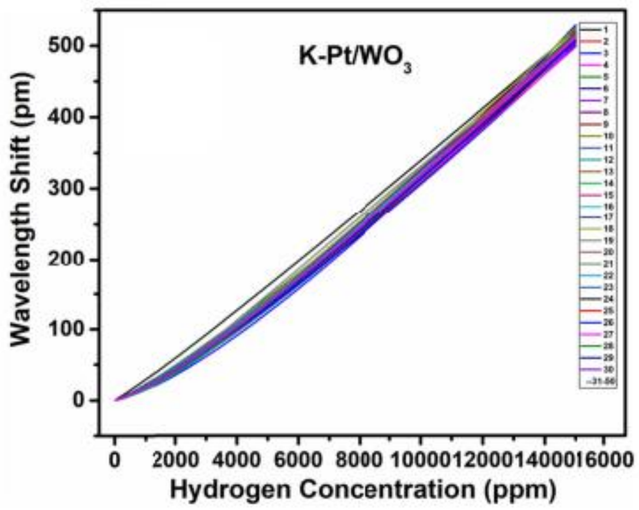
(2) Metal oxides coating: cat./WO_3





S. Yang et al., 2018[1] :
Use of FBG at 1554 nm
Detection of H_2 in air





Detection:

-$t_{s90} \approx$ 20 to 30 s *(detection of 90 % max signal)*

-LOD= 100 ppm v/v H_2

-s= 340 pm/% v/v H_2 in air (K)

-s= 170 pm/% v/v H_2 in air (C, ref)

[1] *S. Yang et al., 2018, Opt. Mater. Express, « Pt nanoparticles encapsulated in mesoporous tungsten oxide to enhance the repeatability of a FBG hydrogen sensor »*

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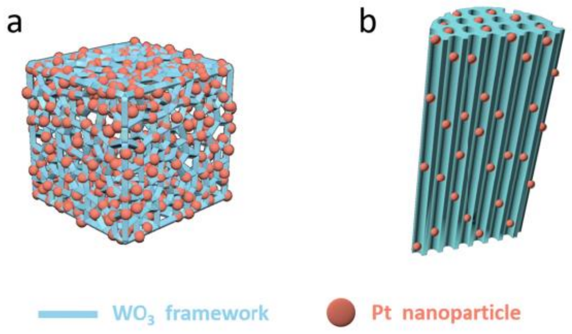

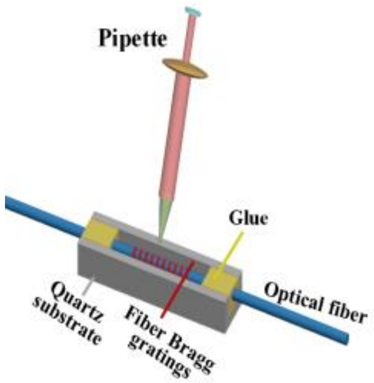

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-t_{s90} ≈ 20 to 30 s *(detection of 90 % max signal)*

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-s= 170 pm/% v/v H_2 dans l'air (C, ref)

-Increase in the specific surface area

Calcination[2]:

Sp=83.94 m²/g (300 °C) ; Sp=11.2 m²/g (500 °C) ; Sp= 4.51 m²/g (600 °C)

S. Yang et al., 2018 : Sp=53.4 m²/g (K)

[1] S. Yang et al., 2018, Opt. Mater. Express, *« Pt nanoparticles encapsulated in mesoporous tungsten oxide to enhance the repeatability of a FBG hydrogen sensor »*

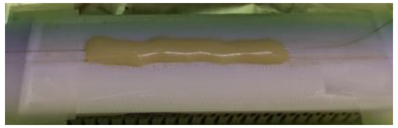
[2] D. Susanti et al., 2014, Front. Chem. Sci. Eng., *« WO3 nanomaterials synthesized via a sol-gel method and calcination for use as a CO gas sensor »*

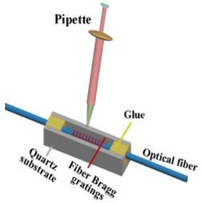
4. Results:

H_2 detection performances:

How to get good performances?

1) Coating thickness[1]:





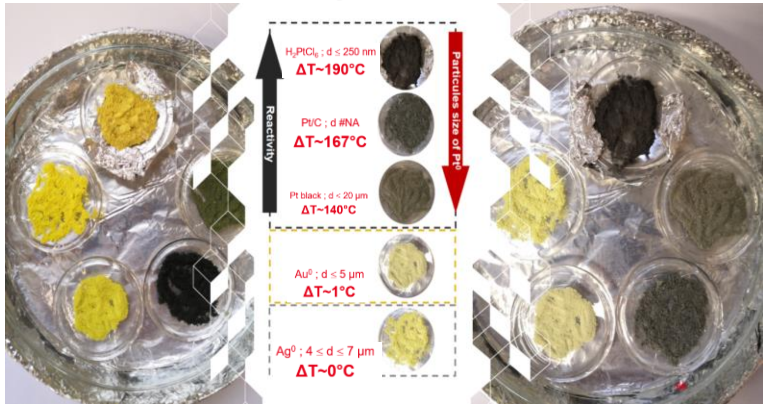
Mold method

[1] S. Yang et al., 2018, Opt. Mater. Express.

4. Results:

H_2 detection performances:

2) Chemical composition optimization



-Tests with other metal oxide, and catalysts
-Decrease of catalyst concentration 7.2 → 0.1 at.%

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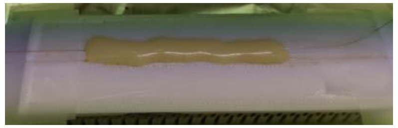

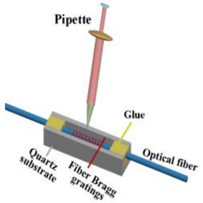

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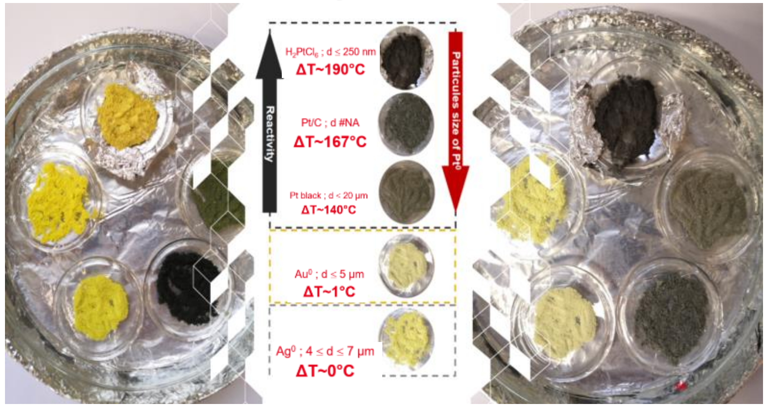

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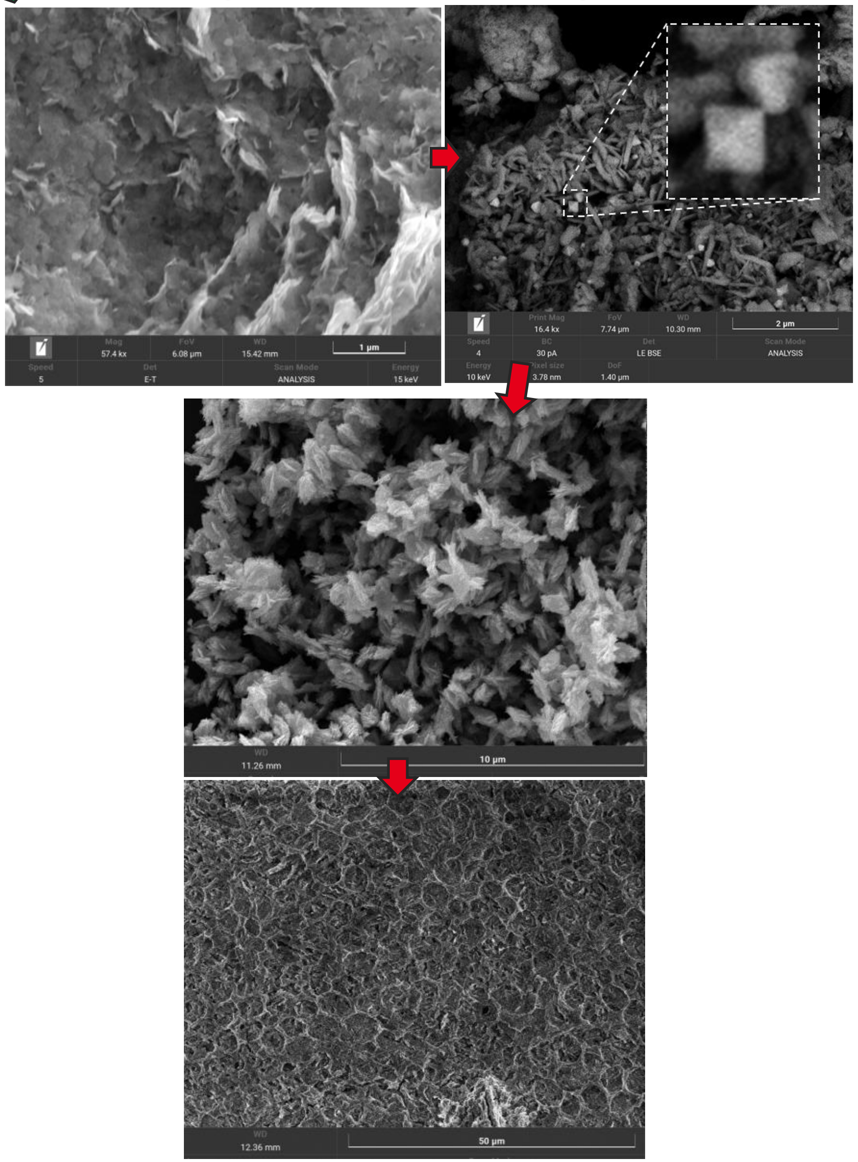

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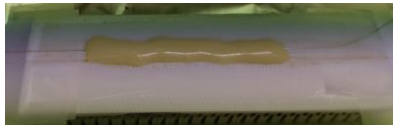

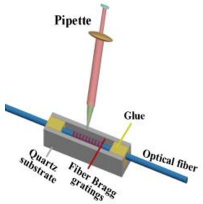

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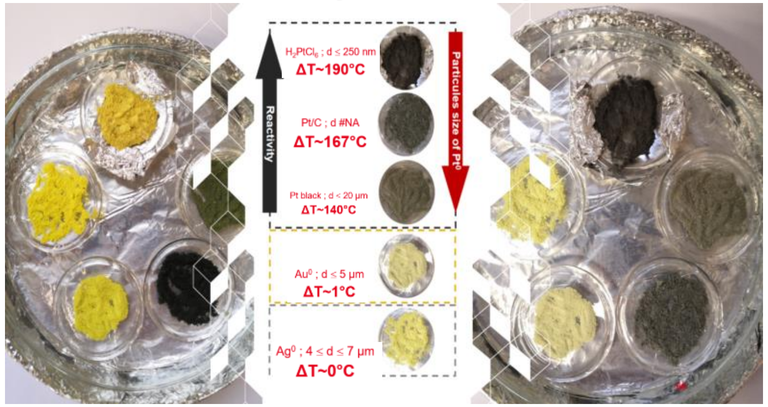

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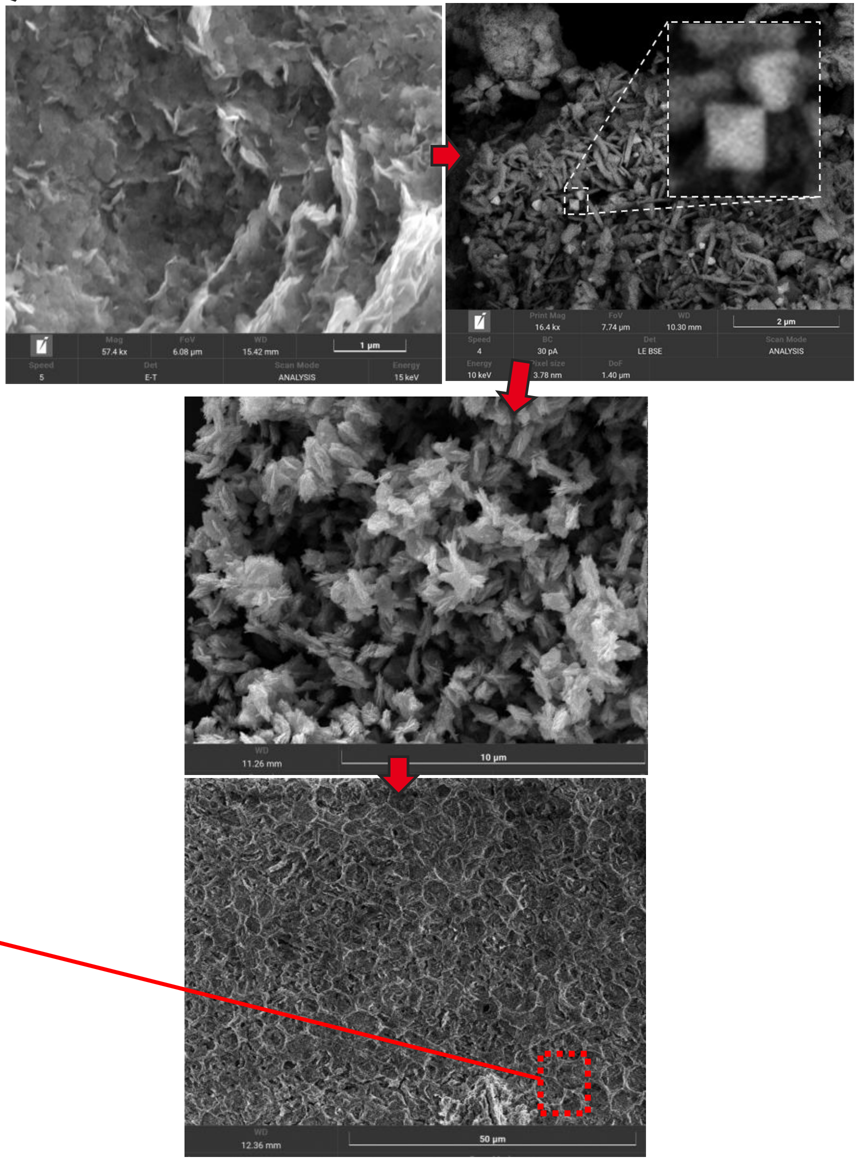

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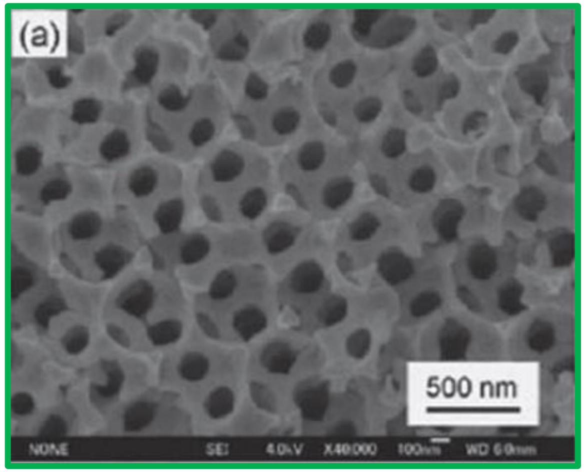

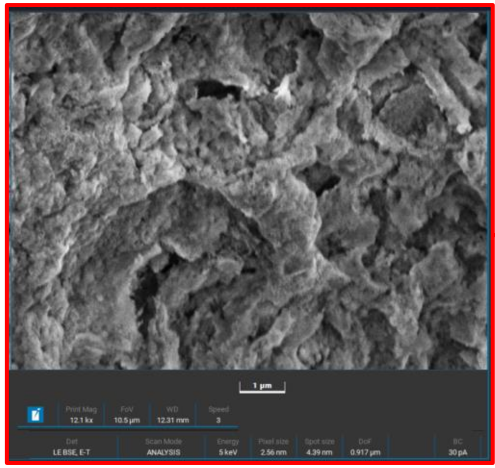

[2] H. Zheng et al., 2011, Adv. Funct. Mater.

[1] S. Yang et al., 2018, Opt. Mater. Express.

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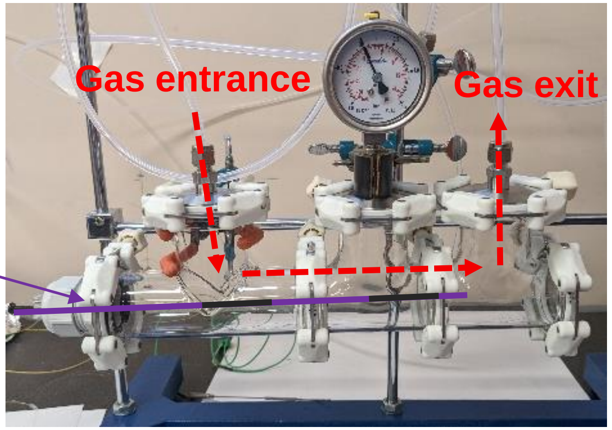
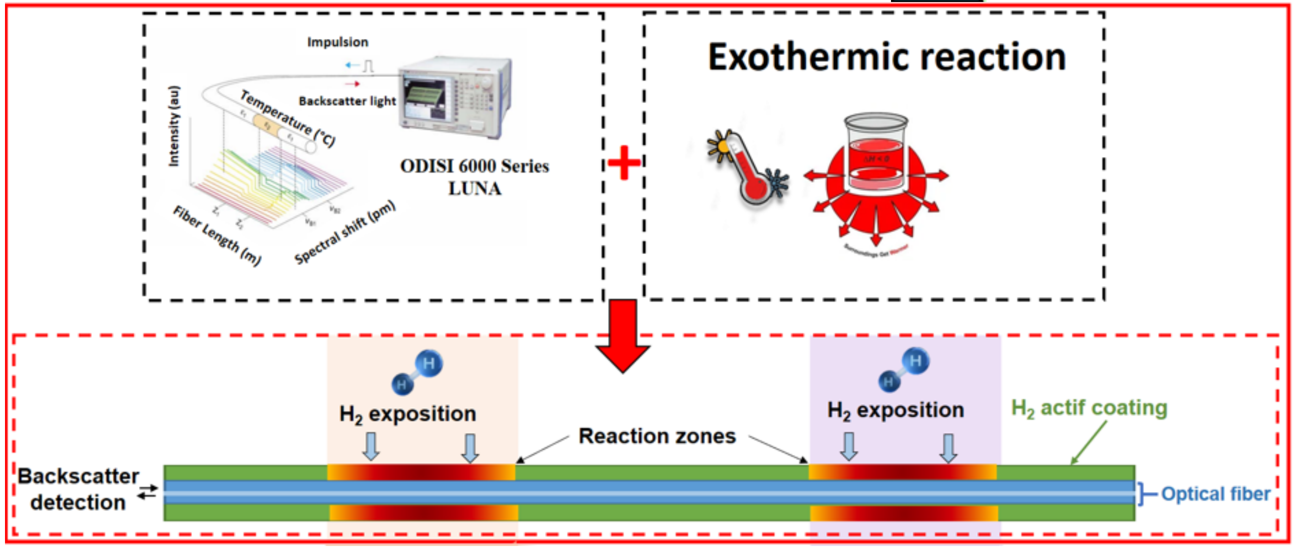
H_2 distributed detection:

Experimental bench:





Measurements:

-1.81 % v/v of H_2 in the air

-Spatial resolution : 1.2 mm

-Gas flow= 830 mL/min (minimal)

-t_{1VCell} = 45 s

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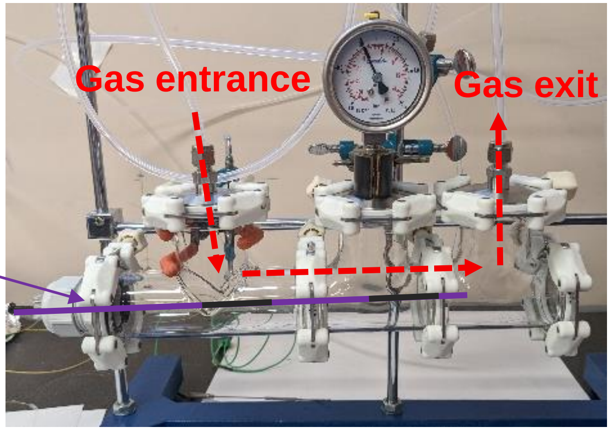
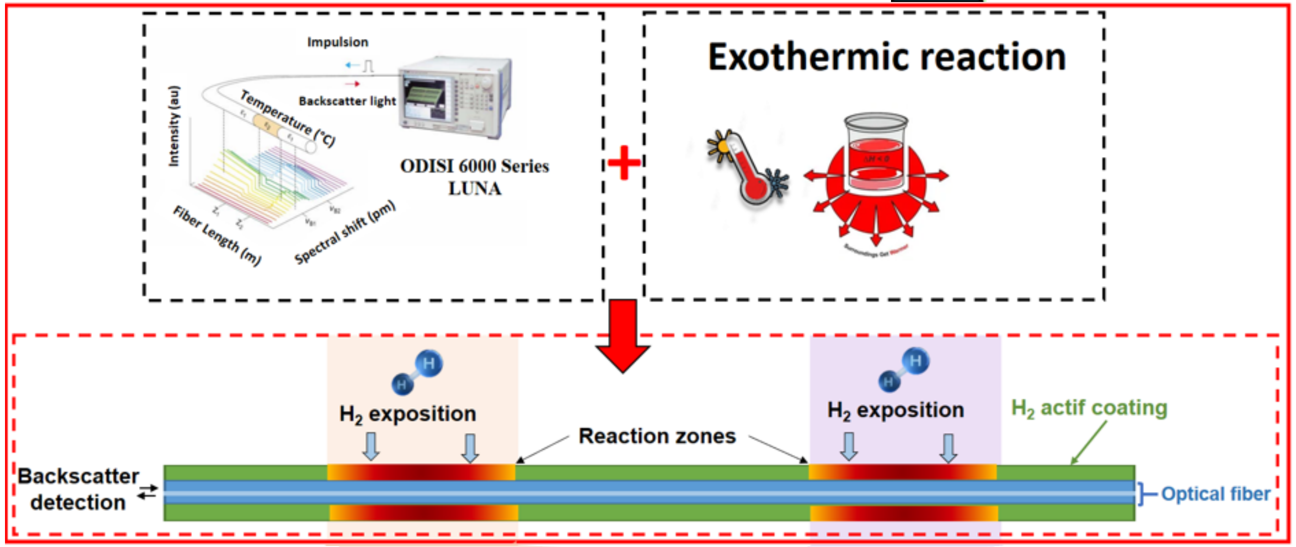
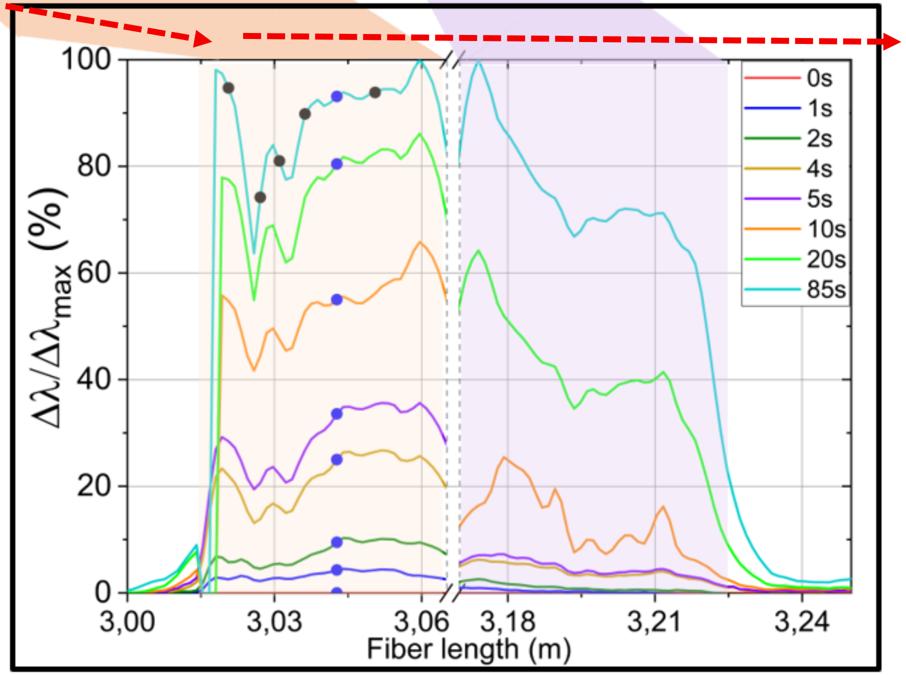




Gas entrance

Gas exit



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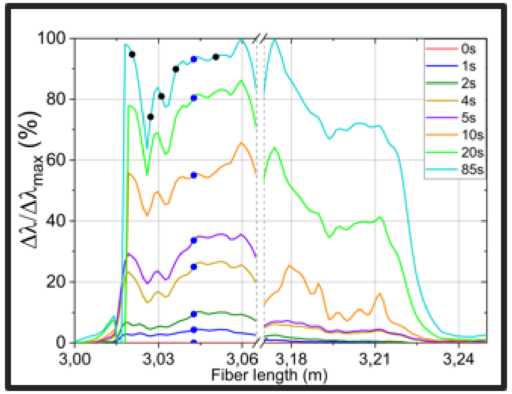

Parameters:

- R=1.2 mm
- **H_2 from 1.81 to 0.009 % v/v (air)**
- **F=5 L/min (maximal)**

[1] S. Yang et al., 2018, Opt. Mater. Express.

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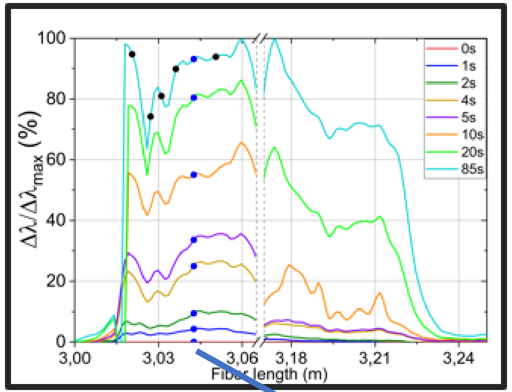

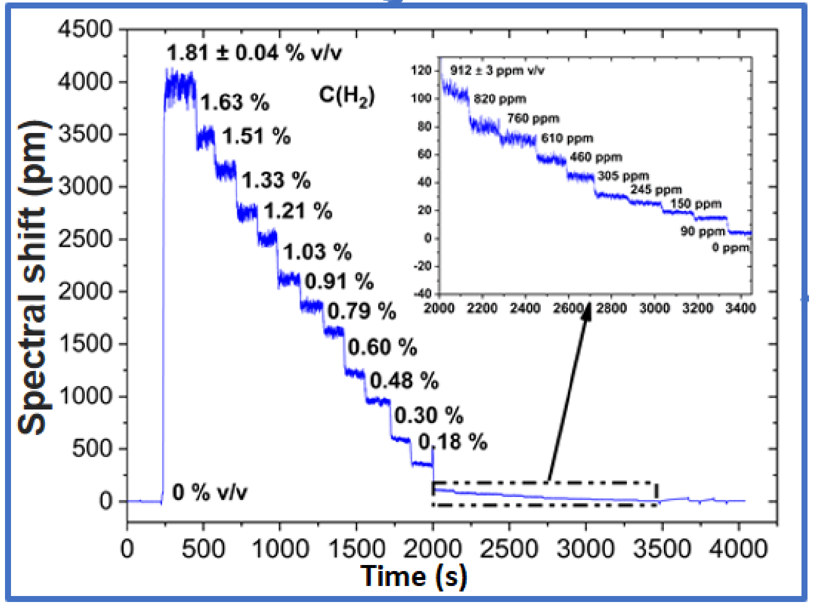

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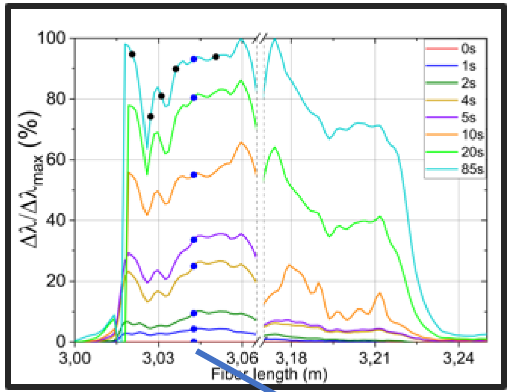
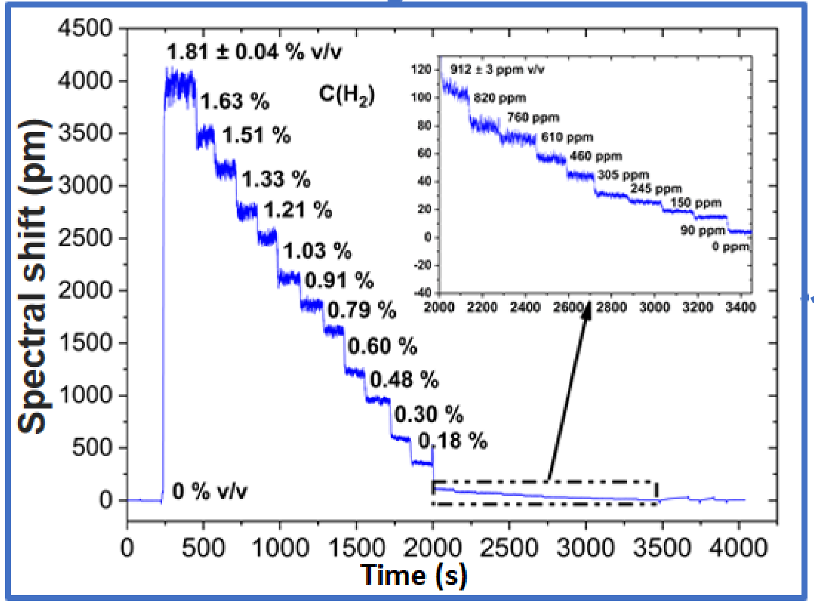
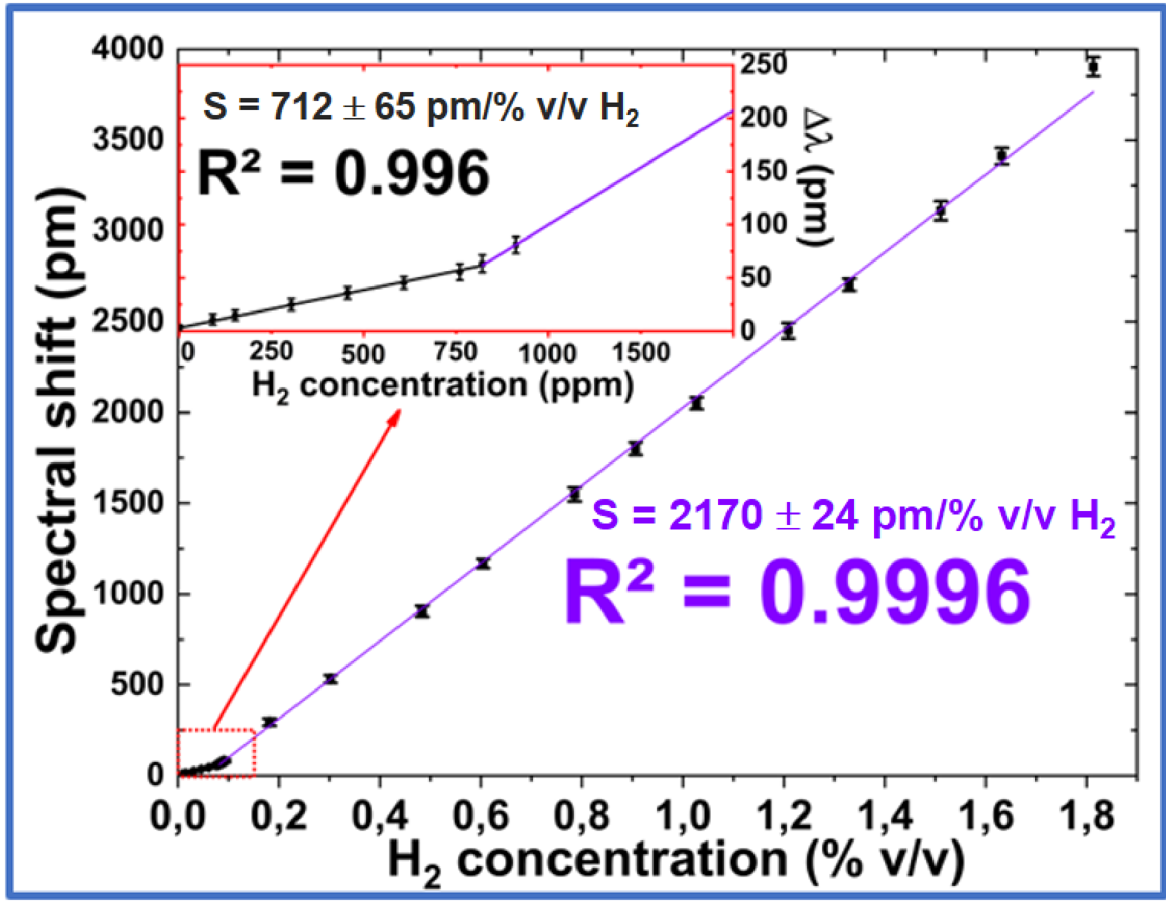


Performances:
- ✓ Sensitive! (X 6.4).[1]
- ✓ Repeatable (preexposition > 10 h)
- ✓ **$t_{90\%}$ few seconds** (<10 s 90 % S_{max})
- ✓ No hysteresis





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4. Results:

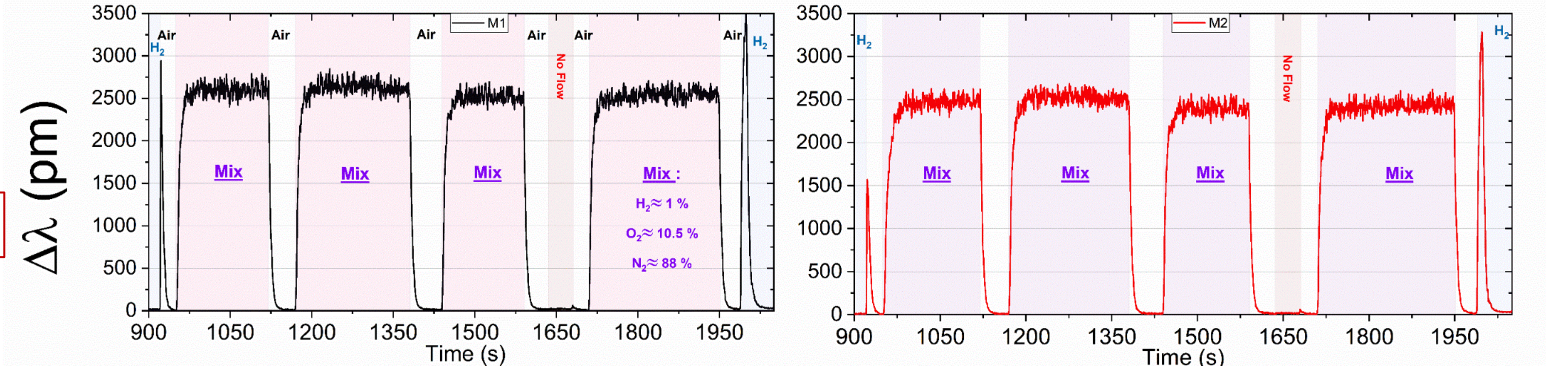
Detection performances with low O_2 concentration:

Thermal sensitivity ≈ 10 pm/°C

Mix [Air/H_2(N_2)]:

- $H_2 \approx 0.9$ % ; **$O_2 \approx 10.5$ %** ; $N_2 \approx 88$ %

- slope = 2544 ± 150 pm/% H_2 (RSD=6 %; 4 repetitions)



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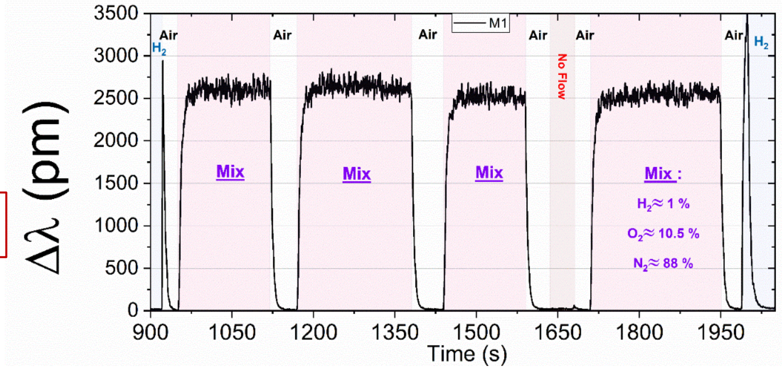
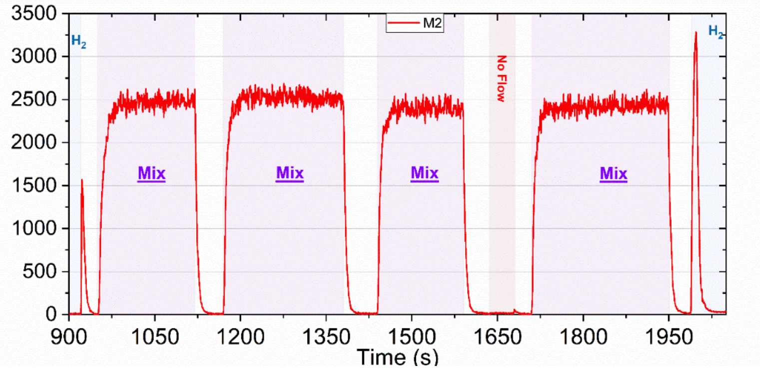
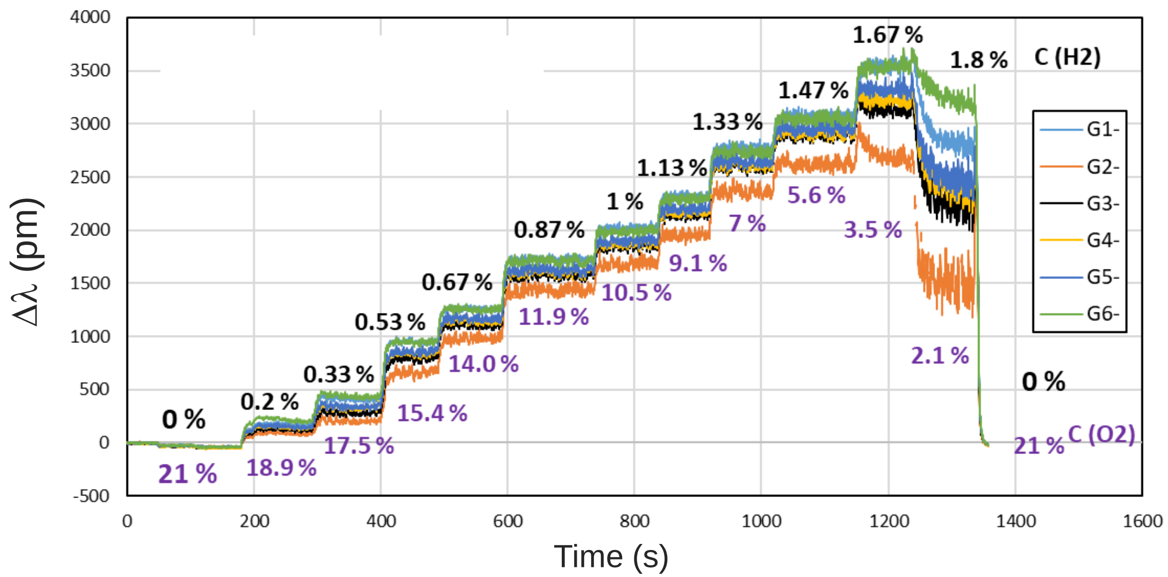






Mixes [Air/H_2(N_2)]:

- $H_2 \in [1.8 ; 0]$ % ; **$O_2 \in [2.1 ; 21]$ %**

Mix [Air/H_2(N_2)]:

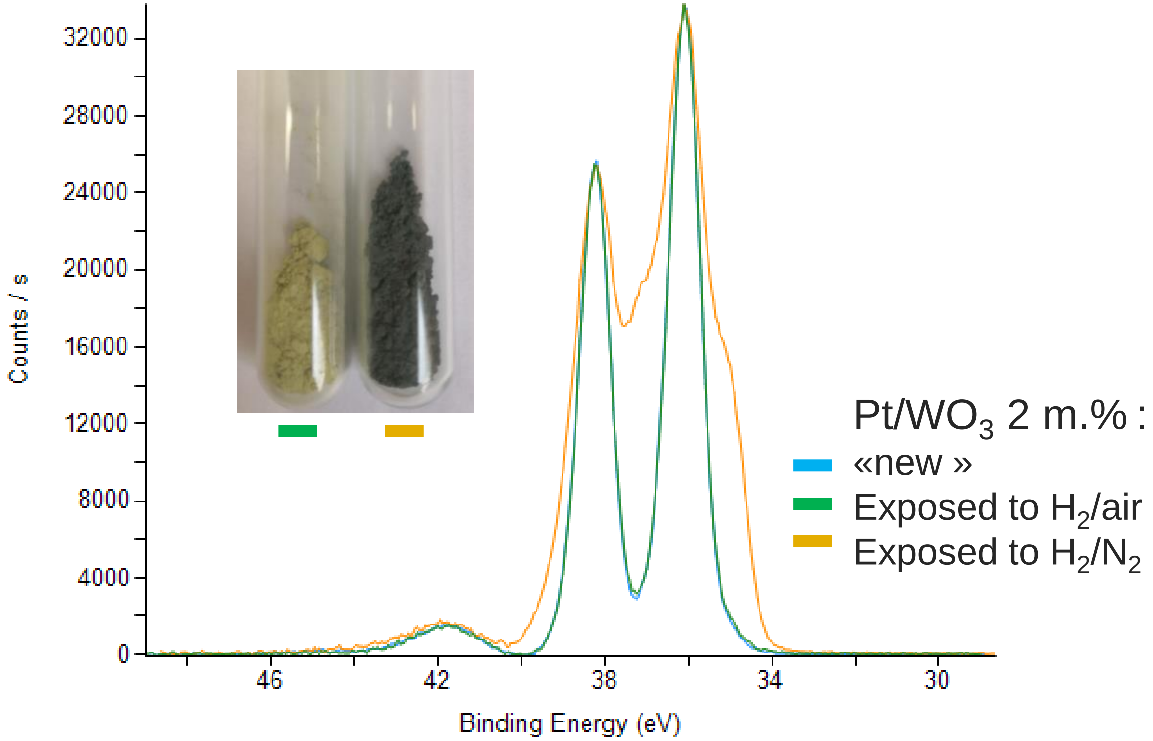
- slope = 2080 ± 125 pm/% H_2 (RSD=6 %; 4 repetitions)

[1] S. Yang et al., 2018, Opt. Mater. Express.

4. Results:

Coating characterization:

Reduction of Pt/WO_3 2 m.% in H_2/N_2 2 m.%



XPS W4f comparison of the same compound Pt/WO_3 with 2%$_{mol}$ before and after exposition to H_2 in N_2 to 2%$_{mol}$

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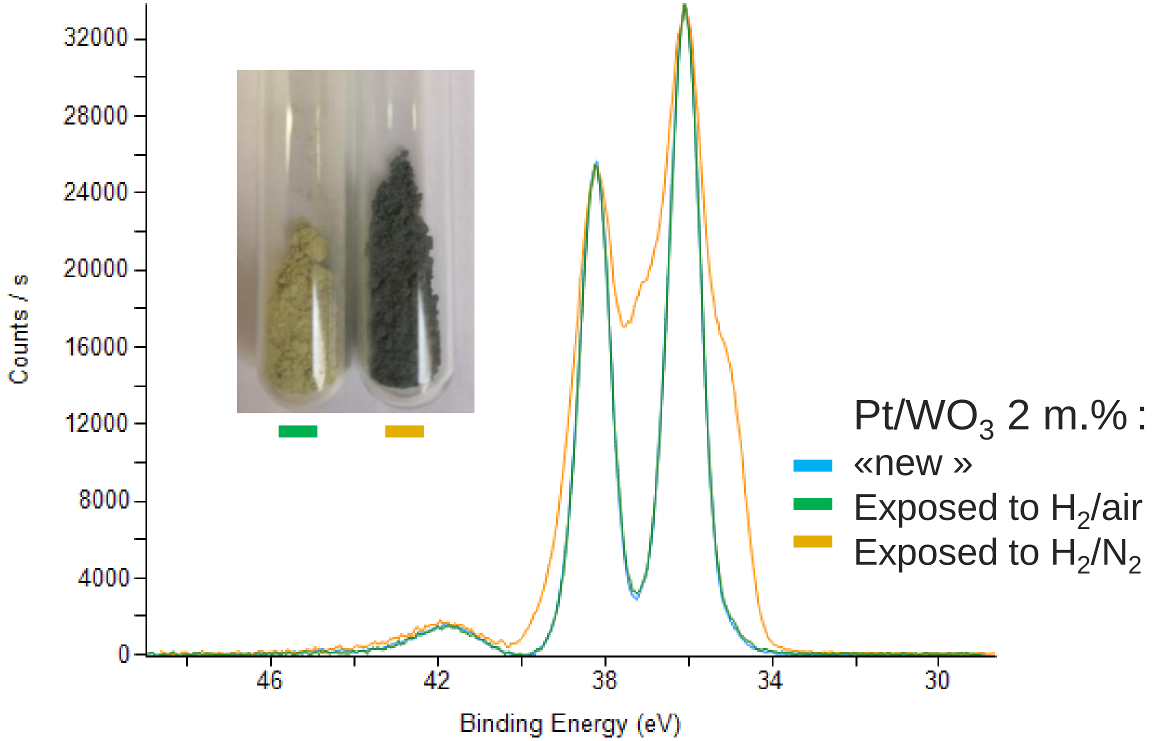

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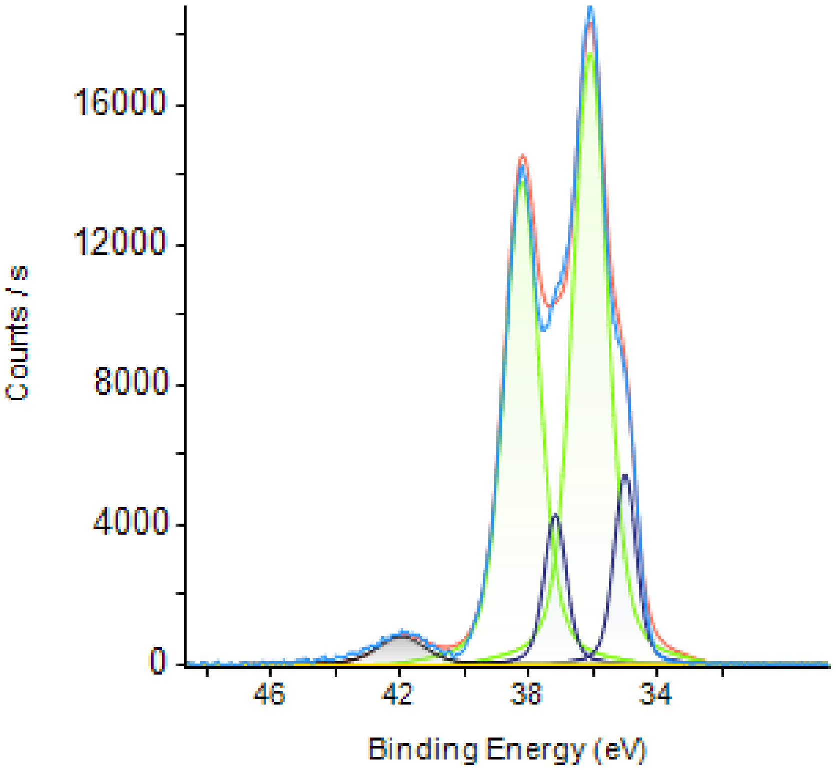

XPS W4f spectrum fit for Pt/WO_3 with $2\%_{mol}$ after exposition to H_2 in N_2 with $2\%_{mol}$

Pt/WO_3 $2\%_{mol}$	BE (eV)	species
W $4f_{7/2}$	36,10	WO_3
W $4f_{5/2}$	38,27	WO_3
W $4f_{7/2}$	34,90	$WO_{2.72}$
W $4f_{5/2}$	37,07	$WO_{2.72}$
W 4f	41,75	loss

NIST XPS Database

Firmin BOURGES

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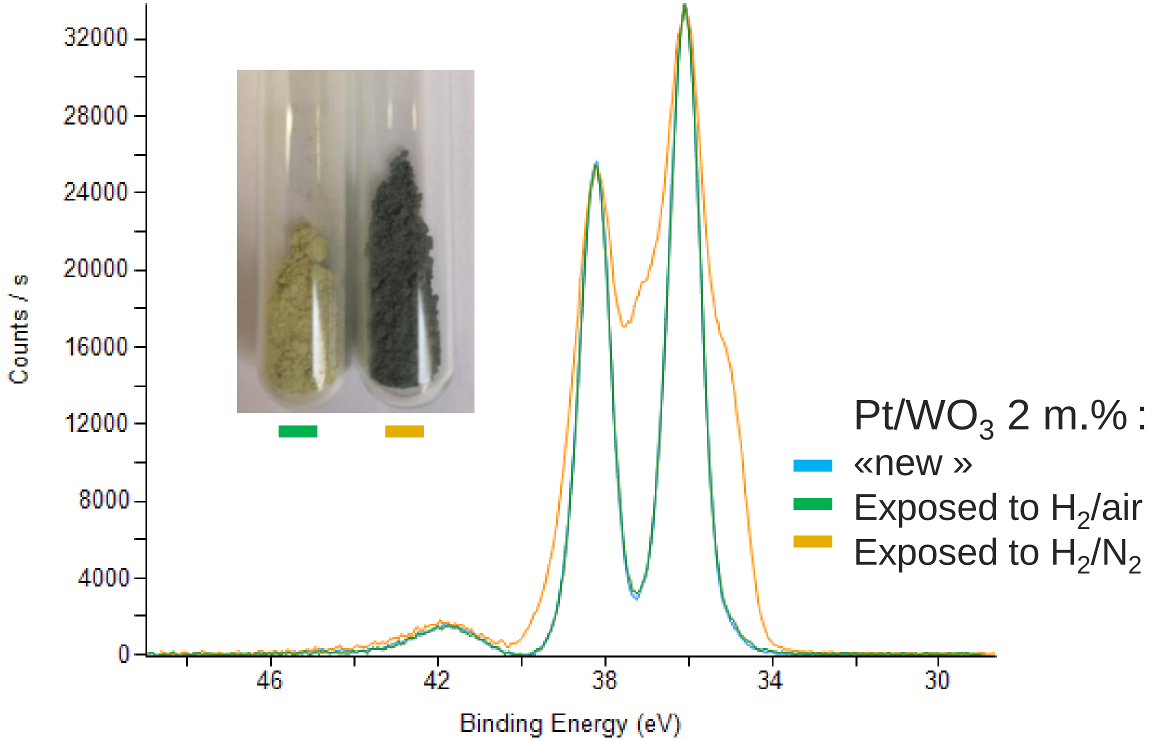

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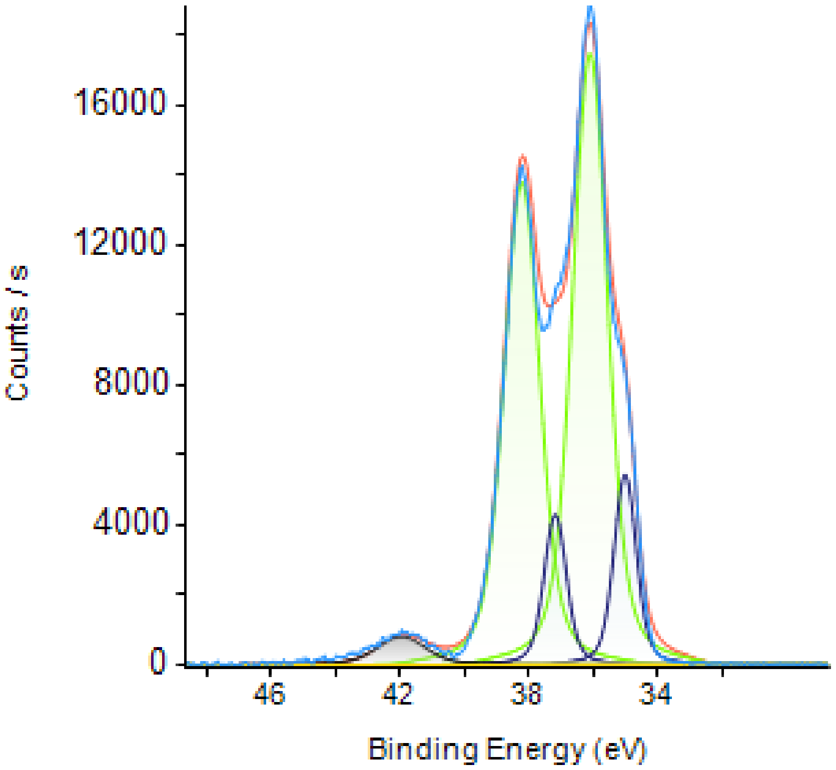

XPS W4f spectrum fit for Pt/WO_3 with $2\%_{mol}$ after exposition to H_2 in N_2 with $2\%_{mol}$

<u>Partial reduction of WO_3 in WO_{3-x}:</u>

- ✓ **Reduced to $WO_{2.72}$**
- ❑ XRD verification
- ❑ In situ Raman measurements

Pt/WO_3 $2\%_{mol}$	BE (eV)	species
W $4f_{7/2}$	36,10	WO_3
W $4f_{5/2}$	38,27	WO_3
W $4f_{7/2}$	34,90	$WO_{2.72}$
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NIST XPS Database

Firmin BOURGES

5. Conclusion and perspectives:

Conclusion:

- ✓ Distributed measures(Rayleigh OFDR) → Hydrogen detection in the air from **1.8 % to 90 ppm**
- ✓ **Important sensitivity** (**X 6.4** : *S. Yang et al., 2018*)
- ✓ Response time of few seconds
- ✓ Reversibility, no hysteresis, good repeatability
- ✓ Selectivity (~~CH_4~~)
- ✓ Detection perform with other metal oxide

- ✓ Test of differents compositions and forms of catalysts

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Perspectives:

-Test with different humidity levels

-Tests under 33 % of H_2 (shortly)

-Measurements with CO and NH_3

For your attention, Thank you

Any questions, do you have ?

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