DOE Hydrogen Program

2007 DOE Hydrogen Program Review
Platinum Recycling Technology Development

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Project ID #
FC29

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Overview

DOE Hydrogen Program

Timeline

- Project start Aug 2003
- Project end Aug 2008
- Percent complete: 70%

Budget

- Total project $3.31M
  - DOE share: $2.65 M
  - Contractor: $0.66 M
- FY06: $0.63 M
- FY07: $0.56 M

Barriers

- Barriers addressed
  - B: Stack Materials and Manufacturing Costs
  - A: 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 equal to the PGM

Approach

- Use solvents to “dissolve” ionomer and physically separate catalyst from ionomer solution in 1-5 sq meter batch sizes.
- Make best attempt to re-manufacture catalyst coated membrane with recovered materials; although may not be commercially acceptable
- Will learn failure modes of MEA materials used in fuel cells; ionomer and catalyst
- Use analytical techniques to determine the differences between used and virgin materials
- Determine the limits of separation technologies
- Economic analysis at pilot scale equipment will be used to determine feasibility of approach.
- Value of recovered NAFION® will likely be found in different application other than fuel cells; e.g. acid catalysis for organic synthesis.

Technical Accomplishments/ Progress/Results

- Scaled up a new proprietary method that can effectively remove foreign cations from the recovered ionomer.
- Scaled up process and offered a 10 gallon 10% solution to DuPont for re-casting into re-manufactured membrane
- Designed and purchased 4000 psi 50 gallon scaled up reactor, install scheduled May 07.
- Purchased and installed and demonstrated scale up equipment:
  - Continuous centrifuge
  - 40 gallon mixing/separation tank
  - “Tower of Purity” for ionomer enrichment/solvent recovery process
  - Used multiple small reactor batches to gather enough material

Technical Accomplishments Previously discussed

- End-of-life NAFION® once cleaned up has properties similar to fresh NAFION®

- Catalyst vitality tests in the presence of small amounts of NAFION® is working at Del State University Subcontractor

- Separation Techniques at small scale are working

Scale-up of Autoclave

DOE Hydrogen Program

Process Energy Analysis
Worst Case Scenario

Reactor size: 300 Liters
- 1550 kg metal mass
- Charge with 145 kg solvent, 30 kg MEAs (with GDL, Typical Lot)
- Delta T: 250 C
- 60 kW-Hr required to heat Metal and contents
- Centrifuge:1 kW for 1 Hr (Estimated)

Output: $54,500 / day ➔ Will reduce by 10x if DOE 2015 goals are met➔ $5,400
- Pt: Typical of Today's MEAs at 0.8 mg Pt/cm2 total
  - 2-3% of MEA weight (0.75 kg) = $27,500 ($1100/try oz)
- NAFION® : 30% of MEA weight (9 kg) = $27,000 ($300/sq meter)
- GDL : 65% of MEA Weight (20 kg) = No value

Costs : Total: $656/day
- Energy: $0.10/kW-hr : $6.10
- Man-Power: Estimate 8 Man-hours : $300
- Solvents And Acids can be re-used; Estimate : $100 cost
- Capital Equipment Costs : ~ $500k; or $250/day (if used each day)

Slide from 2006 Review

We purchased this reactor. Currently being modified at a local ASTM code Shop. Will be inspected by State of Del. Div. Of Boiler Safety. Scheduled start-up May 07

Scale up of NAFION®/Catalyst Separation Process

Operation:

Load Reactor discharge into Mixing Tank

Keep circulation liquid until it exits tank clear

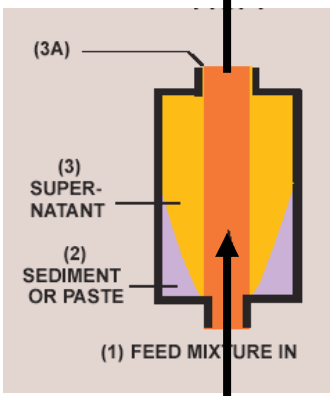
➔ Catalyst in centrifuge

➔ Clear NAFION® solution drained for recovery

40 Gallon Mixing Tank

Coarse Screen

2 Liter Continuous Centrifuge



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Scale up Example

100 MEAs representing 3 sq meters at 1.0 mg PGM/cm^2

Gas Diffusion media removed separately and analyzed for PGM

Processing Step	Total material	Total PGM Collected	Expressed as PGM Loading on MEA	%
Acid & DI water wash :	1.5 Liters Acid	1.67 grams PGM,	0.11 mg PGM/cm^2	5.5%
Centrifuge top Discharge:	2.04 kg Liquid "tinted"	0.6 grams PGM,	0.02 mg PGM/cm^2	2%
Centrifuge bowl solids :	108 grams powder	27 grams PGM,	0.90 mg PGM/cm^2	88.2%
Centrifuge Bowl Liquids :	1.045 kg black liquid	0.75 grams PGM	0.025 mg PGM/cm^2	2.3%
Diffusion Media:	0.64 kg Carbon Paper	0.6 grams PGM	0.02 mg PGM/cm^2	2%
TOTAL:		30.6 grams PGM	1.075 mg PGM/cm^2	100%

Issues at Scale-up

- Does the separation equipment work?
- NAFION® solution is too dilute to be useful
- Screen gets plugged up with GDM fibers
- NAFION® solution is contaminated with Pt/C

Centrifuge Issues

- The centrifuge recovers 98% of the PGM fed to it
- The Catalyst can be scrapped as a solid cake from the centrifuge bowl
- The bowl capacity holds 5 sq meters worth of PGM, larger and/or continuous solids discharge centrifuges are available
- Centrifuge process time was 9 mins which is short compared to the reactor time of 4 hours
- NAFION® solution is contaminated with 2100 ppm of PGM. Will reduce feed rate and concentration to further reduce this.

Mixer Tank Issues

- NAFION® solution is too dilute
  - We need large volumes of liquid in order to get good mixing. E.g. 610 grams of MEAs requires 10 liters of liquid ➔ 1% NAFION® solution
- We have developed a proprietary enrichment / solvent recycle system to solve this problem

Issues Converting recovered NAFION® to a film

- Good film casting is achieved only from alcohol based solutions; however alcohols in the presence of catalysts is problematic
- Water based recycling processing is preferred, makes low viscosity solutions
- Conversion from water based to good film with good mechanical still a challenge.
- Will work with team member DuPont to assist in conversion of recovered solution into a mechanically robust film.

Project Summary

- Recovery and separation work at scale-up is being demonstrated. Challenges are being discovered and overcome.

Future Work

- Remainder of FY 2007:
  - Demonstration of vitality of separated materials
    - Incorporate membrane manufactured by DuPont into MEAs and build into fuel cell stacks and operate for performance and lifetime.
- FY 2007-2008:
  - Operate process equipment on larger scale to get real numbers on process costs.
  - Develop a robust process that is not dependent on type of membranes recovered
  - Develop applications that can purchase recovered end-of-life polymer