Report of the DOE Workshop on Hydrogen Separations and Purification

September 8-9, 2004
Arlington, VA

U.S. Department of Energy
Office of Hydrogen, Fuel Cells & Infrastructure Technologies
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INTRODUCTION

In September 2004, the U.S. Department of Energy held a Workshop on Hydrogen Separations and Purification Technologies to explore technical approaches for lowering the cost of high-purity hydrogen. Specifically, the workshop examined the potential to significantly improve the economics of hydrogen production in the near and mid term using either of two production pathways:

- Distributed (less than 1,500 kg H\textsubscript{2}/day) reforming of natural gas and renewable liquid fuels (e.g., ethanol, methanol)
- Semi-centralized or centralized gasification/pyrolysis of biomass or coal.

At the workshop, representatives from industry and the research community provided input on various separation technology options and defined the research and development (R&D) required to help achieve DOE’s 2010-2015 cost goals for delivered hydrogen. The workshop was sponsored by the DOE Office of Hydrogen, Fuel Cells and Infrastructure Technologies (OHFCIT) in cooperation with the Office of Fossil Energy (FE), which are working together to coordinate R&D activities in this area. This report presents the results of the workshop.

Background

The President’s Hydrogen Fuel Initiative provides funding to accelerate R&D of hydrogen fuel and infrastructure technology. The goal is to enable industry to make commercialization decisions by 2015, so that hydrogen fuel cell vehicles can be available for purchase in showrooms by 2020. Success will require hydrogen production and distribution at a price that is competitive with gasoline, as well as hydrogen fuel cell vehicles that are competitive with gasoline and electric-hybrid vehicles.

Hydrogen production costs are currently too high. The estimated cost for hydrogen generated through the on-site reforming of natural gas at refueling stations using currently available technology is projected to be $3.00-$5.00/kg.\textsuperscript{2} The long term DOE goal is to develop technologies to be able to produce and deliver hydrogen utilizing a variety of domestic resources in a manner that results in near-zero net greenhouse gas and other emissions. The net fuel cost of operating the hydrogen powered vehicle ($/mile) must be equivalent to alternative vehicles for the hydrogen vehicles to be competitive in the marketplace. The DOE is researching a broad range of hydrogen production and delivery options including on-site reforming of ethanol or methanol; centralized coal gasification (with carbon sequestration) and biomass gasification with

\textsuperscript{1} $1.50/kg for distributed production from natural gas (untaxed, at the pump by 2010), based on a natural gas price of $4.50/MM Btu; less than $2.60/kg for central production from biomass gasification/pyrolysis (untaxed, at the pump by 2015); and $1.80/kg for central production from coal gasification/pyrolysis (untaxed, at the pump by 2015). The $1.50/kg for distributed natural gas target is currently under review by the FreedomCAR and Fuels Partnership.

hydrogen delivery via truck or pipeline, electrolysis, photolytic hydrogen production, and high temperature thermochemical cycle production using solar or nuclear energy. Major technology advances are needed to lower equipment and operating costs for these technologies to meet the low cost required to be competitive.

For the distributed natural gas reforming pathway, a sensitivity analysis identified three critical components of hydrogen cost. Based on current cost estimates, major progress will be required in all three of these component areas to meet the 2010 cost goal of $1.50/kg. As shown in Table 1, capital costs will need to come down by 53%, operating and maintenance (O&M) costs will have to decrease by 39%, and system efficiency will need to improve by 10%. Similar challenges face the technology development pathways for distributed liquid fuel reforming and centralized coal or biomass gasification. Advanced hydrogen separation technology is a promising option for simultaneously lowering capital equipment and operating costs and improving overall system efficiency.

| Table 1. Key Cost Factors for Distributed Steam Methane Reforming of Natural Gas (1,500kgH₂/day) |
|-----------------------------------------------|---------------------|--------------------------|
| Cost Factor                              | 2005 | Proposed 2010 Target for $1.50/kg H₂ | % Change from 2005 |
| Capital Costs ($MM)                        | $3.2 | $1.4 | -56% |
| Non-Feedstock O&M Costs ($/kg)             | $0.80 | $0.48 | -40% |
| Energy Efficiency (%)                      | 65   | 75  | +16% |

Current Hydrogen Separation Technology

The hydrogen-rich gas mixture produced via reforming or gasification contains 40-70% hydrogen and a variety of contaminants including carbon monoxide, carbon dioxide, nitrogen, methane, water, sulfur, and possibly tar and ash. The necessary purity of hydrogen in a polymer electrolyte membrane (PEM) fuel cell is not clearly elucidated by the percentage; while some chemicals do not affect membrane performance even in large amounts, others can cause detrimental effects in small quantities. Common safe chemicals (such as nitrogen) are considered diluting agents and must be removed simply to reduce compression and storage volumes. Dangerous agents (such as carbon monoxide, sulfur, and ammonia) are considered impurities and must be almost entirely removed. PEMs are poisoned by CO at more than 10 parts per million and by sulfur at the parts-per-billion level. Based on current available PEM fuel cell information, the tentative contaminant targets are: <10ppb sulfur, <1 ppm carbon monoxide, <100 ppm carbon dioxide, < 1 ppm ammonia, < 100 ppm non-methane hydrocarbons on a C-1 basis, oxygen, nitrogen and argon can not exceed 2% in total, particulate levels must meet ISO standard 14787. Future information on contaminant limits for on-board storage may add additional constraints. By contrast, solid-oxide fuel cells (SOFCs) for stationary applications can accept a less pure hydrogen feed, since they are not poisoned by carbon monoxide (CO) and can tolerate some sulfur.

Hydrogen separation technology separates hydrogen molecules from a mixed gas stream (such as a synthesis gas from natural gas, coal, or biomass) and produces a purified hydrogen gas stream.
Current technology for large-scale, industrial hydrogen production typically employs a two-step system for purifying hydrogen from the feed gas:

1) One or two water-gas-shift (WGS) reactor(s) generate additional hydrogen from the CO in the gas mixture

2) A pressure-swing adsorption (PSA) unit removes impurities such as CO, CO\(_2\), CH\(_4\), H\(_2\)O, H\(_2\)S, etc.

Depending on the hydrogen purity required, polishing filters may also be employed to remove specific trace impurities. Each of these steps adds capital and operating costs to the system and affects overall system efficiency. PSA systems increase the capital cost of the overall reforming system by up to 10% and represent a parasitic power loss. Integrating or replacing the WGS reactor(s) and/or PSA unit with advanced hydrogen integrated reaction and separation membrane modules may significantly reduce hydrogen production costs by reducing capital costs, lowering O&M costs, and improving system efficiency.

Hydrogen separation membrane technologies may offer a number of advantages over current hydrogen separation technology:

- Lower capital costs
- Smaller physical space requirements
- Fewer moving parts
- Higher hydrogen recovery rates
- Ability to produce hydrogen at a steady state (a single-unit PSA system produces hydrogen in batches)
- Ability to produce high-purity hydrogen without polishing filters
- Potential for integration with hydrogen generation technologies so that hydrogen shift and purification or possibly reforming, shift and purification are carried out in a single, simplified, compact “membrane reactor” system
- Improved thermal efficiency by eliminating the need to cool and reheat gases for gas clean-up and shift reactions

**Hydrogen Membrane Separation Technologies**

Currently, no membrane separation technology can simultaneously meet all of the performance criteria for a hydrogen fuel production system, including high hydrogen flux at low pressure drops; tolerance to contaminants; low cost; operation at system temperatures of 250-600°C; durability; and robust performance under harsh operating environments. The variety of membrane transport mechanism used across the membrane: molecular, atomic, and ionic (i.e., proton transport).

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Molecular Transport Membranes

Molecular transport (microporous) membranes are essentially micro-filters. The pores of the membrane are sized to enable the very small hydrogen molecule (at 2.89Å) to move through the membrane while larger gas molecules are left behind. Selectivity is based not only on molecular size but also on how the molecule moves through the medium (mean free path) and on the viscosity of the gas stream. Permeation rate is affected by the pore size, thickness, tortuosity, and total porosity (pore volume) of the membrane. For a membrane to be defined as microporous, its pores must be less than 20Å in diameter; however, hydrogen separation generally requires pores smaller than 10Å. The rate of hydrogen permeation (flux) is directly proportional to the pressure, and is positively affected by increasing temperature. These membranes have the potential to produce hydrogen with a purity of up to 99% in a single pass, and can be made from a number of materials, including ceramics, carbon, and metals. Purity levels of greater than 99% (e.g., 99.99%) can only be achieved by using multiple microporous membrane stages, or perhaps by developing a hybrid membrane that combines a microporous and a dense metallic layer. Commonly used materials are zeolites, SiC, metal composites, silica, and alumina. Ceramics are particularly attractive in this application, because they provide significant thermal and chemical stability in harsh operating environments.4

Atomic Transport

Atomic transport membranes (also referred to as dense metallic membranes) transport hydrogen atoms that have dissolved into a dense metal matrix. These membranes are comprised of a thin, dense metallic layer (usually palladium or palladium alloys) supported on a porous layer. Upon coming into contact with the metal film, molecular hydrogen dissociates into atoms that then pass through the film and recombine into hydrogen molecules. The metal layer is typically formed from metal composites, thin palladium, or a palladium-alloy metal that is supported on an inexpensive, mechanically strong support. The hydrogen diffuses to the metal surface, where dissociative chemisorption occurs, followed by absorption into the bulk metal, diffusion through the metal lattice, recombination into molecular hydrogen at the opposite surface, and finally diffusion away from the metal membrane. These micro-thin metallic films can be poisoned by gaseous impurities like sulfur compounds and carbon monoxide, and at high temperatures they can undergo phase changes that significantly reduce the hydrogen flux. Alloying with other metals like copper and silver reduces this phase change propensity. The flux for these membranes is proportional to the difference of the square roots of the partial pressure of the hydrogen across the membrane. Because only hydrogen is transported through the membrane, it is possible to produce a 100%-pure hydrogen permeate.

Proton Transport Membranes

Proton transport membranes have the ability to conduct protons (or cations) and electrons independently, such that hydrogen is dissociated on one side and re-constituted on the other. There are two types of proton transport membranes: those that consist of one material phase and those that consist of two.

1) Purely Mixed Conducting Membranes: This type of dense membrane is made of one material phase—ceramic (usually a perovskites/brownmillerite/pyrochlores oxide

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composition)—that is capable of conducting both protons and electrons to provide adequate fluxes at high temperatures (>800°C). This type of membrane has demonstrated low electron conductivities, but doping can mitigate this problem. However, the hydrogen flux in purely mixed, conducting ceramic membranes is still rather low for practical hydrogen separation applications. By changing the materials that make up the membrane, oxygen can be selectively transported instead of hydrogen; this approach has been highly successful in oxygen transfer systems for air separation.

2) **Cermet Membranes:** One approach to increase the hydrogen flux is to fabricate cermet membranes. This type of membrane consists of both a composite, dense-phase ceramic and a metallic phase, hence the term “cermet.” There are two general approaches to cermet membrane configuration. In the first, an electron-conducting metallic phase is combined with a dense conducting ceramic matrix phase. The purpose of the metal in this type (often nickel) is to increase the electron conduction and thereby increase the overall hydrogen flux. In the second approach, a hydrogen-permeable metallic phase is combined with the dense-phase ceramic. This metallic phase (like Pd or Pd/Ag) functions in the same way as the atomic transport membrane mentioned previously.

In both of these types of proton transport membranes, the hydrogen flux depends on both the electron and proton conductivities of the membrane. This is because every two protons transferred must react with two electrons to produce molecular hydrogen. Also, the flux is proportional to the natural log of the hydrogen partial pressure gradient across the membrane, and generally increases with operating temperature. In general, proton transport membranes have a lower hydrogen flux and a more limited operating range than the other two membrane options.
HYDROGEN MEMBRANE SEPARATION
PERFORMANCE TARGETS

Tables 2, 3, and 4 (below) show the performance targets that have been established to date for hydrogen membrane separation technologies. These targets will also be published in the updated version of the DOE Office of Hydrogen, Fuel Cells and Infrastructure Technologies’ Multi-Year Research and Development Plan (January 2005). These targets will be revised as more information is gathered on the capabilities and performance of membrane systems, and on the specific conditions and configurations in which they are expected to operate. Workshop participants offered a variety of suggestions for modifying the performance targets. Key suggestions are summarized in Table 5 and will be considered as the targets are revised.

Table 2. Technical Targets: Dense Metallic Membranes for Hydrogen Separation and Purification

<table>
<thead>
<tr>
<th>Performance Criteria(a)</th>
<th>Units</th>
<th>Calendar Year</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>2003 Status(b)</td>
</tr>
<tr>
<td>Flux(c)</td>
<td>scfh/ft(^2)</td>
<td>60</td>
</tr>
<tr>
<td>Membrane Material and All Module Costs(d)</td>
<td>$/ft(^2) of membrane</td>
<td>$2,000</td>
</tr>
<tr>
<td>Durability</td>
<td>Years(e)</td>
<td>&lt;1(f)</td>
</tr>
<tr>
<td>ΔP Operating Capability(g)</td>
<td>psi</td>
<td>100</td>
</tr>
<tr>
<td>Hydrogen Recovery</td>
<td>% of total gas</td>
<td>60</td>
</tr>
<tr>
<td>Hydrogen Purity(h)</td>
<td>% of total (dry) gas</td>
<td>&gt;99.9</td>
</tr>
</tbody>
</table>

\(a\) The membranes must be tolerant to impurities. This will be application specific. Common impurities include, sulfur and carbon monoxide.

\(b\) Based on membrane shift reactor with syngas.

\(c\) Flux at 20 psi hydrogen partial pressure differential with a minimum permeate side pressure of 15 psi, preferably >50psi, and 400°C

\(d\) The membrane support structure is approximately three times membrane material costs.

\(e\) Intervals between membrane replacement.

\(f\) Hydrogen membranes have not been demonstrated to date, only laboratory tested.

\(g\) ΔP operating capability is application dependent. There are many applications that may only require 400 psi or less. For coal gasification 1000 psi is the target.

\(h\) Based on current available PEM fuel cell information, the tentative contaminant targets are: <10 ppb sulfur; <1 ppm carbon monoxide; <100 ppm carbon dioxide; < 1 ppm ammonia; < 100 ppm non-methane hydrocarbons on a C-1 basis; oxygen, nitrogen and argon cannot exceed 2% in total; particulate levels must meet ISO standard 14787. Future information on contaminant limits for on-board storage may add additional constraints.

Notes: Revised targets take into consideration input received at the September 2004 Hydrogen Separations Workshop. These targets are undergoing detailed engineering analysis. Membrane systems should be demonstrated within a temperature range of 250-1,000°C. Also, parasitic power requirements (power used to recompress the hydrogen downstream of the membrane due to potential pressure drops across the membrane) should be minimized.

Table 3. Technical Targets: Microporous Membranes for Hydrogen Separation and Purification

<table>
<thead>
<tr>
<th>Performance Criteria&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Units</th>
<th>2003 Status</th>
<th>2005 Target</th>
<th>2010&lt;sup&gt;b&lt;/sup&gt; Target</th>
<th>2015&lt;sup&gt;b&lt;/sup&gt; Target</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flux&lt;sup&gt;c&lt;/sup&gt;</td>
<td>scfh/ft&lt;sup&gt;2&lt;/sup&gt;</td>
<td>100</td>
<td>100</td>
<td>200</td>
<td>300</td>
</tr>
<tr>
<td>Membrane Material and All Module Costs&lt;sup&gt;d&lt;/sup&gt;</td>
<td>$/ft&lt;sup&gt;2&lt;/sup&gt; of membrane</td>
<td>$450-$600</td>
<td>$400</td>
<td>$200</td>
<td>&lt;$100</td>
</tr>
<tr>
<td>Durability</td>
<td>Years&lt;sup&gt;e&lt;/sup&gt;</td>
<td>&lt;1&lt;sup&gt;f&lt;/sup&gt;</td>
<td>1</td>
<td>3</td>
<td>&gt;5</td>
</tr>
<tr>
<td>∆P Operating Capability&lt;sup&gt;g&lt;/sup&gt;</td>
<td>psi</td>
<td>100</td>
<td>200</td>
<td>400</td>
<td>400-1000</td>
</tr>
<tr>
<td>Hydrogen Recovery</td>
<td>% of total gas</td>
<td>60</td>
<td>&gt;70</td>
<td>&gt;80</td>
<td>&gt;90</td>
</tr>
<tr>
<td>Hydrogen Purity&lt;sup&gt;h&lt;/sup&gt;</td>
<td>% of total (dry) gas</td>
<td>&gt;90%</td>
<td>95%</td>
<td>99.5%</td>
<td>99.99%</td>
</tr>
</tbody>
</table>

<sup>a</sup> The membranes must be tolerant to impurities. This will be application specific. Common impurities include, sulfur and carbon monoxide.

<sup>b</sup> Assumes a two-stage membrane system or a membrane + PSA

<sup>c</sup> Flux at 20 psi hydrogen partial pressure differential with a minimum permeate side total pressure of 15 psi, preferably >50 psi and 400 °C.

<sup>d</sup> The membrane support structure is approximately three times membrane material costs

<sup>e</sup> Intervals between membrane replacement.

<sup>f</sup> Hydrogen membranes have not been demonstrated to date, only laboratory tested.

<sup>g</sup> Delta P operating capability is application dependent. There are many applications that may only require 400 psi or less. For coal gasification 1000 psi is the target.

<sup>h</sup> Based on current available PEM fuel cell information, the tentative contaminant targets are: <10 ppb sulfur; <1 ppm carbon monoxide; <100 ppm carbon dioxide; < 1 ppm ammonia; < 100 ppm non-methane hydrocarbons on a C-1 basis; oxygen, nitrogen and argon cannot exceed 2% in total; particulate levels must meet ISO standard 14787. Future information on contaminant limits for on-board storage may add additional constraints.

Note: Revised targets take into consideration input received at the September 2004 Hydrogen Separations Workshop. These targets are undergoing detailed engineering analysis. Membrane systems should be demonstrated within a temperature range of 250-1,000°C. Also, parasitic power requirements (power used to recompress the hydrogen downstream of the membrane due to potential pressure drops across the membrane) should be minimized.
### Table 4. Technical Targets: Proton Transport Membranes for Hydrogen Separation and Purification

<table>
<thead>
<tr>
<th>Performance Criteria(^a)</th>
<th>Units</th>
<th>2003 Status</th>
<th>2005 Target</th>
<th>2010 Target</th>
<th>2015 Target</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flux(^b)</td>
<td>scfh/ft(^2)</td>
<td>60</td>
<td>100</td>
<td>150</td>
<td>200(^c)</td>
</tr>
<tr>
<td>Cost(^d)</td>
<td>$/ft(^2) of membrane</td>
<td>$1,000</td>
<td>$500</td>
<td>$200</td>
<td>&lt;$100</td>
</tr>
<tr>
<td>Durability</td>
<td>Years(^e)</td>
<td>&lt;1</td>
<td>1</td>
<td>3</td>
<td>&gt;5</td>
</tr>
<tr>
<td>(\Delta P) Operating Capability(^d)</td>
<td>psi</td>
<td>200</td>
<td>300</td>
<td>400</td>
<td>400-1000</td>
</tr>
<tr>
<td>Hydrogen Recovery</td>
<td>% of total gas</td>
<td>Data Not Available</td>
<td>80</td>
<td>90</td>
<td>100</td>
</tr>
<tr>
<td>Hydrogen Purity(^h)</td>
<td>% of total (dry) gas</td>
<td>&gt;90%</td>
<td>95%</td>
<td>99.5%</td>
<td>99.99%</td>
</tr>
</tbody>
</table>

\(^a\) The membranes must be tolerant to impurities. This will be application specific. Common impurities include, sulfur and carbon monoxide.

\(^b\) Flux at 20 psi hydrogen partial pressure differential with a minimum permeate side total pressure of 15 psi, preferably >50 psi. and 400\(^o\)C.

\(^c\) Flux upper limit for Proton Transport Membranes.

\(^d\) Cost of the membrane area available for H\(_2\) extraction including material and module support structure.

\(^e\) Intervals between membrane replacement.

\(^f\) Hydrogen membranes have not been demonstrated to date, only laboratory tested.

\(^g\) Delta P operating capability is application dependent. There are many applications that may only require 400 psi or less. For coal gasification 1000 psi is the target.

\(^h\) Based on current available PEM fuel cell information, the tentative contaminant targets are: <10 ppb sulfur; <1 ppm carbon monoxide; <100 ppm carbon dioxide; < 1 ppm ammonia; < 100 ppm non-methane hydrocarbons on a C-1 basis; oxygen, nitrogen and argon cannot exceed 2% in total; particulate levels must meet ISO standard 14787. Future information on contaminant limits for on-board storage may add additional constraints.

Note: Revised targets take into consideration input received at the September 2004 Hydrogen Separations Workshop. These targets are undergoing detailed engineering analysis. Membrane systems should be demonstrated within a temperature range of 250-1,000\(^o\)C. Also, parasitic power requirements (power used to recompress the hydrogen downstream of the membrane due to potential pressure drops across the membrane) should be minimized.
Table 5. Suggestions for Modifying Performance Targets for Hydrogen Separation Membranes

<table>
<thead>
<tr>
<th>Flux</th>
<th>• Change to flow rate (kg H₂/day), which will vary by application.</th>
</tr>
</thead>
</table>
| Cost          | • Define whether the cost is for the membrane only, the membrane plus supports, or the entire membrane module (membrane, module, and housing). Define targets for both capital and operating & maintenance costs over a specified lifetime.  
• Cost targets should be $/kg H₂ |
| Operating Temperature | • Define specific, more narrow temperature ranges expected for different applications so that membranes can be tailored and tested for those conditions.  
• Specify feed and permeate temperatures by application. |
| Hydrogen Purity | • Define the hydrogen purity and contaminant levels that are required for specific end-use applications |
| Overall        | • Establish an overarching goal of $/scf (or kg) of hydrogen that all membrane separation technologies will need to meet. |
There are many technical barriers that currently limit the ability to meet the technical and economic performance targets for hydrogen membrane separation systems, as summarized below. The detailed results of the workshop’s breakout discussion sessions are presented in Appendix D.

<table>
<thead>
<tr>
<th>Key Technology Barriers</th>
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<tbody>
<tr>
<td><strong>Membrane Materials</strong></td>
</tr>
<tr>
<td>♦ Inadequate, slow screening and testing methods for identification of new materials</td>
</tr>
<tr>
<td>♦ Lack of novel materials (alloys, ceramics, composites, hybrids) that provide optimum diffusivity, flux, and contaminant resistance</td>
</tr>
<tr>
<td><strong>Membrane Stability/Durability</strong></td>
</tr>
<tr>
<td>♦ Poor resistance to thermal cycling</td>
</tr>
<tr>
<td>♦ Inverse relationship of durability and flux</td>
</tr>
<tr>
<td>♦ Inadequate or unknown resistance to contaminants in the syngas</td>
</tr>
<tr>
<td>♦ Lack of standardized, accelerated aging tests</td>
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<tr>
<td><strong>Fundamental Understanding</strong></td>
</tr>
<tr>
<td>♦ Poor understanding of mass transport diffusion through membranes</td>
</tr>
<tr>
<td>♦ Poor understanding of microstructural evolution during membrane operation and effect on permeance and selectivity</td>
</tr>
<tr>
<td><strong>Membrane Fabrication and Defect Management</strong></td>
</tr>
<tr>
<td>♦ Lack of optimal methodologies for large-scale production/fabrication of defect-free, thin film membranes</td>
</tr>
<tr>
<td>♦ Lack of in-situ membrane repair or regeneration (defect healing) methodologies</td>
</tr>
<tr>
<td>♦ Lack of companies who have experience fabricating membranes</td>
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<tr>
<td><strong>Membrane Supports</strong></td>
</tr>
<tr>
<td>♦ Lack of light-weight, low cost supports that can achieve target operating performance</td>
</tr>
<tr>
<td><strong>Membrane Module Construction and Testing</strong></td>
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<tr>
<td>♦ Inadequate module designs</td>
</tr>
<tr>
<td>♦ Lack of cost-effective large scale manufacturing methodologies that incorporate modular design, compact assemble, and life time cost minimization</td>
</tr>
<tr>
<td>♦ Lack of sealing and joining technology</td>
</tr>
<tr>
<td>♦ Lack of benchmarks for module testing (standardized testing protocols, detailed gas feedstock specifications, accelerated aging tests, etc.)</td>
</tr>
<tr>
<td><strong>System Integration/Process Intensification</strong></td>
</tr>
<tr>
<td>♦ Incomplete understanding of how a water-gas-shift reactor might be integrated with a hydrogen membrane separation system</td>
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<tr>
<td>♦ Lack of comparative studies/system analysis of alternative system configurations</td>
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<tr>
<td>♦ Poor understanding of mass transfer limitations in integrated systems</td>
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</tbody>
</table>
A number of high-priority R&D needs have been identified for hydrogen separation technologies, as summarized below. Recurring themes among the top-priority R&D needs are:

1) Standard, application-specific test methods for membrane and modules are needed to effectively focus R&D and provide a clear means for making go/no-go decisions and for comparing the performance of alternatives
2) Investigations are needed that pursue an integrated approach to the development of the membrane, support, and module design/construction
3) Predictive models are needed for assessing the technical and economic performance of alternative membrane/module designs and systems configurations

The detailed results of the workshop’s breakout discussion sessions on R&D needs are presented in Appendix D.

| Develop Advanced Membrane Materials and Rapid Testing Methods | Develop and make publicly available rapid-screening tools to discover novel materials (e.g., novel metal alloys, new families of ceramics, nano-composites, novel hybrids) |
| | – establish key performance metrics and standard testing protocols |
| | – link choice of material to membrane thickness requirements |
| | Conduct root cause analysis of membrane degradation and failure mechanisms in order to understand the effects of contaminants, thermal and pressure cycling, etc., and use this knowledge to develop more robust, durable membranes |
| | Perform systematic study of the effects of feedstock gas constituents on membrane stability, flux, and permeate purity |
| | Conduct fundamental research to improve understanding of mass transport mechanisms and kinetic/thermodynamic impacts of feedstock constituents on the membrane |
| | Develop models to predict performance and failure modes of membrane materials under varying operating conditions |
| | Establish membrane test-bed facilities accessible to industry supplied with standard feed gas samples |

| Develop Cost-Effective Membrane Fabrication Methods | Develop membrane fabrication methods for economically producing defect free, thin film membranes in a large-scale production mode |
| | – develop in-situ techniques to minimize or plug oversize pores |
| | conduct systematic study to compare alternative methods for thin-film deposition (e.g., solgel, PVD, CVD, conventional) |

| Develop High-Performance, Low-Cost Support Materials | Develop compact, low-cost membrane support materials that are practical and cost-effective: |
| | – operate at required temperature ranges |
| | – chemically resistant |
| | – easy to join and connect |
- provide smooth surface with high surface area
- provide required mechanical strength and are dimensionally stable
- provide high flux and low mass transfer resistance
- resist intermetallic diffusion
- offer a compact design for small and large flow applications
- Conduct R&D to improve understanding of interrelationship between membrane and support materials under operating conditions
  - create narrower pore size distribution
- Prevent intermetallic diffusion, thermal expansion, etc.

| Develop Optimal Module Designs and Construction and Testing Methods | Develop and demonstrate optimized module designs
- optimize fluid mechanics
- address all components of the module: membrane, support, sealing and joining
- demonstrate ability to achieve module performance targets for $/kg hydrogen, durability, etc.
- incorporate “design for manufacturing” principles
- test module designs (for go/no-go decisions) using standard testing protocols that are application specific (e.g., accelerated aging and durability, temperature cycling, membrane adherence to supports, feedstock composition and flow rates, operating temperatures and partial pressures, etc.)
- conduct root-cause analysis of degradation and failure mechanisms
- develop models to guide and test module designs and to conduct scenario analysis and sensitivity analysis of alternative membrane module configurations
- Develop methods for large-scale manufacturing of membrane modules |

| Investigate Opportunities For and Benefits of System Integration/Process Intensification | Conduct performance reaction engineering studies to develop candidate designs for system integration
- Perform detailed system analysis and develop models to predict performance of integrated systems |

| Pursue a Multi-Disciplinary, Stage-Gate R&D Strategy | Encourage multi-disciplinary R&D teams to develop and test complete membrane systems
- Include experts in membranes, supports, module construction, process design, manufacturing, operations, and catalysis
- Begin with the system that is most likely to succeed (technically and economically) and extend knowledge to more difficult systems (i.e., start with natural gas system and proceed from there)
- Conduct system analysis and modeling to better understand optimal system configurations and economic efficiencies of hydrogen membrane separation and integrated catalytic membrane reactor systems |
APPENDIX A. WORKSHOP AGENDA

DOE Workshop on HYDROGEN SEPARATION AND PURIFICATION TECHNOLOGIES
September 8–9, 2004
Hyatt Regency Crystal City, Arlington, VA

Wednesday, September 8, 2004

7:30 am  Registration and Continental Breakfast
8:30 am  Welcome and Opening Remarks, Peter Devlin, OHFCIT
8:35 am  Meeting Objectives and Purpose, Shawna McQueen, Energetics

Federal R&D Targets and Status

8:45 am  DOE, Hydrogen, Fuel Cells and Infrastructure Technologies Program,
         Arlene Anderson
9:00 am  DOE, Office of Fossil Energy, Ed Schmetz
9:15 am  DOC/NIST, Advanced Technology Program, Jason Huang
9:30 am  Break

Plenary Presentations and Discussion Sessions on Current Status of Hydrogen Separation Systems

9:45 am  Panel Session 1: Molecular Transport/Microporous Membranes
         Timothy Armstrong, Oak Ridge National Laboratory  (Panel Chair)
         Brian Bischoff, Oak Ridge National Laboratory
         Margaret Welk, Sandia National Laboratory
11:00 am Panel Session 2: Atomic Transport/Dense Metallic Membranes
         Richard Killmeyer, National Energy Technology Lab (Panel Chair)
         David Edlund, IdaTech, LLC
12:15 pm Lunch (Provided)
1:15 pm  Panel Session 3: Proton Transport Membranes
         Balu Balachandran, Argonne National Laboratory (Panel Chair)
         Tony Sammells, Eltron Research, Inc.
2:30 pm  Break
2:45–5:00 pm  **Breakout Groups**

1) Atomic Transport/Dense Metallic Separation Systems
2) Molecular Transport/Microporous Separation Systems
3) Proton Transport Separation Systems

Concurrent breakout groups will address the following questions.
- What are the key performance goals and operating issues for hydrogen separation systems?
- What hydrogen separation technology options show promise for reducing the overall cost of hydrogen production via (a) distributed reforming of natural gas and/or (b) central production from biomass or coal gasification?
- What technical barriers need to be overcome?
- What R&D needs to be done to overcome the barriers?

5:00 pm  Adjourn

**Thursday, September 9, 2004**

7:30 am  Continental Breakfast
8:30 am  **Breakout Groups Complete Prioritization of Technical Barriers and R&D Needs**
12:00 pm  Lunch (on your own)
1:00 pm  **Prepare Breakout Group Presentations for Summary Session**
1:45 pm  **Plenary Summary Session:  Reports from Breakout Groups**
2:30 pm  **Closing Comments and Next Steps**
3:00 pm  Adjourn
APPENDIX B. LIST OF WORKSHOP PARTICIPANTS

DOE Workshop on Hydrogen Separations & Purification Technologies
List of Attendees

Jim Acquaviva
Product Manager
Pall Corporation
2200 Northern Blvd.
East Hills, NY 11548
Phone: 516-801-9572
Email: jim_acquaviva@pall.com

Arlene Anderson
Technology Development Manager
U.S. DOE
1000 Independence Ave
EE-2H
Washington DC 20585
Phone: 202-586-3818
Email: arlene.anderson@ee.doe.gov

Timothy Armstrong
Program Manager
Oak Ridge National Laboratory
1 Bethel Valley Road
Oak Ridge, TN 37831
Phone: 865-574-7996
Email: armstrongt@ornl.gov

Marianne Asaro
Senior Scientist
SRI International
333 Ravenswood Avenue
Menlo Park, CA 94025
Phone: 650-859-2086
Email: marianna.asaro@sri.com

U. (Balu) Balachandran
Manager, Ceramics Section
Argonne National Laboratory
Energy Technology Division
Building# 212
9700 S. Cass Ave
Argonne, IL 60439
Phone: 630-252-4250
Email: balu@anl.gov

Kathryn Berchtold
Technical Staff Member
Los Alamos National Laboratory
Materials Science and Technology
Polymers and Coatings (MST-7)
MS E549
Los Alamos, NM 87545
Phone: 505-665-7841
Email: berchtold@lanl.gov

Brian Bischoff
Oak Ridge National Laboratory
1 Bethel Valley Road
Oak Ridge, TN 37831
Phone: 865-241-3172
Email: bischoffbl@ornl.gov

Peter Bossard
President
Power & Energy, Inc
106 Railroad Dr.
Ivyland, PA 18974
Phone: 215-942-4600 X 14
Email: peter@purehydrogen.com

Ross Brindle
Facilitator
Energetics, Inc.
7164 Gateway Drive
Columbia, MD 21046
Phone: 410-953-6239
Email: rbrindle@energetics.com

David Calabro
Advanced Scientific Associate
ExxonMobil Research & Engineering
1545 Route 22 East
Annandale, NJ 08801
Phone: 908-730-3713
Email: dcalabro@exxonmobil.com
Mark Cervi
Chemical Engineer
NAVSEA
Code 9823
5001 S. Broad Street
Philadelphia, PA 19112
Phone: 215-897-7068
Fax: 215-897-7874
Email: cervimc@nswccd.navy.mil

Anand Chellappa
Director Technology
Intelligent Energy
1001 Menaul Blvd, NE
Albuquerque, NM 87107
Phone: 505-314-8170
Fax: 505-314-8144
Email: anand.chellappa@intelligent-energy.com

Wilson Chu
Marketing and New Business Manager
Johnson Matthey Fuel Cells
1397 King Road
West Chester, PA 19380
Phone: 610-232-1987
Email: chuw@jmusa.com

Chris Cornelius
PMTS
Sandia National Laboratories
PO Box 5800
Albuquerque, NM 87185
Phone: 505-844-6192
Email: cjcorne@sandia.gov

Ashok Damle
Senior Research Chemical Engineer
RTI International
3040 Cornwallis Road
P.O. Box 12194
Research Triangle Park, NC 27709
Phone: 919-541-6146
Email: adamle@rti.org

David Edlund
Chief Technology Officer
IdaTech
63160 Britta Street
Bend, OR 97701
Phone: 541-322-1026
Email: dedlund@idatech.com

Sean Emerson
Sr. Research Engineer
United Technologies
411 Silver Lane
MS 129-30
East Hartford, CT 06108
Phone: 860-610-7524
Fax: 860-660-9093
Email: emersosc@utrc.utc.com

Greg Fleming
Research Director
Air Liquide
305 Water Street
Newport, DE 19804
Phone: 302-225-2114
Email: greg.fleming@airliquide.com

Nancy Garland
Technology Development Manager
U. S. DOE
1000 Independence Avenue. EE-2H
Washington, DC 20585
Phone: 202-586-5673
Email: nancy.garland@ee.doe.gov

Robert Glass
Group Leader
Lawrence Livermore National Laboratory
L-644
P. O. Box 808
7000 East Avenue
Livermore, CA 94550
Phone: 925-423-7140
Email: glass3@llnl.gov

Robert Goldsmith
President
CeraMem Corporation
12 Clematis Avenue
Waltham, MA 02453
Phone: 781-899-4495 ext 24
Email: goldsmith@ceramem.com

Dr. Hugh Hamilton
Johnson Matthey Technology Centre
Blounts Court
Sonning Common
Reading, United Kingdom
Phone: (0)118-9242241
Email: hamilh@matthey.com
Joseph Hartvigsen  
Senior. Engineer  
SOFC & Hydrogen Technologies  
Cerametec, Inc.  
Phone: 801-978-2163  
Email: jjh@ceramatec.com

Richard Higgins  
Vice President  
CeraMem Corporation  
12 Clematis Avenue  
Waltham, MA 02453  
Phone: 781-899-4495  
Email: higgins@ceramem.com

Jason Huang  
Program Manager  
ATP/NIST  
100 Bureau Drive  
Gaithersburg, MD 20899  
Phone: 301-975-4197  
Email: jason.huang@nist.gov

Katie Jereza  
Facilitator  
Energetics, Incorporated  
7164 Gateway Drive  
Columbia, MD 21046  
Phone: 410-290-0370  
Email: kjereza@energetics.com

Sai Katikaneni  
Manager, Fuel Processing  
FuelCell Energy  
3 Great Pasture Road  
Danbury, CT 06813  
Phone: 203-825-6067  
Email: skatikaneni@fce.com

Rich Killmeyer  
Research Group Leader  
U.S. DOE--NETL  
P.O. Box 10940  
Pittsburgh, PA 15236  
Phone: 412-386-6409  
Email: richard.killmeyer@netl.doe.gov

David King  
Senior Research Scientist  
Battelle, PNNL  
902 Battelle Blvd.  
P.O. Box 999  
MSIN K2-44  
Richland, WA 99352  
Phone: 509-375-3908  
Email: david.king@pnl.gov

Curtis Krause  
Program Manager - Fuel Processing  
ChevronTexaco  
3901 Briarpark  
Houston, TX 77042-5301  
Phone: 713-954-6343  
Email: CKrause@ChevronTexaco.com

Charles Krueger  
R&D Manager  
Hy9 Corporation  
4 Colby Street  
Room 148  
Medford, MA 02155  
Phone: 617-627-2338  
Email: ckrueger@hy9corp.com

Romesh Kumar  
Head, Fuel Cell Department  
Argonne National Laboratory  
9700 S Cass Ave, Bldg. 205  
Argonne, IL 60439-4837  
Phone: 630-252-4342  
Email: kumar@cmt.anl.gov

Wei Liu  
Senior Research Scientist  
Corning, Inc.  
Surfaces & Interfaces Core Technology  
Sullivan Park, SP-FR-05-1  
Corning, NY 14831  
Phone: 607-974-9324  
Fax: 607-974-2188  
Email: liuw@corning.com

Yi Hua Ma  
Worcester Polytechnic Institute  
100 Institute Road  
Chemical Engineering Dept  
Worcester, MA 01609  
Phone: 508-831-5398  
Email: yhma@wpi.edu
Richard Marinangeli  
Manager, New Directions  
UOP  
Box 5016  
DesPlaines, IL 60017-5016  
Phone: 847-391-3327  
Email: richard.marinangeli@uop.com

Andreas N. Matzakos  
Staff Research Engineer  
Shell International Exploration and Production Inc.  
3333 Highway 6 South  
Houston, TX 77082  
Phone: 281-544-8886  
Email: andreas.matzakos@shell.com

Ken McCarley  
Technical Coordinator  
ConocoPhillips  
760 Adams Building  
411 S. Keeler Ave.  
Bartlesville, OK 74004  
Phone: 918-661-9776  
Email: ken.c.mccarley@conocophillips.com

Shawna McQueen  
Senior Facilitator  
Energetics  
7164 Gateway Drive  
Columbia, MD 21046  
Phone: 410-953-6235  
Email: smcqueen@energetics.com

J. William Medlin  
Assistant Professor  
Chemical and Biological Engineering  
University of Colorado  
ECCH111, 424 UCB  
Boulder, CO 80309  
Phone: 303-492-2418  
Email: will.medlin@colorado.edu

William J. Mettes  
Senior Scientist  
Power + Energy  
660 Gillaspie Drive  
Boulder, CO 80305  
Phone: 720-304-2653  
Fax: 720-304-2653  
Email: mettesbtt@cs.com

Melanie Miller  
Chemical Facilitator  
Energetics, Inc.  
7164 Gateway Drive  
Columbia, MD 21046  
Phone: 410-953-6240  
Email: mmiller@energetics.com

Bryan Morreale  
Research Engineer  
NETL  
PO Box 618  
Pittsburgh, PA 15129  
Phone: 412-386-5929  
Email: bryan.morreale@netl.doe.gov

Kevin O’Brien  
New Business Development  
Lawrence Livermore National Laboratory  
7000 East Ave.  
L-223  
Livermore, CA 94551  
Phone: 925-422-7782  
Email: obrien14@llnl.gov

Stephen Paglieri  
Technical Staff Member  
Los Alamos National Laboratory  
P.O. Box 1663, MS-C348  
Los Alamos, NM 87545  
Phone: 505-667-0652  
Email: steve.paglieri@lanl.gov

Mark Paster  
Technology Development Manager  
U.S. DOE  
1000 Independence Avenue  
Washington, DC 20585  
Phone: 202-596-2821  
Email: mark.paster@ee.doe.gov

Dilo Paul  
Senior Scientist  
Science Applications International Corporation  
626 Cochran Mill Road  
MS 922-306A  
Pittsburgh, PA 15236  
Phone: 412-386-6110  
Email: paul@sa.netl.doe.gov
Amy Taylor  
Chemical Engineer  
U.S. Department of Energy  
1000 Independence Ave., SW  
NE-20 Germanton Building  
Washington, DC 20585  
Phone: 301-903-7722  
Fax: 301-903-5057  
Email: amy.taylor@hq.doe.gov

Lawrence Van Bibber  
Assistant Vice President  
SAIC  
626 Cochrans Mill Road  
M/S 922-174B  
Pittsburgh, PA, 15236  
Phone: 412-386-4853  
Email: Larry.VanBibber@sa.netl.doe.gov

Margaret Welk  
Post Doc  
Sandia National Laboratories  
P.O. Box 5800  
Org. 6245 MS-0734  
Albuquerque, NM 87185-0734  
Phone: 505-284-9630  
Email: mewelk@sandia.gov

David Winkel  
Facilitator  
Energetics, Incorporated  
7164 Gateway Drive  
Columbia, MD 21046  
Phone: 410-290-0370  
Email: dwinkel@energetics.com

Rick Zalesky  
President, Hydrogen  
ChevronTexaco Technology Ventures  
3901 Briarpark Dr  
Houston, TX 77042  
Phone: 713-954-6102  
Email: rickzalesky@chevrontexaco.com
## APPENDIX C. BREAKOUT GROUP MEMBERS

### Proton Transport Breakout Group

<table>
<thead>
<tr>
<th>Name</th>
<th>Organization</th>
</tr>
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<tbody>
<tr>
<td>Balachandran, U. (Balu)</td>
<td>Argonne National Laboratory</td>
</tr>
<tr>
<td>Cornelius, Chris</td>
<td>Sandia National Laboratories</td>
</tr>
<tr>
<td>Fleming, Greg</td>
<td>Air Liquide</td>
</tr>
<tr>
<td>Glass, Robert</td>
<td>Lawrence Livermore National Laboratory</td>
</tr>
<tr>
<td>Hartvigsen, Joseph</td>
<td>Cerametec, inc.</td>
</tr>
<tr>
<td>Higgins, Richard</td>
<td>CeraMem Corporation</td>
</tr>
<tr>
<td>King, David</td>
<td>Battelle, PNNL</td>
</tr>
<tr>
<td>Paster, Mark</td>
<td>DOE</td>
</tr>
<tr>
<td>Paul, Dilo</td>
<td>Science Applications International Corporation</td>
</tr>
<tr>
<td>Robbins, John</td>
<td>ExxonMobil</td>
</tr>
<tr>
<td>Sammells, Anthony</td>
<td>Eltron Research Inc.</td>
</tr>
<tr>
<td>Schwartz, Michael</td>
<td>ITN Energy Systems</td>
</tr>
<tr>
<td>Schinski, Bill</td>
<td>ChevronTexaco</td>
</tr>
<tr>
<td>Smith, Ronald</td>
<td>SRI Consulting</td>
</tr>
<tr>
<td>Van Bibber, Lawrence</td>
<td>SAIC</td>
</tr>
<tr>
<td>Zalesky, Rick</td>
<td>ChevronTexaco Technology Ventures</td>
</tr>
</tbody>
</table>

### Atomic Transport/Dense Metallic Breakout Group

<table>
<thead>
<tr>
<th>Name</th>
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<tbody>
<tr>
<td>Cervi, Mark</td>
<td>NAVSEA</td>
</tr>
<tr>
<td>Calabro, David</td>
<td>ExxonMobil Research &amp; Engineering</td>
</tr>
<tr>
<td>Chellappa, Anand</td>
<td>Intelligent Energy (Meso Fuel)</td>
</tr>
<tr>
<td>Chu, Wilson</td>
<td>Johnson Matthey Fuel Cells</td>
</tr>
<tr>
<td>Damle, Ashok</td>
<td>RTI International</td>
</tr>
<tr>
<td>Edlund, David</td>
<td>IdaTech</td>
</tr>
<tr>
<td>Emerson, Sean</td>
<td>UT Research Center</td>
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<td>Goldsmith, Robert</td>
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<td>Hamilton, Hugh</td>
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<tr>
<td>Killmeyer, Rich</td>
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<tr>
<td>Krueger, Charles</td>
<td>Hy9 Corporation</td>
</tr>
<tr>
<td>Kumar, Romesh</td>
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</tr>
<tr>
<td>Ma, Yi Hua</td>
<td>Worcester Polytechnic Institute</td>
</tr>
<tr>
<td>Matzakos, Andreas N.</td>
<td>Shell International Exploration &amp; Production Inc.</td>
</tr>
<tr>
<td>Medlin, Will</td>
<td>University of Colorado</td>
</tr>
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<td>Mettes, Jacques</td>
<td>Power &amp; Energy</td>
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<td>Morreale, Bryan</td>
<td>NETL</td>
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<td>O'Brien, Kevin</td>
<td>Lawrence Livermore National Laboratory</td>
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<tr>
<td>Rubenstein</td>
<td>Shell Hydrogen</td>
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<td>Schwartz, Joe</td>
<td>Praxair</td>
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</table>
## Molecular Transport/Microporous Breakout Group

<table>
<thead>
<tr>
<th>Name</th>
<th>Institution</th>
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<tbody>
<tr>
<td>Acquaviva, Jim</td>
<td>Pall Corporation</td>
</tr>
<tr>
<td>Armstrong, Timothy</td>
<td>Oak Ridge National Laboratory</td>
</tr>
<tr>
<td>Asaro, Marianne</td>
<td>SRI International</td>
</tr>
<tr>
<td>Berchtold, Kathryn</td>
<td>Los Alamos National Laboratory</td>
</tr>
<tr>
<td>Bischoff, Brian</td>
<td>Oak Ridge National Laboratory</td>
</tr>
<tr>
<td>Cornelius, Chris</td>
<td>Sandia National Laboratory</td>
</tr>
<tr>
<td>Huang, Jason</td>
<td>ATP/NIST</td>
</tr>
<tr>
<td>Katikaneni, Sai</td>
<td>FuelCell Energy</td>
</tr>
<tr>
<td>Krause, Curtis</td>
<td>ChevronTexaco</td>
</tr>
<tr>
<td>Marinangeli, Richard</td>
<td>UOP</td>
</tr>
<tr>
<td>McCarley, Ken</td>
<td>ConocoPhillips</td>
</tr>
<tr>
<td>Perrin, Jerome</td>
<td>Air Liquide</td>
</tr>
<tr>
<td>Ratcliff, Matt</td>
<td>NREL</td>
</tr>
<tr>
<td>Schlasner, Steven</td>
<td>ConocoPhillips</td>
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<tr>
<td>Shen, John</td>
<td>U.S. Department of Energy</td>
</tr>
<tr>
<td>Staiger, Chad</td>
<td>Sandia National Laboratories</td>
</tr>
<tr>
<td>Taylor, Amy</td>
<td>U.S. Department of Energy</td>
</tr>
<tr>
<td>Welk, Margaret</td>
<td>Sandia National Laboratories</td>
</tr>
</tbody>
</table>

**DOE Workshop on Hydrogen Separation and Purification Technologies**
Appendix D. Detailed Breakout Group Results

TABLE D-1 TECHNICAL BARRIERS TO PROTON TRANSPORT MEMBRANE SYSTEMS  
(Priority votes shown in parentheses)

<table>
<thead>
<tr>
<th>CENTRAL/SEMI-CENTRAL SYSTEMS</th>
<th>DISTRIBUTED SYSTEMS</th>
<th>BOTH CENTRAL AND DISTRIBUTED SYSTEMS</th>
<th>CROSS-CUTTING RESEARCH ACTIVITIES</th>
</tr>
</thead>
</table>
| • Difficulty fabricating membranes at large scale (scalability); (12)  
  - Complexity of the fabrication process  
  - Lack of skilled craftsmen  
  - Minimal industrial base for fabricating membranes at large scale  
  - Problems inherent to making thin sheets of brittle material on a large scale (supports, etc)  
  - Large amount of preconditioning required for output streams from coal gasification (9)  
  - Syngas cleanup requires thermal oscillations (heating up and cooling down), which lead to greater recompression energy requirements to bring stream up to temperature for ITM operation (7)  
  - Complexity of control systems (3)  
  - Larger national security threat (target)  
  - Perceived risk to general public from "transport" of hydrogen | • Significant challenges inherent to integrated membrane reactor concepts (incomplete understanding of WGS integrated with ITM systems; uncertain theoretical limits for ITM) (10)  
• Uncertain capital efficiency of integrated membrane reactor design (4)  
• Increased cycling requirements for fueling stations (will require more robust membranes to deal with turning on, turning off systems, replacement of modules by low-tech staff, etc) (4)  
• Mass transfer and kinetics barriers associated with catalyst and temperature regimes in WGS (2)  
• Difficulty in sequestering CO2 from small units at fueling stations (possible climate change implications) | • Adverse public perceptions of hydrogen as a fuel  
• Limited funds at individual companies to pursue high risk R&D  
• Limited collaboration among R&D performers and industry | • Inadequate and slow screening and testing methods for identifying new materials (note: the goal for materials screening is to optimize capital efficiency and achieve process intensification) (12)  
  - Inability to optimize composition of membranes  
  - Difficulty testing for protonic conductivity in parallel  
  • Flux limitations (i.e., low flux) especially with H2 ITMs (7)  
  • Cost of sealing membrane module (5)  
  • ITMs tend to be less robust than other membranes (3)  
  • Susceptibility of ITMs to electrochemical poisoning (3)  
  • Instability of ITMs in presence of water (3)  
  • Cost of pressure loss (pressure must be increased downstream to ship product to users)  
  • Membrane catalyst design issues (oxidation and reforming) (2)  
  • Carbonate formation, especially with H2 ITM (1) |

DOE Workshop on Hydrogen Separation and Purification Technologies
# TABLE D-2 R&D NEEDS FOR PROTON TRANSPORT MEMBRANE SYSTEMS

*(Priority votes shown in parentheses)*

<table>
<thead>
<tr>
<th>CENTRAL/SEMI-CENTRAL SYSTEMS</th>
<th>DISTRIBUTED SYSTEMS</th>
<th>CROSS-CUTTING RESEARCH ACTIVITIES</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Process models for large-scale systems (e.g., hydrogen production). These should cover all components of the production process and be linked to the separation module models. (7)</td>
<td>• Modular concepts that are uniquely suited for distributed systems (7)</td>
<td>• Rapid screening methods to enable exploration of entirely new membrane material concepts, e.g., totally new family of ceramics (reduce time to test on all parameters; develop high throughput testing methods; establish metrics for testing; screen for conductivity first, then for a multitude of performance parameters; stability, reaction with CO2 and steam, chemical expansion/contraction, creep rate, sintering. (14)</td>
</tr>
<tr>
<td>• Hot gas cleanup (evaluate and coordinate with ongoing work in this area; study impacts of trace metals, as well as potential for sulfur electrochemical poisoning on surface. (6)</td>
<td>• Reliability engineering for modular systems (5)</td>
<td>• System robustness (accelerated aging tests for membranes, to test mechanical durability; failure mode prediction; reliability analysis). If membranes are to last for upwards of 5 years, mechanical durability will be an issue. (8)</td>
</tr>
<tr>
<td>• Process models for pre-conditioning of streams from coal gasification; coal is the dirtiest yet cheapest feed, and will require substantial preconditioning. (5)</td>
<td>• Explore and address challenges unique to integrated reactor design, especially mass transfer issues and kinetics (WGS with membrane, reformer plus membrane) (4)</td>
<td>• Process modeling (process and devices); include development of better properties data; prediction of thermal cycling performance of membrane materials; thermal gradients under heating and cooling; integration of WGS with membrane process to determine efficacy; use these models to looks for better materials with thermal and chemical stability, so that designs can then be adjusted. (5)</td>
</tr>
<tr>
<td>• Large membrane test bed facilities, accessible to industry users; this might be built at a national laboratory, such as the wind tunnel test facilities built by NASA (5)</td>
<td>• Safety, health and security issues unique to hydrogen systems</td>
<td>• Economic models for processes and manufacturing of membranes (3)</td>
</tr>
<tr>
<td>• Telemetry for large scale system performance; this includes the complex sensors and controls needed to monitor performance of large-scale membrane systems.</td>
<td>• Telemetry for large scale system performance; this includes the complex sensors and controls needed to monitor performance of large-scale membrane systems.</td>
<td>• Improved seals for the membrane module (1)</td>
</tr>
</tbody>
</table>

---

**Notes:**

- WGS = Water Gas Shift (a chemical reaction that produces hydrogen from carbon monoxide and water)
### TABLE D-3. TECHNICAL BARRIERS TO ATOMIC/DENSE METALLIC SYSTEMS
(Priority votes shown in parentheses)

<table>
<thead>
<tr>
<th>Membrane Material</th>
<th>Supports</th>
<th>Module Construction</th>
<th>Module Testing</th>
<th>Process Integration</th>
<th>System Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Lack of improved fabrication methods (10)</td>
<td>• Lack of lightweight, low cost support that achieves (9)</td>
<td>• Lack of economical, large-scale manufacturing (8)</td>
<td>• Lack of benchmark or standard for durability (membrane vs. module) (7)</td>
<td>• Lack of understanding of the interactions between the catalyst and membrane (3)</td>
<td>• Lack of comparative studies on alternative system configurations – system analysis (5)</td>
</tr>
<tr>
<td>- Novel alloys</td>
<td>- Operating T range</td>
<td>- Feedstock gas specifications</td>
<td>- Lack of good contacting between WGS/reform catalyst with membrane/support for good utilization of catalyst and membrane (2)</td>
<td>- Existing high-T heaters are not compact, don’t fit well in process intensification (1)</td>
<td>- Lack of work on reducing the cost of hydrogen compression (3)</td>
</tr>
<tr>
<td>- Multi-comp. alloys</td>
<td>- Resists migration of thick membrane</td>
<td>- Small vs. large modules</td>
<td>- Don’t know how systems will operate in field – start up/shut-down (1)</td>
<td>- Temperature mismatch between different processes (1)</td>
<td>- Produces a low pressure hydrogen product (compressor required) (1)</td>
</tr>
<tr>
<td>- Depositing uniform, thin, effect-free membrane</td>
<td>- Provides required mechanical strength and matches TEC of membrane</td>
<td>- Life time cost minimization</td>
<td>- Lack of long duration testing using realistic syn. gas and operation</td>
<td>- Optimize utilization of each</td>
<td>- Lack of innovative ideas for use of membranes in a system (1)</td>
</tr>
<tr>
<td>- Large-scale production of defect-free membranes</td>
<td>- Smooth</td>
<td>- Lack of sealing and joint technology (2)</td>
<td>- Need to understanding if recycling is cost-viable</td>
<td>- How to recycle membranes</td>
<td>- Holistic approach</td>
</tr>
<tr>
<td>- Defect healing</td>
<td>- High surface area</td>
<td>- Module design for recyclability (2)</td>
<td>- Designs which minimize boundary layer effects and loss of driving force (1)</td>
<td>- Temperature mismatch between different processes (1)</td>
<td>- Lack of system designs that incorporate failure detection methods and failure scenarios plans (1)</td>
</tr>
<tr>
<td>Membrane durability (7)</td>
<td>Impact of support on thin membrane flux (6)</td>
<td>- Narrower support pore size distribution at low cost</td>
<td>- Lack of feed samples that contain gas contaminants</td>
<td>- Don’t know where module fits in the system</td>
<td>- Don’t know where module fits in the system</td>
</tr>
<tr>
<td>- Lack of resistance to thermal cycling</td>
<td>- Low support mass transfer resistance</td>
<td>- Need to understanding if recycling is cost-viable</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Effects of contaminants (poisoning)</td>
<td>- Understanding of interrelations of support, pore size and film information</td>
<td>- How to recycle membranes</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>• Lack of novel alloys for contaminant resistance – discovery (6)</td>
<td>• Intermetallic diffusion (5)</td>
<td>- Designs which minimize boundary layer effects and loss of driving force (1)</td>
<td>- Design which minimize boundary layer effects and loss of driving force (1)</td>
<td>- Don’t know where module fits in the system</td>
<td></td>
</tr>
<tr>
<td>- Optimum alloy for diffusivity and flux</td>
<td>- Allow high flux</td>
<td></td>
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</tr>
<tr>
<td>• Lack of understanding of microstructural evolution in operation and its effect on permanence and selectivity (5)</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>- Temperature</td>
<td></td>
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<tr>
<td>- Gas comp.</td>
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<tr>
<td>- Trace</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Contaminants</td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>- Grain growth</td>
<td></td>
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<td></td>
<td></td>
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<tr>
<td>- Cluster growth as function of</td>
<td></td>
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</tr>
<tr>
<td>• Lack of identification of all contaminants and concentrations in feed to develop resistant membranes (2)</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>- Heavy metals?</td>
<td></td>
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<td></td>
<td></td>
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</tr>
<tr>
<td>- Mercury, etc.?</td>
<td></td>
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</tr>
<tr>
<td>Material and support mismatches due to hydrogen expansion</td>
<td></td>
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</tr>
<tr>
<td>• Non-Pd membranes have a hard time dissociating hydrogen</td>
<td></td>
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</tr>
<tr>
<td>• Lack of understanding of the mechanisms that lead to degradation and failure of membranes/modules</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Aging</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>- Poisoning</td>
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</tr>
</tbody>
</table>

DOE Workshop on Hydrogen Separation and Purification Technologies
### TABLE D-4. R&D NEEDS FOR ATOMIC/DENSE METALLIC SYSTEMS

<table>
<thead>
<tr>
<th><strong>MEMBRANE MATERIAL</strong></th>
<th><strong>SUPPORTS</strong></th>
<th><strong>MODULE CONSTRUCTION</strong></th>
<th><strong>MODULE TESTING</strong></th>
<th><strong>PROCESS INTENSIFICATION</strong></th>
<th><strong>SYSTEM ANALYSIS</strong></th>
<th><strong>MISCELLANEOUS</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Develop improved membrane fabrication methods</td>
<td>Develop compact, low cost support materials and structures that are practical and cost effective</td>
<td>Develop and demonstrate optimized module design</td>
<td>Develop standard testing protocols that are application-specific</td>
<td>Performance reaction engineering studies to integrate reformer and membrane or WGS</td>
<td>Conduct comparative studies on alternative system configurations</td>
<td>Encourage Multi-disciplinary R&amp;D approaches to R&amp;D teams to develop and test membrane systems. Experts</td>
</tr>
<tr>
<td>– For defect – free membranes</td>
<td>– Operate at temperature</td>
<td>– Combine membrane, support, and module R&amp;D</td>
<td>– Validate</td>
<td>– Identify cost reductions by implementing membranes</td>
<td></td>
<td>Membrane</td>
</tr>
<tr>
<td>– For large-scale production</td>
<td>– Take deposits</td>
<td>– That meets targets for $/Kg H₂, durability, etc.</td>
<td>– Design the system optimally</td>
<td></td>
<td></td>
<td>Support</td>
</tr>
<tr>
<td>– Integrating novel alloys</td>
<td>– Smooth</td>
<td>– Incorporate “DFM” (design for manufacturing)</td>
<td>– Conduct experiments and modeling for system design</td>
<td></td>
<td></td>
<td>Module const.</td>
</tr>
<tr>
<td>– Meeting cost targets</td>
<td>– Have high flux</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Process design</td>
</tr>
<tr>
<td>– Depositing uniform, thin films</td>
<td>– Are chemically resistant</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Manufacturing</td>
</tr>
<tr>
<td>– Both foils and deposition methods</td>
<td>– Are easy; to join and connect</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Operational</td>
</tr>
<tr>
<td>Conduct root cause analysis of degradation and failure mechanisms</td>
<td>– Have high surface area</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Catalysis (for process intensi</td>
</tr>
<tr>
<td>– Understand effects of contaminants</td>
<td>– Are dimensionally stable</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>rxn engineering)</td>
</tr>
<tr>
<td>– Good membrane characterization</td>
<td>– Compact design for small and large flow applications</td>
<td></td>
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</tr>
<tr>
<td>Develop more durable membranes</td>
<td>Develop improved understanding of inter-relationships between membrane and support</td>
<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>– Cycling Pₕ, thermal</td>
<td>– Create narrower pore size distribution</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>– Customize membranes to feed specification</td>
<td>– Reduce mass transfer resistance</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>– Effect of contaminants (and definition)</td>
<td>– Prevent intermetallic diffusion, thermal expansion, etc.</td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Develop alloy compositions that optimize membrane performance and durability</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>– Link alloy choice to membrane thickness</td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>– Provide contaminant resistance for specific feeds</td>
<td></td>
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</tr>
<tr>
<td>– Utilize techniques for combinatorial screening</td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>
## TABLE D-5. TECHNICAL BARRIERS TO MICROPOROUS MEMBRANE SYSTEMS
*(Priority votes shown in parentheses)*

<table>
<thead>
<tr>
<th>STABILITY/ DURABILITY</th>
<th>SUPPORT ISSUES</th>
<th>FUNDAMENTALS</th>
<th>FABRICATION AND DEFECT MANAGEMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inadequate or unknown hydrothermal and chemical stability (11)</td>
<td>Lack of understanding of how the quality of support effects the quality of membranes (6)</td>
<td>Poor understanding of mass transport diffusion through membranes (11)</td>
<td>Inability to manage defects in membrane (10)</td>
</tr>
<tr>
<td>Lack of durability of thin membrane film (10)</td>
<td>Inability to form quality interfaces between two materials for supports or hybrid systems (1)</td>
<td>- Gas molecules through small pores</td>
<td>- Inability to form defect-free membranes</td>
</tr>
<tr>
<td>- Balance of durability with flux</td>
<td>- Inability to predict the thermal/chemical compatibility of membrane with substrate (10)</td>
<td>- Lack of transport models (e.g. multi-scale, comprehensive, cycling, shock)</td>
<td>Difficulty with scale-up to manufacturing, including fabrication issues (10)</td>
</tr>
<tr>
<td>Inability to predict the thermal/chemical compatibility of membrane with substrate (10)</td>
<td>Material and support stability/ durability (5)</td>
<td>Separation factors are variable and not well understood (1)</td>
<td>Lack of in-situ membrane repair or regeneration (6)</td>
</tr>
<tr>
<td>- Especially with broader gas compositions with impurities</td>
<td>- Selective pore size</td>
<td>Limitations of current separation mechanisms (may reach theoretical limits) (1)</td>
<td>- The ability to isolate and repair a problem section of membrane is unknown</td>
</tr>
<tr>
<td>Material and support stability/ durability (5)</td>
<td>Unproven ability to deal with cycling (3)</td>
<td>Lack of strategy for new or selective membrane development (1)</td>
<td>Membrane synthesis process is not robust or reasonable</td>
</tr>
<tr>
<td>Inability to deal with cycling (3)</td>
<td>Lower durability due to thermal cycling, on/off operation (for distributed production) (2)</td>
<td>“We don’t know what we don’t know”</td>
<td></td>
</tr>
<tr>
<td>- Improvement of cold start times</td>
<td>Tolerance to contaminants with respect to stability</td>
<td></td>
<td></td>
</tr>
<tr>
<td>- The ability to isolate and repair a problem section of membrane is unknown</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*DOE Workshop on Hydrogen Separation and Purification Technologies*
<table>
<thead>
<tr>
<th>CHARACTERIZATION AND PERFORMANCE TESTING</th>
<th>SYSTEM/MODULE ISSUES</th>
<th>PERFORMANCE</th>
</tr>
</thead>
</table>
| • Lack of standardized accelerated aging tests (13) | • Inadequate module designs (10)  
- Designs must be completed through length of membrane  
- Lack of optimization of fluid mechanics within module  
- Designs do not address all components in module/system (e.g. seals)  
• Fouling (8)  
• Particulates and physical erosion (3)  
- Inability to protect membrane from fine dust and carbonaceous contaminants  
• Lack of process integration (2)  
- It is yet to be determined if the membrane should dictate the process or vice versa  
- What to do with retentate  
• Lack of understanding of parasitic losses (2)  
• Potentially low H₂ recovery (2)  
• It is unknown if WGS and membrane integration is feasible (2)  
• Inability to identify most/least expensive part of system (2)  
• Lack of understanding of best/optimal geometry for each application (1)  
• Cost target too high  
- replacement costs may be needed | • Maximum purity achievable may not meet purity goals (8)  
• Optimization of selectivity and flux tradeoff yet to be determined (8)  
• Flux and temperature regimes are too low for use in centralized production  
• Inability to design a material at optimum conditions for WGS (e.g. what temperature to design for)  
• Inability of a single membrane to work for entire range of targets and applications |
| • Inability to test membrane performance under reaction conditions (2) | | |
| • Lack of system-wide characterization tools (publicly available) (1) | | |
| • Lack of uniform/constant feed gas composition that could be used for testing (1) | | |
| • Lack of quick and reliable failure analysis tools | | |
### TABLE D-6. R&D NEEDS FOR MICROPOROUS MEMBRANE SYSTEM
(Priority votes shown in parentheses)

<table>
<thead>
<tr>
<th>MATERIALS AND STRUCTURES DESIGN</th>
<th>PROPERTY AND PERFORMANCE CHARACTERIZATION</th>
<th>MODELING</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Develop new materials. (7)</td>
<td>• Perform systematic study of fouling contaminants and gas composite effects on stability, flux, purity. (10)</td>
<td>• Conduct fundamental research to address top barriers such as diffusion transport mechanisms. (9)</td>
</tr>
<tr>
<td>- Supports and membrane</td>
<td>• Investigate thermochemical properties along the length of the membrane. (8)</td>
<td>• Perform kinetic/thermodynamics studies on stability in presence of water. (5)</td>
</tr>
<tr>
<td>- Nano-composites</td>
<td>• Test other membranes on real streams (3)</td>
<td>• Develop better physical characterization of membranes to help guide modeling. (2)</td>
</tr>
<tr>
<td>- Hybrid materials</td>
<td>- With CO2 removal</td>
<td>- Incorporate the experimental component to modeling for development and evaluation.</td>
</tr>
<tr>
<td>• Paradigm shift in materials membrane platform. (5)</td>
<td>- Use various feed streams</td>
<td>- Investigate in-situ methods.</td>
</tr>
<tr>
<td>- Develop new platforms, e.g. mixed matrix</td>
<td>• Develop methods for thermomechanical property measurement. (3)</td>
<td>- Investigate measurement of gas molecule flow through pores.</td>
</tr>
<tr>
<td>- Research selectivity vs. flux</td>
<td>- Establish standardized tests</td>
<td>• Develop first principle model understanding to correlate membrane substrate structures with performance and stability. (2)</td>
</tr>
<tr>
<td>- Explore new separation mechanisms</td>
<td>- Variables include temperature and pressure</td>
<td>• Structure activity analysis relationships for ceramics for H2/CO2/CO separations. (1)</td>
</tr>
<tr>
<td>• Develop sequential programs, system/module design, and materials design. (5)</td>
<td>• Development of hybrid systems with different coefficients of expansion, stresses. (1)</td>
<td>- Follow-up with combi.</td>
</tr>
<tr>
<td>• Develop methods to stabilize pore structures in nano-composites via thin films. (3)</td>
<td>- Characterize interfacial stability of hybrid systems.</td>
<td>- Qualitative or quantitative of ceramic porosity</td>
</tr>
<tr>
<td>• Develop combi-chem program for zeolites, which has already has a substantially large database, and extend to other materials such as ceramics. (2)</td>
<td>• Explore combi techniques (virtual, high-throughput materials screening techniques) for screening of membrane materials and synthesis methods. (1)</td>
<td>- Investigate use of additives to control pore size.</td>
</tr>
<tr>
<td>• Differentiate among membrane systems to make push to 2010 targets.</td>
<td>• Develop methods for basic thermal expansion and stress measurements.(1)</td>
<td>• Research into fundamental knowledge to generate a model or software and to establish training. (1)</td>
</tr>
<tr>
<td>• Define correlation of membrane life with performance or selectivity.</td>
<td>• Acquire more membrane performance and characterization data.</td>
<td>- Include an extensive variable study.</td>
</tr>
<tr>
<td></td>
<td>- Include an extensive variable study.</td>
<td>• Conduct NDE to validate produced membranes.</td>
</tr>
<tr>
<td><strong>FABRICATION PROCESS</strong></td>
<td><strong>MODULE/SYSTEM DESIGN AND INTEGRATION</strong></td>
<td><strong>DEVELOP STANDARD TESTS</strong></td>
</tr>
<tr>
<td>------------------------</td>
<td>------------------------------------------</td>
<td>---------------------------</td>
</tr>
</tbody>
</table>
| • Develop hybrid-based systems to achieve 99.99% (microporous alone won’t get there). (8)  
• Develop generic separation techniques to minimize or plug oversize pores. (7)  
- Possibly in-situ  
• Explore comparison of physical/chemical techniques (solgel, PVD, CVD, conventional) for thin-film deposition. (5)  
• Develop manufacturing technology (separate from material science). (5)  
- Low cost  
- Thin film  
• Research large-scale production of membranes. (2) | • Continue research on integrated membrane reactors (membrane as part of system/module). (7)  
- Integrated with WGS (upstream) or fuel reactor (downstream)  
• Further develop and then optimize system/module design. (7)  
- Start with end in mind  
- Modeling  
- Overall process H₂ production and separation  
- Existing membranes may already work  
• Develop hybrid systems to add benefits beyond reforming process (e.g. CO₂ sequestration, H₂ storage). (5)  
• Order of magnitude thermodynamics, kinetics, and process economics of membrane reactor and WGS (2)  
• Increase communication between researcher and user. (1)  
- Narrow operating conditions and guidelines  
• Rethink targets and include language for multi-step processes. | • Develop standardized, universal testing for membranes. (11)  
- Aging and durability tests  
- Tests for both centralized and distributed production  
- Temperature cycling tests  
- Tests on membranes losing adherence to support  
• Develop, publicize, and standardize membrane and screening tools (e.g. accelerated aging, fouling, rapid screening tools, etc.). (5)  
• Develop standard erosion testing method. |
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