



Project Review (FE0031633)

DEVELOPMENT AND TESTING OF A HIGH-TEMPERATURE PBI HOLLOW-FIBER MEMBRANE TECHNOLOGY FOR PRE-COMBUSTION CO₂ CAPTURE

Presented by Elisabeth Perea

Advanced Technology and Systems Division

SRI International



Project Team

Enerfex, Inc.



Energy Commercialization, LLC



August 26-30, 2019 • Convention Center • Pittsburgh, Pennsylvania

Disclaimer

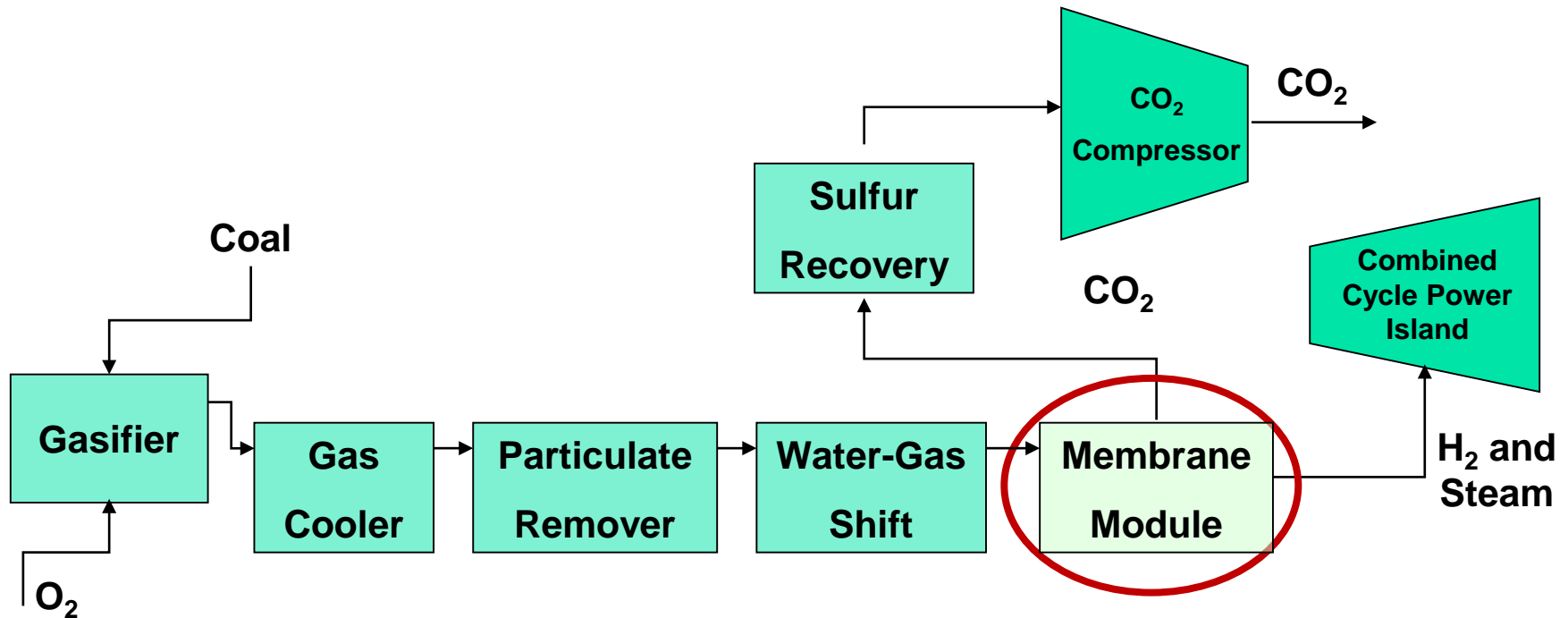
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Technology Background

Membranes for Pre-Combustion CO₂ Capture

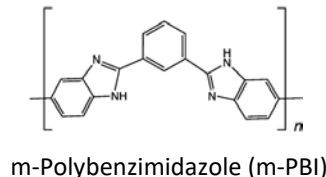
Advantages of High-Temperature Membranes for Separation of CO₂



Note: PBI hollow fiber membrane (HFM) is a H₂O and H₂ transporting membrane

Advantages of Membrane-Based Separation

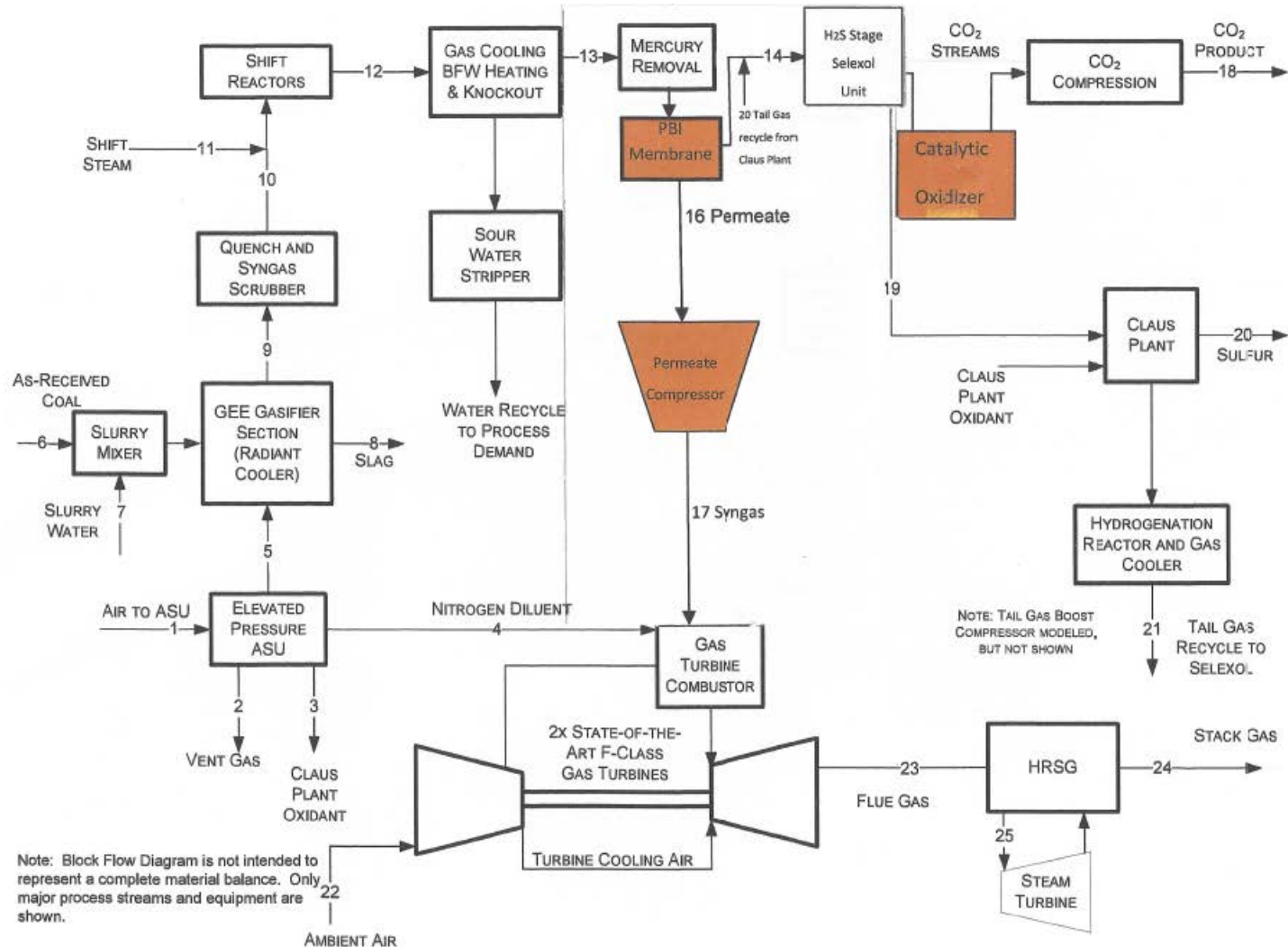
- Reduced costs for syngas cooling
- Reduced CO₂ compression costs
- Emission free, i.e., no solvents
- Decreased capital costs
- Low maintenance
- Modular



Characteristics of PBI Membranes

- Attractive combination of throughput (permeance) and separation (selectivity)
- Thermally stable up to ~ 300°C and sulfur tolerant
- Tested up to 225°C with simulated gases and with real syngas

Modification of Case B5B, GEE IGCC with CO₂ Capture



Techno Economic Analysis

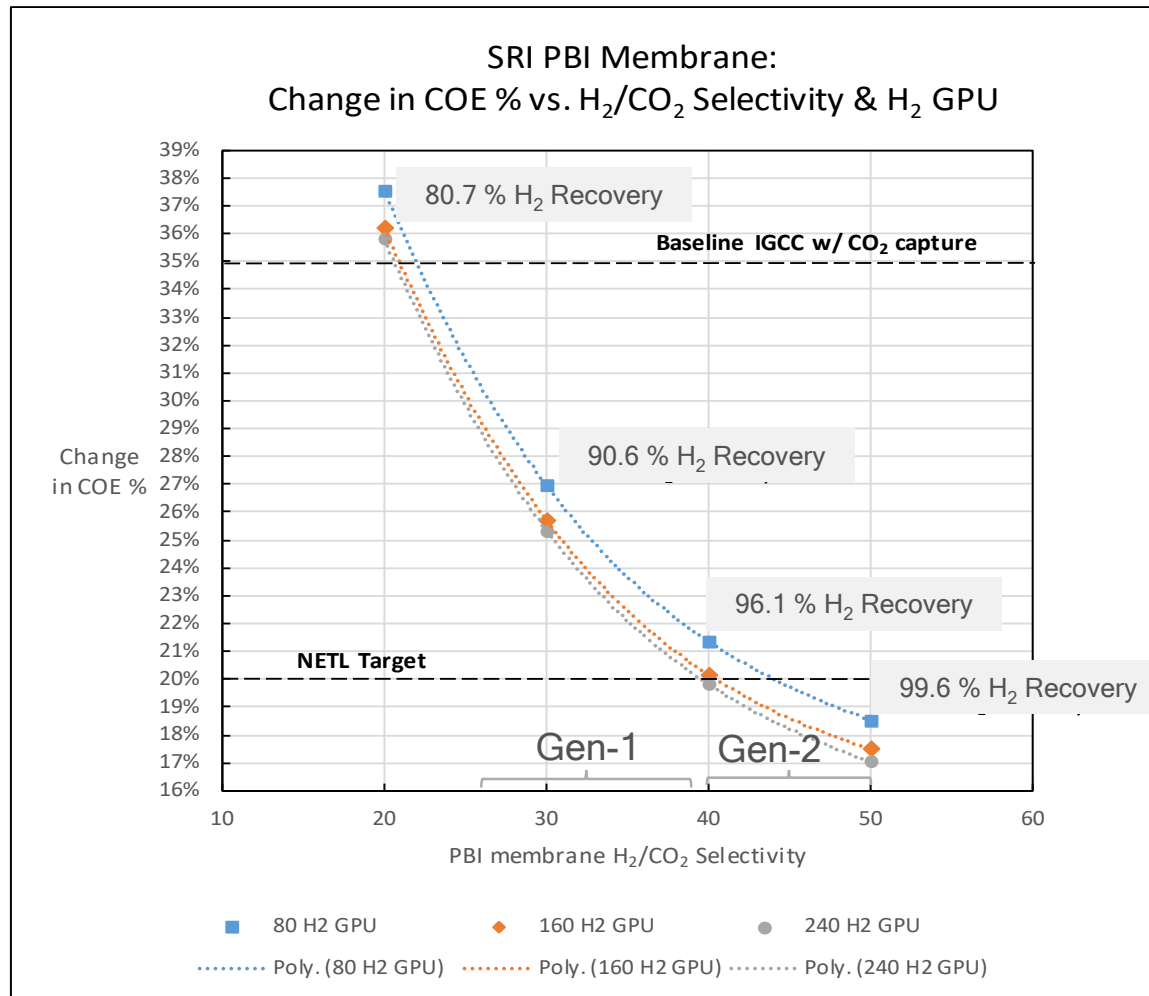
Developed by Enerfex

Case Name	IGCC-B1A Baseline No Capture	IGCC-B5B ¹ Baseline CO ₂ Capture	Gen-1 Membrane CO ₂ Capture	Gen-2 Membrane CO ₂ Capture
CO ₂ removal	No	Selexol	PBI membrane	
CO ₂ purification	No		Yes	
Sulfur removal	Sulfinol	Selexol		
Performance and Economic Summary				
H ₂ /CO ₂ Selectivity	n/a	n/a	40	50
H ₂ GPU	n/a	n/a	80	80
CO ₂ capture	n/a	92.25%	90.0%	90.0%
CO ₂ purity	n/a	99.48%	95.6%	95.6%
H ₂ recovery	n/a	99.98%	96.1%	99.6%
HHV plant efficiency	42.1%	32.6%	32.6%	34.4%
LHV plant efficiency	43.7%	33.8%	33.8%	35.6%
COE w/o T&S (\$/MWh)	\$107	\$135	\$121	\$118
COE w/ T&S (\$/MWh)	\$107	\$144	\$130	\$127
% Increase in COE	0.0%	34.9%	21.3%	18.5%

¹ Cost and Performance Baseline for Fossil Energy Plants Volume 1b: Revision 2b, July 31, 2015

COE vs H₂/CO₂ Selectivity and H₂ GPU

90% CO₂ Capture



Reduced CO₂ Capture Options:

- 40 selectivity and 100 GPU recovers 99% H₂ with 88% CO₂ capture
- 25 selectivity and 150 GPU recovers 99% H₂ with 82 % CO₂ capture

T= 225°C, Pressure ratio= ~12

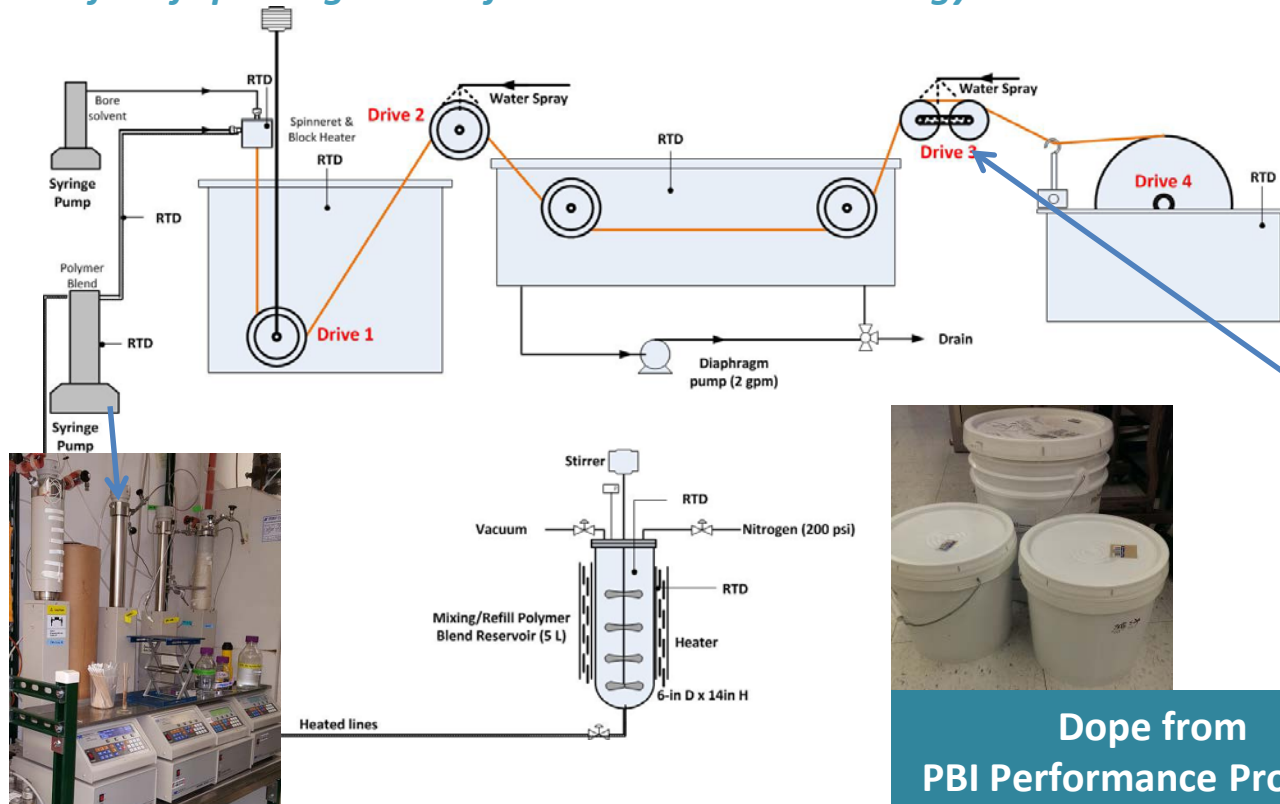
Illustration of the reduction in COE in moving from Gen- 1 to Gen-2 performance.



Advancements in Spinning Kilometer Lengths of Asymmetric PBI Hollow Fibers with Minimal Defects

First Spinning Line Installed at SRI in 2015

Transfer of spinning hollow fibers critical to Technology Maturation



Photograph of Drive 3

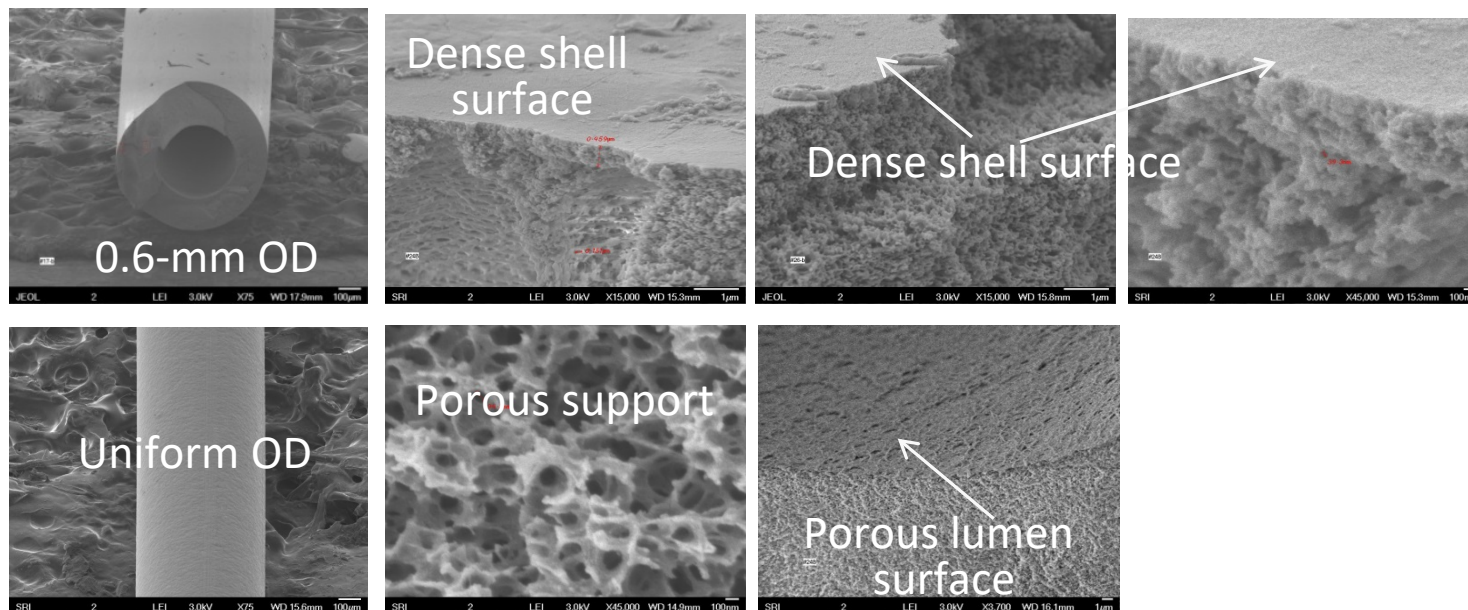
Dope from
PBI Performance Products

The new spinning line was crucial for developing an improved and robust spinning process that can be transferred to industry. The line enabled:

- Use of multiple coagulation solvents
- Increased productivity (1 gal reservoir)
- Process monitoring and data collection
- Precise flow controls and draw ratios
- Optimization of fiber diameter
- Optimization of the fiber dense-layer thickness

Fabrication of Fibers with Good Reproducibility

Quality control is the KEY to success when scaling-up



- Developed protocols for spinning $< 0.3\text{-}\mu\text{m}$ dense layer hollow-fiber membranes with membrane OD 450 to 650 μm . *ABOVE*: $\sim 0.1\text{-}\mu\text{m}$ fibers with $\sim 600\text{-}\mu\text{m}$ OD.
- Tested more than 100 fiber bundles (1-in) for fiber-spinning optimization of OD and dense layer
- Fabricated Gen-1 hollow-fiber membrane with a very thin, dense layer ($< 0.3\text{ }\mu\text{m}$) in kilometer lengths with very good reproducibility
- Spun $> 100\text{ km}$ of Gen-1 fibers for both Generon and SRI modules (4-in)

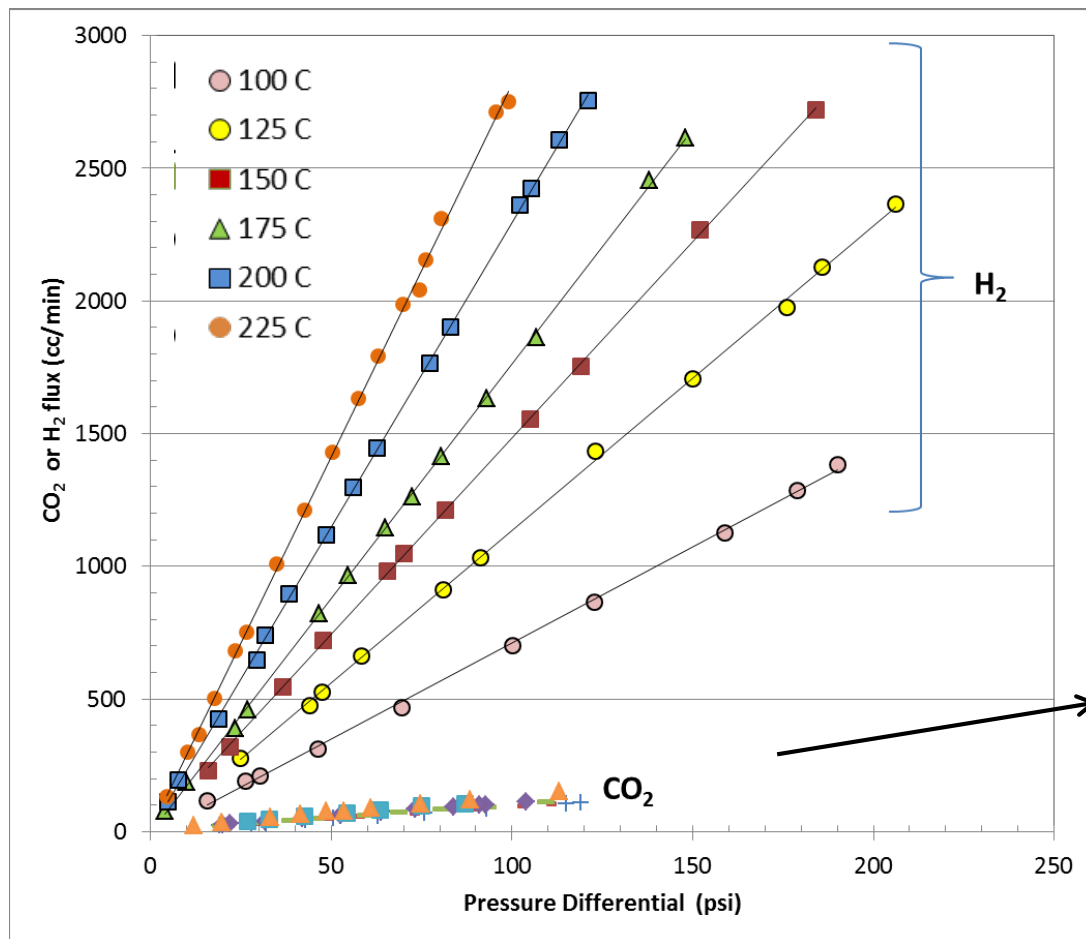
Achievements:

- Dense-layer thickness reduced from $1\text{ }\mu\text{m}$ to $< 0.3\text{ }\mu\text{m}$
- Fiber diameter reduced from 1 mm to less than $600\text{ }\mu\text{m}$

Gen-2:

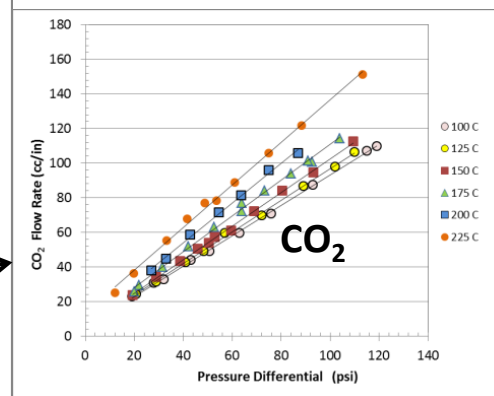
$0.5\text{ }\mu\text{m}$ dense layer
Reduced defect potential

PBI Fiber Withstands High Pressures & High Temperatures



Mixed Gases

	Selectivity
H ₂ /CO ₂	40
H ₂ /N ₂	98
H ₂ /CO	103
H ₂ /H ₂ S	>200
225 °C and 200 psi ΔP	



Observation: H₂/CO₂ selectivity increases with temperature up to 225 °C.

Fabrication of Large Modules at SRI: 4-inch



Photograph of 5000 fibers (5 m²) arranged for potting at SRI



4-in sleeve for fiber potting



Potted 4-in fiber module cross-section
(early design)

SRI fiber modules are designed for:

- Easy fabrication
- Easy handling
- Easy drop-in replacement

Critical Asset: Membrane Testing Skid

Installed and Tested at the NCCC



***Photograph of the skid installed at the NCCC
(April 2017)***



***Photograph of the skid installed at SRI
(December 2018)***

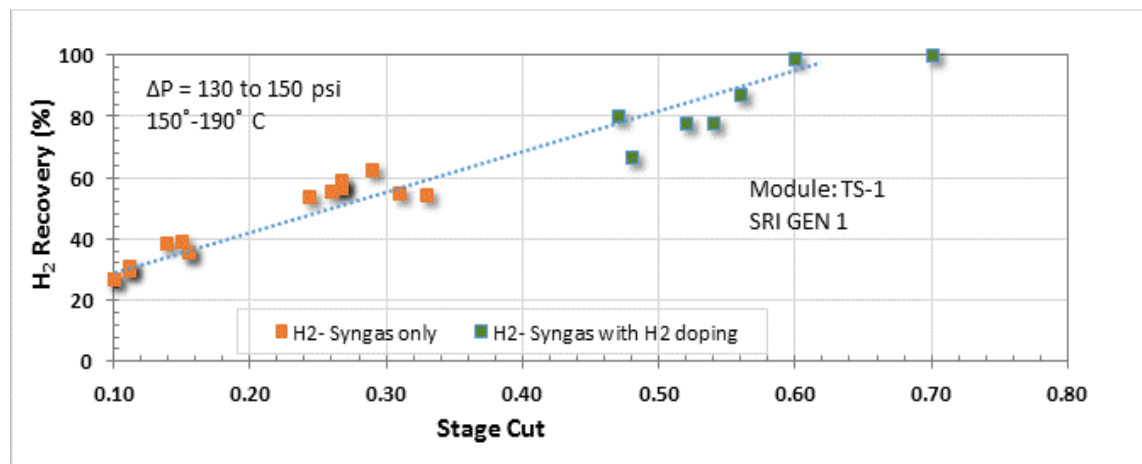
- PBI membrane skid transferred to the NCCC in March 2017
- Test campaign at NCCC conducted in April 2017 at 50 kWth scale
- Skid was removed from the host-site and returned to SRI in March 2018 for inspection and preservation
- Skid will be used in current work

Test Results at NCCC

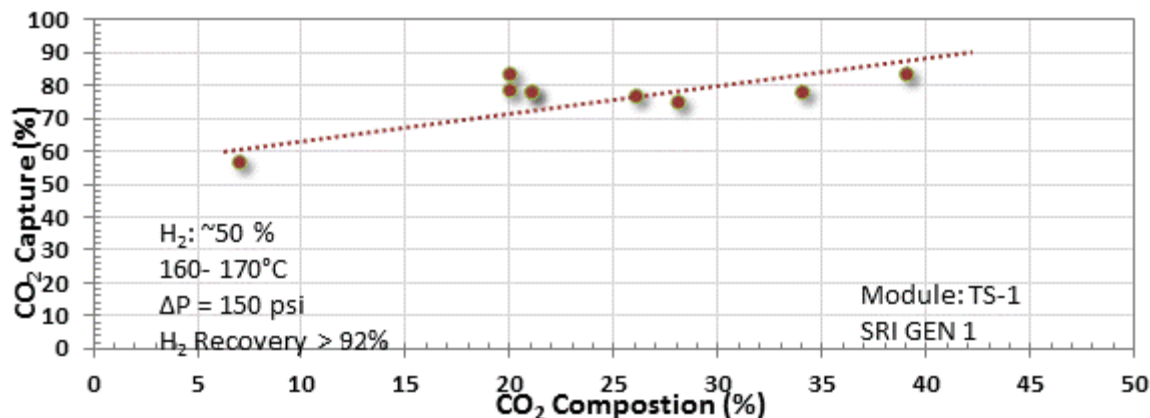
Air-blown Gasifier

Sample parametric matrix

Test Parameter	Range	Unit
Temperature	80 to 215	°C
Pressure	50 to 170	psig
Gas composition	Variable	slpm
Stage cut	0.2-0.7	
H ₂ in syngas	12 to 50	%
CO ₂ in syngas	5 to 40	%



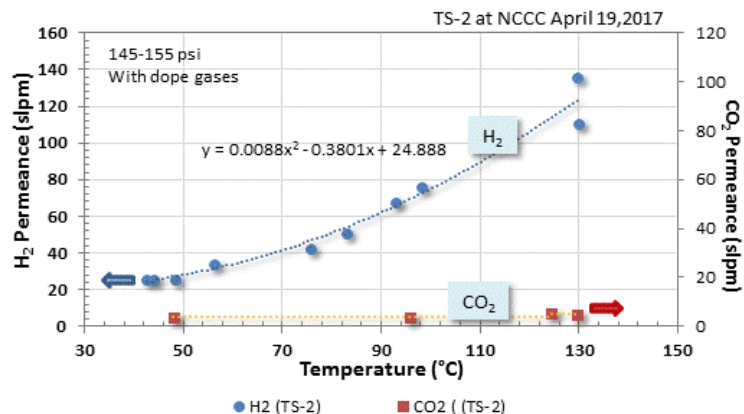
Observed hydrogen recovery with varying stage cuts in the temperature range 150° – 190°C and pressure differentials of 130 to 150 psi for the syngas-only condition and for syngas with doped with H₂



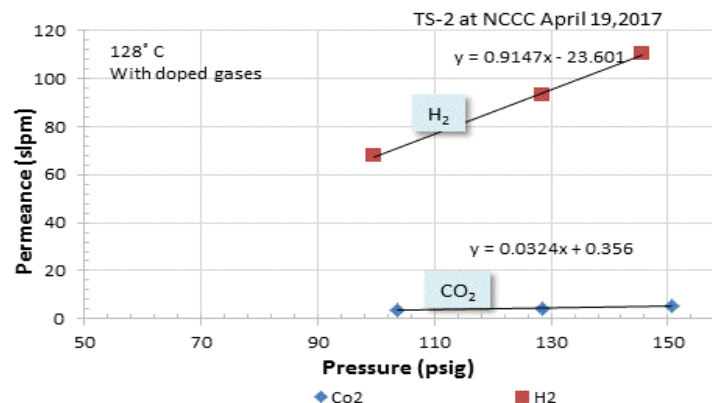
Observed CO₂ capture with varying feed CO₂ composition under fixed hydrogen composition at the temperature range 150 – 190°C and a pressure differential ~ 150 psi for the TS-1 membrane

600 hours of performance data collected from PBI skid

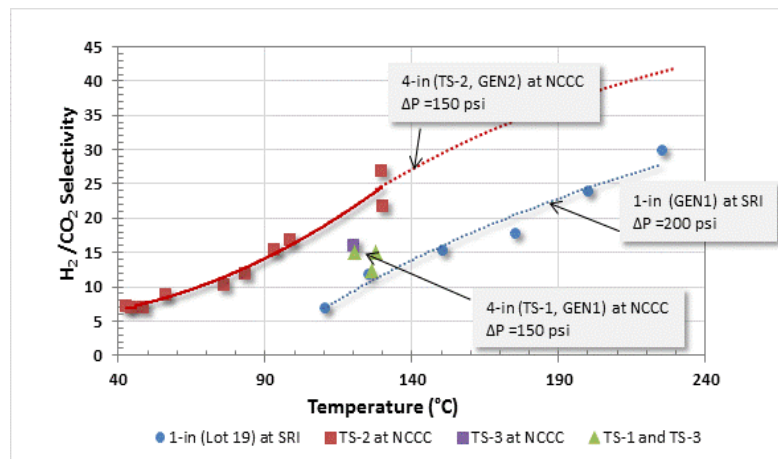
Gen-2 at NCCC: Temperature and Pressure Effect on Permeance



Measured H₂ and CO₂ permeances at the NCCC for the TS-2 (GEN-2) module at varying temperatures under a pressure differential of 145 to 155 psi.



Measured H₂ and CO₂ permeances at the NCCC for the TS-2 (GEN-2) module with varying pressures at 128°C



Comparison of measured H₂/CO₂ selectivity for GEN-1 and GEN-2 modules

Modules tested at NCCC:

- Membrane element TS-1 consisting of SRI GEN-1 fibers (GPU~150, H₂/CO₂ selectivity ~ 25 at 150°C) for ~ 500 hr
- Membrane element TS-2 consisting of SRI GEN-2 fibers (GPU ~ 100 , H₂/CO₂ selectivity ~ 40 at 200°C, 200 psi) for ~ 48 hr

Performance of GEN-2 at SRI: GPU ~ 80, H₂/CO₂ selectivity ~ 50 at 200°C, 200 psi)

PBI Gen-2 Offers Significant Advantages over PBI Gen-1



Current Project Details

Project Budget and Team for DE-FE0031633

- **Cooperative agreement grant with U.S. DOE Period of Performance:**
 - BP1: 10-1-2018 to 03-31-20
 - BP2: 04-01-20 to 09-30-21
- **Funding:**
 - U.S. Department of Energy: \$2.007 million
 - Cost share: \$0.505 million (20.1%)
 - Total: \$2.512 million
- **NETL Project Manager:**
 - Andrew Jones

NETL

- Funding and technology oversight

SRI

- Gen-2 PBI membrane Spinning
- Module fabrication
- Skid installation & testing

PBI Performance Products, Inc.

- PBI Dope and industry perspective

Enerfex, Inc.

- Membrane system modeling and Techno-economic analysis

Energy Commercialization

- Commercialization analysis

UKy CAER

- Gasifier facility test site

- Current Spending: \$222,527 (as of 27 July 2019)

Project Objectives

- Demonstrate that Gen-2 PBI based hollow fiber membranes provide a pathway to achieving DOE's pre-combustion capture targets
 - Bench-scale testing (50 kWth) of Gen-2 with actual syngas feed stream from an OXYGEN BLOWN gasifier
 - Evaluate Techno-economics based on bench-scale test results
- Leverage assets and knowledge generated from previous projects
 - PBI Hollow fiber membrane spinning line
 - Potting of fibers
 - Construction of modules based on PBI fibers
 - Test skid utilized for testing Gen-1 (50 kWe) at the NCCC on an AIR BLOWN gasifier
 - Production of Gen-2 fibers with improved selectivity over Gen-1

Project Tasks

Budget Period 1

Task #	BP	Task	Status
1	1	Project Management and Planning	On going
1	1	Preliminary Technology Maturation Plan Program Management Plan Preliminary TEA	Submitted
1	1	Installation of Partner Agreements and Sub-awards	Completed
2	1	Modification of the 50 kWth Test Unit - Refurbish and upgrading of the existing skid system - Fabrication of Gen-2 Fibers - Module design and installation of the Modules (4 to 6-in diameter) - Membrane performance testing at SRI - HAZOP and PI&D Review at CAER	Started
3	1	Modeling - Modeling of the Module arrangement - Modeling of the skid performance - Preliminary TEA	Started

Project Tasks

Budget Period 2

Task #	BP	Task	Status
4	2	Operation of the Test Unit at a Field - Skid Transport and Installation at the Site - Development of a test plan - Operation of the skid and data collection - Analysis of the data form the skid	Not Started
5	2	EH&S, TEA and other Related Reports - Techno-Economic Analysis - Update the State Point Data Table - Technology Gap Analysis - Preparation of Technology Maturation Plan - Environmental Health and Safety Assessment (EH&S)	Not Started
6	2	Skid Decommissioning - Skid decommissioning and Transport - Skid Postmortem and Storage	Not Started

Project Milestone Plan

Budget Period 1

Task/ Subtask	Milestone Description	Planned Completion	Status	Verification Method
1	Updated PMP	10/31/2018	Completed	PMP file
1	Kick-off Meeting	1/31/2019	Completed	Presentation file
1	Technology Maturation Plan	12/31/2018	Completed	TMP file
3.3	Completion and submission of preliminary TEA	12/31/2018	Completed	Topical report
2.1 to 2.3	Complete the production of at least 100 km of Gen-2 fibers that provide a H ₂ /CO ₂ selectivity of about 40 and H ₂ permeance of 80-120 GPU at > 150°C at a 150 psi pressure differential	11/30/2019	Started	Results reported in QR
2.1	Completion of the updated Basic Engineering Design package for modification of 50 kWth skid	12/30/2019	Started	Results reported in QR
2.1 to 2.4	Completion of bench skid acceptance testing for 50 hrs at SRI and achieve H ₂ /CO ₂ selectivity > 35	3/31/2020	Not started	Results reported in QR

We are on schedule to complete the project milestones

Project Tasks and Schedule

BP1: 18 months BP2:18 months			BP1								BP2				
			10/1/18	1/1/19	4/1/19	7/1/19	10/1/19	1/1/20	4/1/20	7/1/20	10/1/20	1/1/21	4/1/21	7/1/21	12/31/21
Task	Start Date	End Date	Q1	Q2	Q3	Q4	Q5	Q6	Q7	Q8	Q9	Q10	Q11	Q12	
Development and Testing of a High Temperature PBI Hollow Fiber Membrane Technology for Pre-Combustion CO2 Capture	10/1/2018	9/30/2021													
Task 1.0 (BP1 & BP2) - Project Management and Planning	10/1/2018	9/30/2021													
Subtask 1.1 - Project coordination and reporting	10/1/2018	9/30/2021													
Subtask 1.2 - Preparation and Submission of Continuation Report	3/1/2021	3/31/2020													
Milestones			a	b	c				f,h						
Task 2.0 (BP1): Modification of the 50 Kwth Test Unit for the Field Test	10/1/2018	3/31/2020													
Subtask 2.1 - Refurbish and upgrading of the existing skid system	10/1/2018	8/31/2019													
Subtask 2.2 - Fabrication of SRI Generation 2 (GEN 2) Fibers	1/1/2019	3/31/2020													
Subtask 2.3 - Module design and installation of the Modules (4 to 6-in diameter)	4/1/2019	3/31/2020													
Subtask 2.4 - Membrane performance testing at SRI	1/1/2019	3/31/2020													
Subtask 2.5 - HAZOP and PI&D Review at CAER	4/1/2020	3/31/2020													
Milestones									d,e						
Task 3.0 (BP1) -Modeling	10/1/2018	3/31/2020													
Subtask 3.1 - Modeling of the Module arrangement	1/1/2019	3/31/2020													
Subtask 3.2 - Modeling of the skid performance	4/1/2019	3/31/2020													
Subtask 3.3 - Preliminary TEA	10/1/2018	12/31/2018													
Milestones									g						
Task 4.0 (BP2) - Operation of the Test Unit at a Field Site	4/1/2020	9/30/2021													
Subtask 4.1 -Skid Transport and Installation at the Site	4/1/2020	3/31/2021													
Subtask 4.2 - Development of a test plan	10/1/2020	12/31/2020													
Subtask 4.3 - Operation of the skid and data collection	1/1/2021	3/31/2021													
Subtask 4.4 - Analysis of the data form the skid	1/1/2021	6/30/2021													
Milestones											i	j	k		
Task5.0 (BP2) - EH&S, TEA and other Related Reports	1/1/2021	9/30/2021													
Subtask 5.1 - Techno-Economic Analysis	1/1/2021	6/30/2021													
Subtask 5.2 - Update the State Point Data Table	7/1/2021	9/30/2021													
Subtask 5.3 - Technology Gap Analysis	7/1/2021	9/30/2021													
Subtask 5.4 - Preparation of Technology Maturation Plan	10/1/2018	12/31/2018													
Subtask 5.5 - Environmental Health and Safety Assessment (EH&S)	7/1/2021	9/30/2021													
Submit the State Point Data Table	9/1/2021	9/30/2021													
Submit the Technology Gap Analysis	9/1/2021	9/30/2021													
Submit the Technology Maturation Plan	9/1/2021	9/30/2021													
Milestones															
Task 6.0 (BP2) - Skid Decommissioning	9/1/2021	9/30/2021													
Subtask 6.1 - Skid decommissioning and Transport															
Subtask 6.2 - Skid Postmortem and Storage															
Milestones															q,r
Project Reviews															
Final Report Submission	10/1/2021	12/31/2021													

UKy-CAER Pilot Facility

Gasification Unit

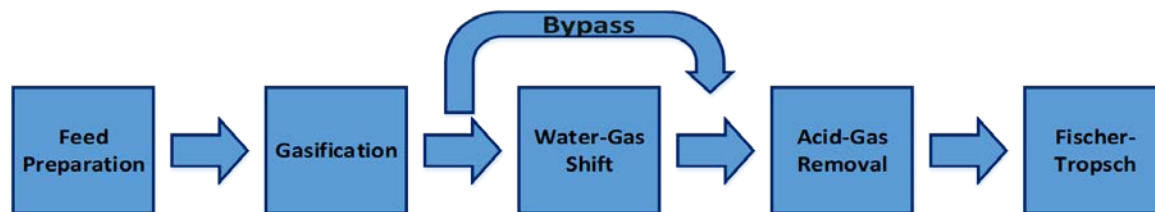
- Multi-burner, entrained flow, oxygen blown, slagging type
- 1 ton/day coal consumption
- Syngas production rate: $\sim 80 \text{ m}^3/\text{hr}$
- H_2/CO : $\sim .80$

Water-Gas Shift

- Packed bed
- Sulfur tolerant sour shift catalyst
- H_2/CO : up to 11/1

Syngas Compressor

- Metal Diaphragm Compressor
- 450 psi max outlet pressure



Gasifier



Gasifier Operating Parameters	
Temperature (°C)	1350
Pressure (MPag)	0.1
CWS Solid (%)	53.0
Syngas (vol%)	
H ₂	24.51
N ₂	2.93
CO	28.94
CO ₂	40.89
H ₂ O	2.54
H ₂ S	0.18
COS	0.02

WGS



Compressor





Current project work update

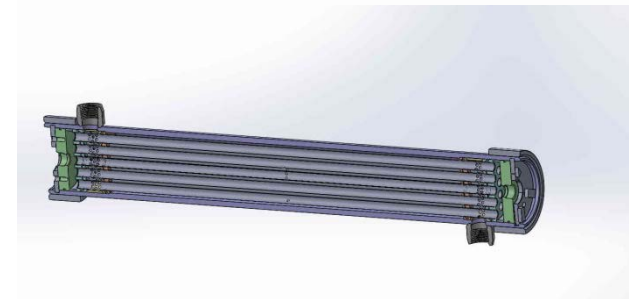
Improved Module Design

Reliable fiber module performance enables maximum collection of high-quality data at UKy-CAER

Previous tube sheet
module design



New tube sheet
module design



Fast module swapping and reduced gas bypass

In Progress: Production of Gen-2 Fibers

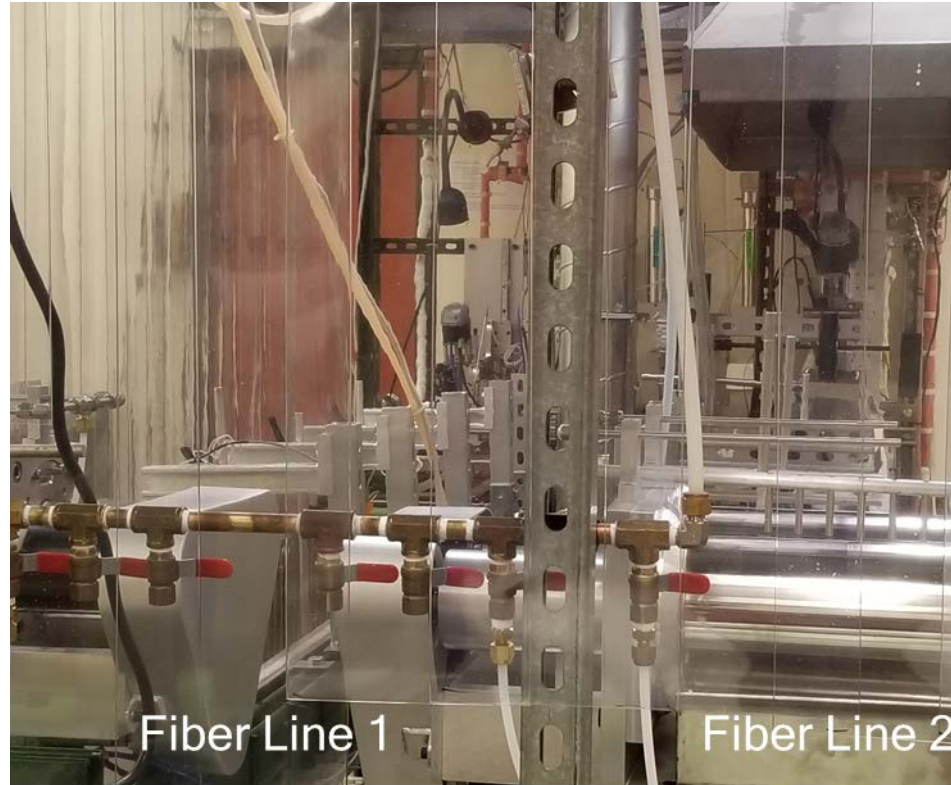
Reproducible spinning process to provide abundant hollow fibers for installation in skid



>10 km Gen-2 fibers produced to date

A Second Spinning Line Installed at SRI in 2019

Transfer of spinning hollow fibers critical to Technology Maturation



Photographs of the new fiber spinning line. Line 1 is the old line, and Line 2 is the newly installed line.

Fiber Spinning Capacity Doubled



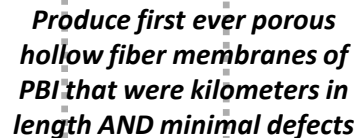
Technology Maturation

DOE enabled “first-of-a kind” hollow fiber membranes of PBI in kilometer lengths

DE-FC26-07NE43090

***Producing Gen-1 PBI
based membrane
modules***

Testing POTTED Gen-1 fiber bundles



Dope preparation

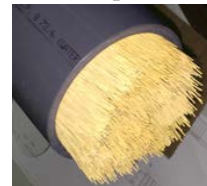
High temperature potting

DE-FE0012965

***Producing and testing
Gen-1 based membrane
modules at NCCC***

Developing Gen-2 PBI based fibers

Surface area $\sim 5 \text{ m}^2$



***Consistent multi-fiber
bundles that are
kilometers in length of
Gen-1 and Gen-2 with
minimal defects***

DE-FE0031633

**Producing and testing
Gen-2 based membrane
modules at UKy-CAER**

**Higher selectivity and
new module design**

Surface area $\sim 20 \text{ m}^2$



Doubled spin capacity

Production of 100 km of Gen-2 with minimal defects

2018 SRI International

26

2006 2008 2010 2012 2014 2016 2018 2020 2022

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- Andrew Jones, Jose Figueroa, Elaine Everitt, Lynn Brickett, and others at NETL
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- Richard Callahan (Enerfex, Inc.)
- Kevin O'Brien (Energy Commercialization, LLC)
- Greg Copeland and Mike Gruende (PBI Performance Products)
- Kunlei Liu and his team (UKy- CAER)
- John Jensvold and his team (Generon IGS)
- The staff at the NCCC

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Thank You