Pd supported membrane purifier: a comparison with other technologies

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Introduction

Hydrogen is one the bulk gases needed for semiconductor processes; it is widely used to grow epitaxial layers to make devices on silicon and in the compound semiconductor industry. The use of purification maintains the same quality of gas over time and also eliminates any impurity contribution coming from the gas distribution system, eliminates variation in gas batch quality, mitigates impurities introduced during the replacement of gas batches, and other random sources of contamination.

Hydrogen has the potential to become a significant vector of clean energy. All of the major car manufacturers are already involved in the development of cars powered by proton exchange membrane (PEM) fuel cells, with thousands of cars being introduced to the market starting in 2015. This technology will be a great step ahead in the introduction of clean cars because the exhaust consists of only water vapor. A necessary requirement for the mass adoption of this new vehicle technology is the development of a suitable infrastructure capable of filling car tanks at high pressure, 700 bars, with high purity hydrogen. The specification limits for some impurities, such as carbon monoxide (CO) and sulphur compounds, are very tight, down to 100 ppb or even less, because of their ability to deplete the lifetime of the fuel cells.

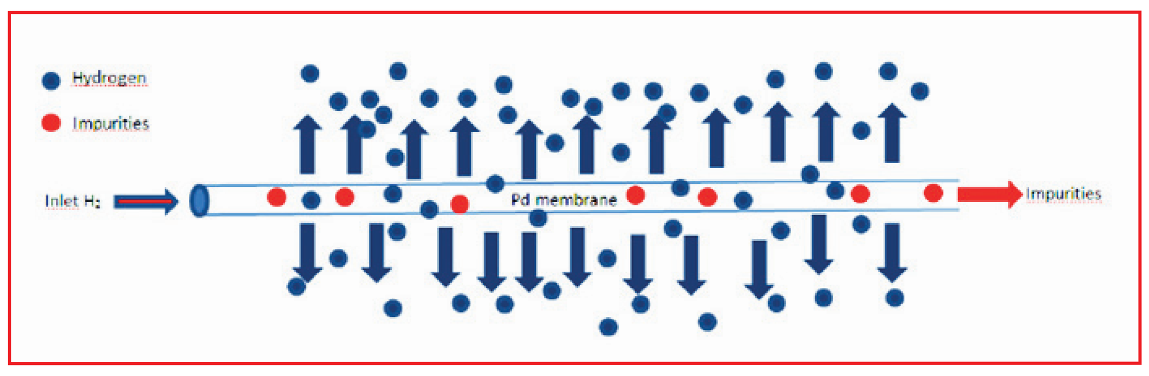
Due to the chemical and physical properties of hydrogen, several purification technologies have been developed over the years, some of them specific only to hydrogen. This article will review the most common technologies used to improve hydrogen quality down to at least 8 nines quality, explaining where each technology has advantages over the others.

Purification technology: adsorber

- Removes: O_2, H_2O, CO, CO_2, NH_3, sulfur compounds and some hydrocarbons
- Transparent to N_2, CH_4, and rare gases
- Inlet gas purity: 3N or better
- Available for flow rates up to 1000 m^3/h
- Low pressure drop
- Typical lifetime of the adsorber column: 1-3 years
- Regenerable offline (no waste of H_2 on site) or inline
- Works at room temperature

Purification technology: self standing Pd membranes

- Removes all impurities: O_2, H_2O, CO, CO_2, N_2, NH_3, organics and inert gases (Ar, He, etc.) down to < 1 ppb
- Only allows hydrogen molecules to pass through to the outlet; infinite efficiency
- Inlet gas purity: 3N or even lower grade
- Available for flow rates up to 100 m^3/h
- About 2-5% of the incoming H_2 is lost to purge out the impurities
- Unlimited lifetime, no regeneration or replacement due to consumable components
- Small footprint and inexpensive facilitization
- High pressure drop
- Work at high temperature



Purification technology: cryogenic

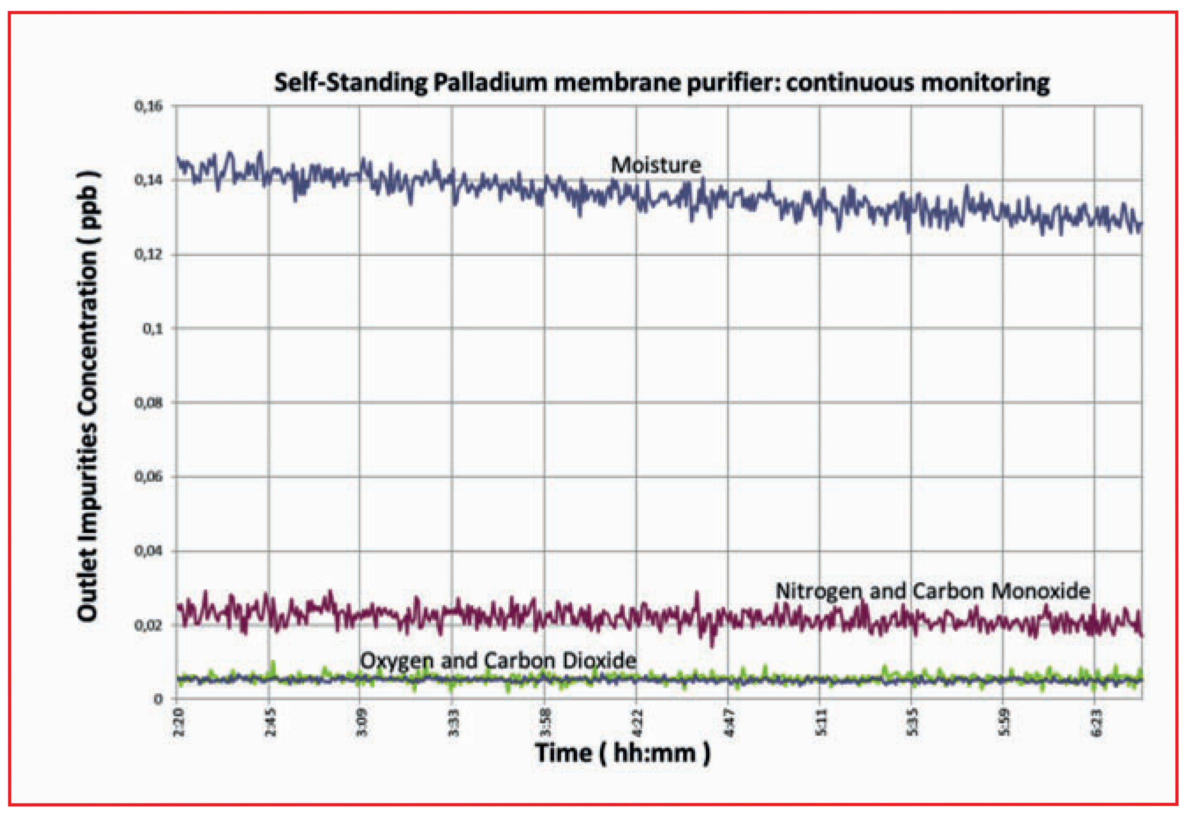
- Removes all impurities: O_2, H_2O, CO, CO_2, N_2, NH_3, organics and inert gases (Ar, Kr etc.) down to < 1 ppb with the exception of He
- Inlet gas purity: 3N or better
- Available for flow rates up to 500 m^3/h
- Low pressure drop
- Dual column for continuous operation
- Need continuous supply of liquid N_2
- Works at cryogenic temperature

Purification technology: supported Pd membranes

It is similar to the self-standing Pd purifiers but it uses thinner layers of Pd. The lower thickness has two main advantages:

- the need of a small quantity of a very expensive precious metal
- high H_2 permeance across the membrane
  - Removes all impurities: O_2, H_2O, CO, CO_2, N_2, NH_3, organics and inert gases (Ar, He, etc.) down to 10-1000 ppb depending on inlet concentration
  - Inlet gas purity: 3N or even lower grade
  - Suitable for flow rates up to 500 m^3/h
  - About 2-5% of the incoming H_2 is lost to purge out the impurities
  - Unlimited lifetime – no regeneration or replacement due to consumable components
  - Small footprint and inexpensive facilitization
  - Medium pressure drop
  - Work at high temperature

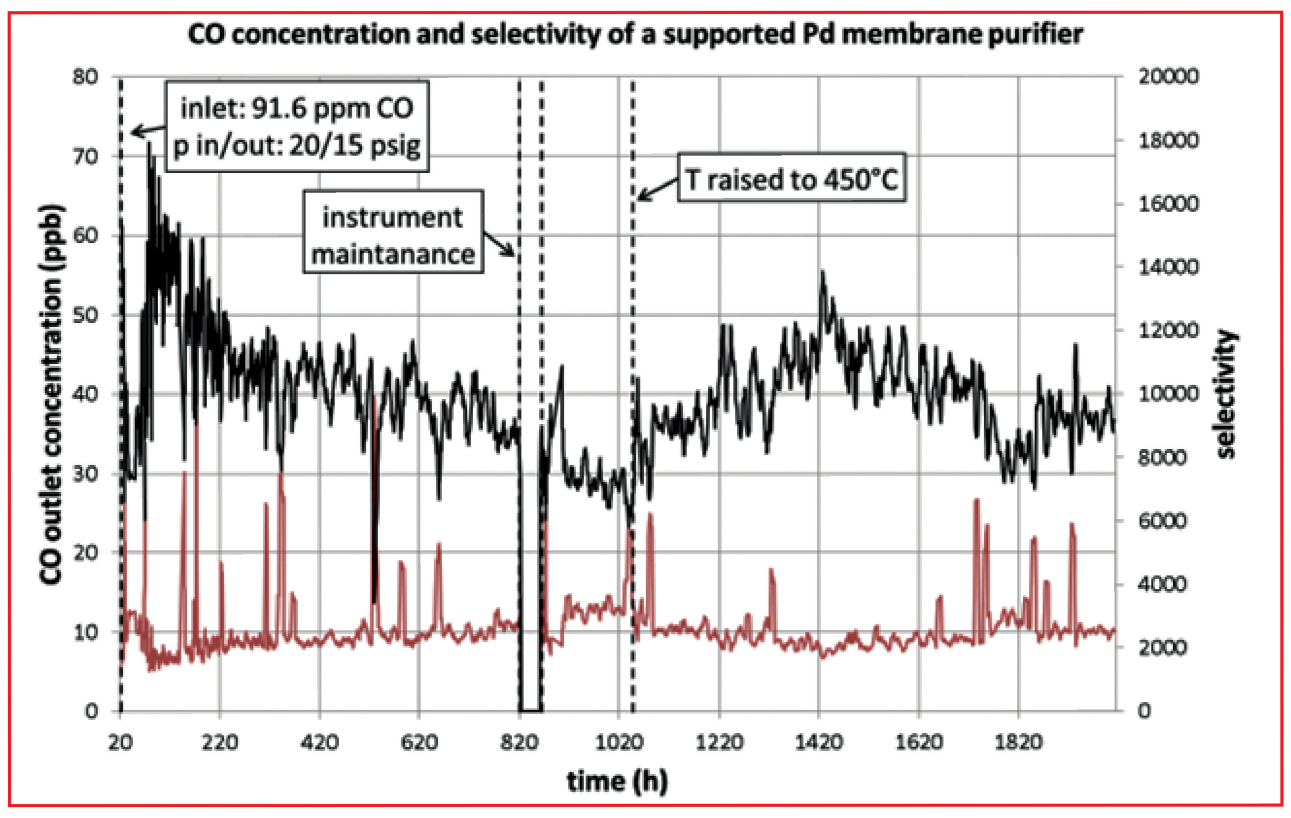
Results



Impurities at the outlet of a self-standing Pd membrane purifier monitored by Atmospheric Pressure Ionization Mass Spectrometer: all readings are close to the detection limit well below 1 ppb. Inlet pressure: 8 barg, operating temperature 400°C, flow rate 20 slpm.

CO and its selectivity at the outlet of a supported Pd membrane purifier prototype monitored by an Agilent 7890A GC equipped with a PDD. Even after 2000 hours of operation at different temperatures, 400°C and 450°C, the CO concentration at the purifier outlet and, thus the selectivity, remained stable with no indication of any degradation of the supported membrane.

Inlet pressure 20 psig, flow rate 0.4 slpm.



Conclusions

The purification technologies that are suitable to purify Hydrogen and reduce the impurities concentration down to the ppb range have been briefly discussed and compared. Each one has its own peculiarities and it is up to the customer to decide on the most appropriate purifier technology for the application based on the inlet hydrogen purity, the desired specifications and the target purity levels.

The reason to install a gas purifier is not only to get a very low concentration of the impurities of concern but to maintain it over time even when the incoming purity of hydrogen is not consistent.

Acknowledgement: The authors gratefully acknowledge Ekain Fernandez, Jon Zuniga, Alfredo Pacheco Tanaka and Josè Luis Viviente, TECNALIA Energy and Environment Division, for providing the Pd supported membranes and their support.