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Platinum Group Metal Recycling Technology Development



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ENGELHARD



Overview

Timeline

- Project start date 11/2003
- Project end date of 10/2008
- Project 30% Complete

Budget

- Total project funding
 - DOE share of \$4.8MM
 - Engelhard share of \$1.2MM
- \$730,000 spent in FY04
- \$1,210,000 budgeted for FY05

Barriers

- N (Cost)
- O (Stack Material and Manufacturing Cost)
- \$45/kw for transportation
- \$400-\$700kw for stationary

Partners

- Virginia Polytechnic Institute
- University of Kansas
- Ceralink



Objectives of DOE Pt Recycling Project (Started 11/2003)

Recycle all PM-containing catalysts in a fuel cell 'system'

- **Develop a commercially-acceptable, environmentally-friendly process for recovering and recycling Pt and Ru from membrane electrode assemblies (MEAs)**
 - **Develop a process that does not emit pollutants, especially HF**
 - Evaluate Ru recovery from MEA's
 - Recycle perfluoropolymer membranes
- **Develop a process for PM recovery from metal monoliths**

*Principal objectives in **bold**.*



Approach

Assumptions driving the Conventional Combustion Approach to MEA Recycling

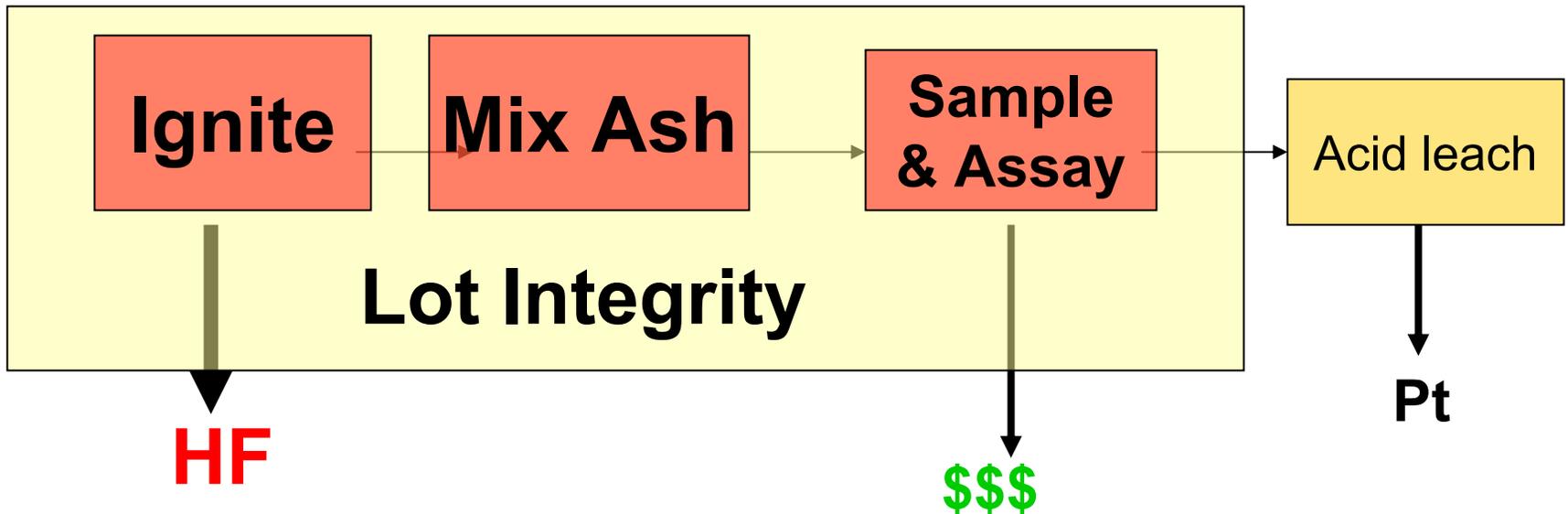
- High value of precious metal (PM) content. Electrode catalysts cannot be re-used; the PM must be purified during the recycling process.
- MEA membrane has minimum value
- Environmental issues need to be addressed

Assumptions driving the EC Approach to MEA Recycling

- HF emissions will be reduced to meet discharge limits
- High value of precious metal (PM) content. Electrode catalysts cannot be re-used; the PM must be refined during the recycling process.
- MEA membrane has value, but polymer cannot be re-used in a fuel cell

Current Combustion Process

- Ignite (GDL?)
- Homogenize
- Sample and assay
- Acid treat ash (>90% Pt)





Technical Approaches Under Investigation for MEA Recycling

- MEA Delamination (with Ceralink)
- Microwave-assisted digestion (with Ceralink)
- Microwave combustion (Virginia Tech)
- Supercritical Water dissolution (University of Kansas)

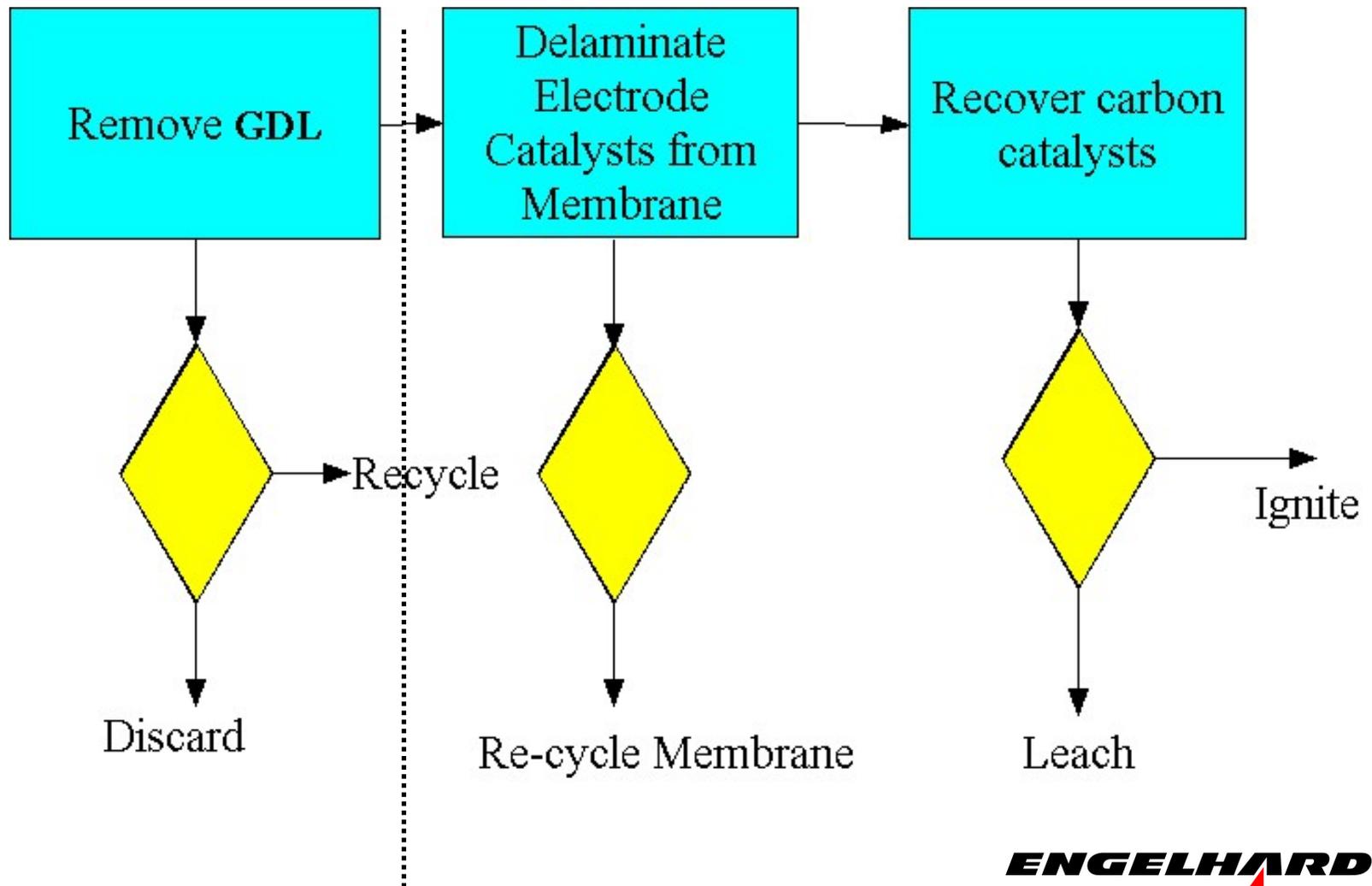
Technical results – MEA Delamination Process

- **Low-capital process**
- **Mechanism for recovery of membrane materials for recycling, not re-use**
- High Pt recovery (>98%) achieved on used MEA's



- Recovery of Pt from unused (i.e. scrap) MEA's dependent on:
 - MEA supplier
 - Preliminary GDL removal
 - Media used

Prototype MEA Recycling Process – Solvent Delamination



Timeline For MEA Delamination Approach

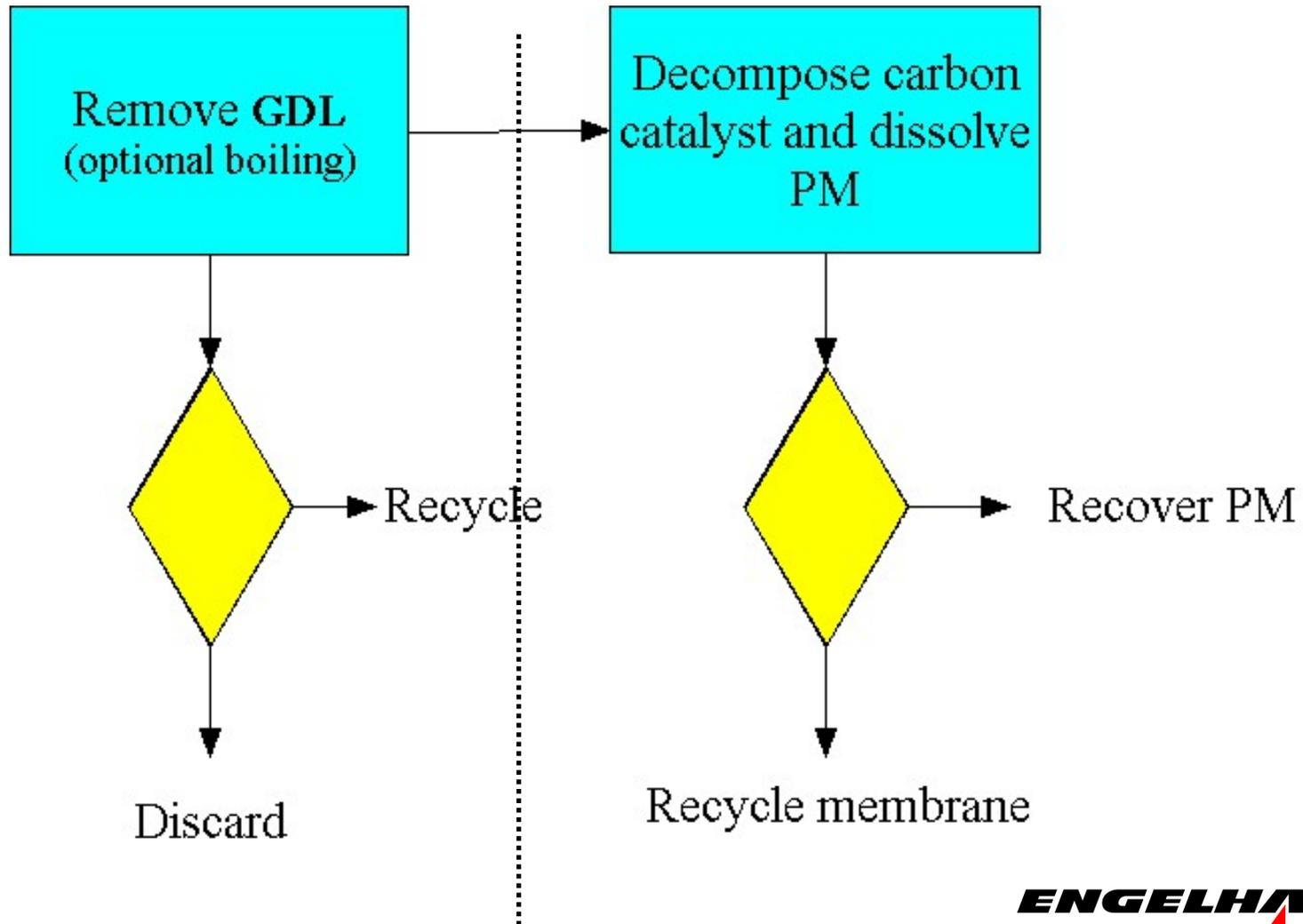
Task	Target date	Status
Separate electrode catalysts from membranes using delamination	5/04	Complete
Determine experimental factors affecting delamination efficiency	3/05	Complete
Age a batch of MEA's at Greenlight Power	7/05	Underway
Evaluate preparation required for used MEA's prior to delamination	10/05	
Test scale-up of delamination process on aged samples	03/06	
Demonstrate suitability of the delamination process for PM recovery	10/06	

Technical Results - Microwave assisted digestion



- **Capital-intensive process combines the electrode catalyst recovery and PM leaching steps of the delamination process.**
- **After holding at elevated temperature and pressure, 98% of Pt (and most of carbon) dissolves.**
- **The membrane is recyclable.**

Prototype MEA Recycling Process – Microwave-Assisted Digestion

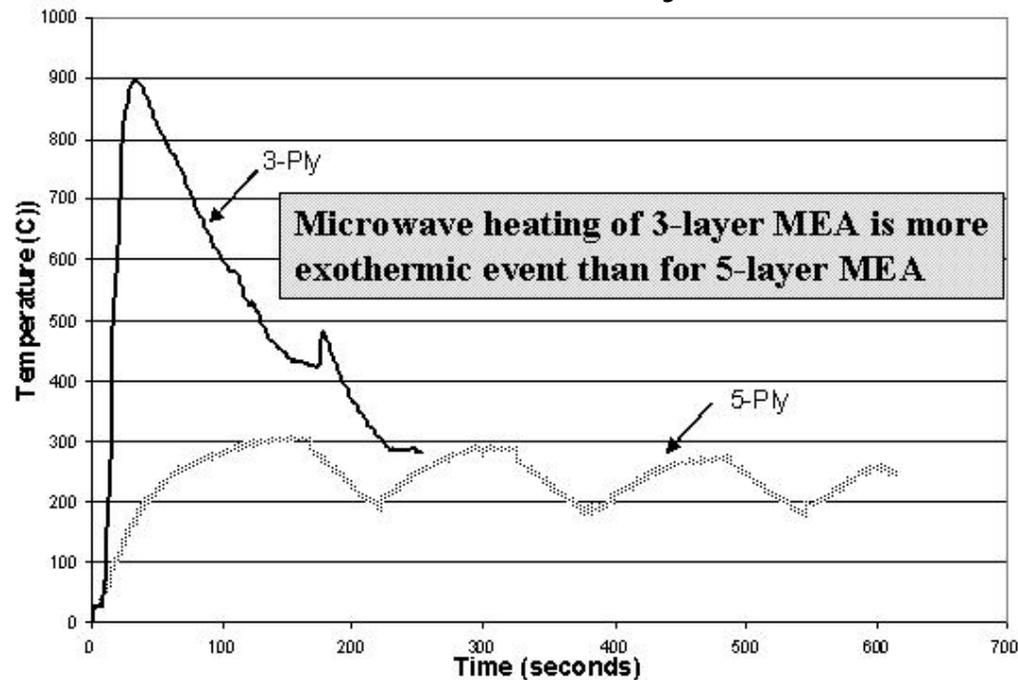


Timeline For Microwave-Assisted Digestion Approach

Task	Target date	Status
Install equipment to develop a microwave digestion process	3/04	Complete
Determine the feasibility of PM recovery using microwave process	12/04	Complete
Age a batch of MEA's at Greenlight Power	7/05	Underway
Test scale-up of microwave process	3/06	Underway
Identify hazardous vapors and liquids generated using microwave-assisted digestion	12/05	
Demonstrate suitability of microwave assisted digestion process for PM recovery	10/06	

Technical Results - Microwave combustion

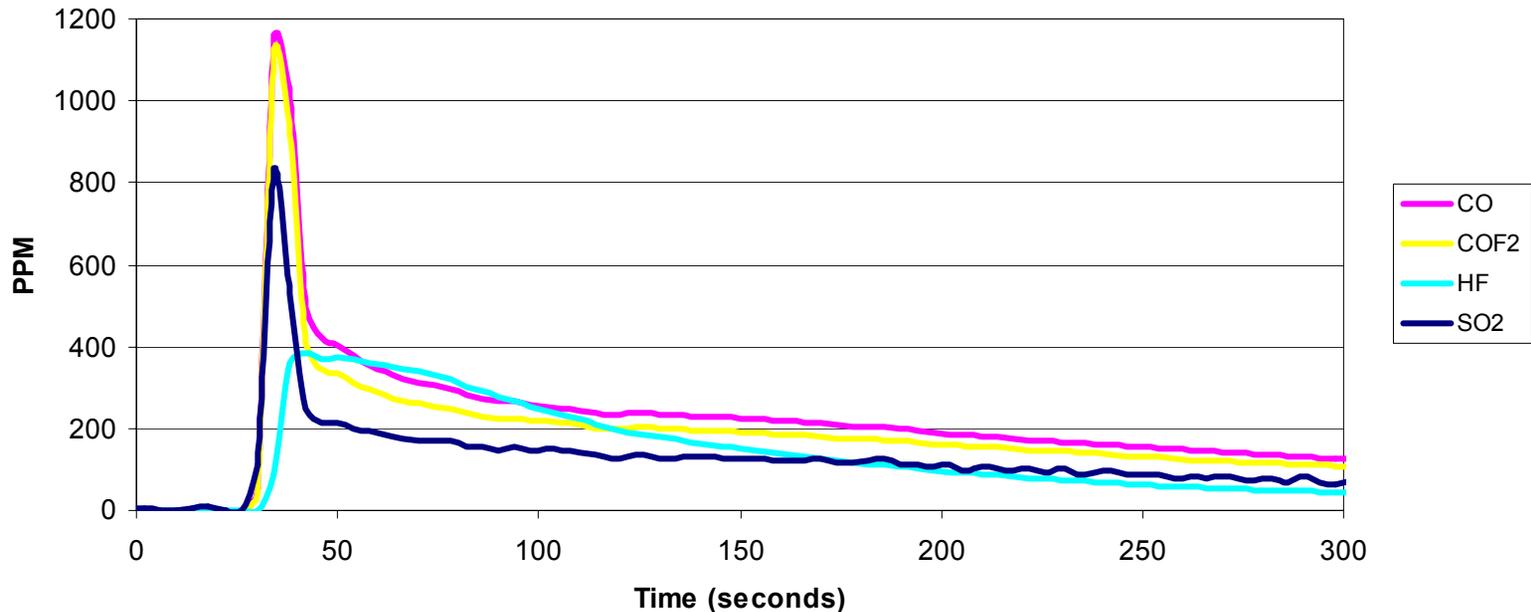
- Microwave Decomposition is highly favored for 3- over 5 –layer MEA's



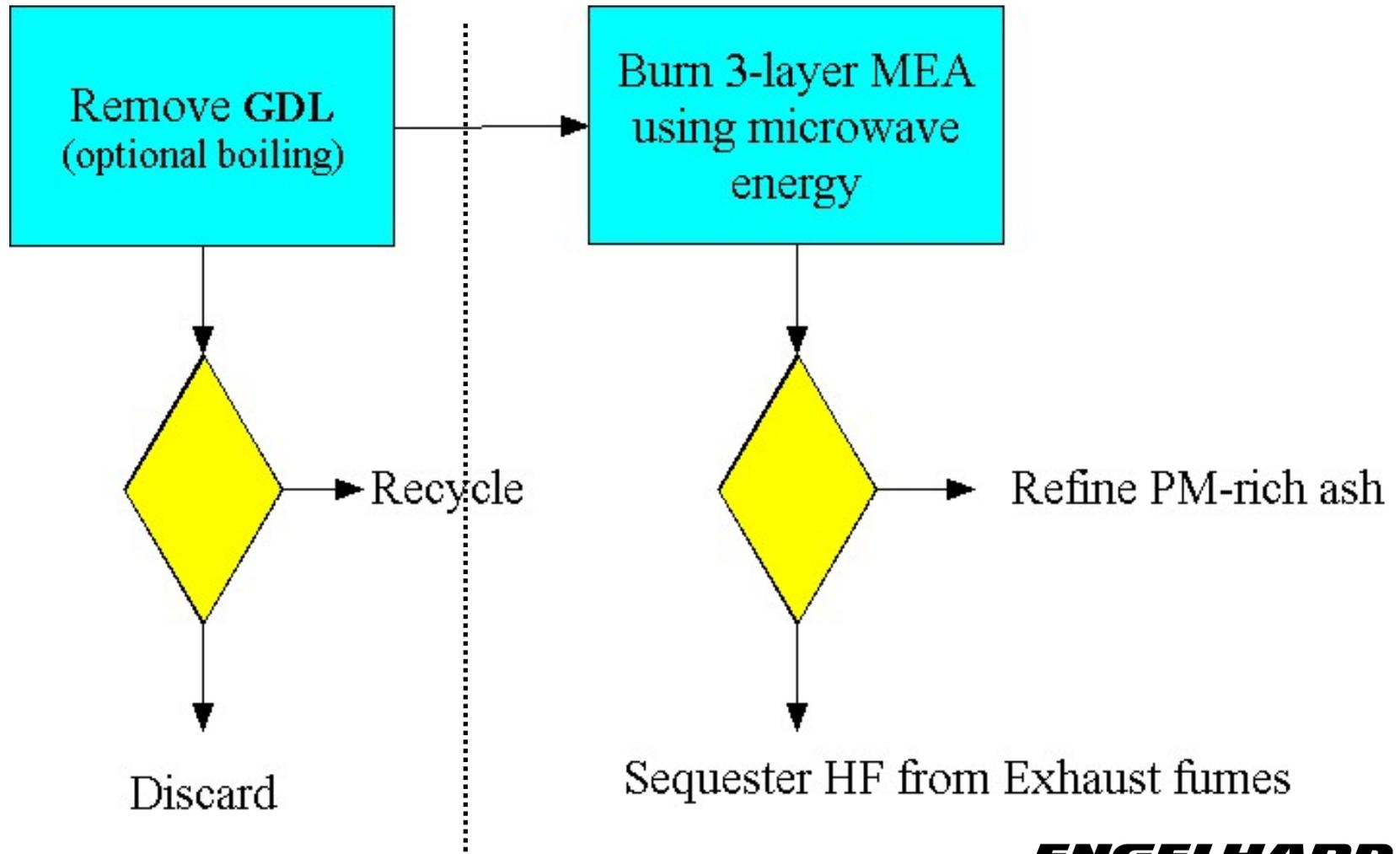
Technical Results - Microwave combustion – cont.

- Nafion® decomposition is accelerated using microwaves
- The major breakdown product, identified by FT-IR, is COF₂

Microwave Timeline for MEA (0.468 g)



Prototype MEA Recycling Process – Microwave Combustion



Timeline For Microwave Combustion Approach

Task	Target date	Status
Build a variable power microwave system	9/04	Complete
Connect FT-IR and monitor emissions composition	3/05	Complete
Build tandem microwave system to treat emissions	6/05	Underway
Identify emissions generated using microwave combustion	9/05	Underway
Develop adsorbent technology for emissions-free process	10/06	
Demonstrate suitability of the microwave tandem process for PM recovery	3/07	



Technical results – Supercritical Fluid Dissolution

- With high pressure and temperature, SC water has been demonstrated to dissolve Nafion®
- Electrode catalyst recovery achieved through filtration
 - SC water does not dissolve any Pt
 - Ionomer still found in carbon catalyst
- Project will be phased out because of both the complexity of the process and the lack of technical innovation to date



Response to Previous Year Reviewers' Comments

- Question 1: Relevance to overall DOE objectives
 - There were a few comments comparing the goal of re-use to the less desirable approach of precious metal reclaiming
 - Continued development of electrode catalysts makes direct re-use undesirable, even if feasible. Also, compositional differences between anode and cathode will make any co-mixture unsuited for re-use.
 - Vendor differentiation will make recycling of membrane material very complicated. A process will need to be instituted to identify the polymer by composition.
 - Unless the issue of membrane degradation is addressed, non-classical uses of reclaimed polymer may be preferred over recycling the membranes into new feedstock.



Response to Previous Year Reviewers' Comments

- Question 5: Approach to and relevance of proposed future research
 - What is the contingency plan if no suitable reclamation technologies are found?
 - The two primary issues are, in order, HF release and PM recovery. We are confident we can address these issues with at least one research approach. If necessary, Engelhard may pursue a pyrometallurgical approach, but that would result in loss of the polymer.
 - Address recycling of Nafion
 - Because of the difference in the structure of the perfluoropolymers used by different manufacturers, a complex sorting system would be required for membrane recycling, degradation mechanisms notwithstanding.
 - Engelhard is investigating a novel, alternative, non-fuel cell application for recycled fluoropolymers.