Towards Industrial Applications: Palladium Membrane Development at Pall

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Presentation Overview

- About Pall
  - Company organization
  - Applicable markets
- Palladium Membrane Wishlist
  - Durability and stability
  - “Easy” large-scale manufacturing
    - Membrane fabrication
    - Substrate support
  - Module design
    - Sealing
    - Addressing concentration polarization
About Pall: Worldwide Presence
About Pall: Finger Lakes Center of Excellence
About Pall: Finger Lakes Center of Excellence
About Pall: Applicable Markets

**Pall Life Sciences**

- BioPharmaceuticals
  - Process
    - Biotech
    - Biologics
    - Pharmaceuticals
    - Process Monitoring
  - Pharma QC
  - Protein Characterization
  - Laboratory Research
- OEM Specialty Materials
- Core Lab
- OEM Diagnostics
- Laboratory QC

- Medical
  - OEM Healthcare
    - Device
    - Cell Therapy
    - Cardiovascular
    - Infusion Therapy
    - Hospital/Consumer Infection Control
  - Water
  - Respiratory Care
  - Cell Therapy
  - Cardiovascular

- Food & Beverage
  - Beer
  - Wine & Spirits
  - Alcohol-free Beverages
  - Food & Dairy
  - Emerging Focus Areas
    - Food Safety
    - Laboratory QC

**Pall Industrial**

- Process Technologies
  - Chemicals
  - Oil & Gas
  - Polymers
  - Refineries
  - Nuclear/Fossil
  - Alternative Energy
  - Water
  - Mining
  - Auto/In-plant
  - Primary Metals
  - Pulp & Paper
  - OEMs

- Aerospace
  - Commercial Aero
  - Military Aero
  - Marine

- Microelectronics
  - Semiconductors
  - Electronic Components
  - Data Storage
  - Displays
  - Graphic Arts
Pd Membrane: Durability and Stability

- Long lasting
  - 1000s of hours
  - Months
  - Years
- Chemical and thermal resistance
  - Application dependent
- Stable/consistent performance
- Able to withstand anticipated temperature and pressure swings/cycling
Pd-alloy for Enhanced Carbon/Sulfur Tolerance

Composition Spread Fabrication: Prof. Bruce van Dover (Cornell)

Fabrication and Testing: Prof. Doug Way (CSM) and Pall

Syngas Exposure: Pall Corporation

Corrosion Assessment: Prof. Meilin Liu (Georgia Tech)
Sputtered Wafers After Syngas Exposure
Pure-gas Testing Summary at 500°C

Hydrogen Permeability x 10^8 (mol-m²/s/Pa⁰.⁵)

- Pd-A-B
- Pd-C-D
- Pd-E-F
- Pd-H-I
- Pd-J-K
- Pd-N-O
- PdAu
Sample Test Sequence

Hydrogen Permeance (mol H₂/m²/s/Pa⁰.⁵)

T = 500°C

- No Carbon Average
- Syngas No Sulfur Average
- Syngas With Sulfur Average

1. Carbon Resistance
2. Sulfur Resistance

1. No Carbon Average
2. Syngas No Sulfur Average
3. Syngas With Sulfur Average
4. Syngas No Sulfur Average

Time (hours)
Testing Results at 500°C, $P_{\text{feed}} = 160$ psig

**Syngas (air blown)**
- $H_2 = 36$ v%
- $CO_2 = 11$ v%
- $CO = 1.3$ v%
- $H_2O = 3$ v%
- $N_2 = 49$ v%

*Note: membranes without the “no carbon” data were tested before the current testing plan was in place.*
Pall Addressing Long-term Stability

- On-off cycling (pressure pulses) and temperature cycling (idle: 275°C to in-use: 400°C)
- 12 on-off cycles per hour for 8 hours/day and idling in the evenings

<table>
<thead>
<tr>
<th>Permeate Purity (% Hydrogen)</th>
<th>Time (hours)</th>
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<tbody>
<tr>
<td>100.0000%</td>
<td>0</td>
</tr>
<tr>
<td>99.9995%</td>
<td>100</td>
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<tr>
<td>99.9990%</td>
<td>200</td>
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<td>99.9975%</td>
<td>500</td>
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<td>99.9970%</td>
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<td>99.9965%</td>
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<td>99.9960%</td>
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<tr>
<td>99.9955%</td>
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Ease of Large-Scale Manufacturing

Membrane Fabrication

- **PVD**
  - Pros
    - Uniform composition
    - Controlled thickness
    - No liquid waste for disposal
  - Cons
    - Initial capital investment
    - Directionality
    - Microstructure

- **Electroless Plating**
  - Pros
    - Inexpensive equipment
    - Ability to plate complex geometries
  - Cons
    - Liquid waste stream
    - Poor control of composition (with co-deposition)
    - Annealing (if depositing pure layers)
Ease of Large-Scale Manufacturing

**Substrate Support Fabrication**

- **Ceramic**
  - YSZ
  - ZrO₂
  - Al₂O₃

- **Metallic with diffusion barrier**
  - Ceramic
  - Oxide layer
  - Zeolite
Module Design

- Sealing membranes into housing
  - Ceramic-to-metal braze
  - Welding
  - Ferrules

- Addressing concentration polarization

- Modeling
  - Used a heat exchanger as an analog

Addressing Concentration Polarization

T = 400°C
P_{\text{feed}} = 75 \text{ psig}
P_{\text{perm}} = \text{atmospheric}
Feed Gas: 50\% \text{ H}_2 / 50\% \text{ Ar}

Pall CP Solution: 1.8x higher, 2.3x higher, 2.6x higher, 4x higher
Not Addressing CP: [Graphical representation]
Collaborating with Pall

- Willing to license technology
- Development through technology stage gates
  - New projects
  - High risk
  - More efficient allocation of resources
- Large analytical lab