Composite Pd and Alloy Porous Stainless Steel Membranes for Hydrogen Production and Process Intensification

Yi Hua MA
Center for Inorganic Membrane Studies (CIMS)
Worcester Polytechnic Institute
Department of Chemical Engineering
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Project ID: PD007

Overview

Timeline

- Start : 5/7/2007
- Finish : 5/6/2011
- 100% Complete

Budget

- Total Project Cost: $ 2,228,851
  - DOE Share: $ 1,756,444
  - Recipient Share: $ 472,407
- Funding Received:
  - FY08: $ 442,785
  - FY09: $ 420,638
  - FY10: $ 392,803
  - FY11: $ 500,218

- DOE Award #: DE-FC26-07NT43058
- DOE Project Manager: Dr. Daniel Driscoll

Subcontractor

- **Adsorption Research Inc. (ARI)**

Barriers

- **Barriers Addressed:**
  - Re-producibility of high H_2 flux targets
  - Setting of Pd thickness and support characteristics to meet 2015 DOE targets
  - Determination optimum temperature WGSR
  - 2D model for CMR simulations, safety and economical analysis
  - Absorbent selection & PSA system build-up, testing completed with syngas+H_2O, H_2S and COS.

- **Technical Targets****

	H_2 Flux§ [scfh/ft^2]	Temp. [°C]	ΔP max. [psi]	H_2 Purity	Sulfur Tolerance
2010	200	300-600	400	99.5%	20 ppm
2015	300	250-500	800-1000	99.99%	>100 ppm
§ @ 100 psi ΔP H_2 partial pressure					
CO Tolerance: Yes; WGS Activity: Yes					

** DOE-NETL Test Protocol v7 – 05/10/2008

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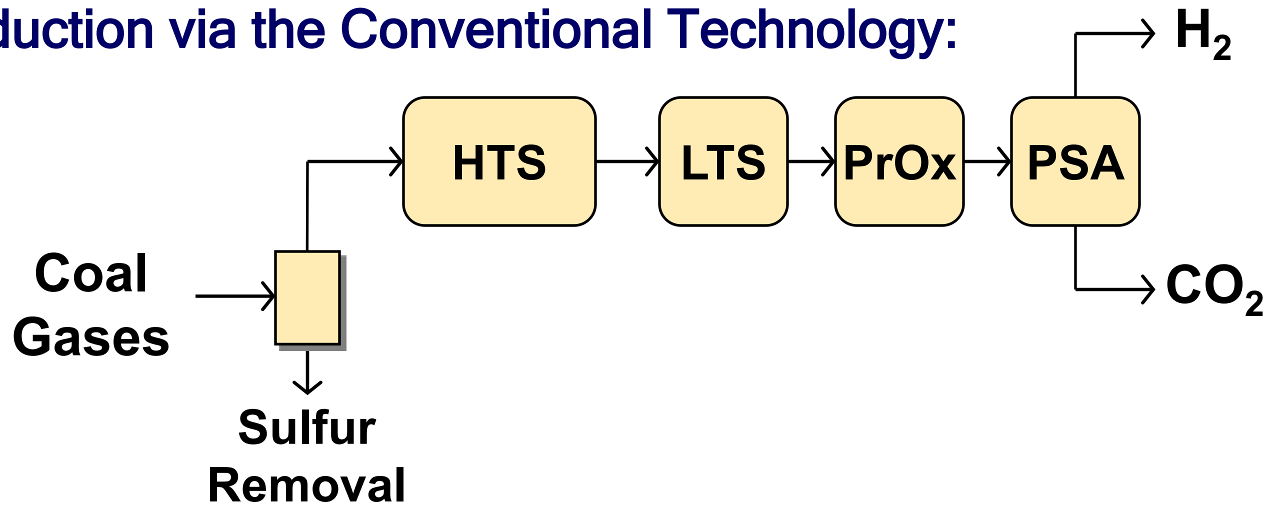
Project Objectives & Relevance

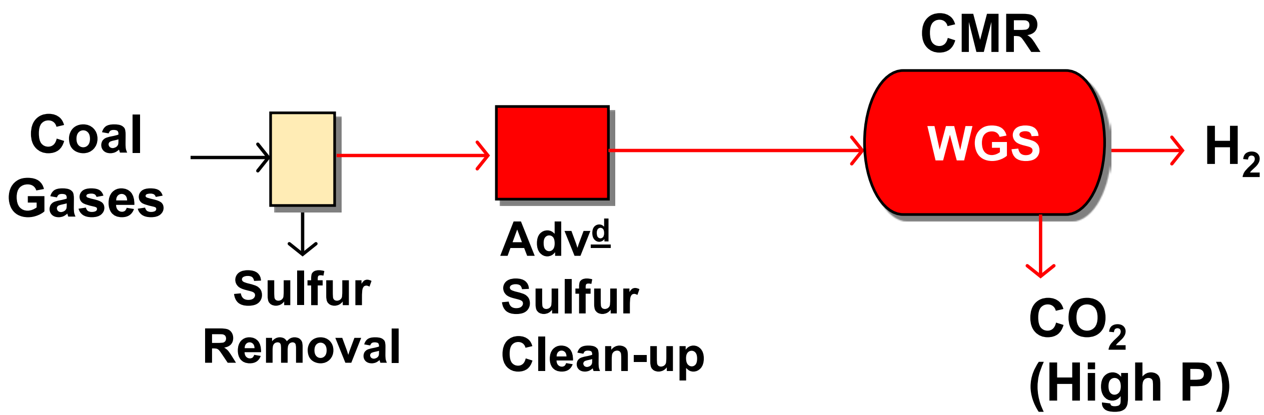
- Synthesis of composite Pd and Pd/alloy porous Inconel membranes for WGS shift reactors with long-term thermal, chemical and mechanical stability with special emphasis on the stability of hydrogen flux and selectivity
- Demonstration of the effectiveness and long-term stability of the WGS membrane shift reactor for the production of fuel-cell quality hydrogen
- Research and development of advanced gas clean-up technologies for sulfur removal to reduce the sulfur compounds to <2 ppm
- Development of a systematic framework towards process intensification to achieve higher efficiencies and enhanced performance at a lower cost
- Rigorous analysis and characterization of the behavior of the resulting overall process system, as well as the design of reliable control and supervision/monitoring systems
- Assessment of the economic viability of the proposed intensification strategy through a comprehensive calculation of the cost of energy output and its determinants (capital cost, operation cost, fuel cost, etc.), followed by comparative studies against other existing pertinent energy technologies

Approach: Coal Gasification & CMR

H_2 Production via the Conventional Technology:



Novel Catalytic Membrane Reactor (CMR):



Project Schedule & Milestones

Tasks	Year 1				Year 2				Year 3				Year 4			
	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4
	Months															
	3	6	9	12	15	18	21	24	27	30	33	36	39	42	45	48
Gas Clean-up & Fast PSA using Structured Adsorbent			M1 ✓		G1 ✓											
						M2 ✓										
										M3 ✓						
Membrane Synthesis		M4 ✓														
				M5 ✓				G2 ✓								
Membrane Characterization & Reactor Performance						M6 ✓										
										M7 ✓						
Membrane Reactor Modeling			M8 ✓													
Process Intensification					M9 ✓											
Process Control System; Design & Implementation								M10 ✓								
Process monitoring System; Design & Implementation										M11 ✓						
Address process safety; risk assessment/management																
Program Management & Reporting																

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Membrane Properties & Permeation Test Set-up

- **Membrane:**

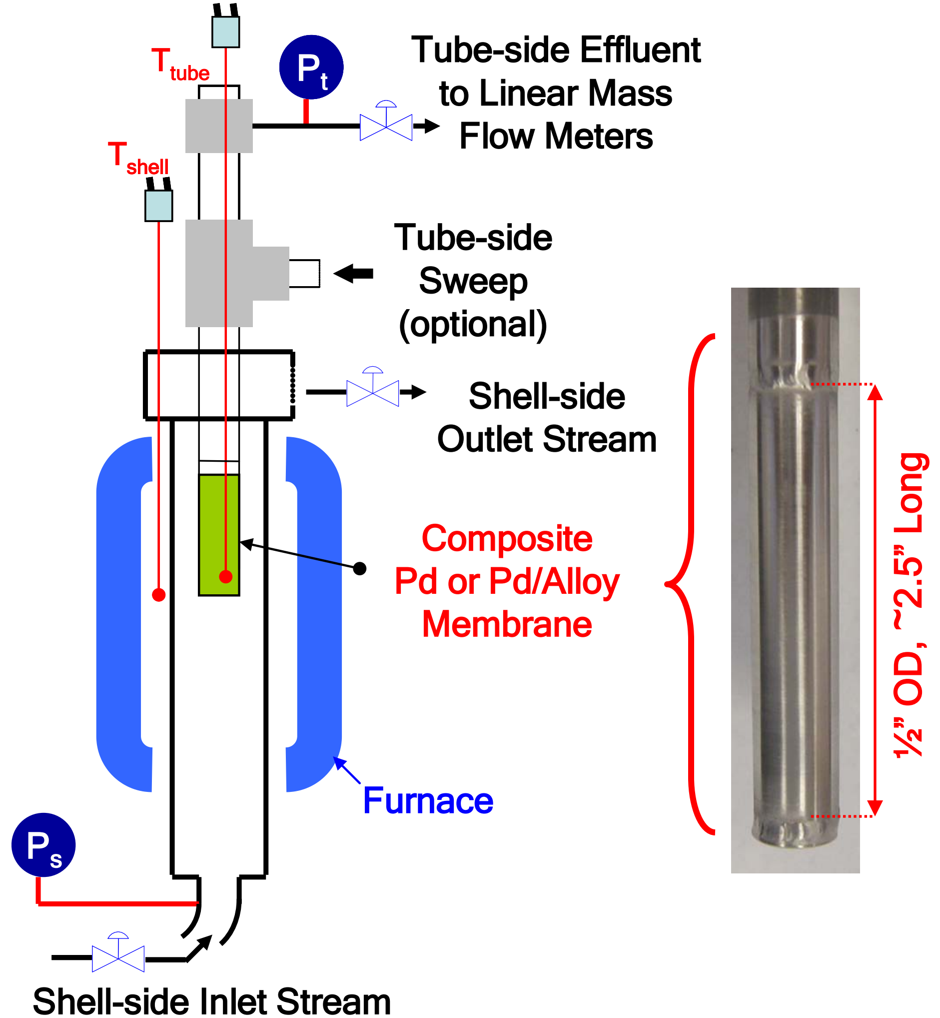
  Pd supported on porous Inconel (media grade 0.1 µm)

- **Method of Preparation:**
  Electroless Plating

- **Geometry:**

  Tubular (Plated on the outside of a tube)

- **Membrane Area ≈** 25 cm^2



Similar setup equipped w/ pre-heater, mixer, cold trap & GC was utilized for the mixed gas & WGS reaction tests

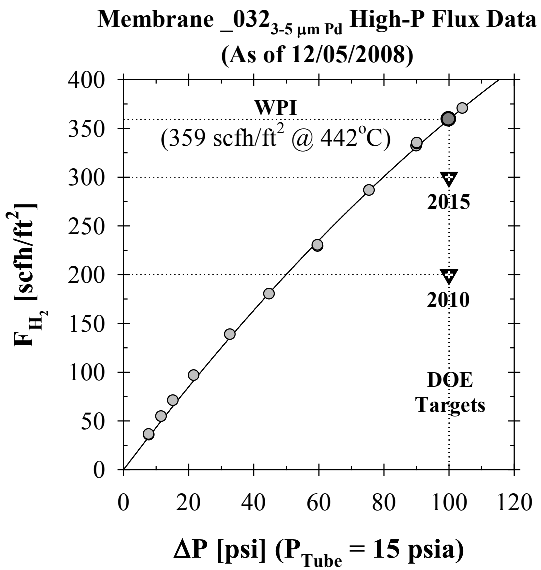
Previous achievements

- Excellent long-term H_2/He selectivity stability was achieved over a total testing period of ~3550 hours (>147 days) (membrane #029)

- Selectivity stability reproduced with membranes #030 / 031 / 033
- The thicknesses of #029/030/031/033 were all higher than 7 µm
- Selectivities in the order of 5000 -10 000 after long test at 450°C

- #032 showed high flux 359 scfh/ft² but low selectivity of 450 (3-5 µm thick)

Membrane	Results
$\#029_{7.6\ \mu m}$	• H_2/He ≅ ∞ at 450°C • H_2 purity: ≥ 99.999% • 3550 hours (>147 days)
$\#030_{7.9\ \mu m}$	• H_2/He ≅ 10000 at 450°C • H_2 purity: ≥ 99.99% • 1400 hours (>58 days)
$\#031_{7.0\ \mu m}$	• H_2/He ≅ 4500 at 450°C • H_2 purity: ≥ 99.99% • 2200 hours (>90 days)
$\#033_{8.7\ \mu m}$	• H_2/He ≅ 9725 at 450°C • H_2 purity: ≥ 99.99% • 800 hours (>32 days)



Progress Towards DOE H_2 Flux targets

Mem.	Support	LPd (μm)	Temp. (°C)	Time (hs)	Permeance / Pd Foil (scfh/ft²-psi$^{0.5}$)	Selectivity (H_2/He)	F at ΔP=100 psi Exp. / Pd Foil (scfh/ft²)
034	0.1 μm/Inc.	8.7	300	200	12 / 24 ▯	-	82 / 164 ▯▯
035	0.1 μm/Inc.	20	300	150	6 / 10	-	82 / 68
036	0.1 μm/Inc.	11.4	450	100	33.4 / 35.2	-	229 / 241
036R	idem	12 (3-5 wt%Au)	450		16 / ~ 33	200	110 / 226
038	0.1 μm/Inc.	6	450	170	38 / 67	330	<u>280</u> / 459
039	0.1 μm/Inc.	7.3	450	165	33 / 55		
040	0.1 μm/ PSS 316L	12.3	450	120	20 / 33		
041	0.1 μm PSS 316L	8.6	450	200	20 / 47		
042	0.1 μm PSS 316L	13.4	450	200	20 / 30		

The presence of mass transfer from support is significant

▯ xx / xx = permeance of the composite membrane / permeance of a free-standing Pd foil having the same thickness
▯▯ XX / XXX flux at ΔP=100 psi of composite membrane / flux at ΔP=100 psi of Pd foil with the same thickness
RED = does not meet DOE's 2010 target
GREEN = exceed DOE's 2010 target but does not meet DOE's 2015 target
BLUE = meet DOE's 2015 flux target
Underlined = actually measured (not underlined = extrapolated from measured permeance)

Several membranes have the potential to meet 2015 DOE's target

Support specifications

	H_2 Flux§ [scfh/ft²]	Temp. [°C]	ΔP max. [psi]	H_2 Purity	Sulfur Tolerance
2010	200	300-600	400	99.5%	20 ppm
2015	300	250-500	800-1000	99.9%	>100 ppm
§ @ 100 psi ΔP H_2 partial pressure (or 6.85 psi $^{0.5}$)					
CO Tolerance: Yes; WGS Activity: Yes					

•Hydrogen permeance target

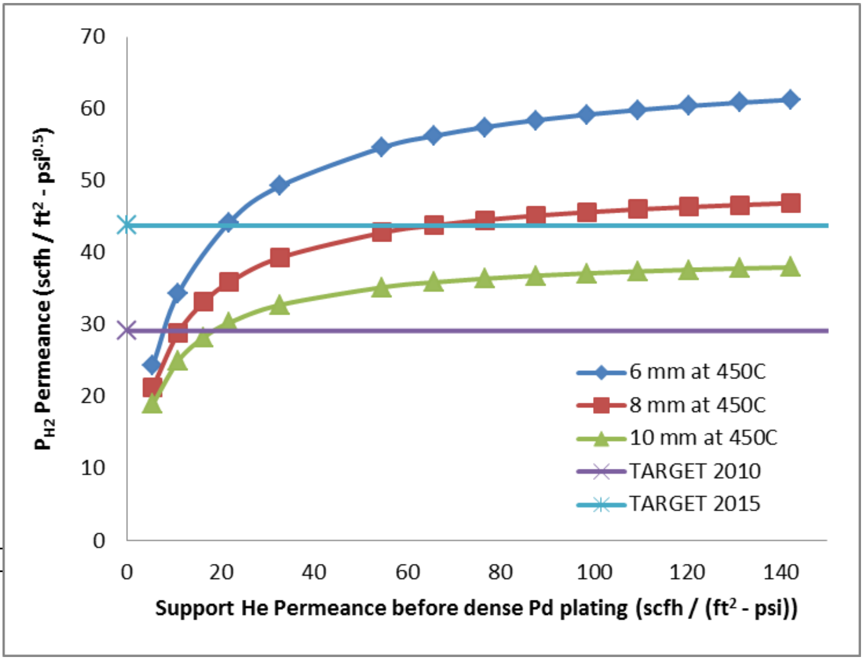
2010 → 29.2 scfh/ft²-psi$^{0.5}$

2015 → 43.8 scfh/ft²-psi$^{0.5}$

•Pd foil targets

→ 11.8 μm at 450 C□

→ 9.2 μm at 450 C□



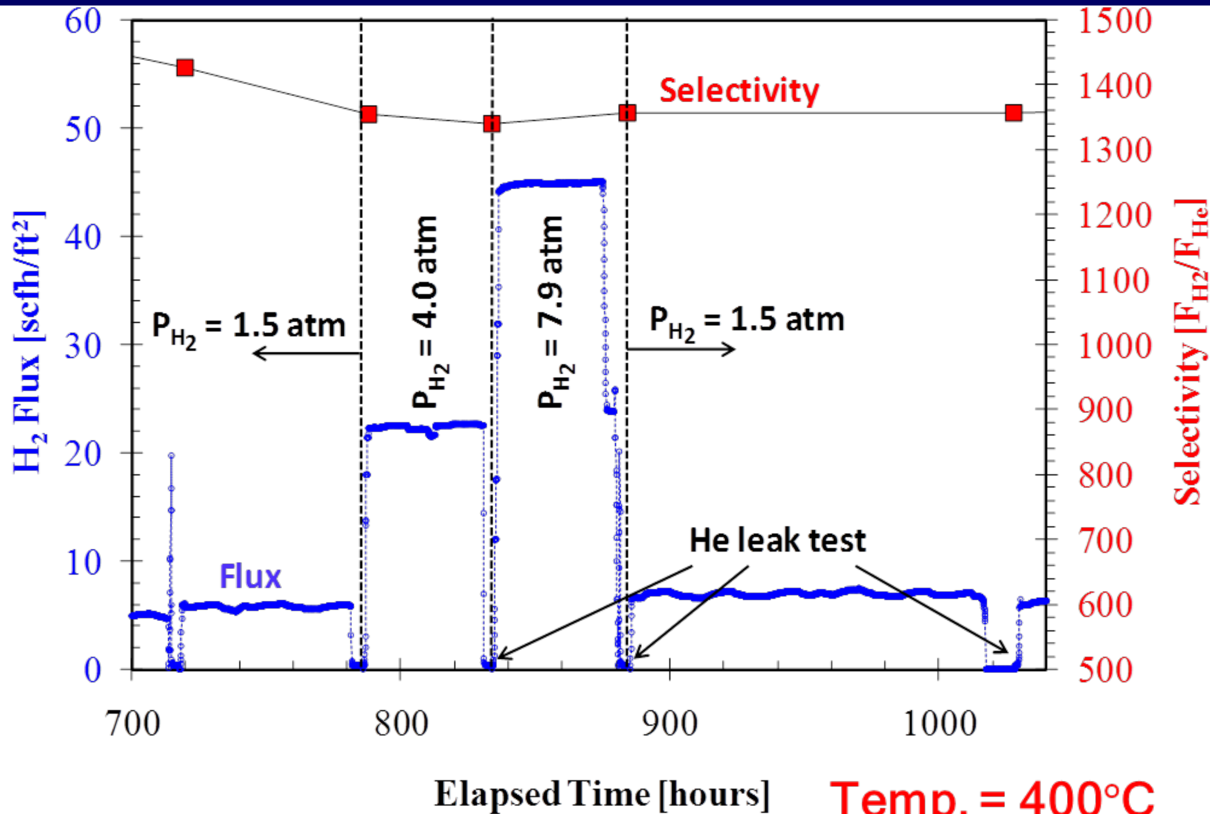
□ 450°C was found to be an optimum temperature for WGS-MR in our group (see later)

•Thickness targeted 7-8 μm (e.g., 029-031, very selective)

•Support He permeance before Pd deposition higher than 65 scfh/(ft² - psi)

•Bare support He permeance should be higher than 130 scfh/(ft² - psi)

High Pressure H_2 Testing/Selectivity stability



Temp. = 400°C

- •Membrane: MA-79
- •PSS support,
- •17 µm Pd/Ag layer,
- •10 µm dense Pd layer

- High pressure H_2 fed to the membrane for 100 hours

- Stable H_2 fluxes of 74.5 and 147 scfh/ft^2 at 60 and 120 psi respectively

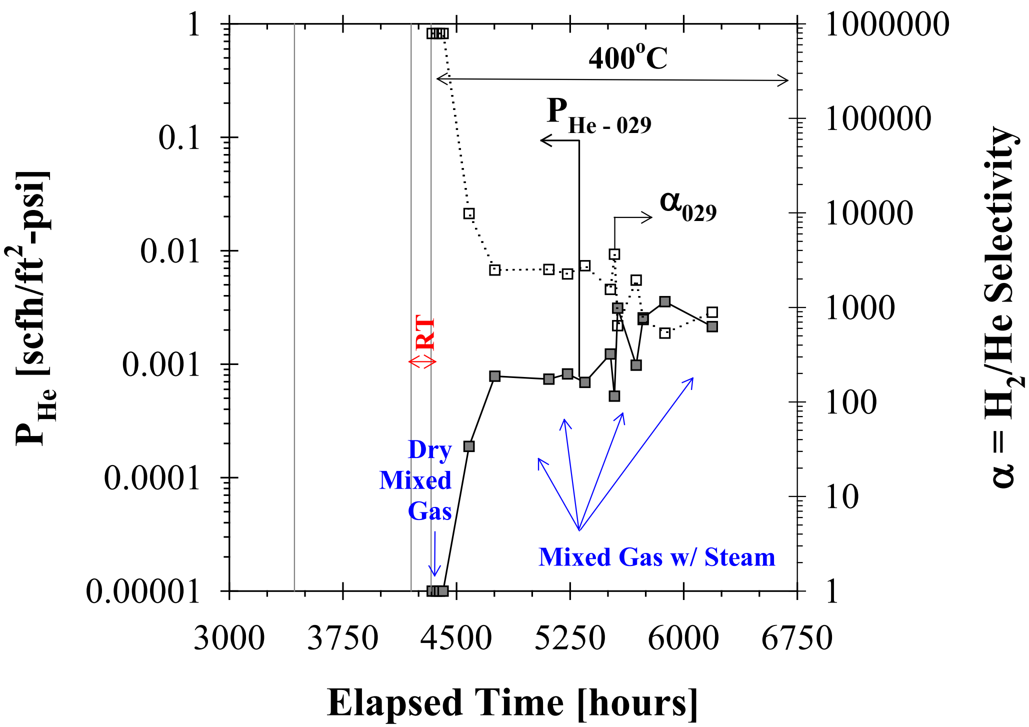
- H_2/He selectivity over 1000 and stable over the entire testing period

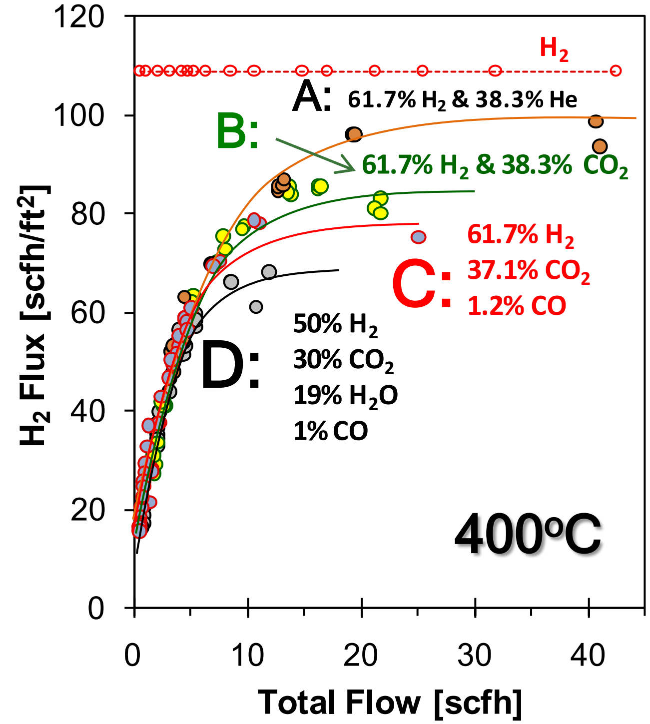
•Pressures of 330 psi have also been tested → H_2/He Selectivity before and after 330 psi. equaled 250

•n(H/Pd) estimated at 0.05 at 330 psi / 450°C → max compression stress -300-500 Mpa

• → **high pressure has a negligible effect on leak formation**

Mixed Gas Testing of Membrane #029 7.6 µm Pd

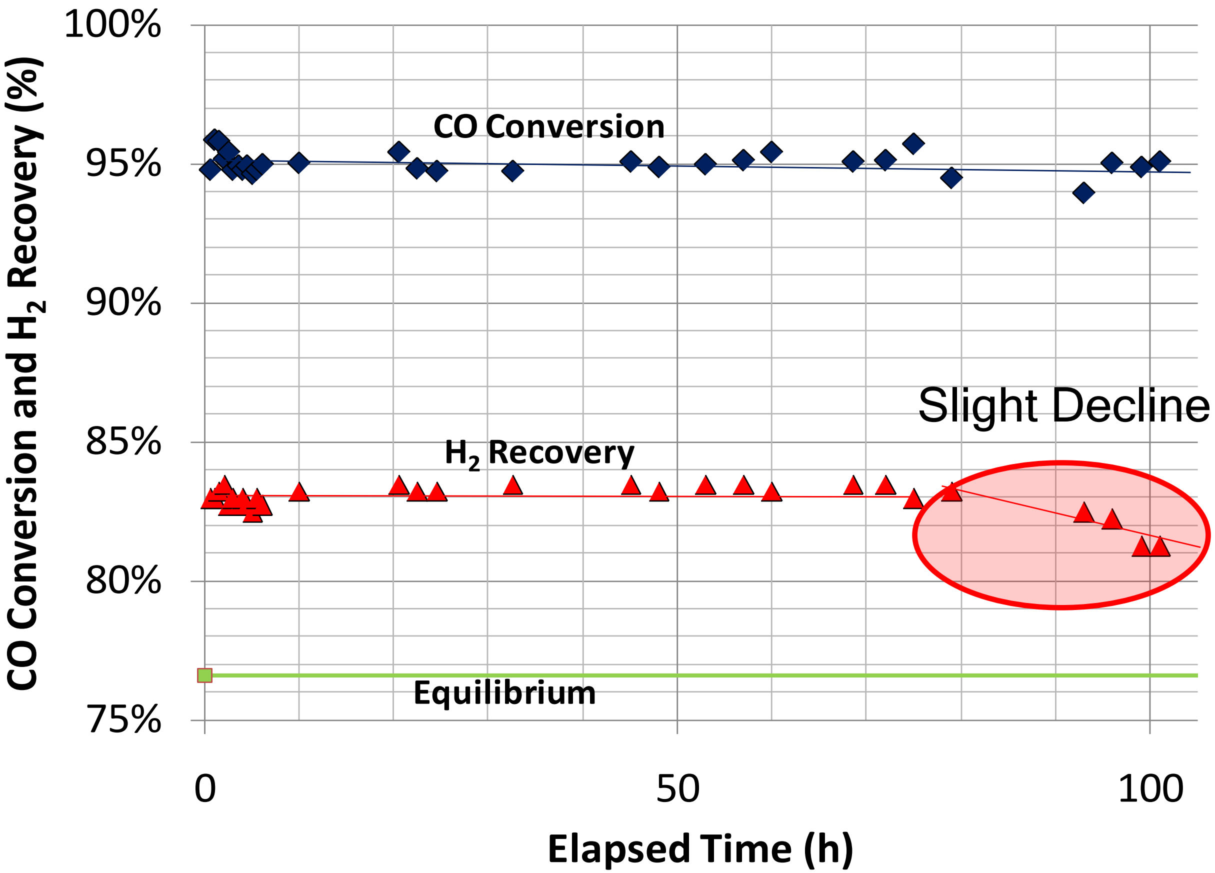




- Mixed gas permeation testing for an additional ~3000 hours at ~400°C & at a ΔP range of 1-14 atm (P_{Low}=1 atm) w/ stable H_2 Flux, H_2/He Selectivity & no significant increase in He leak after successive testing at 400°C
- Below 10 scfh, high recovery (> 90%) and no significant/additional inhibiting effect of ~19% steam or CO on H_2 flux
- Permeate: H_2 only, no other gases were detected
- Retentate: High-pressure CO_2

DOE-NETL Test Protocol v7 - 05/10/2008

Long-term WGS $CMR_{13.1\ \mu m\ Pd}$



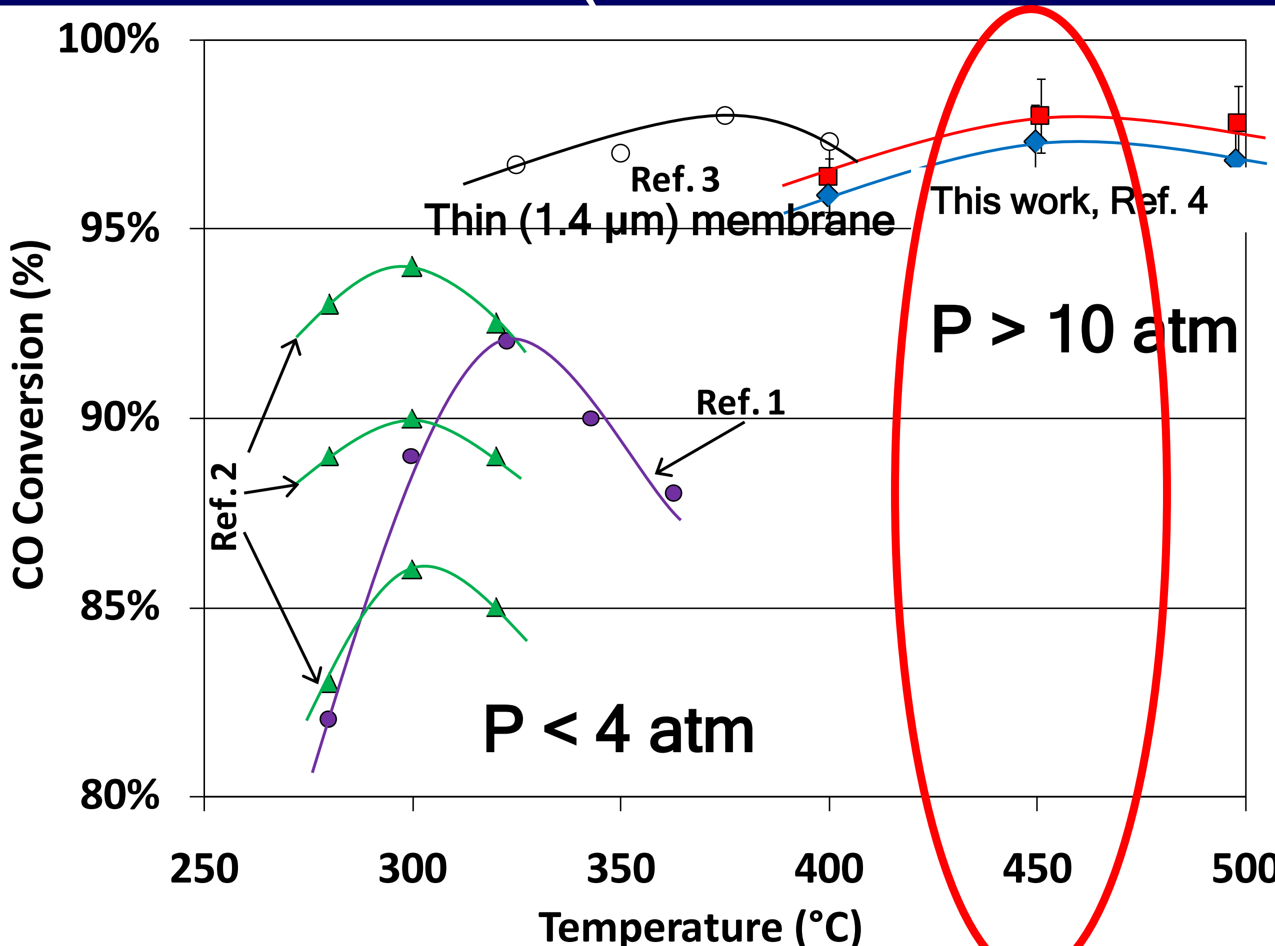
Feed Conditions	
22.7% CO, 22.0% H_2, 9.9% CO_2, 45.4% H_2O	
H2O/CO	2.0
GHSV (h^{-1}):	4500
T (°C):	450

Membrane: AA-8R	
Selectivity (F_{H2}/F_{He})	
Initial	4000
After WGS Experiment	400 (-90%)
Permeance ($scfh/ft^2psi^{0.5}$)	
Initial	27.9
After WGS Experiment	26.6 (-4.6%)

- Stable CO conversion and H_2 recovery were observed for up to 80 hours
- Stable H_2 permeance after WGS test
- Significant selectivity decline after test

Flux @ 450°C = 44.3 $scfh/ft^2$ (ΔP of 14.7 psi)

WGS CMR Temperature Dependence
(existence of a maximum)

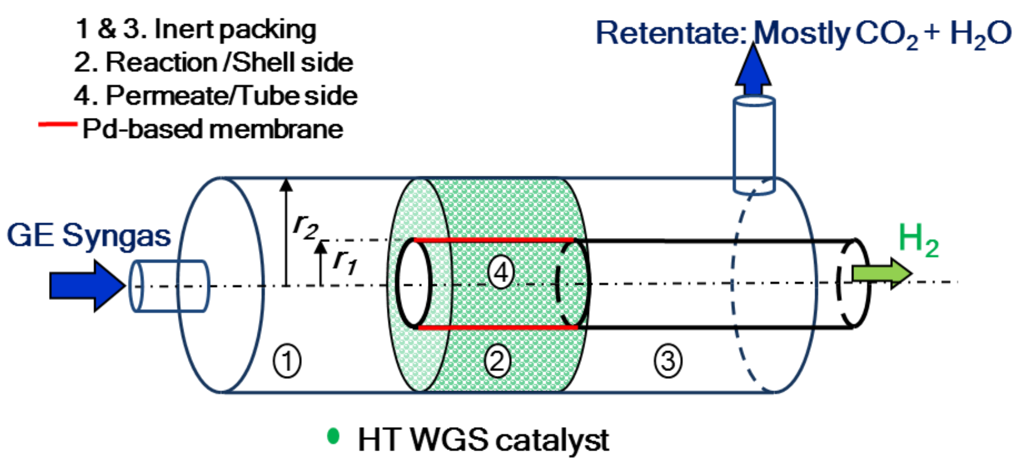


- Maximum in CO conversion results from balance between thermodynamic equil. and reaction kinetic
  - At low temperatures
    - Low catalytic activity
    - High therm. equil. conv.
  - At high temperatures
    - High catalytic activity
    - High permeance
    - Low therm. equil. conv.

- [1]Basile *et al.*, Gas Sep & Pur 1996; 10(4), 243-54.
- [2]Barbieri *et al.*, J Power Sources 2008; 182, 160-7.
- [3]Bi *et al.*, Int J H Energy 2009; 34, 2965-71.
- [4]Augustine et al. Int. J. H. Energy, 2011, accepted for publication. Published online

Existence of a temperature maximum at ≈450°C

2D Membrane Reactor Model



GE Syngas Composition (Dry):

45% CO + 38% H_2 + 17% CO_2

Properties of Pd/Inconel Membrane:

Q_o = 5409 $ft^3.\mu m/(ft^2.h.psi^{0.5})$

Pd Thickness = 10 µm

E_p = 15.6 kJ/mol

Selectivity (H_2/He) = ∞

Mass balance equations at the unsteady state conditions:

$$\frac{\partial}{\partial t}\left(\frac{F_{H_2}}{F_{Total}}\right) = \frac{1}{C_{Total}^{Shell}}\left[-\frac{\partial F_{H_2}}{\partial v^{Shell}} + \frac{1}{\Delta v^{Shell}}\int_{W_y}^{W_{y+\Delta y}} r_{H_2} dw - \frac{Q.A_m}{l.\Delta v^{Shell}}.\Delta P_{H_2}^{0.5}\right]$$

Reaction side (1)

$$\frac{\partial}{\partial t}\left(\frac{F_{H_2}}{F_{Total}}\right) = \frac{1}{C_{Total}^{Tube}}\left[-\frac{\partial F_{H_2}}{\partial v^{Tube}} + \frac{Q.A_m}{l.\Delta v^{Tube}}.\Delta P_{H_2}^{0.5}\right]$$

Permeate side (2)

Energy balance equation in the reaction side at the unsteady state conditions

$$\left(\sum_{i=1}^{n} C_i c_{pi}\right)\frac{\partial T}{\partial t} = -\left(\sum_{i=1}^{n} F_i c_{pi}\right)\frac{\partial T}{\Delta v_{shell}} + (-r_{CO})[-\Delta H_{Rxn}(T)] \quad (3)$$

Two dimensional mass balance equations at the steady state and isothermal conditions:

$$\frac{\partial(u.C_i)}{\partial y} - \frac{D_{ef,i}}{u}\left[\frac{\partial^2(u.C_i)}{\partial r^2} + \frac{1}{r}\frac{\partial(u.C_i)}{\partial r}\right] + \rho_{bulk,cat}R_i = 0 \quad (4)$$

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Process Intensification

➤ Performance target levels: X_{CO} = 98% & R_{H2} = 95%

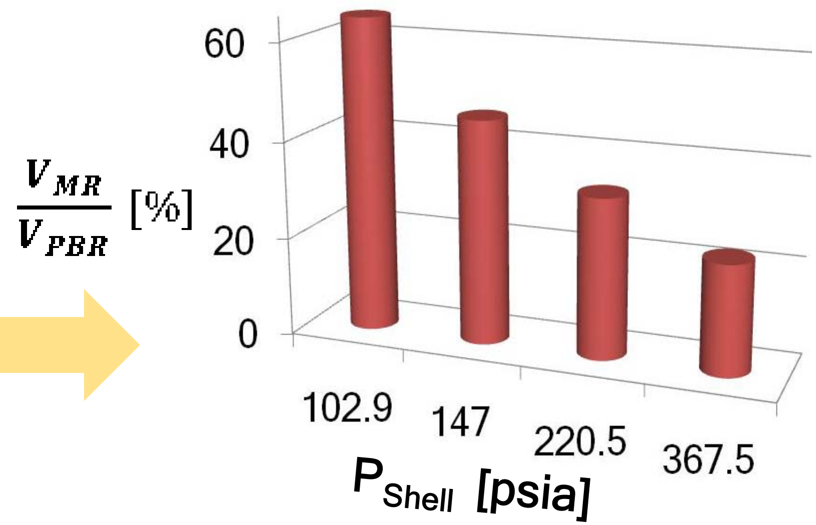
For the non-isothermal MR: $T_{Rxn,Max}$ = 500°C & $T_{Rxn,Min}$ = 300°C for pure Pd

Isothermal Case:

P_{shell} = 220 & $P_{Tube,MR}$ = 6 psia

$F_{Dry\ feed}$=1.1 scfh , T_{Rxn}=400°C

	H_2O:CO	X_{CO} [%]	R_{H2} [%]	F_{H_2} Ratio MR / PBR	$W_{catalyst}$ Ratio
MR	2	99	96	1.2	1
PBR	9	98	-		



Non-isothermal Case:

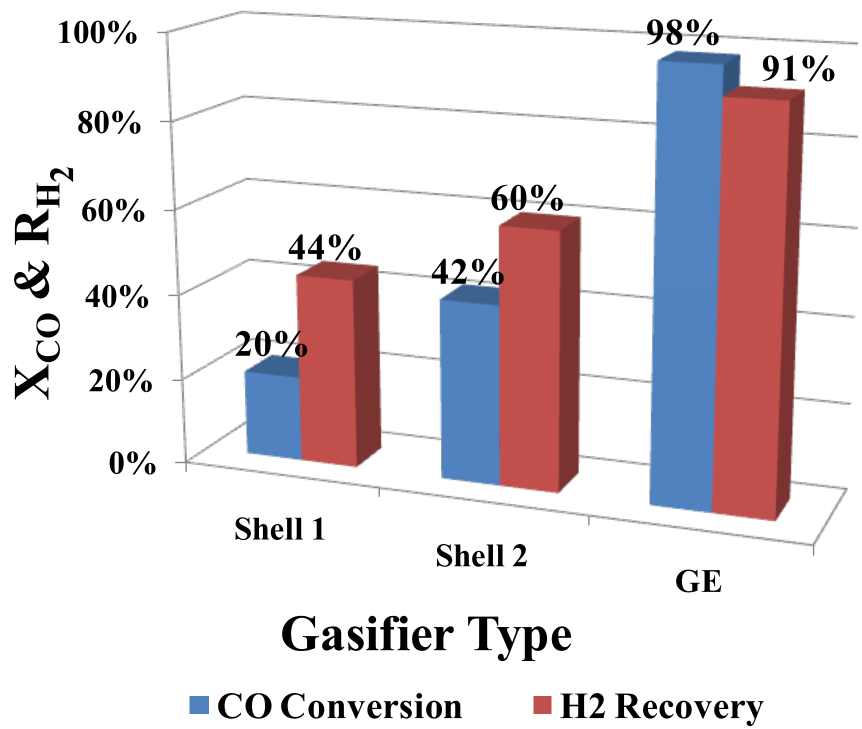
P_{Shell} = 300 & $P_{Tube,MR}$ = 6 psia, $F_{Dry\ feed}$=3.4 scfh

	H_2O:CO	X_{CO} [%]	R_{H_2} [%]	$T_{Rxn,Max}$ [°C]	F_{H_2} Ratio MR / PBR	Volume Ratio MR/PBR	$W_{catalyst}$ Ratio
MR	4	99.5	95	477	1.2	0.6	0.6
PBR	9	98	-	395			

Pd/Alloy Membrane Reactors in IGCC Plants

Table 1. Syngas compositions†

	Shell 1	Shell 2	GE
Scrubber T [°C] →	128	160	243
CO	**56.4**	**49.6**	**15.6**
H_2	29.7	26.3	15.1
CO_2	1.4	1.3	7.3
H_2O	**7**	**18.1**	**61**
N_2	4.53	3.86	0.8
Ar	0.7	0.6	
H_2S	0.24	0.21	0.12
COS	0.02	0.02	
other	0.01	0.01	0.08



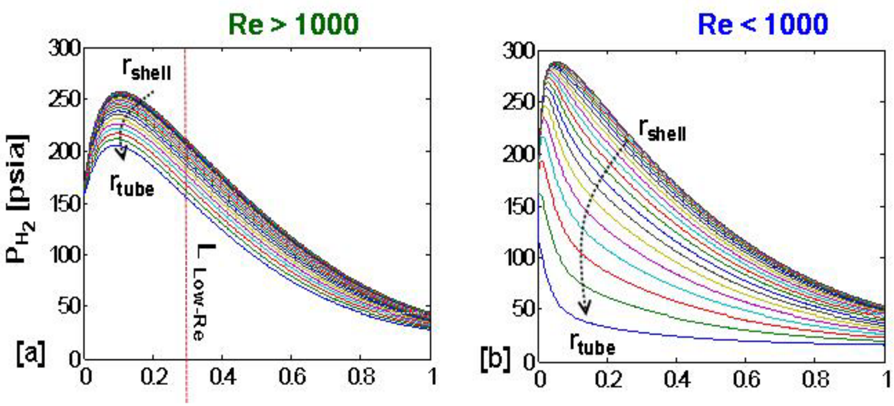
P_{shell} = 220 & $P_{Tube,MR}$ = 15 psia
F_{feed}=1.1 scfh , T_{Rxn}=450°C

GE Gasifier

- Highest H_2O:CO ratio
- Highest gasifier pressure
- Highest X_{CO} and R_{H_2}
- Lowest H_2S +COS concentration

† http://sequestration.mit.edu/pdf/LFEE_2005-002_WP.pdf

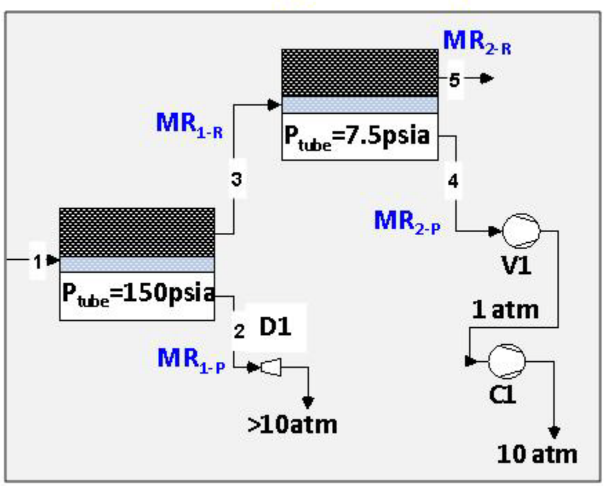
Two-Dimensional Isothermal Model Results

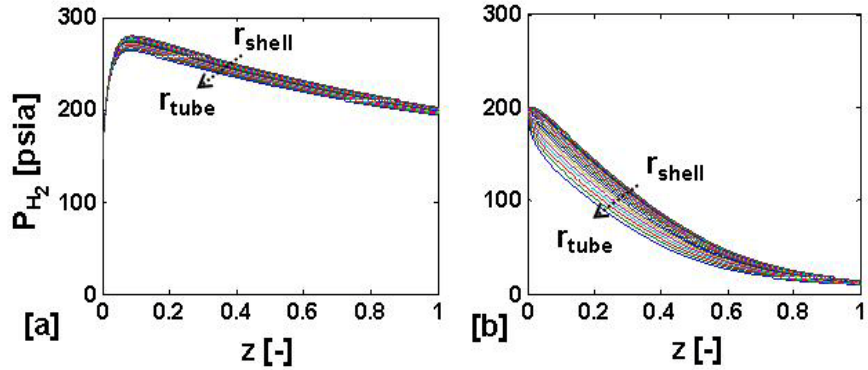


$T = 450°C$, $P_{shell} = 735$ psia, $P_{tube} = 15$ psia, $H_2O:CO = 2$

- Good match between the experimental and modeling results for both X_{CO} & R_{H2}
- Operate Re > 1000, use longer tubes
- Total Pd $Area_{Re>1000}$ < Total Pd $Area_{Re<1000}$

MR Configuration Options





$T = 400°C$, $P_{shell} = 735$ psia, $H_2O:CO = 2$
2 in OD × 31 ft L membrane tubes

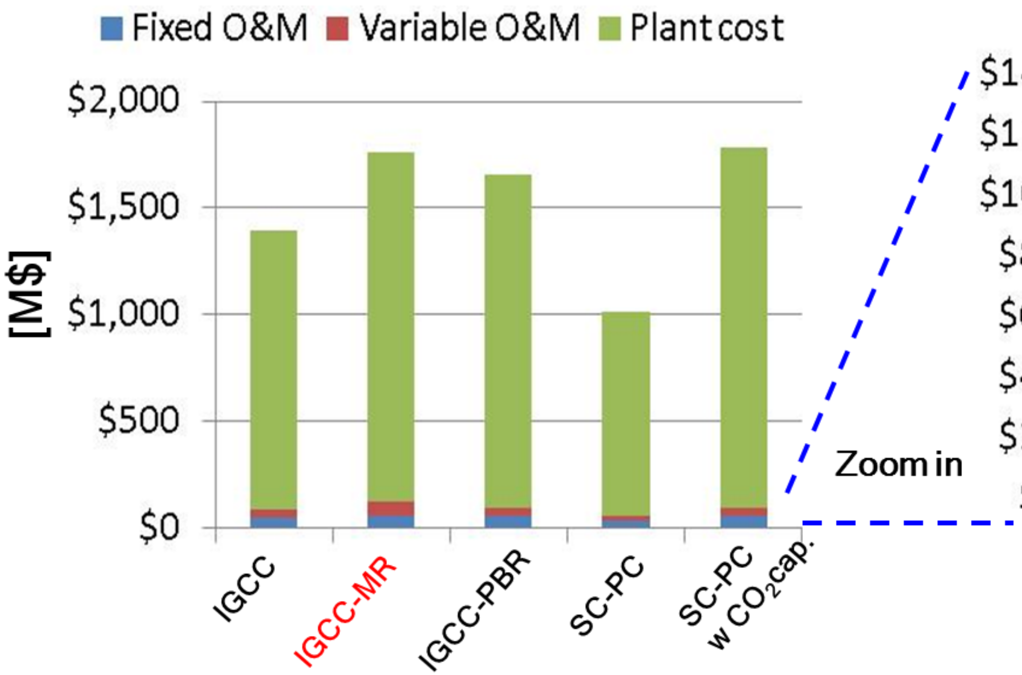
X_{CO} [%]	98.7
R_{H2} [%]	97.1
H_2 tpd	~737

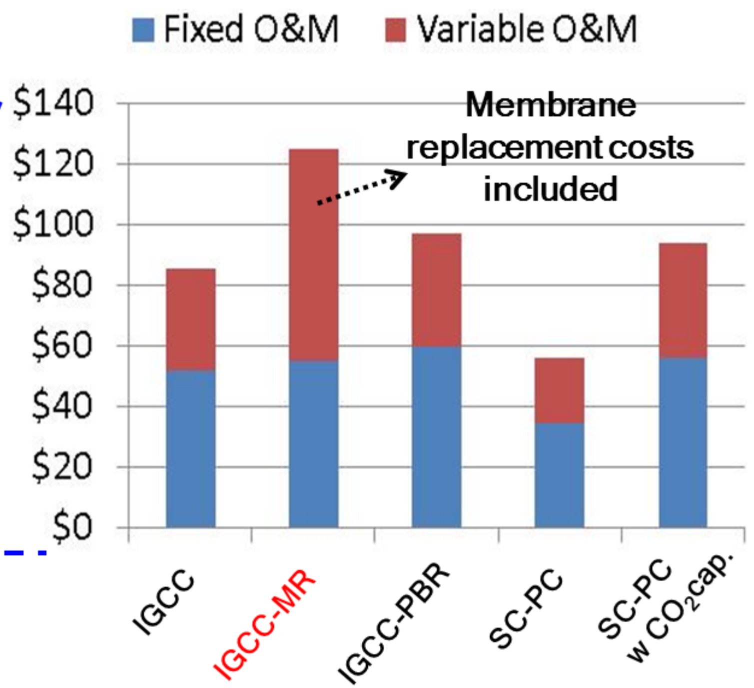
Capital – O&M Costs

Target: Coproduction of hydrogen and power by using Pd-based membrane reactors (Mitretek TR 2002-31)

Cost analysis is based on GEE radiant only IGCC power-plant (DOE/NETL 2010/1397) + Pd/Inconel MR

Assumption : 1 year membrane lifetime





Dimension (each)	2 in OD × 25 ft L
Total Pd Area	~118000 ft^2
Total H_2 Production	744 tonne/day

Membrane reactor module:

Equipment cost : 533 \$/ft^2

Fixed capital investment : 1004 \$/ft^2

Total capital investment : 1254 \$/ft^2

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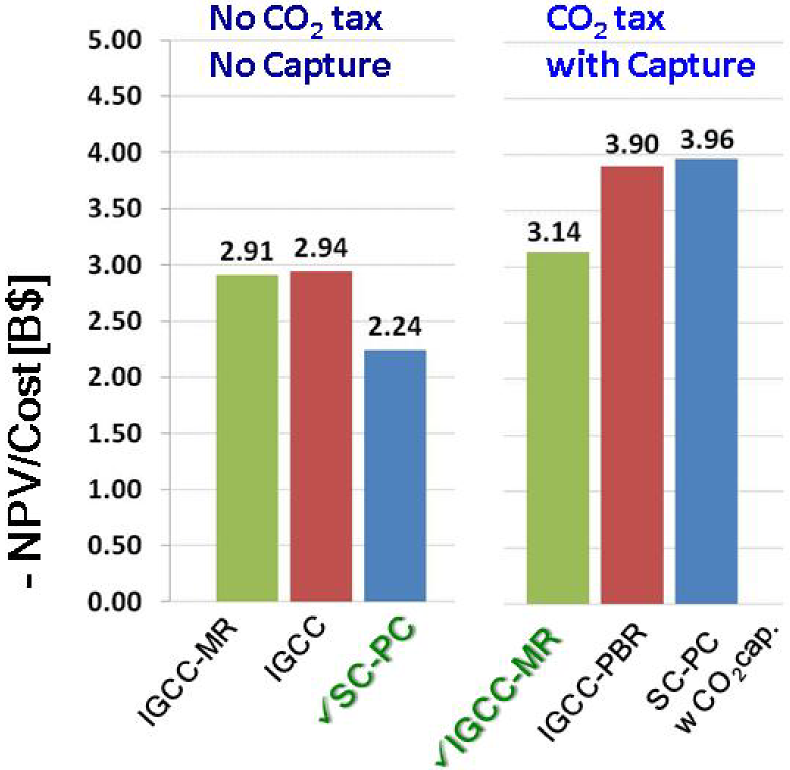
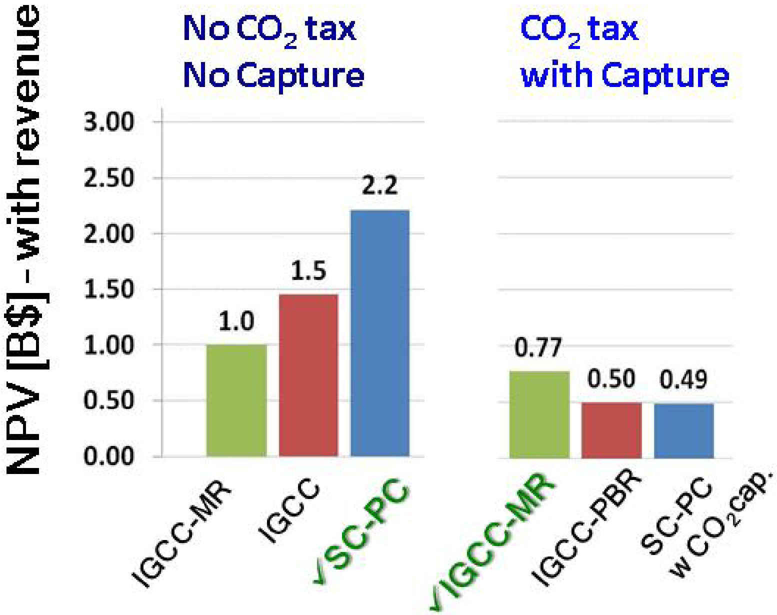
Net Present Value (NPV) Analysis





Assumptions:

- 40 years plant lifetime
- 1 year membrane lifetime
- Discount rate = 9% for MR 6% for the others
- Inflation rate = 2.5% (U.S. Dep. of Lab. Bureau)
- 25$/ tCO_2 tax, starting in 2015 (MIT, "Future of Coal")
- Electricity selling price = 9 ¢/kWh (U.S. Energy Inf. Adm.)

❑ Flaw of averages

❑ Risk in the uncertain inputs

❑ Generate a more realistic distribution of the plant's economic value rather than a single-point/estimate

Monte Carlo simulations for uncertainties effect on NPV

1. Develop the parametric NPV model

2. Determine the uncertain inputs

3. Define realistic distributions for the uncertain inputs

4. Assign random variables for the uncertain variable within the distribution range

Repeat N times ($N=10^4$)

5. Evaluate the NPV model

6. Analyze the results

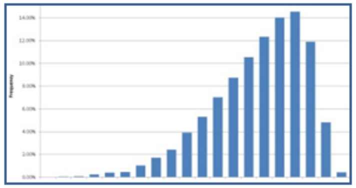
x_1 x_2..........x_i

Inputs (x)

Net Present Value Model

$y = f(x_1, x_2, \ldots x_i)$

NPV (y)



Create Histograms
(Estimate of the probability distribution)

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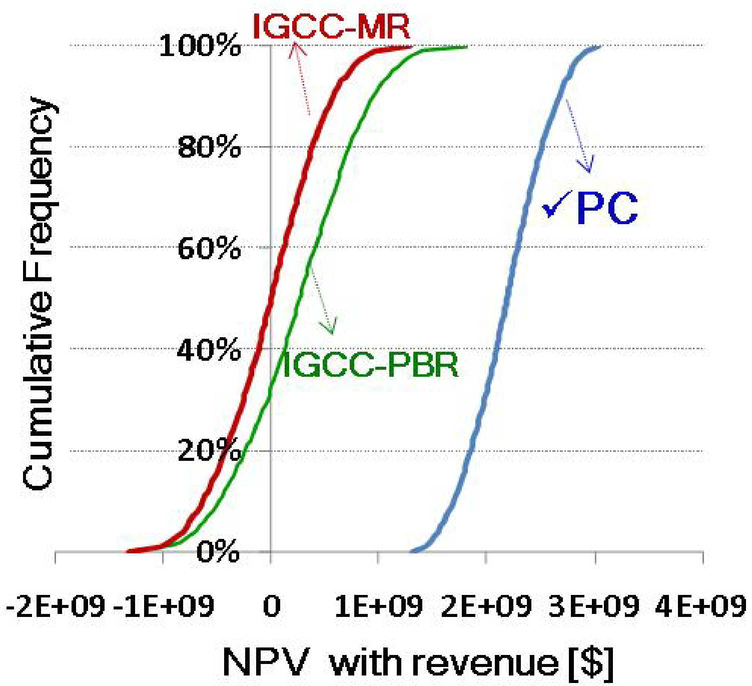
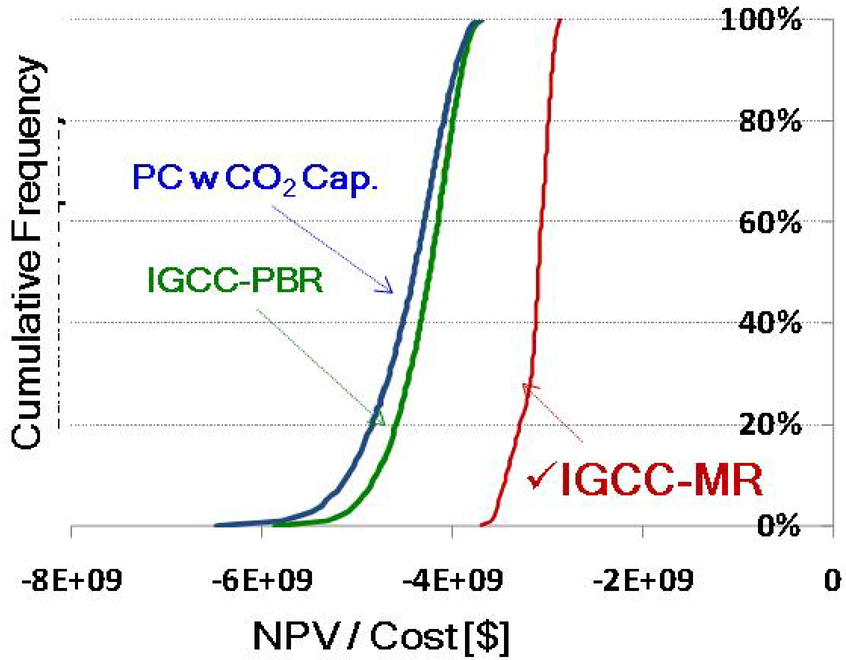
Economic Evaluation in the Presence of Uncertainty with Monte Carlo Simulation





Current Situation

- No CO_2 tax
- PC capacity factor = 85%

Uncertainty

- IGCC-MR & IGCC-PBR capacity factor = 40-80%
- Electricity selling price:
  min =6.6, most likely=8.4, max = 9.9 ¢/kWh

Regulatory action on carbon emissions after 2015

Uncertainty

- Initial CO_2 tax:
  min =0, most likely= 25, max = 100 \$/ tCO_2
- CO_2 tax growth rate:
  min =2%, most likely= 5%, max = 8%
- Capacity factor = 60-85% for IGCC-PBR&MR
  70-85% for PC w CO_2 cap.
- Pd price: Historical data

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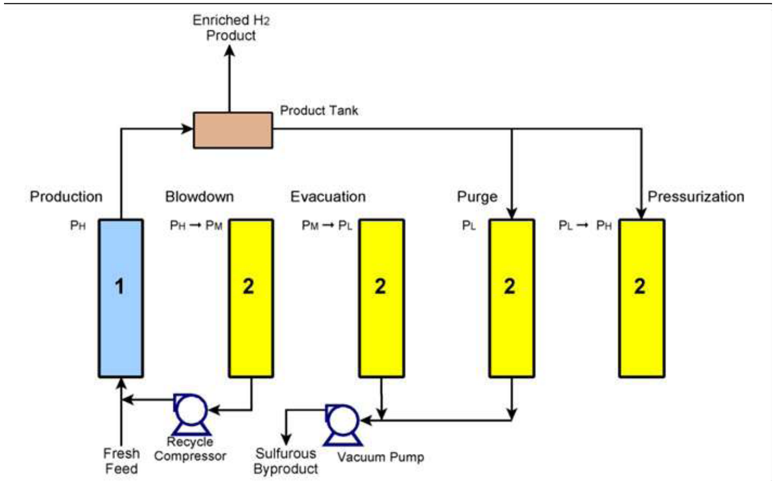
Collaborations
Adsorption Research Inc. (ARI); sub

- Zeolite 5A and Hisiv 3000 were chosen for all tests
- Feed H_2S Concentration: 500 to 1,000 ppm(v)
  - Light Product = 98+% Helium (H_2) Recovery.
  - Light Product = Either <1 or <20 ppm H_2S.
- Feed: 500 ppm H_2S + 500 ppm COS ≈ Same Performance as 1,000 ppm H_2S.
- Feed H_2S Concentration: 10,000 ppm(v)
  - Apparent Working Capacity Was Reduced.
  - Light Product = 97% Helium (H_2) Recovery.
  - Light Product = 1.4 ppm H_2S.
- CO_2 Recovery in Light Product Varied According To The Allowable H_2S Concentration in Light Product.

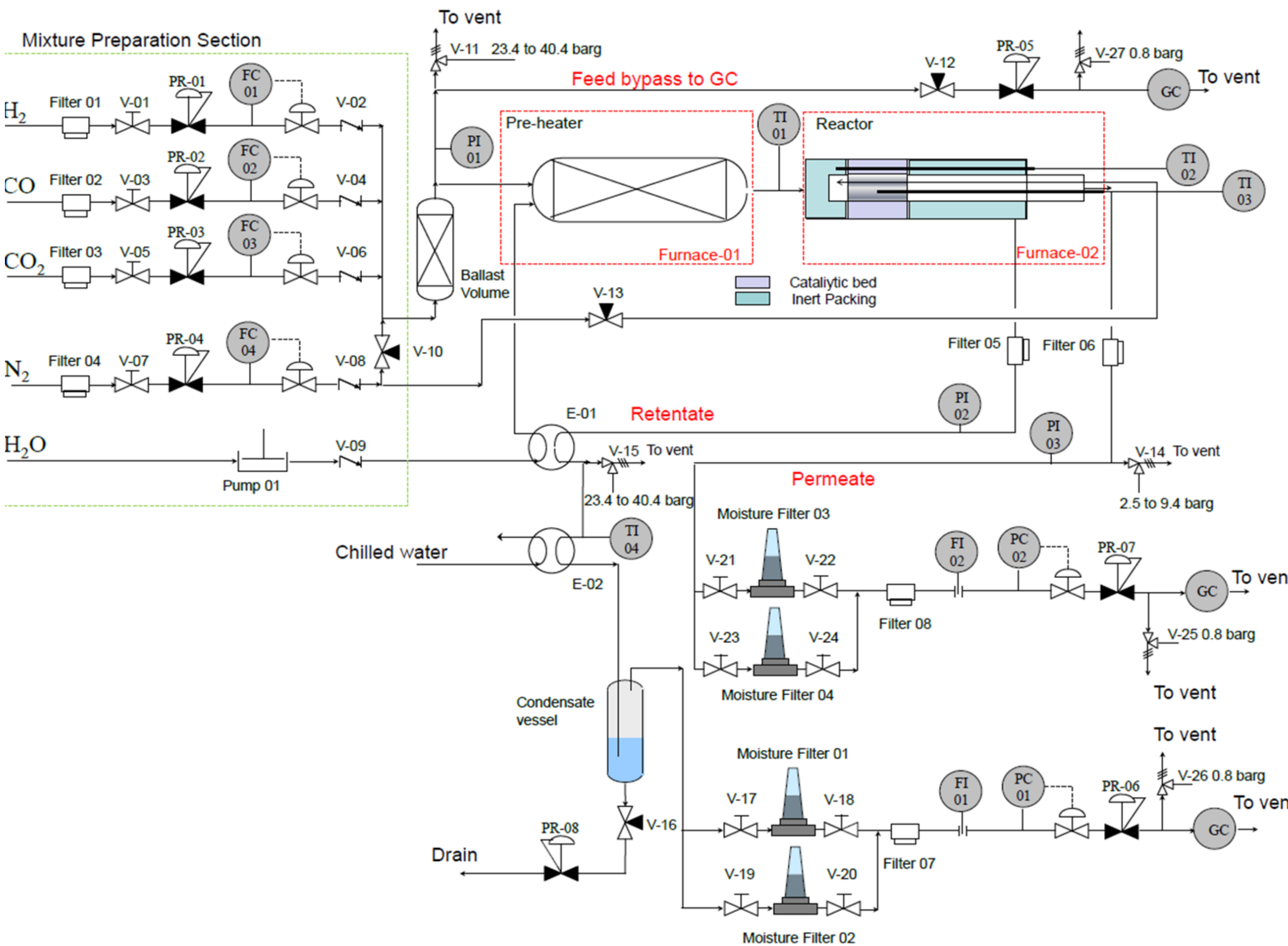
Current PSA Cycle



•General conclusion:

PSA system and the cycle developed are promising however the loss of CO_2 for carbon capture is a prohibiting factor

PID of a new WGS CMR testing Rig at WPI



new WGS CMR testing Rig at WPI

Proposed Future Work (under grant DE-FE0004895)

- Test new grading techniques on bare support surfaces to achieve 8 μm Pd on any PSS support (alumina deposition)
- Test new Pd deposition methods (e. g. electrodeposition) for selectivity stability
- Continue long term WGS reaction study (an 1000 h run is planned) for both ½" OD and 1" OD membranes
- Asses the Syngas effect on selectivity stability
- Continue Pd/Au alloying studies to improve H_2 flux
- Study new coatings for Hydrogen sulfide poisoning inhibition
- Continue long-term sulfur poisoning & recovery experiments

* Table 4 in DOE-NETL Test Protocol v7 – 05/10/2008

Project Summary

- Membranes prepared by sequential annealing/polishing (pAP method) -with thickness higher than 7-8 μm have a stable selectivity over thousands of hours at 400-450°C (029/030/31/033) with permeances limited by mass transfer resistance in the support
- Flux of ~359 scfh/ft^2, which exceeded the DOE's 2015 H_2 flux targets [T=442°C & ΔP of 100 psi (with P_{Low}=15 psia)] was achieved but low selectivity H_2/He (~500) due to very low thickness (3-5 μm)
- Calculation made to establish that the minimum initial He permeance of 130 scfh /(ft^2-psi) for the porous metal supports in order to achieve DOE's 2015 target with a 7-8 μm membrane at 450°C
- Completed the first WGS testing rig for 1"OD membranes at WPI
- Demonstrated that:
  - WGS reaction with high temperature catalyst (HIFUEL) occurs optimally at a temperature of ≈450°C
  - Effect of pressure on leak stability negligible
  - 97% CO conversion and 80% H_2 recovery achieved in an 18.1 µm thick Pd-based CMR operated at 450°C, ΔP=200 psi (P_{Low}=15 psia) and $GHSV_{stp}$ = 2900 h^{-1}, exceeding the equilibrium conversion of 93%.

WPI

Project Summary

- **Successfully completed steady-state MSR & WGS reaction 2D modeling studies & process intensification analysis:**
  - A 2D model was developed to take into account radial gradients. Several configurations were modeled to achieve Xco > 98% and R_{H2} > 95% for the isothermal case.
- **Successfully completed process intensification, safety and economic analysis:**
  - Safety boundaries for WGS feed gas composition, feed temperature, catalyst density have been established for the adiabatic case (in case cooling fails) to achieve Xco > 98% and R_{H2} > 95% keeping membrane temperature lower than 500°C.
  - Capital -O&M costs was calculated to be around 1254 $\$/ft^2$ very close to DOE target (1000 $\$/ft^2$)
  - NPV analysis showed that if CO_2 taxes were implemented, IGCC-MR with carbon capture would be the preferred configuration.
  - Monte-Carlo analysis was used to study the variability of 11 uncertainties and proved that with taxes on CO_2 emission, IGCC-MR configuration would be the preferred choice.
- **Completed all simulations, tests of PSA cycles with commercial adsorbents and feeds including all reactants plus H_2S and COS (Sub, ARI).**
- **PSA for hydrogen sulfide removal is however not included in the process intensification due to the high removal loss of CO_2**

WPI

Project Summary Table: Permeation Results

	DOE Targets		Current WPI Membranes (1/2” OD, 2.5” Length, ~24 cm^2)					
	2010	2015	#025R	#027	#029	#030	#031	#032
Flux [$scfh/ft^2$]	200	300	65.9	36.1	166	178	26.6	359
ΔP (psi) H_2 partial pressure (P_{Low}=15 psia)	100*	100*	15	15	100	102	15	100
Temperature [°C]	300-600	250-500	400	400	450	442	450	442
H_2/He Selectivity	n/a	n/a	~220	~120	∞	10000	~4500	~450
Total Test Duration [hours]	n/a	n/a	1015	~1250	~4500	~1400	~2200	~523
Thickness [μm]	n/a	n/a	4.2 Pd	6.2 Pd/$Au_{5\ wt\%}$	7.6 Pd	7.9 Pd	7.0 Pd	3-5 Pd
WGS Activity	Yes	Yes	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested
CO Tolerance	Yes	Yes	Not tested	Not tested	Yes	Not tested	Not tested	Not tested
S Tolerance [ppm]	20	>100	Not tested	Not tested	Not tested	Not tested	Not tested	Not tested
H_2 Purity	99.5%	99.99%	99.0%	99.5%	≥99.999%	≥99.99%	99.98%	99.8%
ΔP Operating Capability (Max. Sys. Pressure, psi)	400	800-1000	15**	15**	225**	102**	15**	100**

§ DOE-NETL Test Protocol v7 - 05/10/2008, * Standard conditions are 150 psia hydrogen feed pressure and 50 psia hydrogen sweep pressure

** Maximum pressure tested, however, the ΔP can be higher since previous WPI membranes were tested up to 600 psi under MSR reaction conditions

Project Summary Table: Mixed Gas & WGS Reaction Results

	DOE Targets		Current WPI Membranes (1/2” OD, 2.5” Length, ~24 cm^2)				
	2010	2015	AA-4R*	AA-5R*	AA-6R*	AA-7R*	AA-8R*
Flux [scfh/ft^2]	200	300	262.3	108.6	427.0	98.1	96.4
ΔP (psi) H_2 partial pressure (P_{Low}=15 psia)	100**	100**	245.1	71.0	222.7	45.4	37.1
Temperature [°C]	300-600	250-500	400	450	450	450	450
H_2/He Selectivity	n/a	n/a	71,000	2,800	1,100	25	670
Total Test Duration [hours]	n/a	n/a	1,030	1,080	860	350	970
Thickness [μm]	n/a	n/a	14.4	18.1	18.1	14.3	13.4
WGS Activity	Yes	Yes	Not tested	w/ packed catalyst	Not tested	w/ packed catalyst	w/ packed catalyst
CO Tolerance	Yes	Yes	Yes	Yes	Yes	Yes	Yes
S Tolerance [ppm]	20	>100	Not tested	Not tested	Not tested	Not tested	Not tested
H_2 Purity	99.5%	99.99%	99.99%	99.96%	99.91%	96.2%	99.85%
ΔP Operating Capability (Max. System Pressure, psi)	400	800-1000	250	250	250	250	250

§ DOE-NETL Test Protocol v7 - 05/10/2008

* R - repaired by mechanical treatment and Pd plating

** Standard conditions are 150 psia hydrogen feed pressure and 50 psia hydrogen sweep pressure

WPI

Project Summary Table: Mixed Gas & WGS Reaction Results

	DOE Targets		Current WPI Membranes (1/2" OD, 2.5" Length, ~24 cm^2)				
	2010	2015	AA-12R	AA-14	AA-18	AA-21R	AA-22
Flux [scfh/ft^2]	200	300	550.5	76.4	76.0	79.6	86.3
ΔP (psi) H_2 partial pressure (P_{Low}=15 psia)	100**	100**	300	29	57	48	28
Temperature [°C]	300-600	250-500	450	450	450	400	450
H_2/He Selectivity	n/a	n/a	240	190	998	176	80
Total Test Duration [hours]	n/a	n/a	150	360	400	864	144
Thickness [μm]	n/a	n/a	12.7	11.4	19.6	22.0	13.5
WGS Activity	Yes	Yes	Not tested	Not tested	Not tested	Not tested	Not tested
CO Tolerance	Yes	Yes	Not tested	Not tested	Not tested	Not tested	Not tested
S Tolerance [ppm]	20	>100	Not tested	Not tested	Not tested	Not tested	Not tested
H_2 Purity	99.5%	99.99%	99.6%	99.5%	99.9%	99.4%	98.8%
ΔP Operating Capability (Max. System Pressure, psi)	400	800-1000	400	250	250	400	250

§ DOE-NETL Test Protocol v7 - 05/10/2008
* R - repaired by mechanical treatment and Pd plating
** Standard conditions are 150 psia hydrogen feed pressure and 50 psia hydrogen sweep pressure

Project Summary Table: Mixed Gas & WGS Reaction Results

	DOE Targets		Current WPI Membranes (1/2” OD, 2.5” Length, ~24 cm²)				
	2010	2015	AA-24	AA-25	AA-26		
Flux [scfh/ft²]	200	300	84.6	59	51.2		
ΔP (psi) H_2 partial pressure (P_{Low}=15 psia)	100**	100**	37.8	16	23		
Temperature [°C]	300-600	250-500	400	450	500		
H_2/He Selectivity	n/a	n/a	360	78	233		
Total Test Duration [hours]	n/a	n/a	1220	170	288		
Thickness [μm]	n/a	n/a	16.2	20.6	12.8		
WGS Activity	Yes	Yes	Yes	Not tested	Not tested		
CO Tolerance	Yes	Yes	Yes	Not tested	Not tested		
S Tolerance [ppm]	20	>100	Not tested	Not tested	Not tested		
H_2 Purity	99.5%	99.99%	99.7%	98.7%	99.6%		
ΔP Operating Capability (Max. System Pressure, psi)	400	800-1000	250	250	250		

§ DOE-NETL Test Protocol v7 - 05/10/2008
* R - repaired by mechanical treatment and Pd plating
** Standard conditions are 150 psia hydrogen feed pressure and 50 psia hydrogen sweep pressure

WPI

Project Summary Table: Mixed Gas & WGS Reaction Results

	DOE Targets		Current WPI Membranes (1/2" OD, 2.5" Length, ~24 cm^2)				
	2010	2015	RK01	RK02	RK03	RK04	
Flux [scfh/ft^2]	200	300	41.3	82	60.7	83.8	
ΔP (psi) H_2 partial pressure (P_{Low}=15 psia)	100**	100**	15	15	15	15	
Temperature [°C]	300-600	250-500	450	500	450	450	
H_2/He Selectivity	n/a	n/a	∞	112	2189	1000	
Total Test Duration [hours]	n/a	n/a	1023	373	1017	1250	
Thickness [μm]	n/a	n/a	14	30.8	22	13	
WGS Activity	Yes	Yes	Not tested	Not tested	Not tested	Not tested	
CO Tolerance	Yes	Yes	Not tested	Not tested	Not tested	Not tested	
S Tolerance [ppm]	20	>100	Not tested	Not tested	Not tested	Not tested	
H_2 Purity	99.5%	99.99%	99.9999%	99.11%	99.95%	99.90%	
ΔP Operating Capability (Max. System Pressure, psi)	400	800-1000	Not tested	Not tested	Not tested	Not tested	

§ DOE-NETL Test Protocol v7 - 05/10/2008
* R - repaired by mechanical treatment and Pd plating
** Standard conditions are 150 psia hydrogen feed pressure and 50 psia hydrogen sweep pressure

WPI