Recycling of Mixed Precious Metal/Ionomer Materials from Hydrogen Technologies

Department of Energy Manufacturing/Automation &
Recycling for Clean Hydrogen Technologies

May 24-27, 2022

Ion Power, Inc

Ion Power Headquarters in Delaware, USA

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- Ion Power develops, manufactures, and markets value-added products made with Nafion™

- Distribution agreement with ~~DuPont~~ now Chemours, since 1999

  >3000 m^2 of inventory
  for immediate delivery

  3 Locations,
  New Castle, Delaware
  Tyrone, Pennsylvania
  Munich Germany

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The 3 C's of Recycling

Collection

How widely dispersed is material source?

Contamination

How to purify / separate materials of interest?

Cash

Sale of recovered material to finance first 2 C's

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3 C's Examples

Plastic drinking bottles

Collection – Widely dispersed in every trash can in USA

Contamination – Wide variety of contamination on/in bottle

Cash – Low value of recovered material;

needs outlet for material second use – Trex decking

Used Nafion™ membranes

Collection – point source at a few Industrial locations

OEM Service agreements

Contamination – Well characterized contamination

Cash – High value polymer, but needs application

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Material scrap Price examples

Gold : ……$17,500 / lb
Silver : ……..$ 400 / lb
NAFION™ ……..$ 40 / lb (As manufacturing scrap)
Copper :…….. $ 3 / lb
Aluminum: ……$ 0.50/ lb

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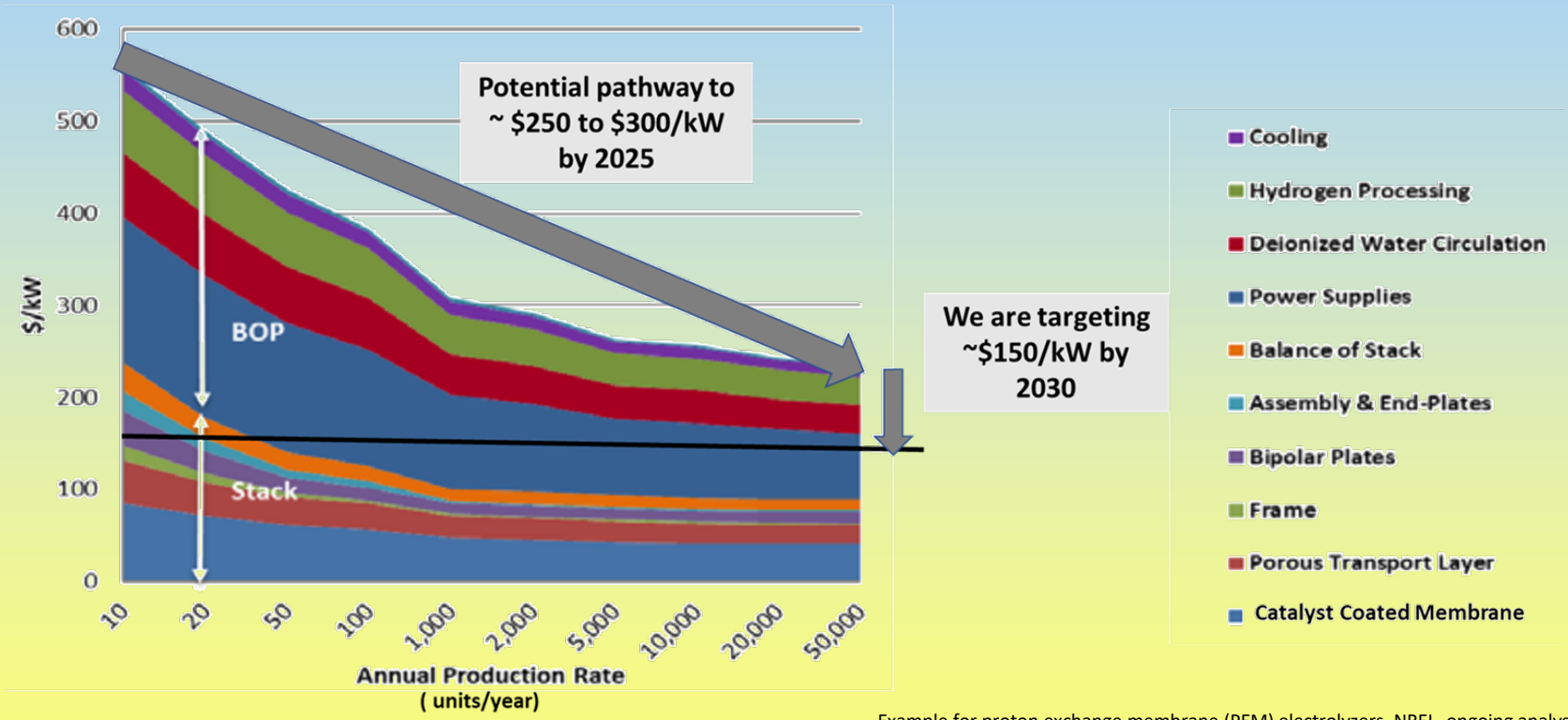
Analysis guides RD&D and cost reduction strategies

Electrolyzer System Cost Reduction Needs – cost reductions needed for stack and balance of plant (BOP) components



Example for proton exchange membrane (PEM) electrolyzers, NREL, ongoing analysis underway
Economies of scale projections for 1 MW per unit

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Scale of the problem

50,000 x 1 MW Electrolyzers/year ➔ 50 GW/year
@ 2 A/cm^2 ➔ 1.4 Million m^2
@ 175 g/m^2 ➔ 175 Tons membrane/year

Scale of the opportunity

50,000 x 1 MW Electrolyzers/year ➔ @ 150/kW ➔ $7.5 Billion
CCM Budget @ 20% of cost ➔ $1.5 B
Membrane @ $200/m^2 ➔ $0.3 B
Iridium @ 5 g/m^2 ➔ $1.1 B

$1.4 Billion opportunity!

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Current state-of-the-art

End-of-life systems:

% that return to OEM?

PGM bearing materials sent to companies that operate PGM recovery services.

Thermal decomposition is used to get rid of Carbon/Fluoropolymer components

$0.3 Billion/year of economic value lost!

Degradation Products of Sulfonic Acid Copolymer

Compound	Evolution Temperature, °C (°F)	Mg/g Sample
SO_2	280 (536)	15
CO_2	300 (572)	30
HF	400 (752)	*
CO	400 (752)	3
R_fCOF	400 (752)	10**
COF_2	400 (752)	3
COS	400 (752)	Trace
R_fOH	400 (752)	Trace

*Significant level, but could not calculate because HF reacts with and absorbs on cell walls.
**Mixture of products

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Opportunity:

How to credit the value of end-of-life materials to customer at time of system purchase?

Customer gets “Service Agreement” that covers maintenance of systems; recovered materials reduce the cost of “Service agreement”

Equipment carries a “Core “ charge payable by the OEM to owner when equipment/component returned.

We have commercialized this approach in our HYDRion™ water electrolysis CCM’s …. generates customer loyalty

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Contamination in used membranes, via XRF

	Water Electrolysis	Chlor-Alkali	De-Contaminated
			
Pt	36.9%		
Ir	32.3%		
Ti	22.0%		
Fe	3.9%	1.43%	0.04%
Mo	2.3%		
Ni	1.4%	0.03%	0.02%
Pb	1%		
Zr		0.33%	0.34%

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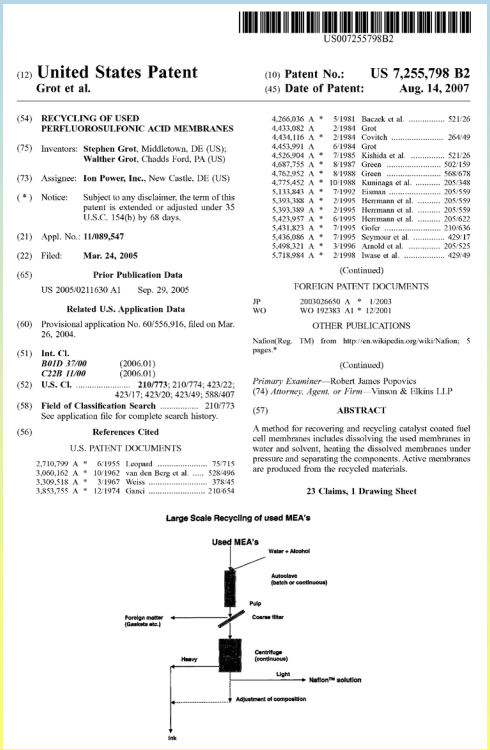
Patented Process



US007255798B2

(12) **United States Patent**
Grot et al.

(10) **Patent No.: US 7,255,798 B2**
(45) **Date of Patent: Aug. 14, 2007**

(54) **RECYCLING OF USED PERFLUOROSULFONIC ACID MEMBRANES**

(75) Inventors: **Stephen Grot**, Middletown, DE (US); **Walther Grot**, Chadds Ford, PA (US)

(73) Assignee: **Ion Power, Inc.**, New Castle, DE (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 68 days.

(21) Appl. No.: **11/089,547**

(22) Filed: **Mar. 24, 2005**

(65) **Prior Publication Data**

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Related U.S. Application Data

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(51) **Int. Cl.**
B01D 37/00 (2006.01)
C22B 11/00 (2006.01)

(52) **U.S. Cl.** **210/773**; 210/774; 423/22; 423/17; 423/20; 423/49; 588/407

(58) **Field of Classification Search** 210/773
See application file for complete search history.

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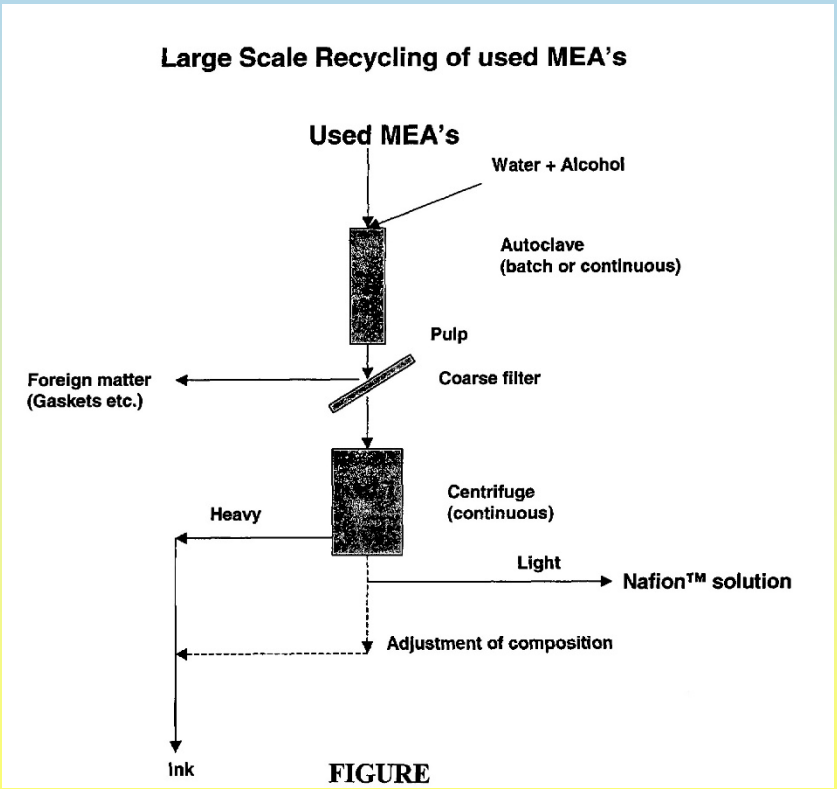
(74) *Attorney, Agent, or Firm*—Vinson & Elkins LLP

(57) **ABSTRACT**

A method for recovering and recycling catalyst coated fuel cell membranes includes dissolving the used membranes in water and solvent, heating the dissolved membranes under pressure and separating the components. Active membranes are produced from the recycled materials.

23 Claims, 1 Drawing Sheet



FIGURE

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After centrifuge and collect ionomer, adjust the solvent and cast films.

For water electrolysis membranes we cast films made with ionomer from used and fresh CCMs

For Chlor Alkali membranes we adjust process so that Carboxylate layer from Chlor-Alkali will not dissolve.

Films were Cast followed by a higher temperature curing step, 160 – 220 C for varying amounts of time

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Study of Recovered ionomer

- Re-cast ionomer into films, test following properties
  - Mechanical Strength
  - EW / Resistivity
  - Water Uptake/expansion
  - Integrity to Dissolution

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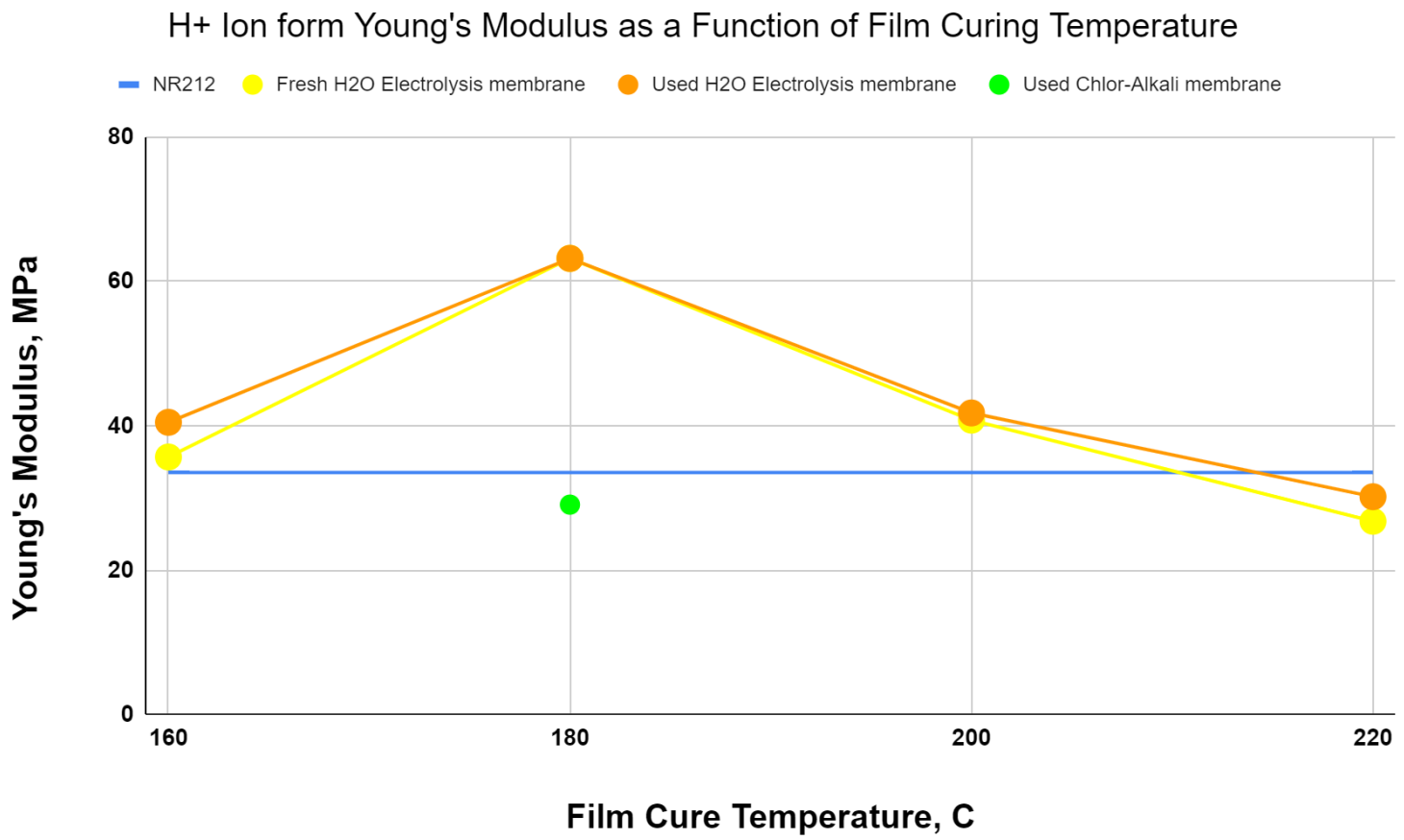

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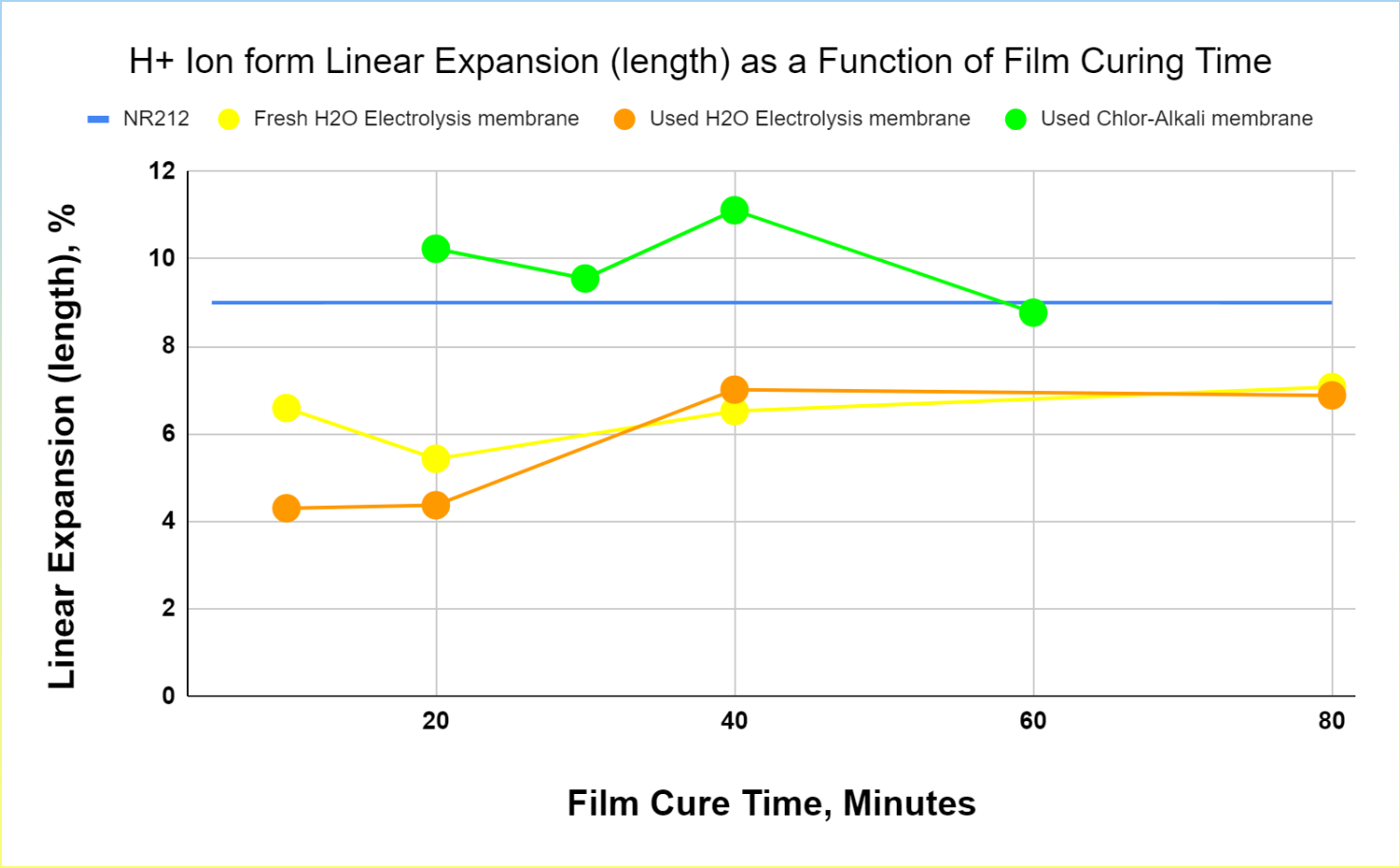

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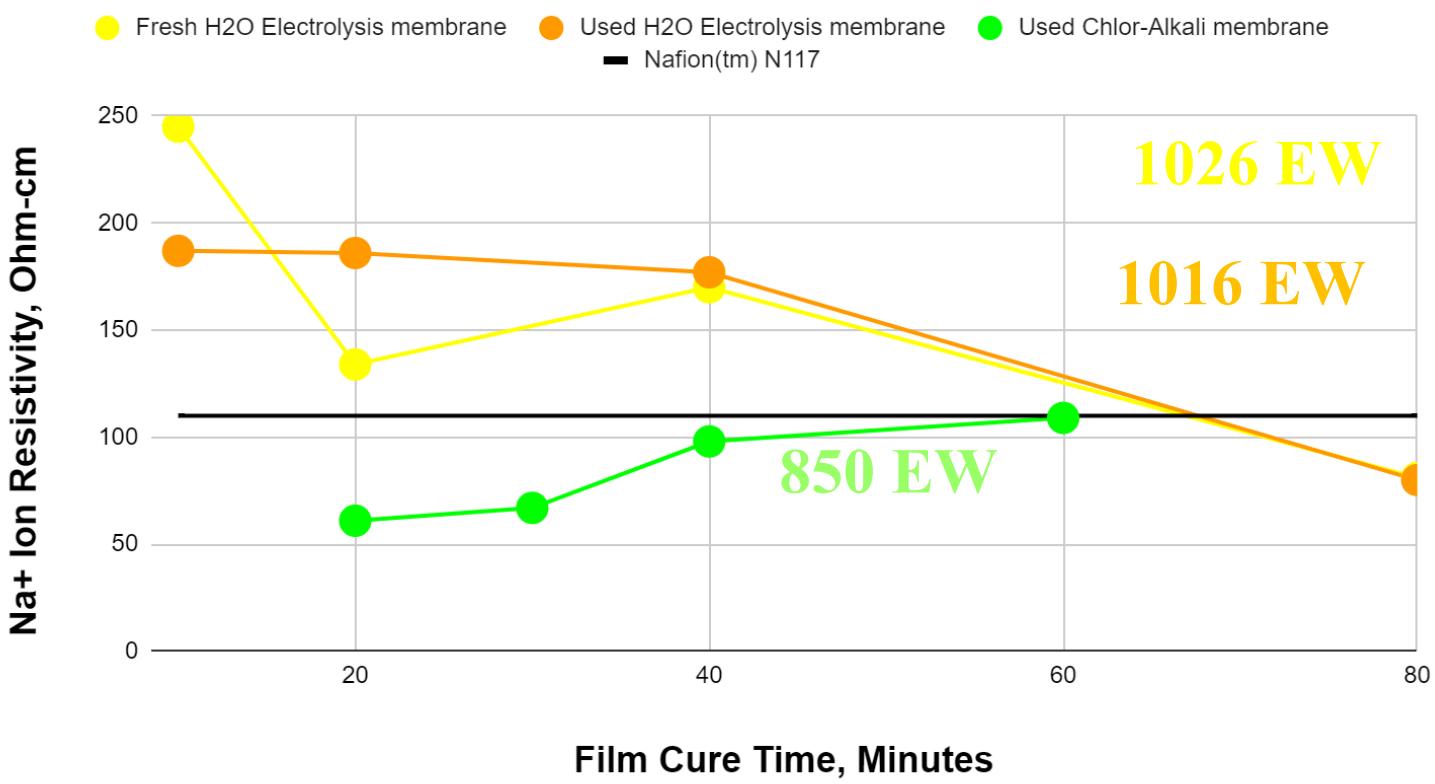
Thru Plane Resistivity as a Function of Film Curing Time



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Integrity to Dissolution

Soak in 1:1 IPA:DI Water (by volume) then bring to boil

Measure weight loss of sample

N115 Nafion™ - 3% weight loss

NR-212 Nafion™ - entire dissolution

< = 200 C cure all dissolved

Fresh MEA recast with 220 C Cure, 6% weight loss

Used MEA – Recast with 220 C cure, 9% weight loss

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Applications for Re-Use

- benefit from unique properties of Nafion™ but hampered by cost of Nafion™
- Does not need all properties
- Can sustain the “Right” demand for material

Examples:

- gas separation membranes, acid catalysis resin, flow battery membranes.

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Composite Membrane for Flow Batteries

Thin coating of PFSA ionomer on porous support

Porous support has strength / allows electrolyte penetration for conductivity

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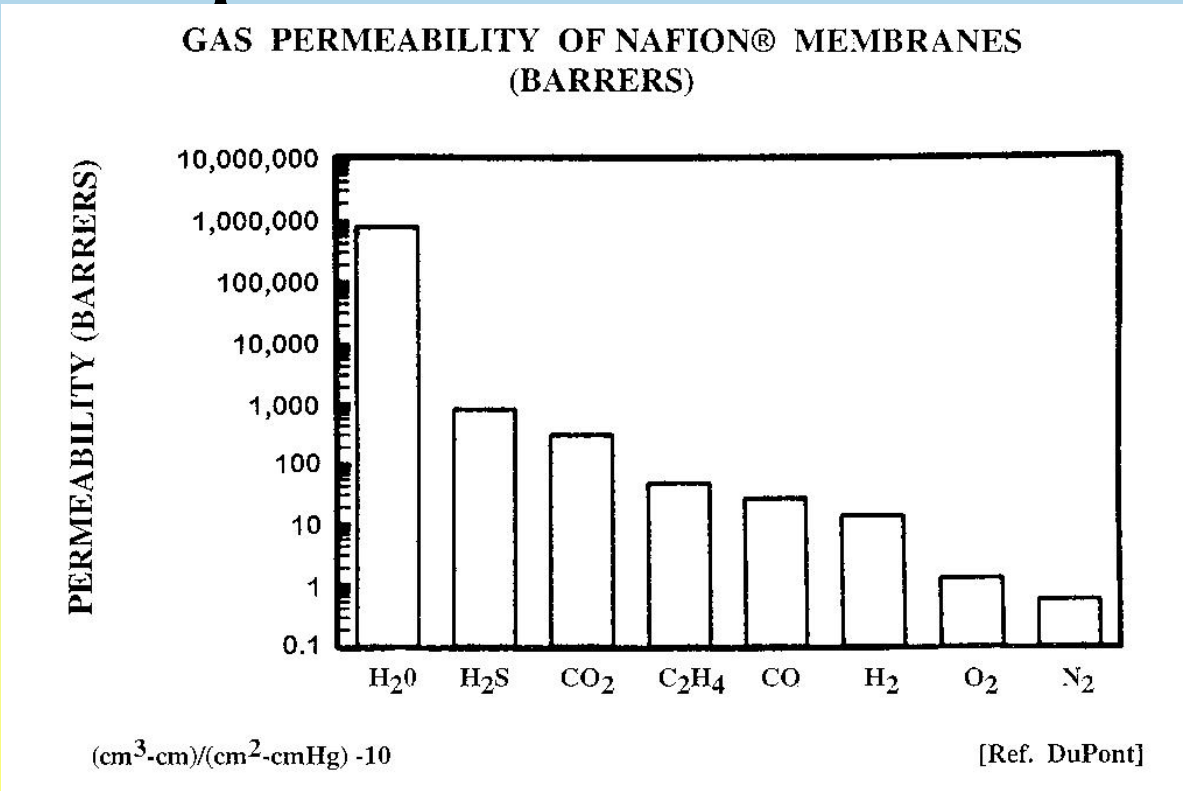
Gas Separation Membranes



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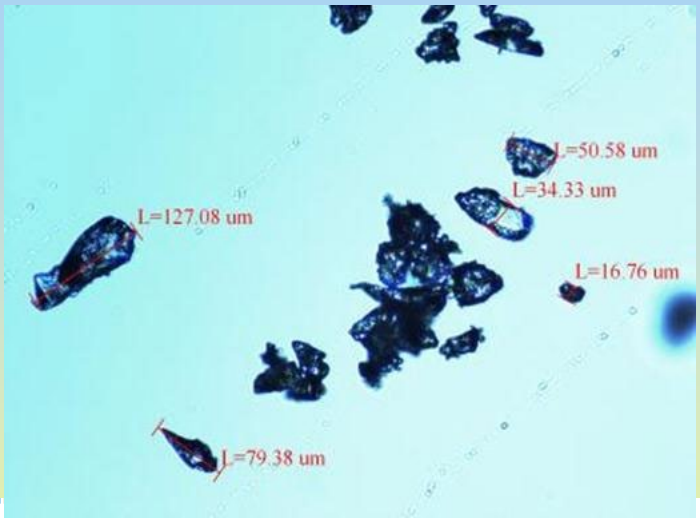
RECYLion™

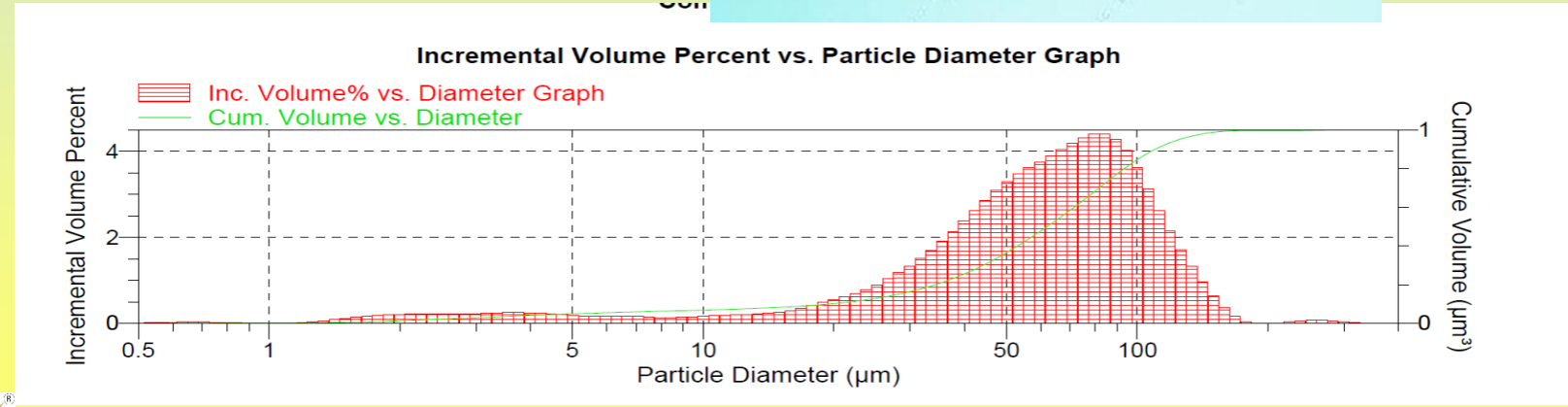
PFSA ionomer granules in a Soluble or insoluble form



0.1 to 1 mm Particle size

< 100 micron size

850 or 1100 EW



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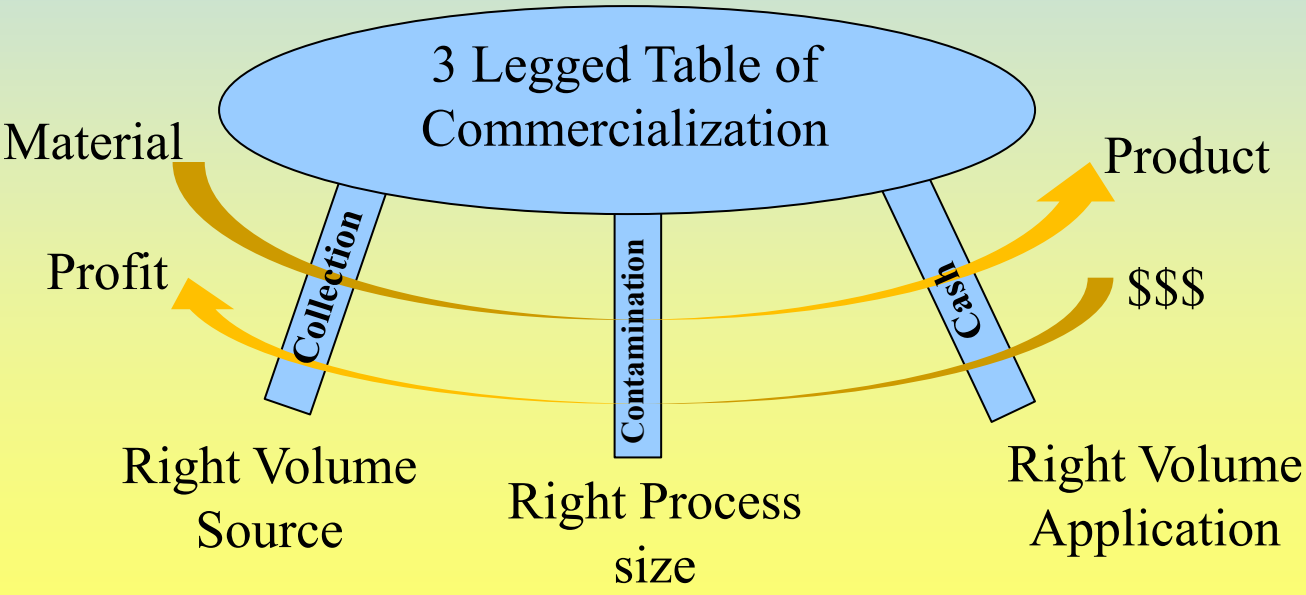
Commercialization

Scale of operation needs to be just right:

too small, and recovered material has high cost

too large, and source of material might not keep up



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Where Can DOE Projects Provide most impact to Commercialization?

Projects that aim to demonstrate/commercialize applications using recovered ionomer in or outside of the Hydrogen Technology space.

Projects can help understand degradation mechanisms of membranes