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Outside-In vs. Inside-Out:
Which method produces cleaner hydrogen?

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The decision to purchase a hydrogen purifier is made easier if you examine the two techniques used by manufacturers of palladium membrane hydrogen purifiers.

COMMON FEATURES

When hydrogen is passed over a heated palladium-alloy membrane, hydrogen molecules are adsorbed onto the surface, where each one dissociates and diffuses through the metal lattice as atomic hydrogen. The molecules recombine on the far side of the lattice to form a hydrogen molecule, which is then desorbed from the palladium membrane.

There are two techniques (Outside-In and Inside-Out) that use drawn PdAg tubing (membrane) as a selective filter for separating ultra-pure hydrogen (99.9999999% pure) from industrial-grade hydrogen gas (99.9% pure). These names refer to which side of the tube the industrial grade hydrogen is presented to. In both cases, the diffusion of hydrogen through the membrane is driven by the pressure gradient. Atomic hydrogen flows from the high-pressure side of the membrane to the low-pressure side. When the feed hydrogen pressure is greater than the pressure of the hydrogen on the ultra-pure hydrogen (UPH) side of the membrane, UPH can be produced. This is because the diffusion rate of atomic hydrogen through PdAg at 400°C is more than nine orders of magnitude faster than any other gas.

The purity of the UPH gas is dependent on the ability to clean the UPH side of the PdAg membrane, proper welding, and the elimination of virtual leaks on the UPH side of the PdAg membrane. We will limit the description below to the methods used to clean the PdAg membrane, which is the dominant factor.

Palladium-silver tubing is made from an ingot of PdAg that is repeatedly drawn through a lubricated tube-drawing die until the required wall thickness and tube I.D. are attained. The carbon-based lubricant coats both sides of the tube, and is necessary to draw the tube. **This lubricant must be completely removed from the UPH side of the tube, or else it will out-gas and contaminate the UPH.**

CLEANING THE SMALL-DIAMETER TUBING

In purifiers using the Outside-In method, crude hydrogen flows perpendicular to the axis of the outside of small diameter tubing. This flow dissociates inward through the tubing wall. In a typical Outside-In PdAg membrane, 25' of PdAg tubing with an inside-diameter of 0.069" must be meticulously cleaned. The diameter must be small, because mechanically the tube is under compression when it is purifying hydrogen. Remember that it is critical to the purity of the UPH that the carbon-based lubricant be removed from the inside surface of the tube when the Outside-In Method is to be used in the diffusion cell. Because of the composition of the lubricant, the small diameter of the tube, and the length of the tube, this cleaning process is very difficult. The current process flushes the tube with one or more solvents. However, this process still leaves a significant amount of contamination on the inside of the tube. After cleaning, the tubing is annealed in a nitrogen belt furnace at approximately 1820°F (approximately 990°C). This process converts the remaining lubricant and solvent into carbon and low-weight carbon compounds. However, much of the lubricant residue remains in the tube due to several factors:

- Only 20% of the tube is in the hot zone of the belt furnace at any one time.
- The tube's small (0.069") diameter.
- The low diffusion rate of carbon in a nitrogen atmosphere along the length of the small diameter tube.

Typically the end of the tube is melted shut and this creates the potential for virtual leaks at the inside end of the tube. Crude hydrogen flow is normal or perpendicular to the axis of the outside of the tubing.

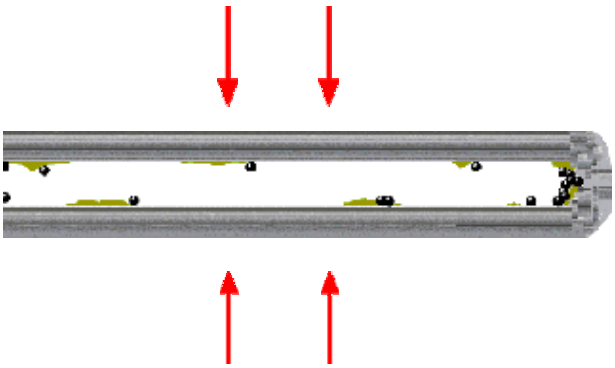

Figure 1: Small-diameter PdAg tube showing the carbonized lubricant contamination left inside the tube after solvent cleaning.

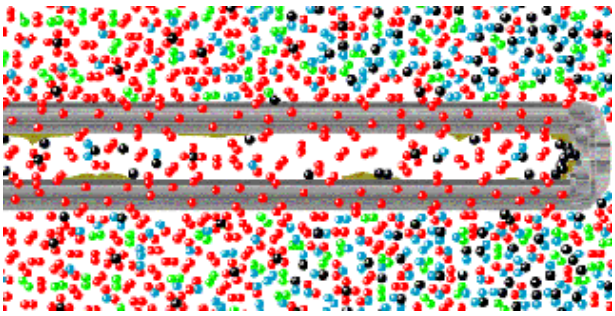

Figure 2: When using the Outside-In method, the inside of the tube is the area where the ultra-pure hydrogen is produced. Shortly after the

Outside-In cell is put into use to generate ultra-pure hydrogen, the hydrogen inside the tube can combine with the residual lubricant contamination remaining on the inside surface of the PdAg tubing to form the contaminates CH4, CO, and CO2. The Outside-In process inherently does not produce ultra-pure hydrogen.

DEAD SPOTS

Contamination builds up on the outside surface of the PdAg tubing because there is very little flow of hydrogen along the surface. In purifiers using the Outside-In method, crude hydrogen flow is normal or perpendicular to the axis of the outside of the small diameter tubing. This flow dissociates inward through the tubing wall. The impurities in the feed hydrogen have a relatively high probability of remaining on the surface of the membrane (dead spots) instead of exiting the cell along with the bleed gas.

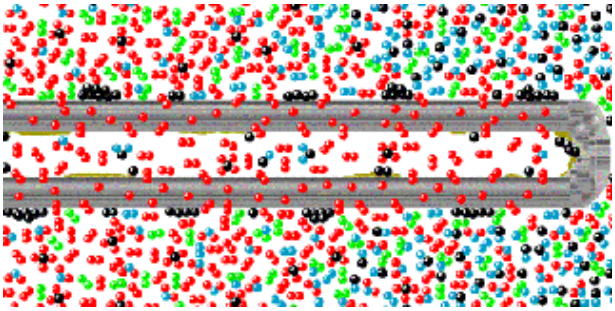

Figure 3: The results of the dead spot effect. The black dots represent impurities that have accumulated on the outside surface of the membrane over time. This contamination lowers the efficiency of the membrane and therefore similarly reduces the amount of UPH available to the user.

THE BETTER WAY: THE INSIDE-OUT METHOD

Power+Energy hydrogen purifiers use the Inside-Out technique. In this process, the feed hydrogen is in contact with the membrane on the inside of the large-diameter (0.160") PdAg tubing. The molecular hydrogen dissociates on the inside surface of the PdAg tubing and diffuses outward through the tubing wall. The hydrogen recombines on the outside wall of the tube to form molecular hydrogen with roughly 1 ppb total impurities.

This method inherently produces cleaner ultra-pure hydrogen because the outside surface of the tubing can be thoroughly cleaned, minimizing dead spots.

The tube can be made larger in diameter because it is in tension, rather than in compression. The tubing interior can be cleaned thoroughly, as the diameter is more than double that of the small-diameter tubing. In addition to the fact that the inside of the tubing used in the Inside-Out method is much easier to clean than the small diameter tubing, only the feed hydrogen is in contact with the inside of the tube, further reducing contamination and dead spots.

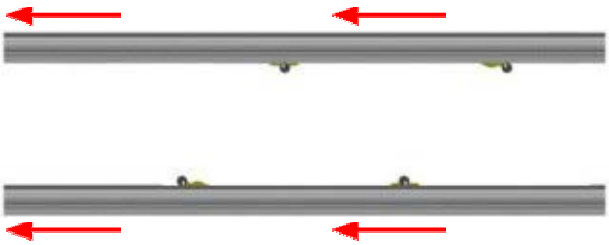

Figure 4: Power+Energy purifiers have very few dead spots on the tubing because flow is parallel to the surface of the tubing interior. Any impurities in the crude hydrogen are swept from the tubing and exit through the bleed port. The illustration also shows how the 120%-larger Power+Energy PdAg tubing allows us to clean the entire tube effectively at 1840°C in a vacuum furnace. There is no residue on the exterior surface, and the UPH is decidedly cleaner.

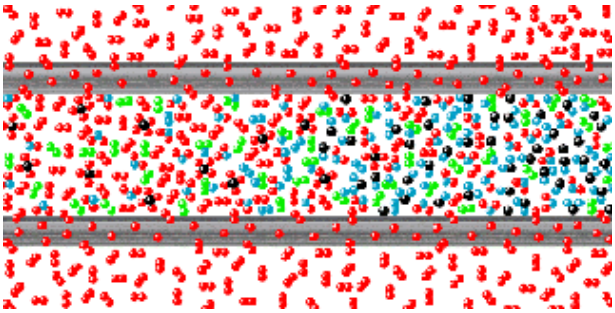

Figure 5: An illustration of the Inside-Out method purifying hydrogen.

Because flow is parallel to the inside of the tubing, impurities are swept out the bleed port and there is little dead spot buildup. The entire PdAg tube is cleaned in a vacuum furnace at 1840°C. All carbonized lubricant contamination is removed from the outside surface of the tubing, resulting in cleaner UPH.