2005 DOE Hydrogen Program Review
Platinum Recycling Technology Development

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This presentation does not contain any proprietary or confidential information

Project ID #
FC20

Overview

Timeline

- Project start Aug 2003
- Project end Aug 2008
- Percent complete: 30%

Budget

- Total project $3.31M
  - DOE share: $2.65 M
  - Contractor: $0.66 M
- FY04: $0.53 M
- FY05: $0.70 M

Barriers

- Barriers addressed
  - O: Stack Materials and Manufacturing Costs
  - P: Durability
    - (vitality measurements of materials recovered from end-of-life components will identify failure modes)

Partners

- DuPont, Delaware State University, NIST, Ballard, BCS Technology, Plug Power, Drexel University

Objectives

- To assist the DOE to demonstrate a cost effective and environmentally friendly recovery and re-use technology for PGM containing materials used in fuel cell systems.
- Use new processes that can also separate and recover valuable ionomer materials
  - DOE 2010 *targets* for membrane costs indicate membrane has value comparable to the PGM

Approach

- Use pilot scale equipment that can operate on 1 sq meter (5 kW) of catalyst coated membranes at one time (200grams)
- 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

Technical Accomplishments/ Progress/Results

- Examined the “design for recyclability” for several real-world fuel cell stack systems.
- Demonstrated the commercial end-of-life NAFION® membrane retains much of its original properties, Further investigation ongoing to fully quantify this.
- Scaled-up the separation equipment, provides materials, and information for cost analysis
- Demonstrated world’s first re-manufactured fuel cell membrane operating in a fuel cell
- Developed some cost metrics for recycling and developed framework for a cost model.

End of Life NAFION®

PURCHASED 18 POUNDS OF END-OF-LIFE MEAS FROM FUEL CELL STACK MANUFACTURER

94.3% of starting materials recovered as separate ionomer from catalyst

The NAFION® was analyzed for acid capacity, conductivity, Stability, & Infrared spectroscopy.

Acid Capacity: 1233 EW (grams/mole) as received, 1171 EW after acid exchange.

Conductivity: 10.3 Ohm-cm as-received 9.9 Ohm-cm after acid exchange

Fenton Test shows similar stability as compared to virgin NAFION® (0.011 meq F/gram)

Infra-red: No Carboxylic acid groups found

Conclusion : Polymer after recovery is close to the reported specifications for acid capacity and conductivity, however likely has contaminates due to color staining and improvements in conductivity achieved by subsequent acid washing. Stability of equal to that of new NAFION® membranes.

Disassembly

Labor: 10 seconds/part

Catalyst

Machine separation work

Ionomer

Machine clean-up work

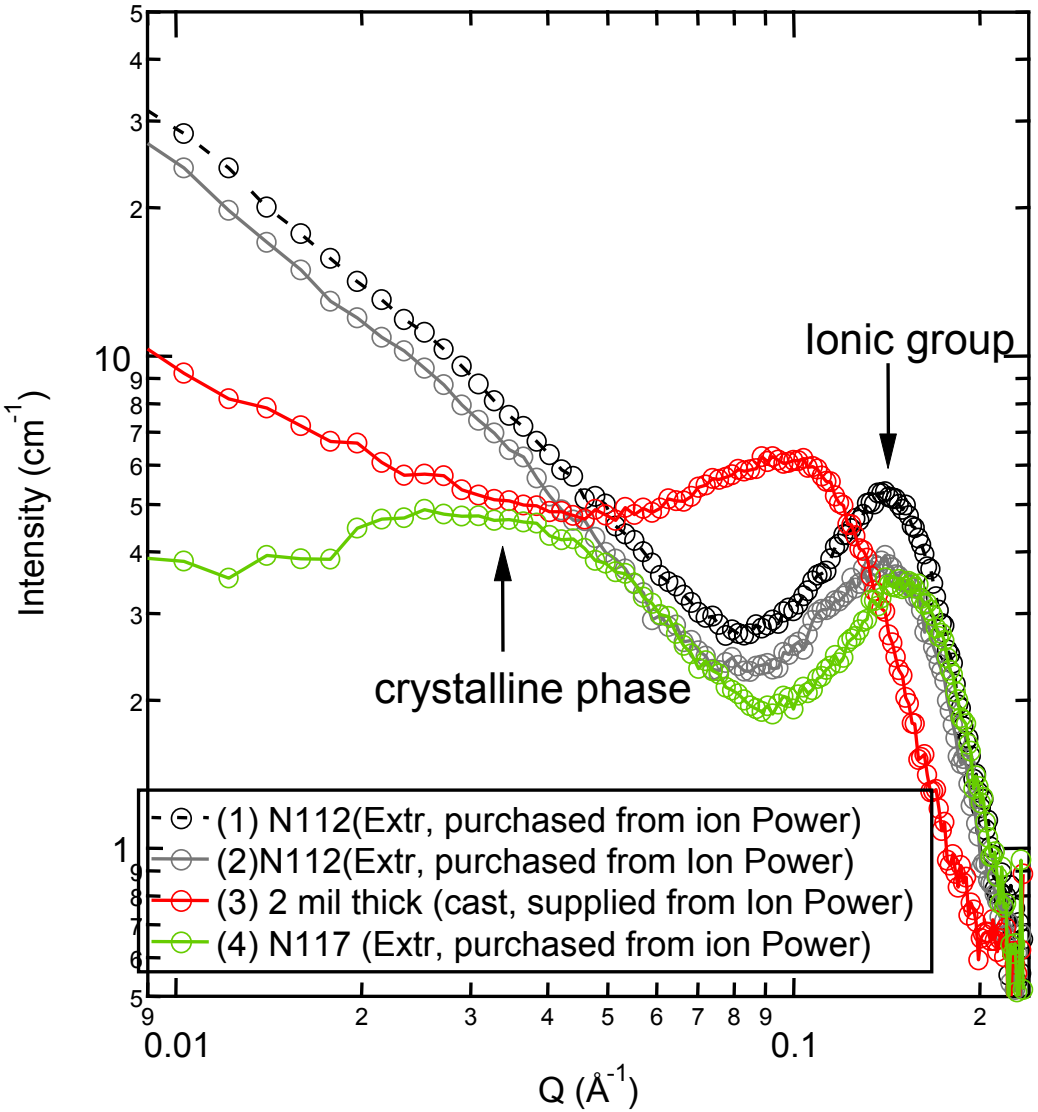
End-of-Life NAFION®

Used NAFION® N112 membrane with catalyst removed, contamination visible at reactant gas inlets on the left and right.

Making a Re-Manufacture Membrane

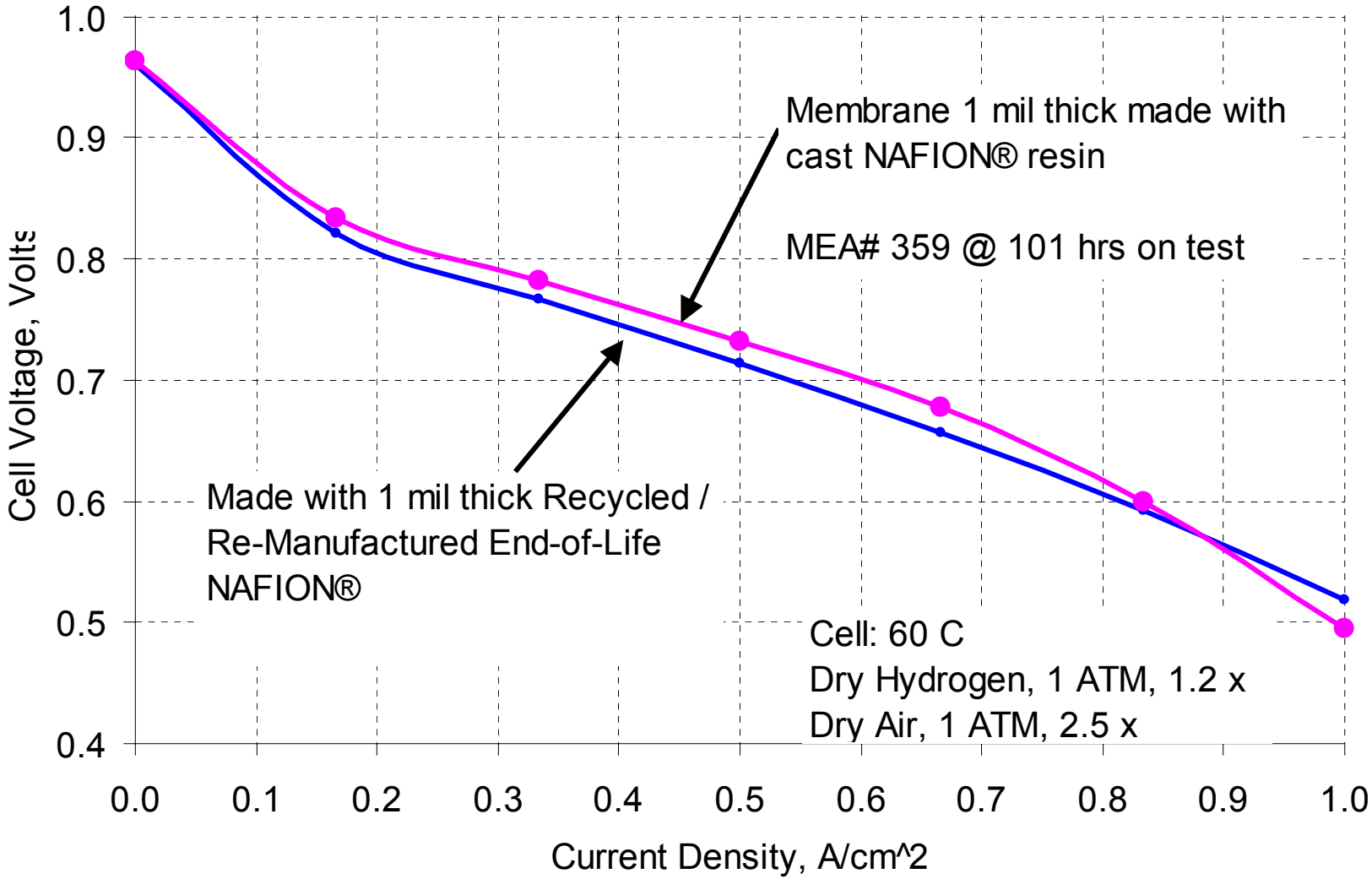
- These Membranes were cleaned and protonated in a new cleaning procedure. Then solubilized into a low viscosity solution where it can be easily filtered, the viscosity is then modified for good film casting.

Nano-phase Structure of Cast films compared to extruded films



Neutron Scattering Tests performed by Man-Ho Kim of NIST, (new Collaboration)

Recycled NAFION® from End-of-life Fuel Cell System has Performance Close to Membrane Containing Virgin NAFION®



Design for Recyclability

-500 Watt 48 cell, organized in 12 4-cell hot-swap packs 62 cm2 active each.

-Hand dissassembled each pack (Very labor intensive, due to numerous screws used for compression)

5.123 grams of catalyst coated membrane were separated 10 MEAs

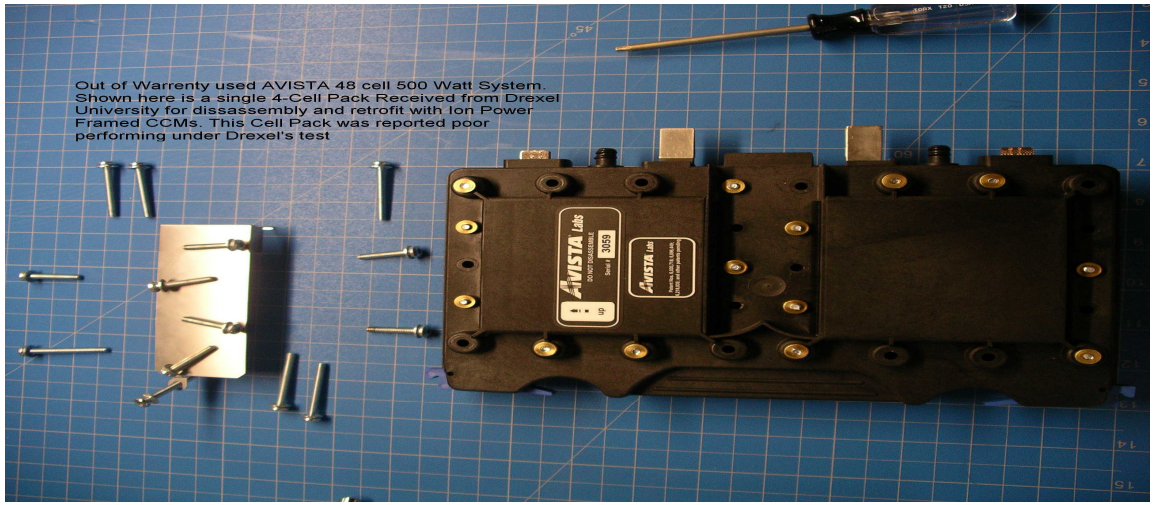
2.46 grams ionomer recovered ➔EW confirmed via ion-exchange at 1033 g/mole

0.866 grams of PTFE support filtered out

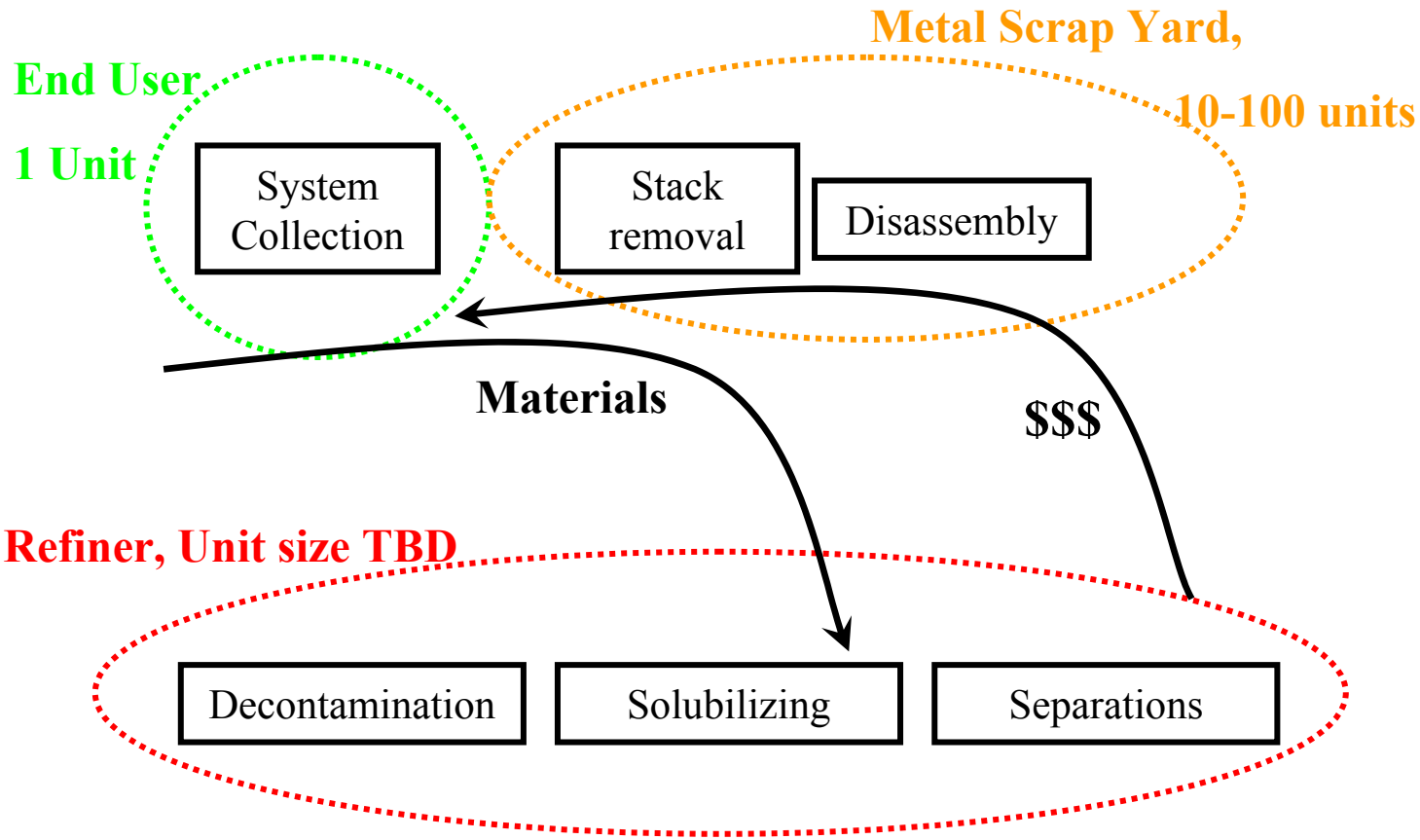
1.23 grams of Pt/C catalyst separated out

0.519 grams of Pt recovered after combustion ➔ Wet Gravimetric assay shows 97.01% or 0.503 gr Pt which agrees with the Manufacture estimation of 0.8 mg Pt/cm2 total loading on 10 MEAs

48 New MEAs were manufactured and supplied to Drexel University. Re-built Stack Performance reached similar performance to the as-received system.



MEA Recycling Cost Model
Multi kW and above stationary and automotive Systems



COSTS

Labor &

Transportation

Capital

Costs

Processing

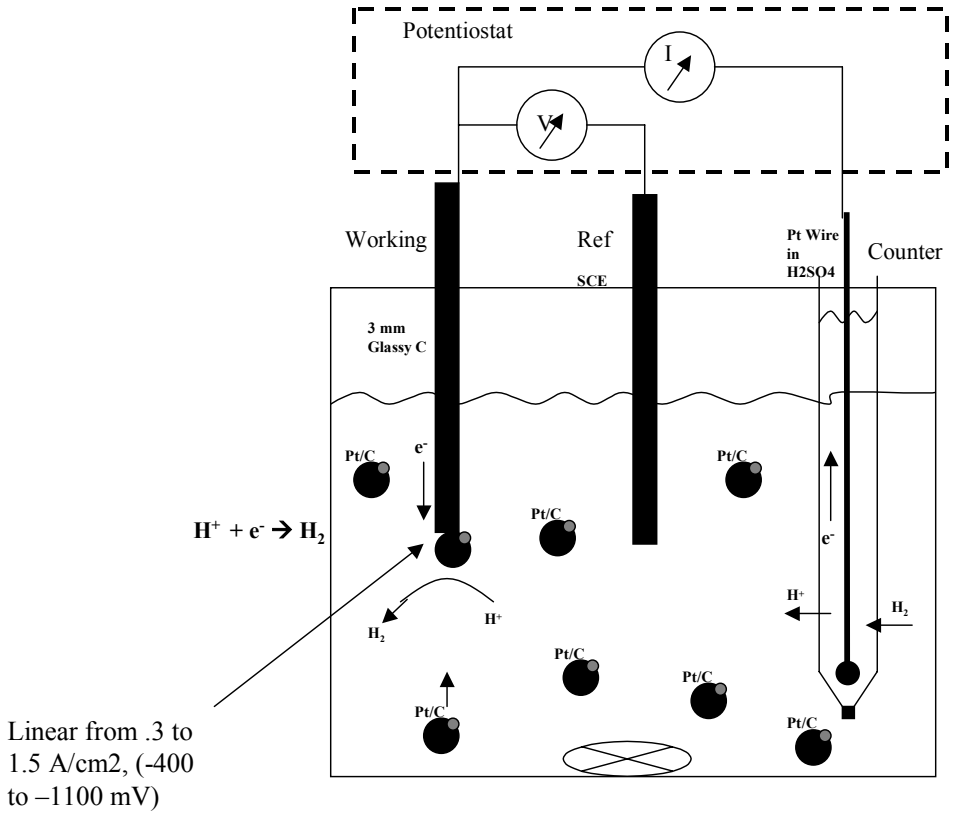
Equipment

Economics rely on an increasing minimum batch size at each step

Catalyst Vitality

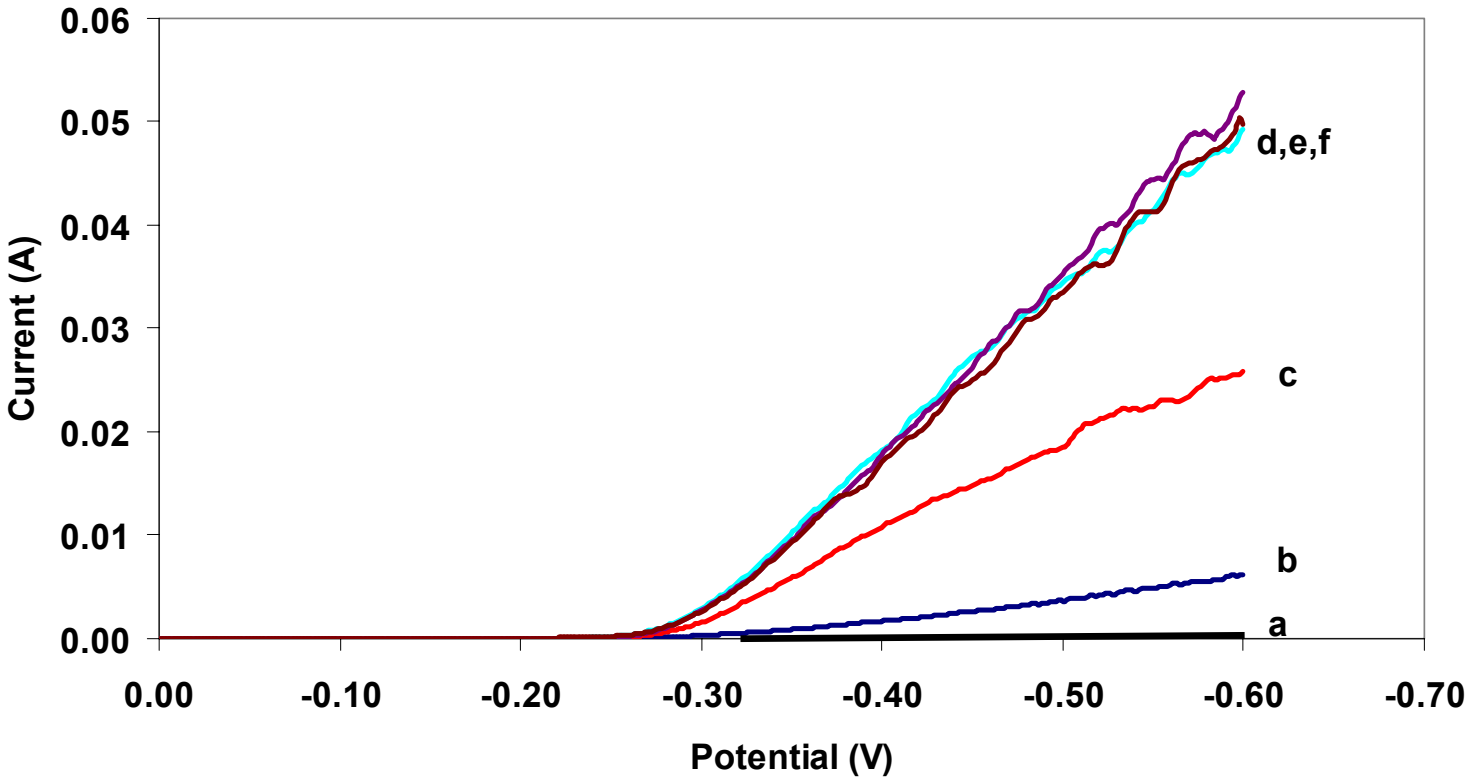
- Existing catalyst vitality tests are effected by the presence of small amounts of PFSA, e.g. CO Chemisorption
- Developed a new test that can be useful in the presence of small amounts of PFSA

Catalyst Vitality



Catalyst Vitality

Linear Sweep Voltammograms of Pt/C (A') Suspensions with Various Concentrations of Pt/C; a = 0.0 g/L, b=0.25 g/L, c=0.5 g/L, d=1.0 g/L, e=5.0 g/L, and f=10 g/L



Responses to Previous Year Reviewers' Comments

- Recycle of ionomer needs better justification
  - Extensive study of ionomer extracted from real-world end-of-life systems.
  - Achieved actual fuel cell operation using re-manufactured end-of-life ionomer
- Needs Better Collaborations
  - Established collaboration with NIST for structure analysis of re-manufactured/end-of-life membranes via Neutron Scattering
  - Ballard will analyze our re-manufactured membranes recovered from two end-of-life NEXA® fuel cell units
  - Contacts made to key stake-holders; Plug Power

Future Work

- Remainder of FY 2005:
  - Demonstration of vitality of separated materials
    - Search for additional sources of End-of-Life MEAs
    - Re-manufacture additional MEAs, test for lifetime
    - Install fuel cell test system 5 kW size to age and re-build
    - Additional Hours on Ballard stack until significant performance lost
- FY 2006-2008:
  - Remanufacture and Test
    - Demonstrate a full stack running on remanufactured CCMs
  - Economic analysis
    - Based on best known pilot scale method estimate scale required to become competitive and profitable over existing methods of PGM recycling.

Publications and Presentations

- Patent filing for Recycling process, March 2005
- B. Workie, et al., *"Simple Electrochemical Procedure to Test the Catalytic Activity of Platinum Supported on Carbon"*, Historically Black College &University-Undergraduate Program, 2005 National Research Conference, Park Plaza Hotel Downtown New Orleans, February 10 – 13, 2005

- Grot, et al. "*Platinum Recycling Technology Development*"; 2004 Fuel Cell Seminar, San Antonio Texas, Nov 2004

Hydrogen Safety

The most significant hydrogen hazard associated with this project is:

- Fuel Cell Testing with Hydrogen. Either single cells or with commercial fuel cell systems.

Hydrogen Safety

Our approach to deal with this hazard is:

All lab single cell fuel cell testing will be equipped with hydrogen sensors, that will shut building hydrogen supply off in the event of hydrogen detection or in the event of detected low cell voltage

Commercial systems will be installed per local building codes.