Johnson Matthey

TECH BULLETIN
O303

Palladium Membrane Hydrogen Purifiers: "Outside-In or Inside-Out" Flow - Which Design is Best for Compound Semiconductors?

Diffusion Process

Palladium (Pd) membrane hydrogen purifiers over the years have proven to be highly effective in MOCVD processes and are becoming the standard in the industry for providing maximum hydrogen purity compared to any other purification technology. Pd membranes continue to be the technology of choice among compound semiconductor engineers, with over 5,000 Pd purifiers installed throughout the world.

The palladium membrane is typically a metallic tube comprising a palladium and silver alloy material possessing the unique property of allowing only monatomic hydrogen to pass through its crystal lattice when it is heated above 300°C. The hydrogen gas molecule, coming into contact with the palladium membrane surface, dissociates into monatomic hydrogen and passes through the membrane. On the other surface of the palladium membrane, the monatomic hydrogen is recombined into molecular hydrogen – the ultrapure hydrogen used in the semiconductor process.

Flow Design

In a tubular membrane configuration, the impure or feed hydrogen can be directed to flow either from the outside of the tube to the inside, or from the inside of the tube to the outside. In an *Inside-Out* design, the Pd tubes are connected at both ends to a manifold. In an *Outside-In* design, only one end of the Pd tube is attached to a manifold and the other end is sealed. While it would seem that there should be no difference in purification effectiveness or product performance whether the design is *Outside-In* or *Inside-Out*, in actual practice there are significant advantages of the *Outside-In* design over the *Inside-Out* design. Johnson Matthey, the inventor of the palladium alloy that made this technology possible for purification over 30 years ago, painstakingly evaluated each of the flow designs before building their first purifier product. They found that the *Outside-In* design provided the highest purity hydrogen and the highest integrity purification product.

Outside-In Design vs. *Inside-Out* Design

- Hydrogen Purity - The objective of any hydrogen purifier design is to deliver the highest purity hydrogen possible. In a Pd membrane cell, the volume space inside of the Pd tubes is always much smaller than that of the volume space surrounding the tubes. In addition, the area surrounding the tubes will naturally have a greater amount of heated surfaces. Therefore, in an *Inside-Out* design, the ultrapure hydrogen will come in contact with many more heated surfaces, which increases the chance of contamination of the ultrapure hydrogen, especially as the purifier ages and the heated surfaces become oxidized. While these heated surfaces can be

electropolished to minimize the potential problem, there is still a greater risk of contamination with this design.

Our competitors claim that in *Outside-In* technology there may be residual lubricant contamination in the draw process. This is not true, however, in Johnson Matthey's *Outside-In* design since a proprietary cleaning process is used to eliminate contamination. APIMS results show contaminants are <1ppb.

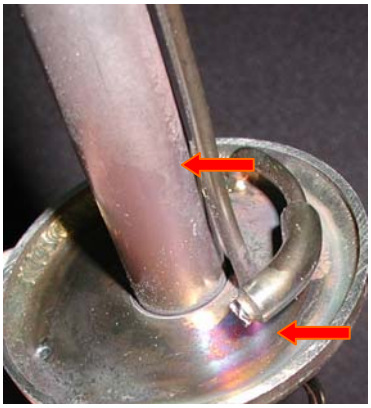

Figure 1 – Interior view of *Inside-Out* Design. The oxidation on the inside vessel body, the pure side, is very obvious

- Membrane Durability – In an *Outside-In* design only one end of the Pd tube is attached to the manifold, while the other end is free. This gives the Pd tube the freedom to expand during thermal cycling thus minimizing stress on the tube. Conversely, the Pd tube, which is attached at both ends in an *Inside-Out* design, is under constant stress throughout the thermal cycles. This stress results in decreased life of the membrane.

Figure 2 – Coiled Pd tubes from *Inside-Out* Design. The Pd membrane tubes become twisted from thermal cycling stress.

In the Johnson Matthey *Outside-In* design, Pd tube integrity is further enhanced with the insertion of pre-cleaned stainless steel springs to prevent collapsing of the tube due to the pressure differential between the outside and the inside of the tube. Suppliers of *Inside-Out* design claim that stainless steel springs are not required since there is hydrogen pressure inside the tube. This, however, is not always the case. Under certain conditions, such as in purging, there is sufficient pressure differential to cause a collapse of the tube if it does not contain the protective spring – especially in large tube diameter *Inside-Out* designs.

- Purging Efficiency – Purging of the Pd membrane purifier is sometimes required as a means of preserving membrane life during situations such as emergency shutdowns due to power failures. The objective of the purging process is to remove the hydrogen as rapidly as possible from the cell and the pure side of the membrane before the Pd membrane cools down. In an *Outside-In* design, the volume of hydrogen is significantly less on the pure side than in an *Inside-Out* design. Because the removal of hydrogen on the pure side relies on back diffusion across the membrane, there is less hydrogen to be purged; the likelihood of achieving a more rapid purge exists with the *Outside-In* design.

Conclusion

It is indisputable that the *Outside-In* design, which provides the highest purity hydrogen possible and enhances the life of the palladium membrane, is the design of choice for the optimum Pd membrane hydrogen purifier. Johnson Matthey, the first to develop and commercialize Pd membrane technology for hydrogen purification for compound semiconductor MOCVD, chose the *Outside-In* design over 30 years ago. They have continued to be the leading supplier of Pd membrane purifiers to the compound semiconductor industry.