

Platinum Recycling Technology Development

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"This presentation does not contain any proprietary or confidential information."

Project Overview/Objectives

- To assist the DOE in demonstrating a cost effective and environmentally friendly recovery and re-use technology for PGM containing materials used in fuel cell systems.
- The initial objectives:
 - Development of lab scale processes for the solubilizing catalyst coated membranes
 - Development of Lab scale processes for the separation of catalyst and ionomer materials
 - Develop test methods to determine vitality of the recovered materials
 - Partner with the key stakeholders in this technology area

Budget

- Total Project Costs: \$3.31 Million
- DOE Share: \$2.65 Million
- Cost Share: \$0.66 M

Technical Barriers and Targets

- DOE Technical Barriers for Fuel Cell Components
 - O. Stack Material and Manufacturing Cost:
 - PGM costs could become unstable if fuel cell systems are introduced without the a recovery industry's ability to meet demands
 - Material costs of Ionomer and PGMs can be driven down during near-term commercialization, could be up to 10x reduction for ionomer
 - P. Durability
 - New separation processes developed here will help identify failure modes of PEM fuel cell materials
- DOE Technical Target for Fuel Cell Stack System for 2010
 - Cost \$35/kW
 - Durability 5000 hours

Approach

- Development of a technology that can re-manufacture spent fuel cell components into fresh fuel cell components.
 - Use pilot scale equipment that can operate on 1 sq meter (5 kW) of CCMs at one time
 - Separate ionomer and catalyst to levels needed for re-manufacture
 - Use analytical techniques to determine the differences between used and virgin materials
 - Will learn failure modes of CCM component
 - Determine the limits of separation technologies

Project Safety

- Pressure relief and over temperature alarms on all pressure vessel equipment
- Implementation of OSHA approved Chemical Hygiene Program
- Regular OSHA inspections of facilities
- H₂ Sensor with e-stop on all H₂ supplied equipment

Project Timeline

ID	Task Name	Start	End	Duration	2003		2004				2005				2006				2007				2008	
					Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2	Q3	Q4	Q1	Q2
1	Task 1 MEA Dissolution	8/7/2003	3/21/2008	241.40w																				
2	SubTask 1.1 Procure and install Autoclave	8/7/2003	12/5/2003	17.40w																				
3	Sub Task 1.2 Develop Decontamination Procedures	10/1/2003	1/1/2008	222w																				
4	Sub Task 1.2.1 Develop Solution Filtration Processes	10/1/2003	7/8/2005	92.60w																				
5	Milestone Task 1 Identify all Significant Contaminates on EOL MEAs	1/1/2008	1/1/2008	0w																				
6	Sub Task 1.2.2 Study to Identify Contaminates in Used MEAs	3/1/2004	3/17/2008	211.20w																				
7	Sub Task 1.3 Develop Dissolution Procedures	12/8/2003	3/21/2008	224w																				
8	Milestone Task 1 Demonstrate Dissolution process on MEA	2/2/2004	2/2/2004	0w																				
9	Task 2 Separation of NAFION(r) from Catalyst	8/7/2003	6/1/2007	199.40w																				
10	Sub Task 2.1 Selection of Separation Method	8/7/2003	6/1/2007	199.40w																				
11	Milestone Task 2 Identification of a Catalyst/Ionomer separation process < 1% ionomer remains with catalyst	8/1/2005	8/1/2005	0w																				
12	Sub Task 2.1.1 Measurement of Separation Selectivity	12/12/2003	5/1/2006	124.40w																				
13	Sub Task 2.1.2 Perform separation of dissolved 1st batch of MEA	2/2/2004	3/3/2004	4.60w																				
14	Sub Task 2.3 Identification of recovered NAFION(r) properties comparison to virgin NAFION(r)	1/1/2004	12/30/2005	104.40w																				
15	Milestone Task 2: Aged, Recovered, Purified Ionomer shows vitality within 10% of virgin ionomer	1/2/2006	1/2/2006	0w																				
16	Task 3 Evaluation of Fresh and End of Life Catalysts	1/1/2004	12/30/2005	104.40w																				
17	SubTask 3.1 Development of Catalyst Characterization Tools	1/1/2004	12/30/2005	104.40w																				
18	SubTask 3.2 Evaluation and Comparison of fresh and end of life catalysts	1/1/2004	12/30/2005	104.40w																				
19	Milestone Task 3 Recovered Catalyst shows vitality within 10% of Virgin Catalyst	1/1/2007	1/1/2007	0w																				
20	Task 4 Re-Manufacture and Test of MEAs	8/7/2003	9/5/2008	265.40w																				
21	Task 4.1 Prepare Site Installation for aging stacks/systems	9/1/2003	3/1/2004	26.20w																				
22	Taks 4.2 Manufacture 1 sq m of MEAs initial for dissolution/separation experiments	8/7/2003	10/2/2003	8.20w																				
23	Task 4.3 Identify stack/system partners	8/7/2003	12/31/2003	21w																				
24	Task 4.4 Remanufacture 1st stack worth of MEAs from separated materials recovered from fresh MEAs	3/4/2004	4/2/2004	4.40w																				
25	Sub Task 4.5 Ongoing real life aging of Fresh and re-manufactured MEAs	4/5/2004	9/5/2008	231w																				
26	Milestone Task 4 Demonstrate Worlds First Re-manufactured MEA	1/3/2005	1/3/2005	0w																				
27	Task 5 Vapor Phase Extraction of PGM	1/1/2004	12/30/2005	104.40w																				
28	Milestone Task 5: Demonstration of Reverse MOCVD Catalyst Stripping Process	1/1/2008	1/1/2008	0w																				
29	Task 6 Process Evaluation	1/3/2005	8/1/2008	187w																				
30	Milestone Task 6 Industrial Scale Cost Estimate of Re-manufacture process	1/1/2008	1/1/2008	0w																				
31	Task 7 Project Management	8/7/2003	9/4/2008	265.20w																				
32	Sub Task 7.1 Develop Subcontracts	8/7/2003	2/2/2004	25.60w																				
33	Sub Task 7.2 Prepare Management Plan	8/7/2003	12/11/2003	18.20w																				
34	Sub Task 7.3 Reporting, Quarterly	8/7/2003	9/4/2008	265.20w																				

Milestone Schedule

Task 1: Demonstration MEA dissolution process

Identification of all significant contaminants in EOL MEAs

Task 2: Identification of a catalyst/NAFION® separation process

Identification of EOL NAFION® vitality

Task 3: Identification of EOL Catalyst vitality

Task 4: Demonstration of world's first Re-Manufactured MEA

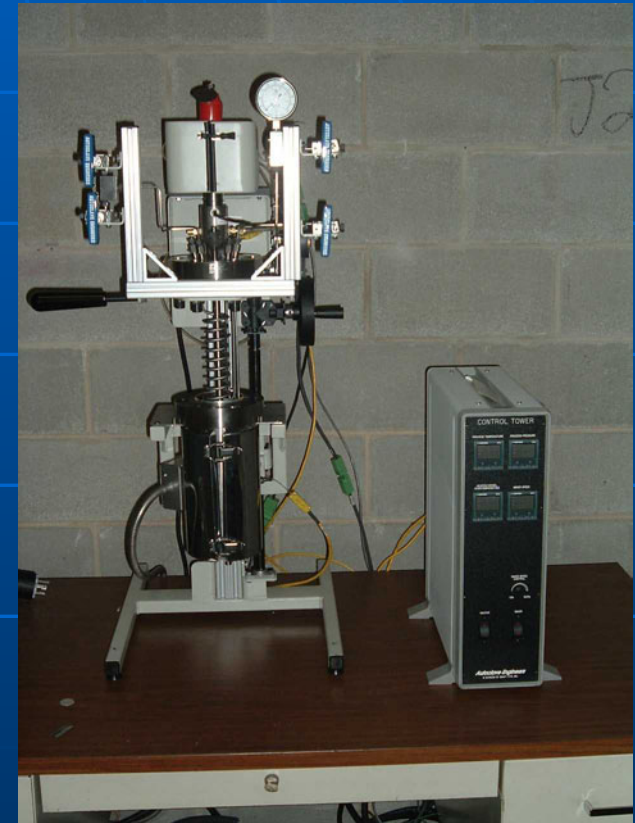
Task 5: Demonstration of Rev. MOCVD catalyst stripping

Task 6: Industrial Scale Cost Estimation of Process

Task 7: Reporting Requirements

TASK 1 MEA Dissolution

- Operated on Fresh CCM, Both Ion Power and DUPONT made
- Solids Decontamination issues, remaining solids such as Kapton frame material easily separated out after run
- Many more decontamination issues will arise once we use actual aged MEAs
- Wait to use Aged MEAs until analytic techniques perfected (Task 3)



**1 Liter Autoclave can process
1 sq meter of MEA at a time**

TASK 2 Separation

- Centrifuge with double Rinse step still leaves some NAFION® in the catalyst powder
 - Target for Re-manufacture is ~ 50wt% NAFION®
 - Target for Recycle is ppm
- Via colormetric methods we estimate Pt/C is removed to 150 ppm from the NAFION®
 - Target for Remanufacture may be adequate; this sample to be film cast and tested

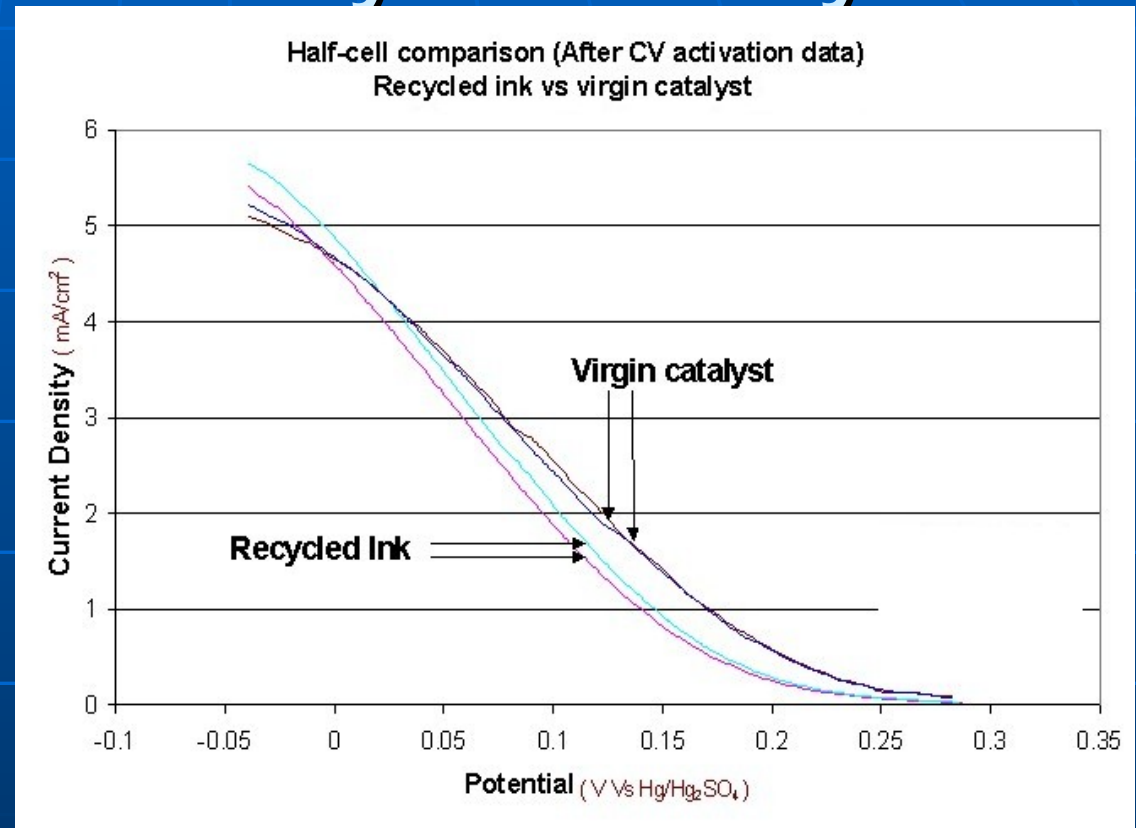


**Sorvall SUPERSPEED 20krpm
Centrifuge, 8 x 30 ml**

TASK 3 Catalyst Vitality

1/2 cell catalyst activity test on virgin versus recovered catalyst.

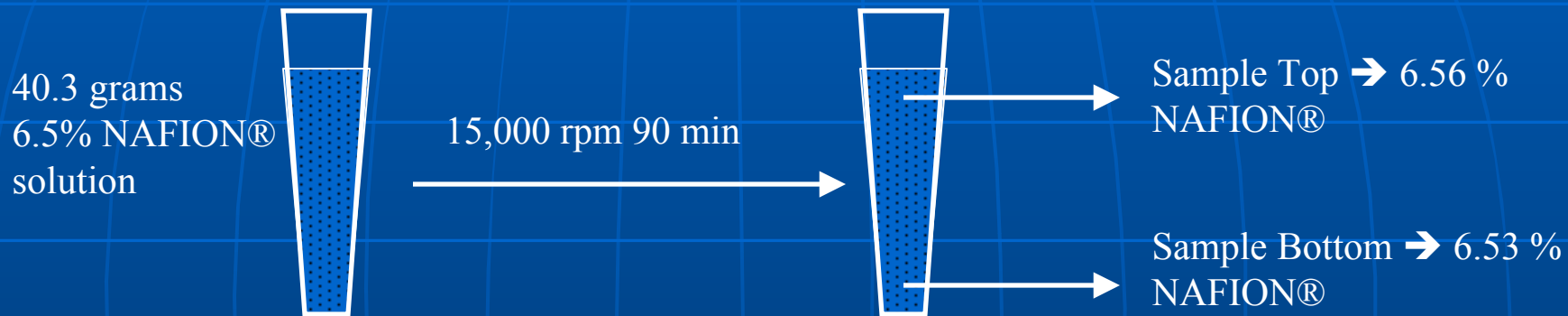
This 1/2 cell test may have some sample preparation effects that limit the activity of the recycle catalyst; still under investigation



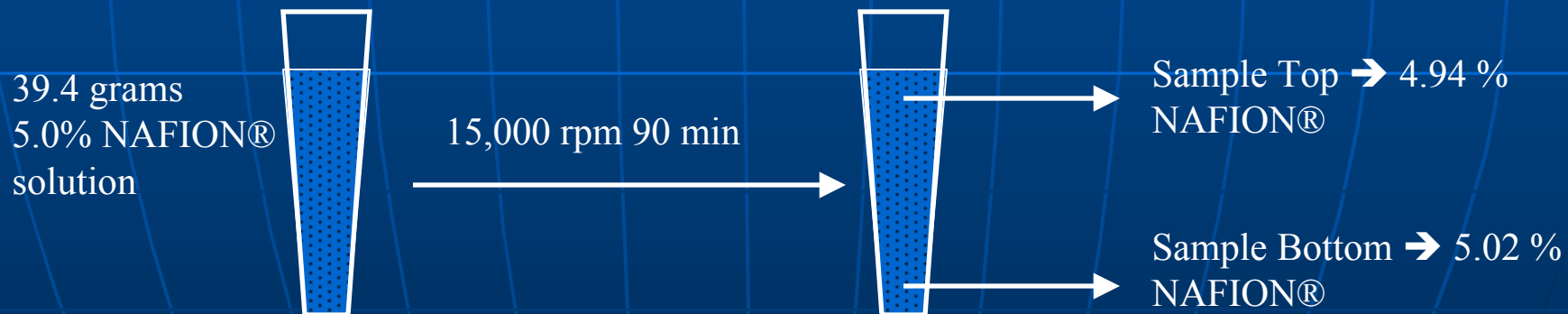
➤ Do NAFION™ particles settle upon Centrifugation?

This would limit effectiveness of Centrifugation as a separation process since NAFION® would settle with Pt

H-Type NAFION® solution, Centrifugation Experiment



L-Type NAFION® solution, Centrifugation Experiment



➤ Conclusion → Ion Power NAFION® solutions do not separate upon Centrifugation

TASK 4 Remanufacture/Test

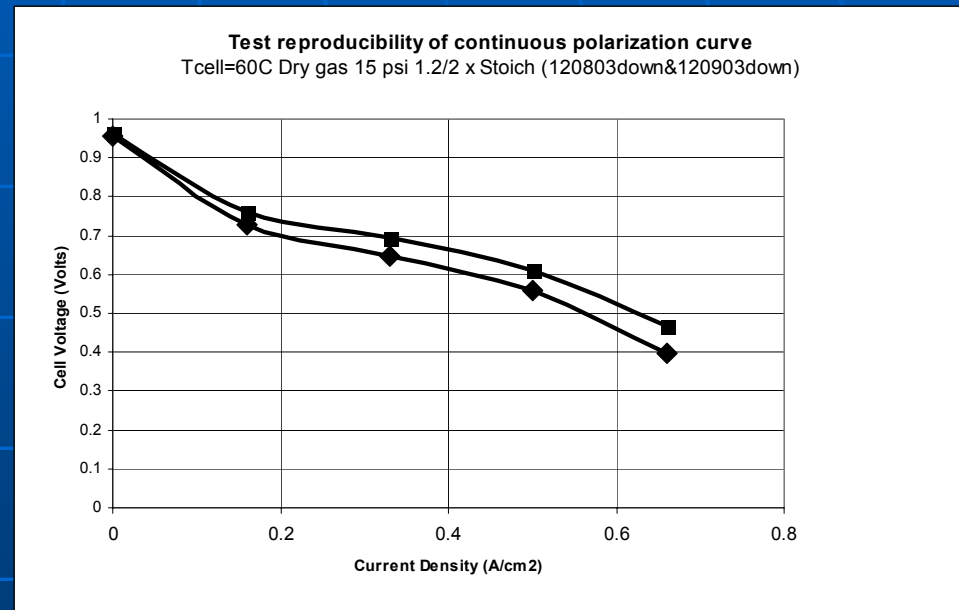
-5 kW Commercial fuel cell Systems Identified to be Purchased and run to failure at local Hydrogen Distribution facility with Program MEAs

-Stainless Stack

-Graphite Stack

-5kW is right size for Process Equipment identified in TASK 2

-Small scale tests identify baseline



Simple test set-up for Fast Aging under Dry Gas Operation, BOL Reproducibility single cell

Interactions & Collaborations

- DuPont:
 - Dennis Kounz; Supply of MEA's new & used materials evaluations

- Delaware State University
 - Prof. Goudy; Materials Characterization Catalyst & NAFION[®] vitality

Future Work

- Remainder of FY 2004:
 - Demonstration of vitality of separated materials
 - establish baseline characterization tools to determine vitality of separated materials
 - Demonstrate the degree of separation that can be achieved
 - Develop ability to controllably aged CCMs
 - Identify partners that can provide equipment to controllably age multi kW quantities of CCMs at once.
- FY 2005-2008:
 - Remanufacture and Test
 - Demonstrate a full stack running on remanufactured CCMs
 - Fuel Processor catalysts
 - Look at state of the art challenges, and identify resolutions to the challenges
 - Economic analysis
 - Based on best known pilot scale method estimate scale required to become competitive and profitable over existing methods of PGM recycling.