UNIVERSITI PUTRA MALAYSIA

RICULTURE • INNOVATION • LIFE

Highly Sensitive Hydrogen Sensor Based on Palladium Coated Tapered Optical Fiber at Room Temperature

Mohammed M. Alkhabet
Saad Girei
Suriati Paiman
Norhana Arsad
Mohd Adzir Mahdi
Mohd Hanif Yaacob

CONTENT

1 INTRODUCTION

2 PROBLEM STATEMENT

3 OBJECTIVES

4 METHODOLOGY

5 RESULTS

6 CONCLUSION

INTRODUCTION

Hydrogen (H_2) has high energy content, making it an ideal clean fuel with several application potentials in different industries.

Optical offers interesting properties, such as light weight, resistance to EI, instability, and rigidity in harsh environments.

Palladium (Pd) is currently receiving interest in many applications, such as H_2, the hydrogenation process, and the detection of H_2.

Semiconducting metal oxides can provide good sensitivity, selectivity and stability sensors.

PROBLEM STATEMENT

- Hydrogen is flammable at concentrations > 4 vol% in the air and can explode at a wider range of 15–59 vol% at standard pressure.

- Currently, there are several types of H_2 sensors available.
  - Electrical sensors (i.e. chemiresistor or microelectronic) are susceptible to electromagnetic interference (EMI) which can affect their response to signals.

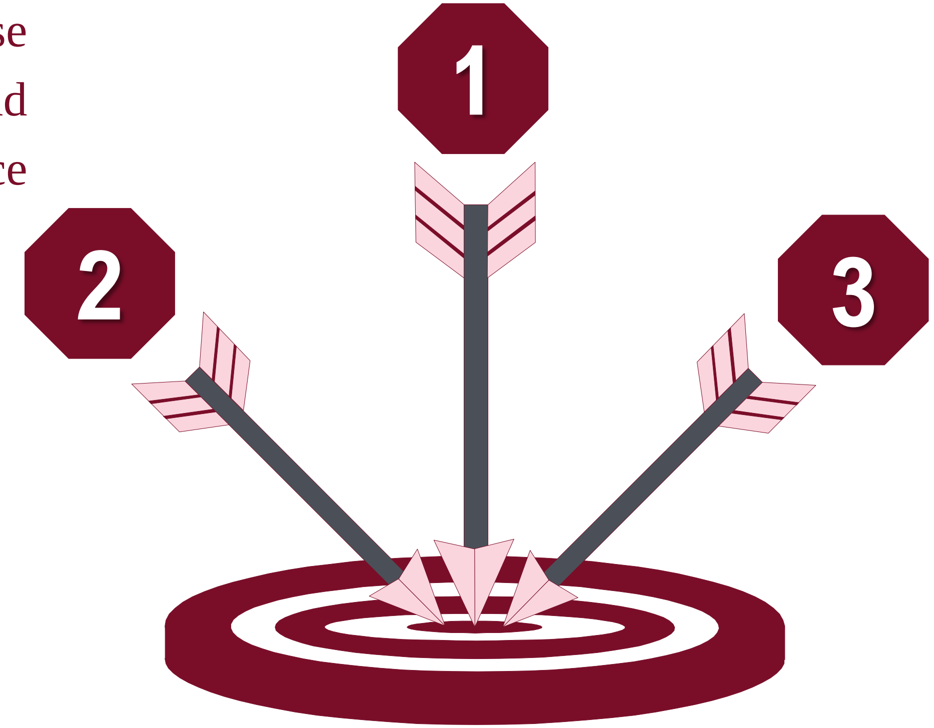
- On the other hand, optical fiber that offers other advantages, such as lightweight, small size, resistance to EMI, non-inductiveness, and ruggedness in harsh environments.
  - These properties make optical fiber an ideal candidate for H_2 detection in rugged environment.

OBJECTIVES

To design and develop hydrogen gas sensors based on Pd NPs coated on tapered optical fiber via drop-casting technique.

To evaluate the optical fiber sensor performance (sensitivity, response and recovery time. repeatability, and selectivity) based on absorbance measurement.

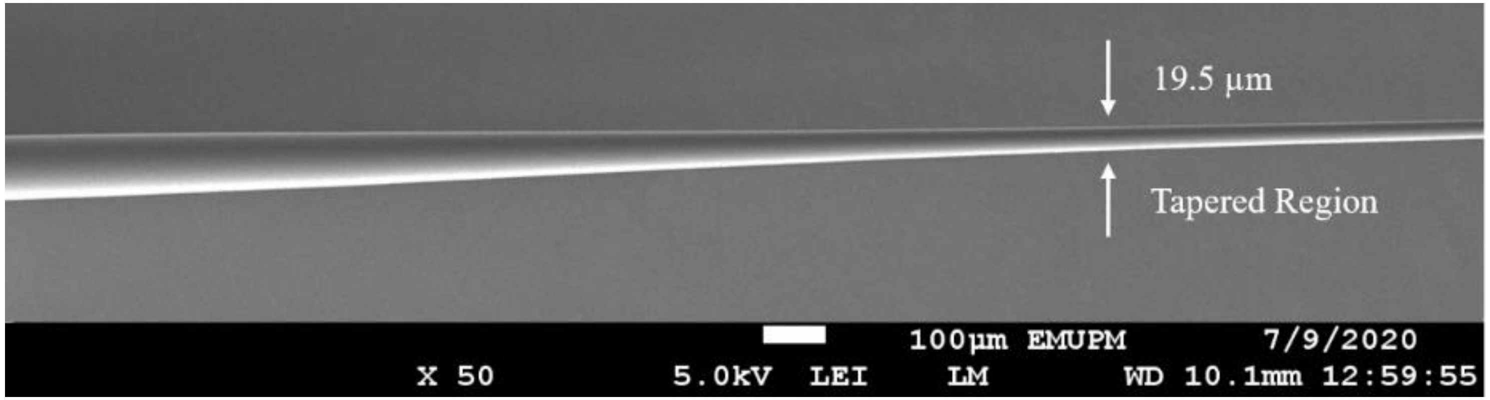
To discuss the sensing mechanism of gas molecules-sensing layer interaction of tapered optical fiber sensor.



METHODOLOGY

Fabrication of Tapered Optical Fiber

- Multimode Optical fiber (MMF) was fabricated with cladding and core diameters of 125 µm and 62.5 µm respectively, as a transducing platform.
- The MMF was tapered from cladding diameter of 125 µm to waist diameter of 20 µm, waist-length of 10 mm, and down taper and up of 5 mm.
- The tapering was done using the Vytran glass processing machine (Vytran GPX-3400).
- The machine works based on a heating and pulling process, using a graphite filament as a heater to achieve the desired geometry of the tapered profile.



METHODOLOGY

Palladium Functionalization of the Tapered Optical Fiber

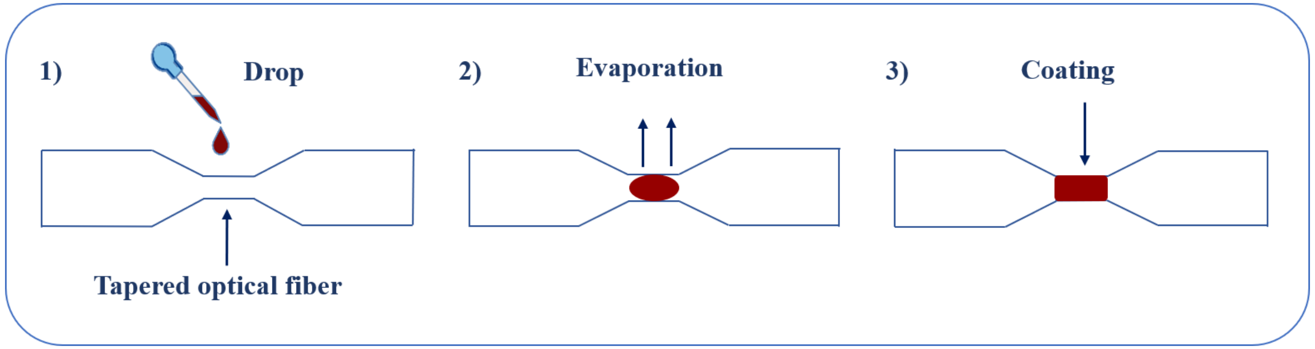
- The Pd sensor was fabricated following a simple one-step process by mixed :

  - 0.1 mL of hydrochloride acid
  - 0.9 mL of palladium chloride ($PdCl_2$)
  - 10 mL of deionized water

  The solution was placed in an ultrasonic bath and left for 15 minutes to homogenize.

- The coating of the tapered optical fiber was done using the drop-casting technique.

  - A drop of the mixture (approx. 10 μL) was dropped into the base of the tapered optical fiber.
  - Heating the sample at 80 °C for 15 minutes in the oven to ensure complete evaporation of the aqueous medium.

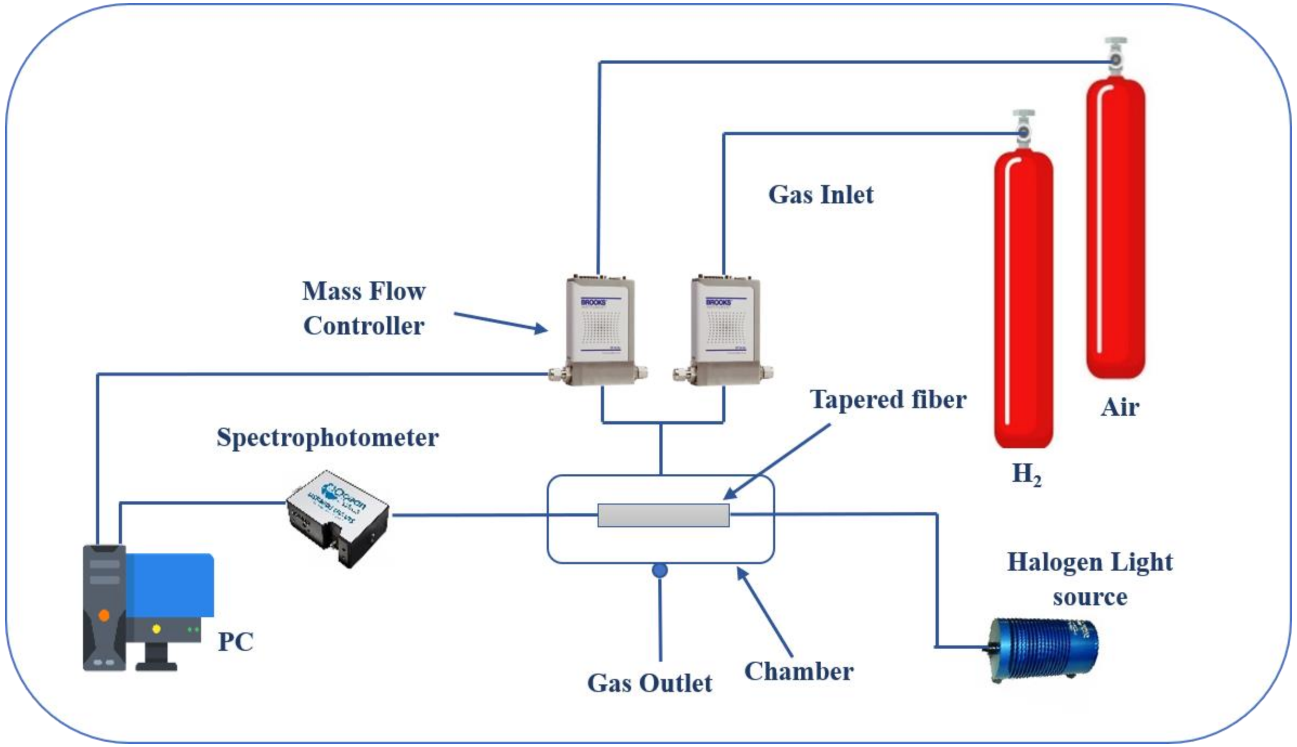


METHODOLOGY

The experimental setup

- The gas optical sensing system consists of a light source (Tungsten Halogen, HL-2000, Ocean Optics USA) with coverage wavelength of 360 to 2500 nm, a spectrophotometer (USB 4000, Ocean Optics USA) with a detection range of 200-1100 for monitoring the optical absorption spectrum, and a dedicated gas chamber.

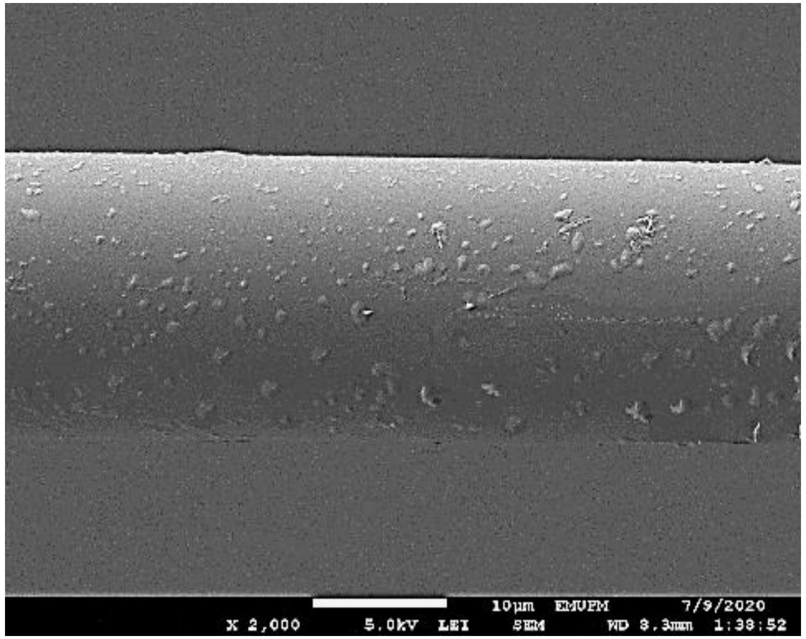
- The Pd coated sensor was placed in a closed gas unit and purged with the centrifuge from a computer-regulated mass flow controller at a gas flow rate of 200 sccm.

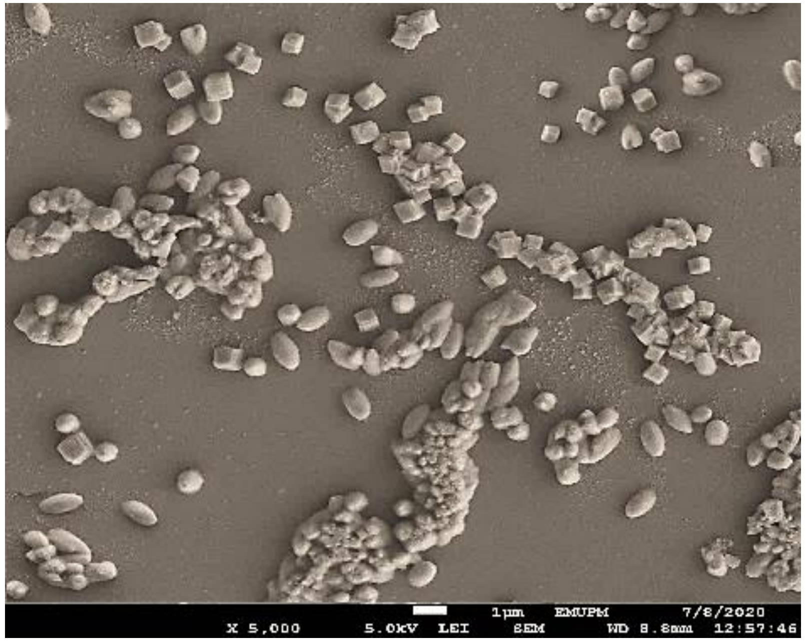


METHODOLOGY

Material Characterization

- The films' morphology was observed using FEESEM (JSM-7600F).
- The FESEM images of Pd nanoparticles show that the Pd NPs are clearly formed and separated.





METHODOLOGY

Material Characterization

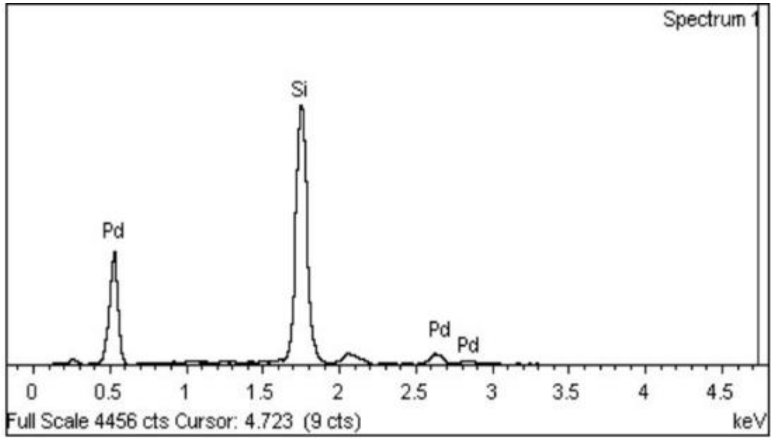
- The elemental composition was determined through an EDX analysis as shown in Figure (a).

- The EDX pattern of Pd revealed that the important elements in Pd films are Pd and O, as evidenced by their respective peaks.

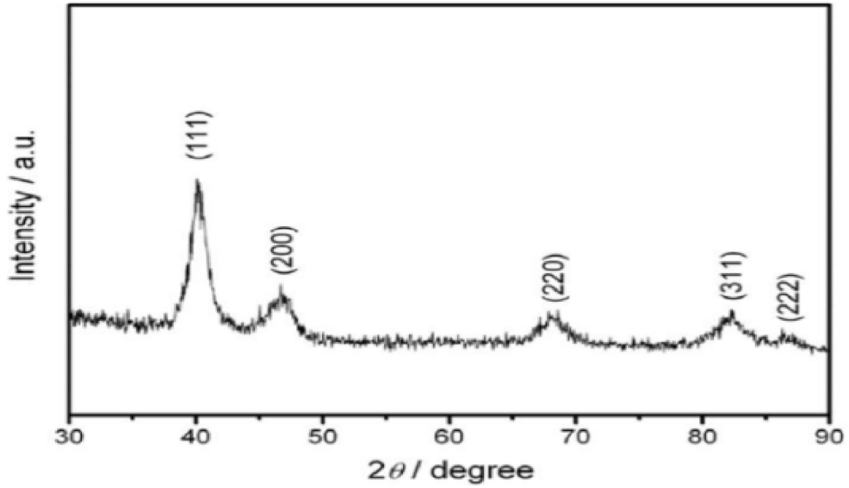
- Material identification, crystallinity, and phase transition of Pd was observed by an XRD analysis (APD 2000) as shown in Figure (b).

- XRD patterns of the Pd-coated sensor recorded in range 2θ, from 30° to 90°.

- There are five distinct reflections in the reflection at 40.02° (111), 46.49° (200), 68.05° (220), 82.74° (311) and 86.27° (222).



(a)



(b)

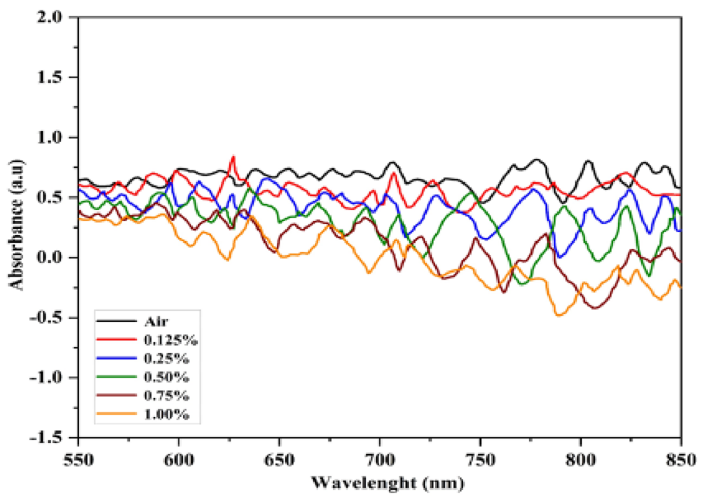
RESULTS AND DISCUSSION

- The absorption spectra of the sensor coated with Pd to synthetic air at room temperature with different concentration 0.125% to 1.00% H_2 .

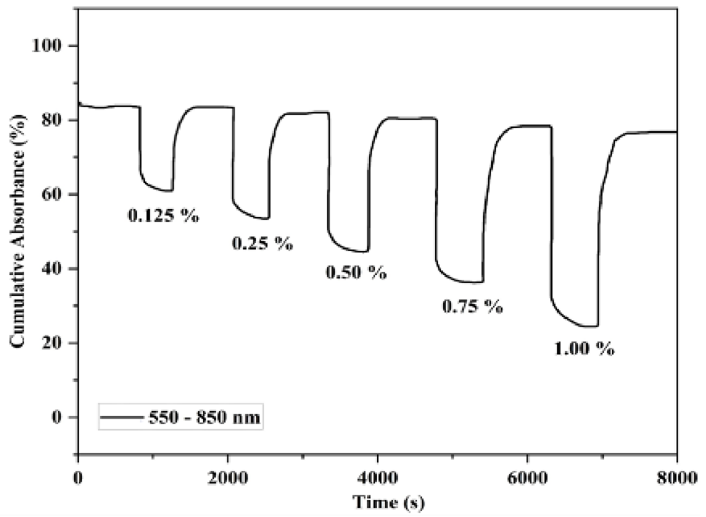
- The Pd sensor demonstrated notable changes in absorbance, especially in the wavelength range of 550-850 nm as shown in Figure (a).

- The response time and recovery time of the Pd costed sensor was 50 sec and 200 sec respectively. Changes in absorption at 0.125% H_2 are about 24% and 52% higher at 1.00% H_2 as shown in Figure (b).

- The Pd coated sensor showed stronger absorbance and recovery of H_2 at higher absorption changes.



(a)



(b)

RESULTS AND DISCUSSION

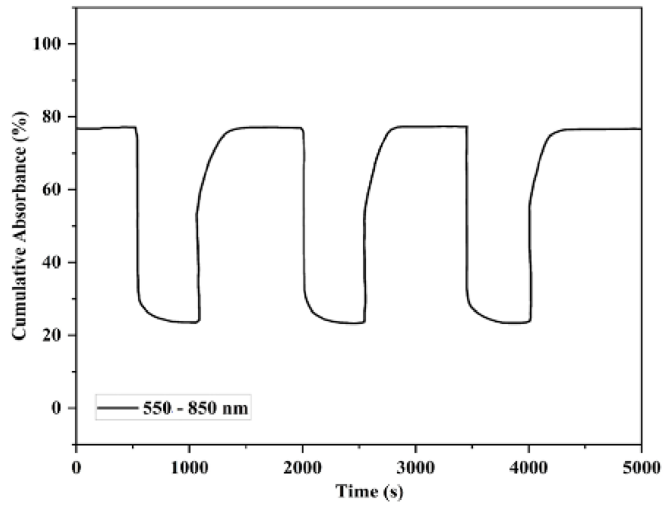
- Sensor repeatability was confirmed by exposure of the sensor to 3 cycles of 1.00% H_2, as shown in Figure (c). Overall, the Pd coated sensor showed a high level of good repeatability of H_2.

- A test for selectivity was done for Pd coated sensor toward NH_3 and CH_4 gas at 1.00% concentration as shown in Figure (b).

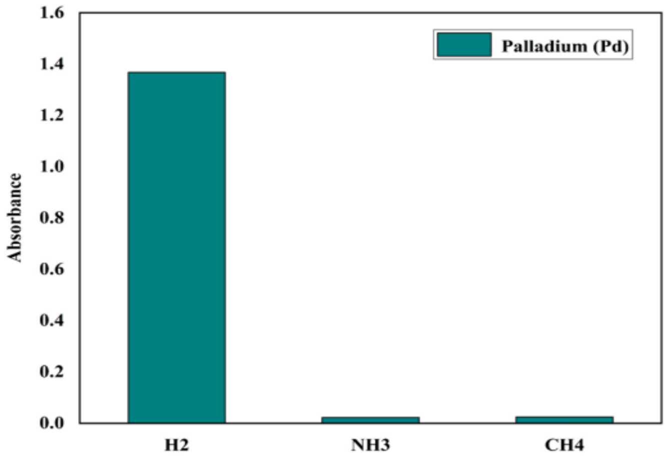
- The Pd coated sensor showed a remarkably high H_2 absorbance response with a weak response for other gases.

- CH_4 gas is a stable gas that requires very high energy to dissociate H from C.

- The sensor is less sensitive toward NH_3, probably because of Pd since it is more suitable for dissociating the H_2 gas.



(c)

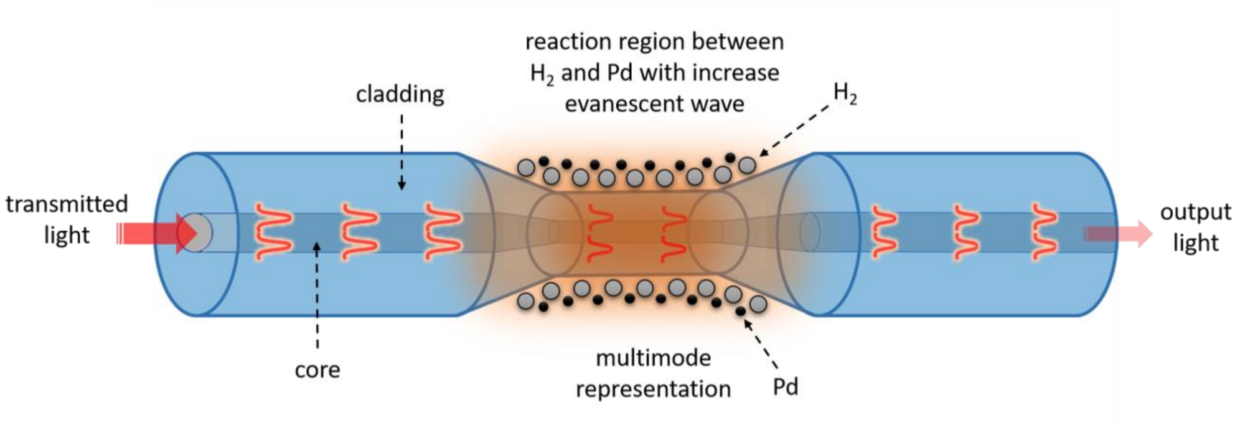


(b)

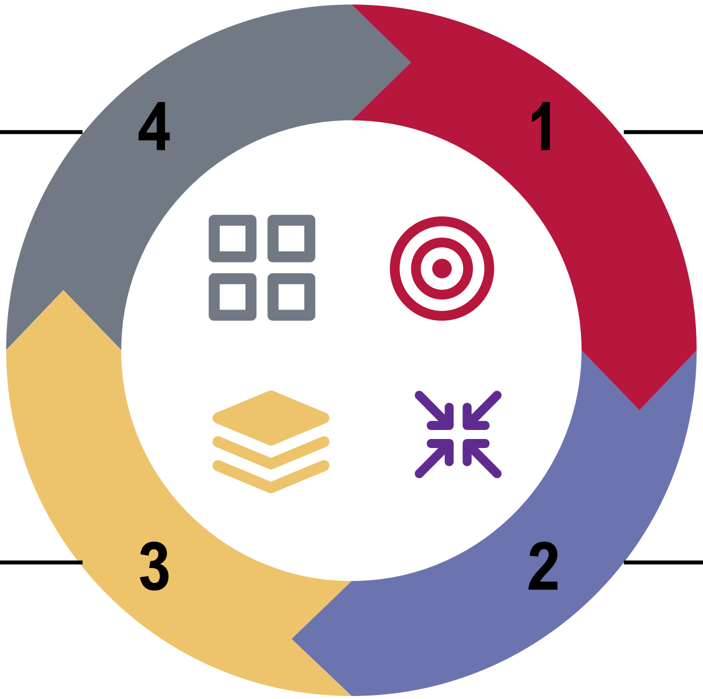
RESULTS AND DISCUSSION

The Sensing Mechanism for Tapered Pd NPs Coated Optical Fibers

- The Pd-coated fiber sensor's optical response occurs because of the reaction of palladium to hydrogen gas.
- Pd absorbs H_2 gas molecules, resulting in it changing into PdHx (where a small percentage expands the Pd particle size).
- The Pd layer increases in thickness and size while absorbing hydrogen, thereby also changing the layer's optical properties.
- The real and imaginary parts alter the permittivity of the Pd layer to result in a corresponding change of boundary conditions on the sensor surface.



CONCLUSION



This study demonstrated that optical fiber sensors could be developed from Pd NPs by employing a drop-casting technique.

The performance of the developed sensor was evaluated in terms of its response at room temperature using different concentrations of H_2 gas.

These evaluations indicated that the Pd-coated sensor exhibited a 52% change in the absorbance response when exposed to 1.00% H_2 in synthetic air.

It is possible to develop an efficient, reliable and reproducible H_2 sensor by using a simple and cost-effective approach under real atmospheric conditions.

THANK YOU