IONICS Category 3 Membrane Testing

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With content from Grigorii Soloveichik (ARPA-E Program Director) and Paul Albertus (Former ARPA-E Program Director)

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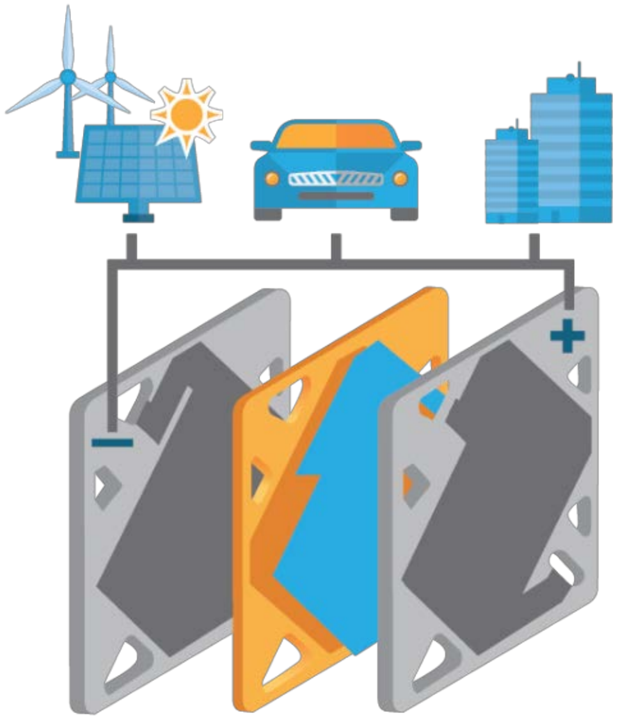
IONICS program mission



IONICS program mission

Create *solid separators* using solid ion conductors to enable transformational performance and cost improvements in electrochemical cells.

Program Directors:
Paul Albertus 2017–2018
Grigorii Soloveichik 2018–

IONICS is focused on the separator component

Typical ARPA-E program

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<u>*Device* metrics:</u>
W/kg, Wh/kg, \$/kW, \$/kWh, durability, mA/cm^2 at a given V, etc.

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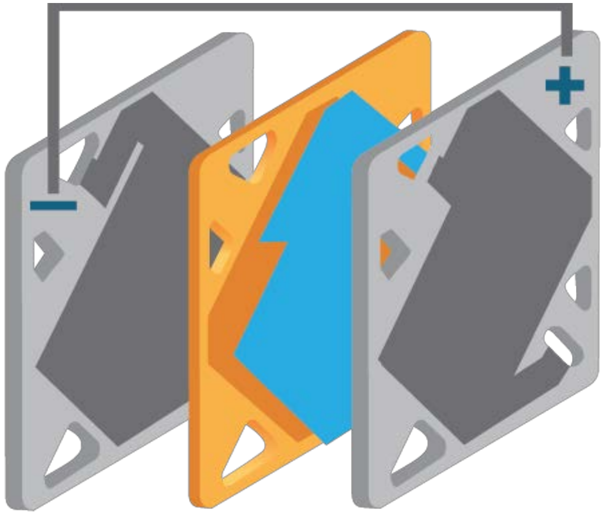
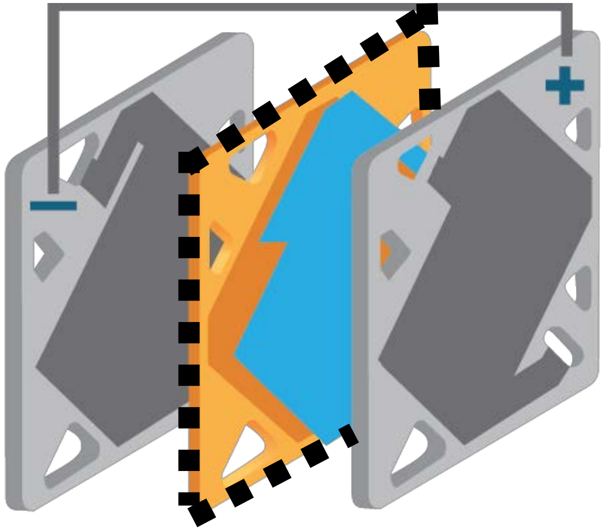
IONICS program

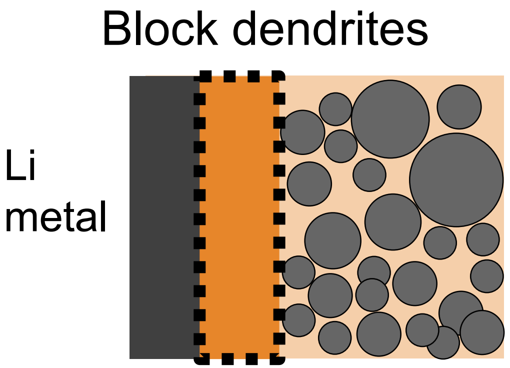


IONICS is focused on the separator component

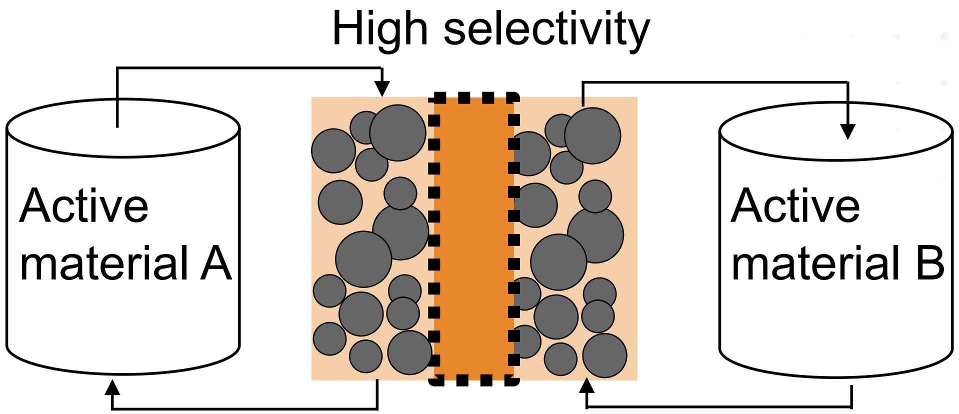
Typical ARPA-E program	IONICS program
	
Device metrics: W/kg, Wh/kg, \$/kW, \$/kWh, durability, mA/cm^2 at a given V, etc.	*Component* metrics in the device context: Selectivity, stability, separator and interfacial ASR, dendrite resistance, $\$/m^2$.

IONICS has three categories

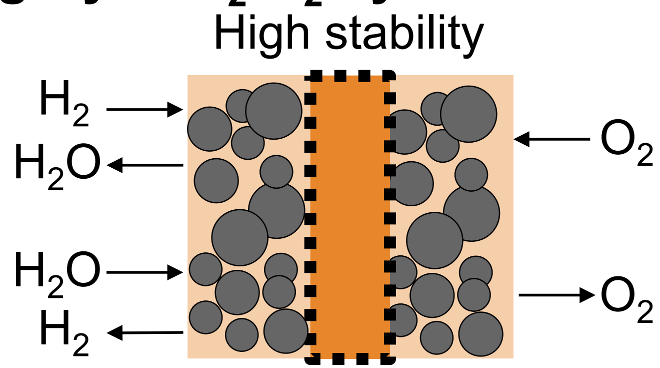
Category 1: Solid Li^+ conductors, and the lithium metal electrode



Category 2: Flow battery

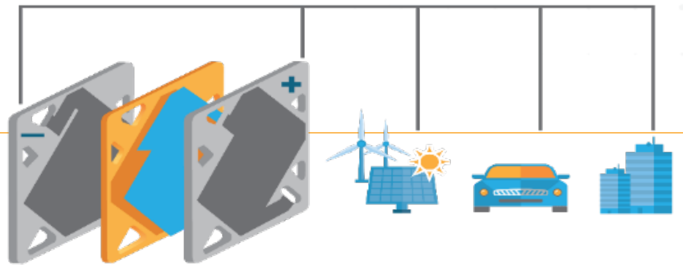


Category 3: H_2/O_2 hydroxide devices



IONICS

16 Project Teams • 3 Technology Areas



1: Li^+ conductors to enable the cycling of Li metal

ionic MATERIALS
POLY PLUS
OAK RIDGE National Laboratory
IOWA STATE UNIVERSITY
University of Colorado Boulder
PennState
24m
SILA NANOTECHNOLOGIES
UC San Diego

2: Separators for flow batteries

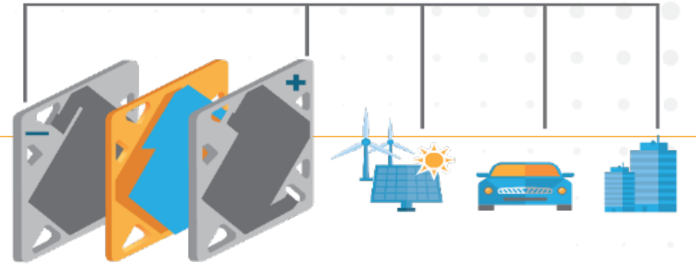
COLORADO SCHOOL OF MINES
United Technologies Research Center
Washington University in St. Louis
University of Colorado Boulder

3: Alkaline conductors

University of Delaware
3M
Rensselaer

OPEN, IDEAS, REFUEL

7 Project Teams



1: Li^+ conductors to enable the cycling of Li metal

University of Maryland
University of Michigan
Cornell University

2: Separators for flow batteries

Oak Ridge National Laboratory (Also working on fuel cells)

3: Alkaline conductors

Dioxide Materials
University of Delaware
Wichita State University
StoragEnergy – Energy Storage Solutions
ecolectro
Tetramer Technologies

IONICS alkaline-exchange membrane metrics (Category 3)

ID	Metric	Value	Rationale
1	Membrane chemical stability (at ≥80°C immersed in a pH≥14 solution)	≥1000 hours with ≤2% loss in IEC, ASR, spectroscopic measures of membrane state, and mechanical properties	Intrinsic membrane stability is a must
2	Area over which properties are achieved with ≥90% uniformity	≥100 cm^2	Relevant area for stack
3	Ionic ASR (hydroxide form, 80°C, liquid equilibrated)	≤0.04 Ohm-cm^2	To meet aggressive device-level ASR targets
4	Ionic ASR (80°C, ≤50% RH, air exposure)	≤0.08 Ohm-cm^2	Performance still needed when operating in air
5	Humidity cycling mechanical durability	≥20,000 RH cycles	For portable power fuel cells
6	Electronic ASR	≥1000 Ohm-cm^2	For high efficiency
7	Humidity Stability Factor	>5	Simple test for mechanical properties
8	Swelling in liquid water at 25°C	<50%	Needed for stack integration
9	Pressure differential (bar)	≥1	~1 bar for fuel cell ≥30 bar desired for electrolyzer
10	H_2 crossover and O_2 crossover	≤25 nmol/cm^2-s	For high efficiency
11	Cost	≤20 \$/m^2	To reach cost target of 30 \$/kW for automotive fuel cell

See IONICS Funding Opportunity Announcement for more details

IONICS alkaline-exchange membrane metrics (Category 3)

- PEM metrics
- Workshop input
- Economics
- Practical limitations (time, tools)

ID	Metric	Value	Rationale
1	Membrane chemical stability ([illegible] pH≥14 solution)	≥1000 hours with ≤2% loss in IEC, ASR, spectroscopic measures of membrane state, and mechanical properties	Intrinsic membrane stability is a must
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arpa·e
CHANGING WHAT'S POSSIBLE

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Specific to fuel cells

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FOA metrics

- One set for both electrolyzer and fuel cell applications
- Membrane testing only – no device testing
- No direct mechanical tests
- Room for inconsistency in procedures between groups

Why not device testing?

- Shouldn't we care about how the actual device performs?

- But no standard devices
  - Difficult to compare performance
  - Electrode incompatibilities or interface issues

- Membrane tests can reveal specific failure modes

Literature conditions

- Variation in
  - Temperature
  - Humidity
  - Counter-ion
  - $[HO^{-}]$
  - Time

- Comparison between research groups is difficult

We knew we needed actual test protocols, not just goals

ARPA-E IONICS Category 3 Recommended Guidelines and Protocols for Alkaline-Exchange Membranes

August 2017

Mark Pouy (Booz Allen Hamilton) and Paul Albertus (ARPA-E), with input from numerous IONICS program members and other experts in AEMs

Anion exchange membranes (AEMs) can be used as hydroxide-conducting electrolytes membrane electrode assemblies (MEAs) in fuel cells and electrolyzers. Polymer AEMs comprise a polymer backbone functionalized with cation groups that facilitate conduction of hydroxide. AEMs would ideally operate under a range of operating conditions while maintaining high conductivity and mechanical robustness. The conditions depend on the application (i.e. power generation or electrolysis). AEMs in fuel cells that power vehicles must be able to withstand winter and summer temperature extremes, as well as humidity variation between ambient and saturated. AEMs in electrolyzers should be able to withstand high pressures and pressure cycling. In both applications, AEMs are subject to degradation by reaction with hydroxide.

IONICS Category 3 testing guidelines

- Test recommendations (in order of importance):
  1. Baseline polymer characterization (MW, spectra, etc)
  2. Base stability assessed with multiple characterization techniques (including **IEC, conductivity, spectra, mechanical properties**).
  3. IEC
  4. Mechanical properties from a stress-strain curve
  5. Conductivity
  6. Water uptake and swelling
  7. Humidity cycling
  8. Pressure and pressure cycling
  9. Gas crossover

Base Stability

- FOA:
  - Temperature
  - pH
  - Before/after conductivity measurements
- Updated guidelines:
  - Inert atmosphere
  - Degassed solvents
  - Solution volume
  - Sample dimensions and mass
  - Polyethylene liner
  - Before/after mechanical tests

Mechanical properties

- FOA: N/A

- Updated guidelines:
  - Temperature 50 °C
  - RH 50%
  - Measure tensile strength, elongation, stress-strain curve

- Need to balance use conditions with practical measurement conditions

Current activities

- Set up centralized testing for IONICS teams at NREL (2018)
  - Lab-to-lab variations observed
  - Not planning a full round-robin
- Standard material?
- Some tests specify hydroxide form – when is this necessary?
- Other degradation mechanisms?
- Extrusion/creep