FARADAY TECHNOLOGY, INC.

LOW TEMPERATURE REDUCTION OF RARE EARTH METALS USING IONIC LIQUIDS

Rare Earth Elements and Critical Minerals Project Review Meeting

Sponsored by:
US DOE
Contract No. DE-SC0019794 / Phase I SBIR

September 15, 2020

Overview: FARADAY TECHNOLOGY, INC.

~ 49 PATENT PORTFOLIO:
ELECTRODEPOSITED COATINGS,
SURFACE FINISHING,
ELECTROCHEMICAL
CONVERSION, POWER,
ENVIRONMENTAL SYSTEMS,
CORROSION MONITORING


"…to be known as the company that changed the focus of electrochemical engineering from the art of complex chemistries to the science of pulse/pulse reverse electric fields…"

- Electrochemical engineering processes and technologies – founded 1991
  - www.FaradayTechnology.com
- Subsidiary of Physical Sciences, Inc. (Boston, MA) – acquired 2008
  - www.psicorp.com
- Collective employment ~225; ~150 MS/PhD/Annual revenue of ~ $70 million
- Outsourced R&D, pre-production scale-up, IP protection, β-test novel manufacturing solutions

The Patented FARADAYIC® Process

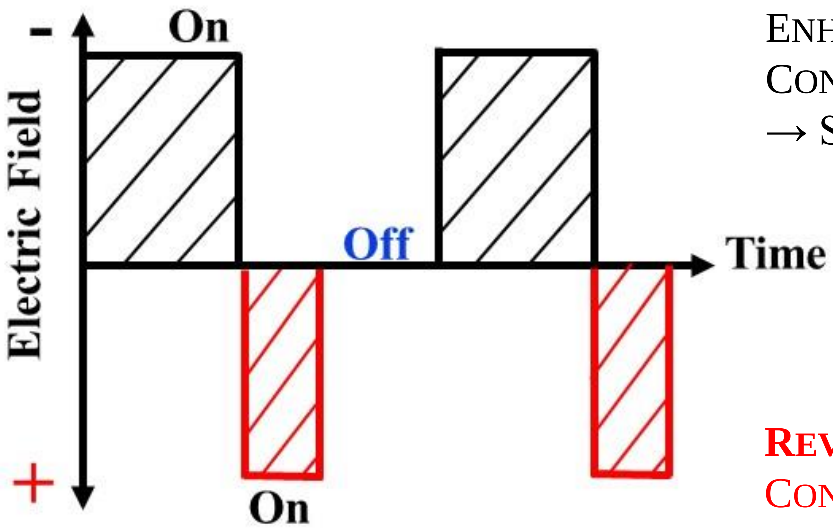
AWARD WINNING ELECTROCHEMICAL PROCESS DEVELOPMENT

ELECTROCHEMICAL MACHINING, POLISHING, DEBURRING, THROUGH-MASK ETCHING

- 2008 Blum Scientific Achievement Award for Pulse Reverse Surface Finishing
- 2016 R&D 100 Finalist for HF-Free Nb SRF Cavity Polishing

ELECTRODEPOSITION/PLATING

- 2011 R&D 100 for Co-Mn Alloy Plating
- 2013 Presidential Green Chemistry Challenge Award for Cr^{+3} Plating



FORWARD:
ENHANCED MASS TRANSPORT
CONTROL CURRENT DISTRIBUTION
→ SIMPLIFIED CHEMISTRY

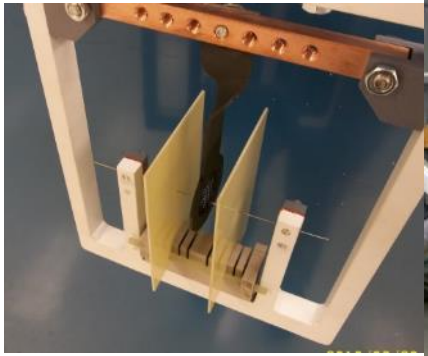
OFF:
REPLENISH REACTION SPECIES,
REMOVAL REACTION PRODUCTS
HEAT DISSIPATION

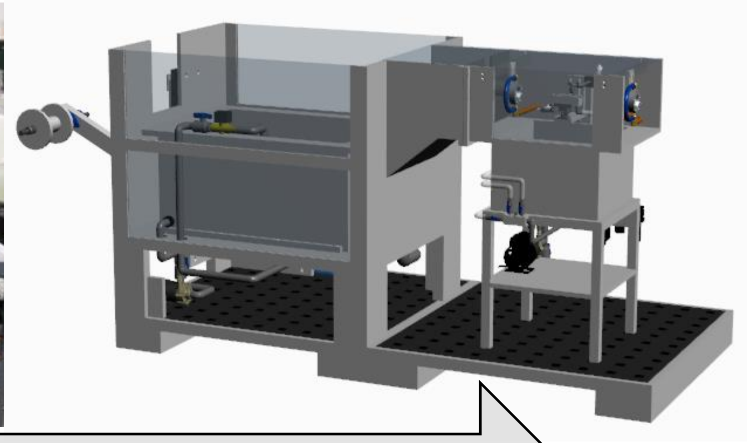
REVERSE:
CONTROL INTERFACIAL CHEMISTRY
→ REMOVE H_2 EFFECTS, OXIDE REMOVAL, SURFACE DEPASSIVATION

FARADAY TECHNOLOGY, INC.

DEM/VAL: α- to β-Scale

Technology development begins conceptually and is demonstrated at the bench-scale and developed through α/β-scale validation. IP alignment with technology.





Development through Production Scale & Technology Scaling to meet Manufacturing Needs

Problem and Opportunity

- **Problem**
  - Rare earth elements (REEs) are industrially important for energy, defense and consumer products. The United States has limited domestic mining capabilities and furthermore, China dominates the market in terms of refinement of the REEs from the ores in which they are found. This leaves the United States reliant on importing REEs.

- **Opportunity**
  - Coal ash (and other byproducts such as acid mine drainage and mine tailings) contain low levels of REEs. Recovery of these valuable elements from these waste streams may present an economically and environmentally attractive feedstock of these materials. Current processing relies on high temperature processing using corrosive solutions.

Technical Approach

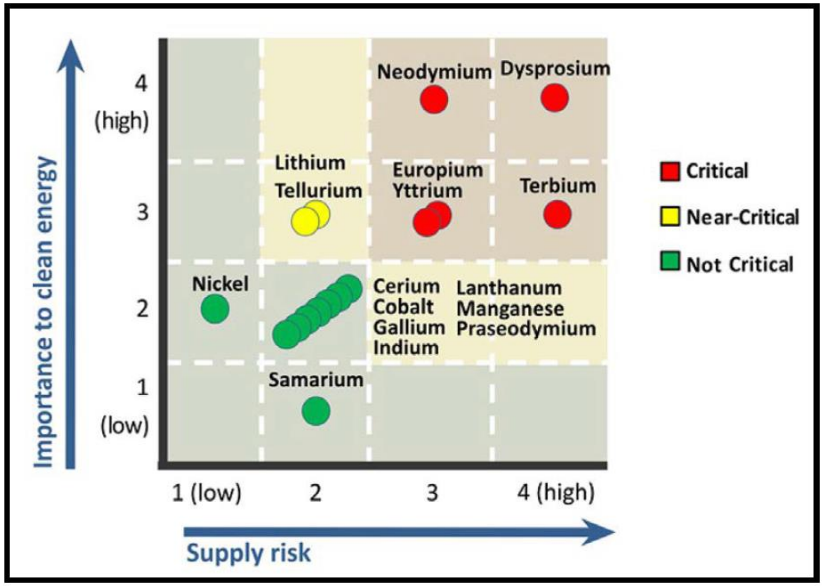
- Low temperature operation: room temperature ionic liquids have electrochemical and thermal stability = replacement for molten salt electrolytes
- Identification of appropriate ionic liquids to facilitate extraction of rare earth elements from coal ash and support electrochemical recovery
- Utilization of novel electrochemical fields (FARADAYIC® Process) proven to improve deposition in ionic liquid electrolytes
- Partner with University of South Alabama for ionic liquid selection and synthesis

Program Objectives

Overall: development of a ***low-temperature*** reduction method for production of ***high purity*** REEs from coal byproduct feedstocks

Phase I Program Goals:

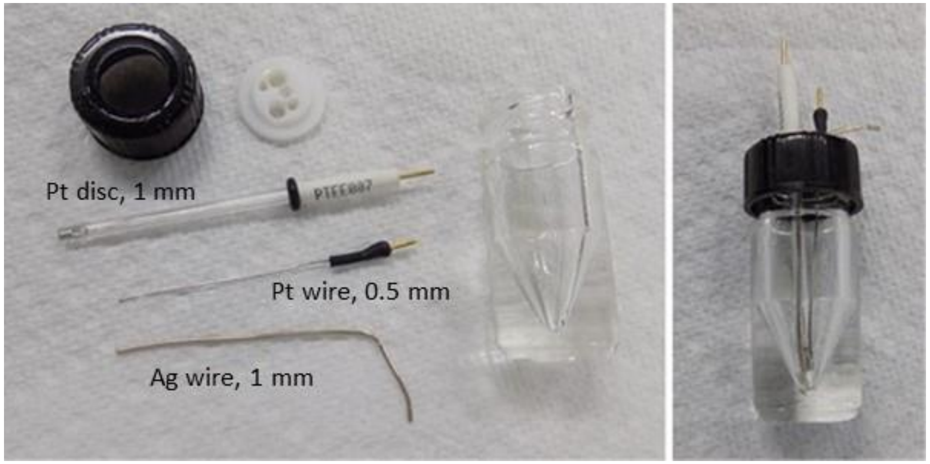
- Explore room temperature ionic liquid (RTIL) electrolytes and processing conditions needed for recovery of REE via low temperature electro-reduction reactions
- Start with model solutions using neodymium oxide
- Transition to coal ash solutions for recovery of REEs in metallic form



DOE Energy Report to Congress, "Report on Rare Earth Elements from Coal and Coal Byproducts", January 2017

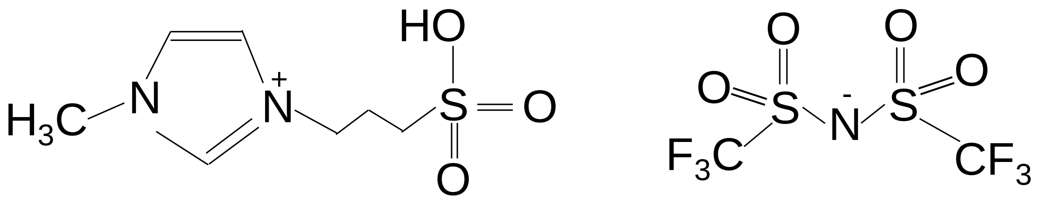
Electrochemical Measurements

- Experiments conducted in small glass vials (2-5 mL)
- Cyclic voltammetry: scanning over a specified potential range and recording the resulting current
  - Increases in current correspond to redox activity in system
  - Appropriate reduction potential for each “system” (electrolyte formulation and electrode setup)
- ElectroRecovery experiments were conducted using 2 wire electrodes, exploring both constant voltage and pulse voltage waveforms

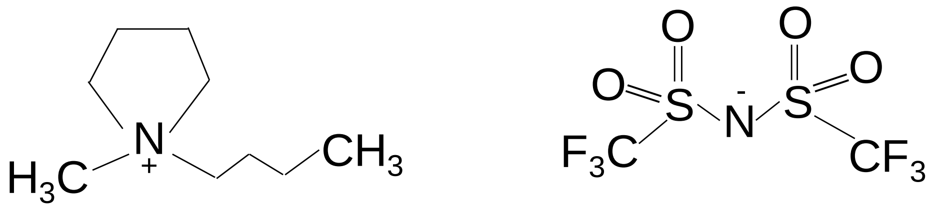


Ionic Liquid Electrolyte Formulation

- Faraday partnered with the University of South Alabama for identification and synthesis of candidate RTIL species:

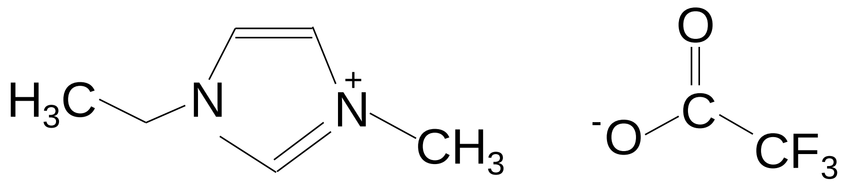


1-methyl-3-propylsulfonyl-imidazolium bis(trifluoromethanesufonyl)imide (MPSIm TFSI)



1-methyl-1-butyl-pyrrolidinium bis(trifluoromethanesufonyl)imide (BMPyrr TFSI)

- The following RTIL was purchased from Iolitec:

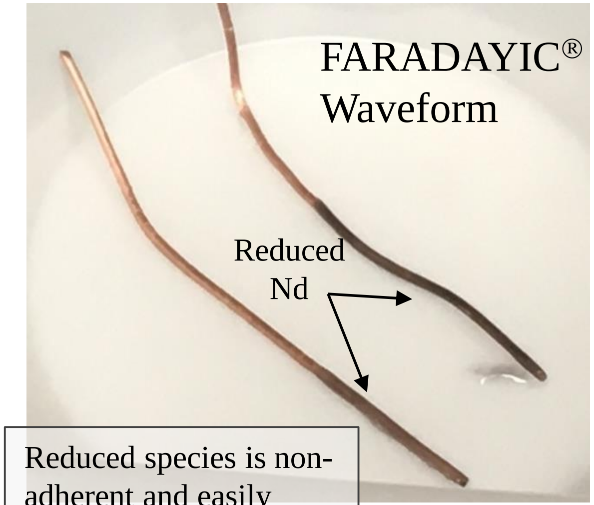


1-ethyl-3-methyl-imidazolium trifluoroacetate(EMIm TFA)

Work Conducted To-Date

- Preliminary work conducted to assess necessary RTIL or RTIL blend for deposition experiments
    - Nd_2O_3 powder used for preliminary characterization and electrolyte formulation studies
    - Focused on RTILs provided by U.S.A. due to larger electrochemical window
    - Evaluated different electrode materials
    - Low temperature (RT - 100 °C) deposition using pulse and constant voltage fields
- Demonstrated low temperature reduction of neodymium from model IL solution using pulse deposition
- Started preliminary electrolyte studies using coal ash using these RTIL solutions and pulse and constant voltage conditions → Poor/No Extraction
- Identification of new partner with proprietary technology with proven extraction of REEs from coal base resources into RTIL solutions

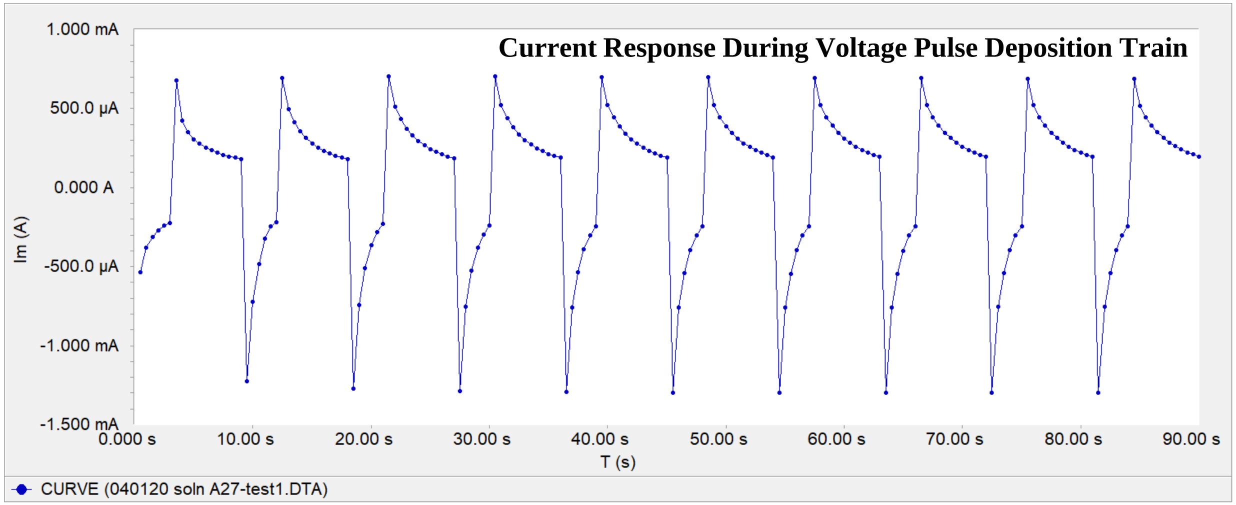
REE ElectroRecovery: Model Solutions



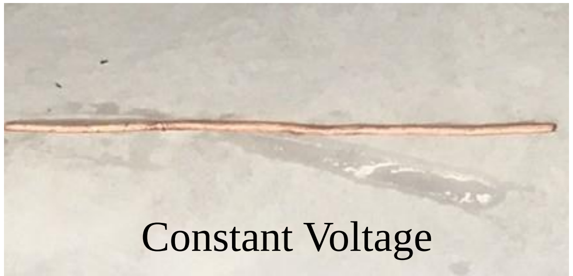
FARADAYIC® Waveform

Reduced Nd

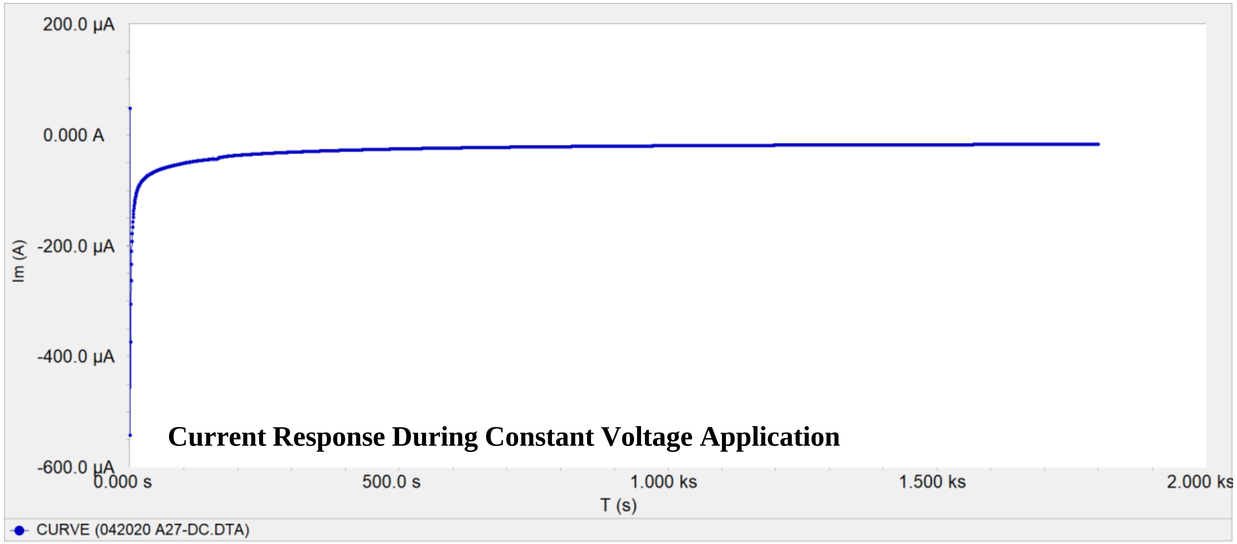
Reduced species is non-adherent and easily removed from substrate



Current Response During Voltage Pulse Deposition Train



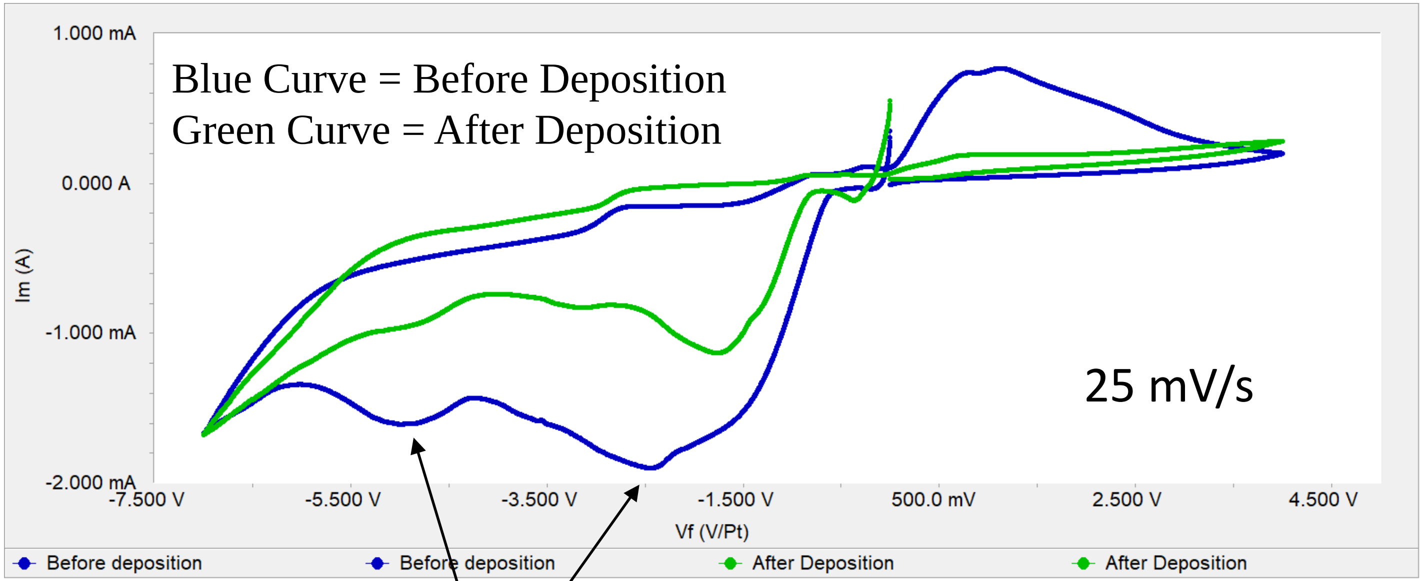
Constant Voltage



Current Response During Constant Voltage Application

REE ElectroRecovery: Model Solutions

Cyclic voltammograms conducted before and after electro-reduction of Nd from oxide/IL solutions (MPSIm TFSI/BMPyrr TFSI mix). Experiments conducted ~ 85 °C.

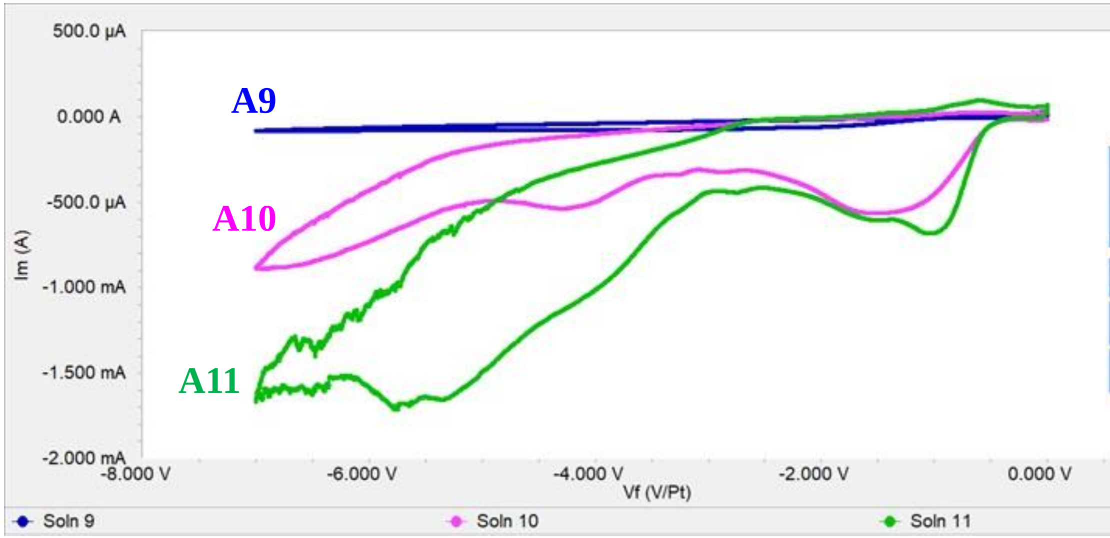


Blue Curve = Before Deposition
Green Curve = After Deposition

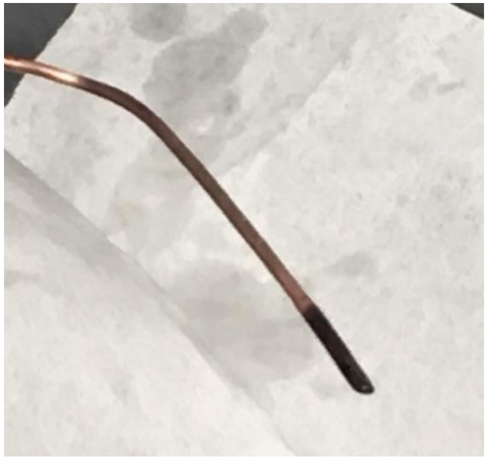
25 mV/s

Nd species reduction peaks

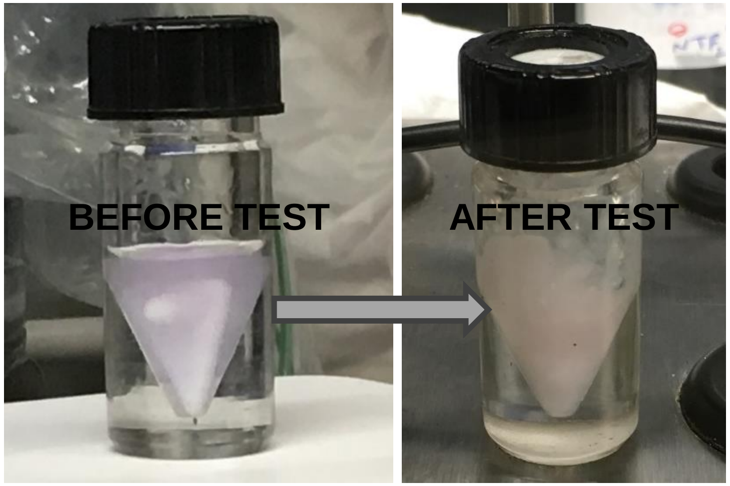
REE ElectroRecovery: Model Solutions



Soln ID	MPSIm TFSI (g)	BMPyrr TFSI (g)	Nd_2O_3 (g)
A9	1.11		~0.07
A10	1.11	0.65	~0.07
A11	1.11	1.18	~0.07

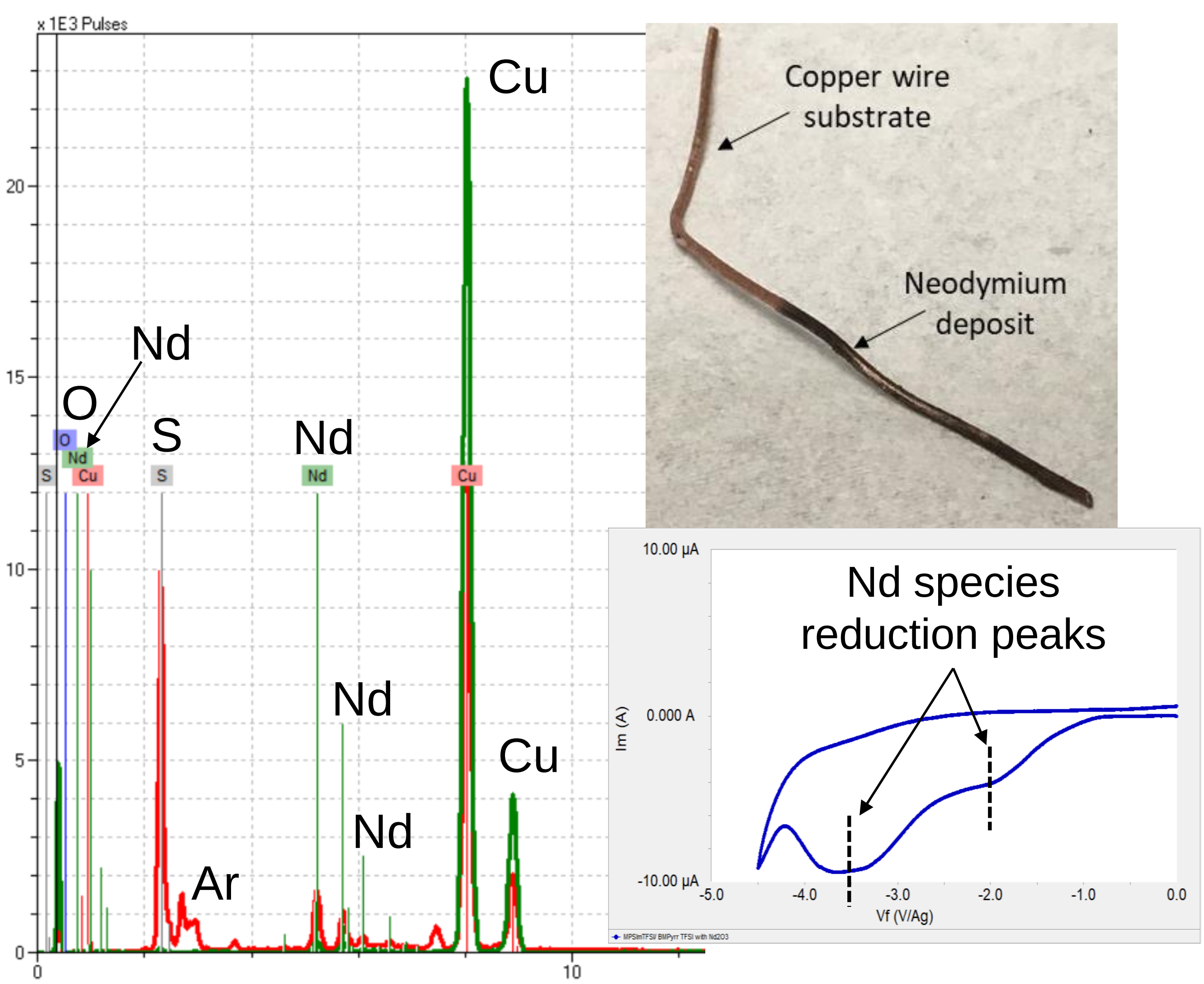


- Electrolyte composition is important: deposition only observed here in solution A11
- Solution color change observed after deposition



REE ElectroRecovery: Model Solutions

- Nd readily electro-reduced and ***recovered*** (on a Cu wire) from oxide based ionic liquid solutions
- Reduction ***only*** observed with FARADAYIC® Waveforms – ***no*** reduction observed with constant voltage



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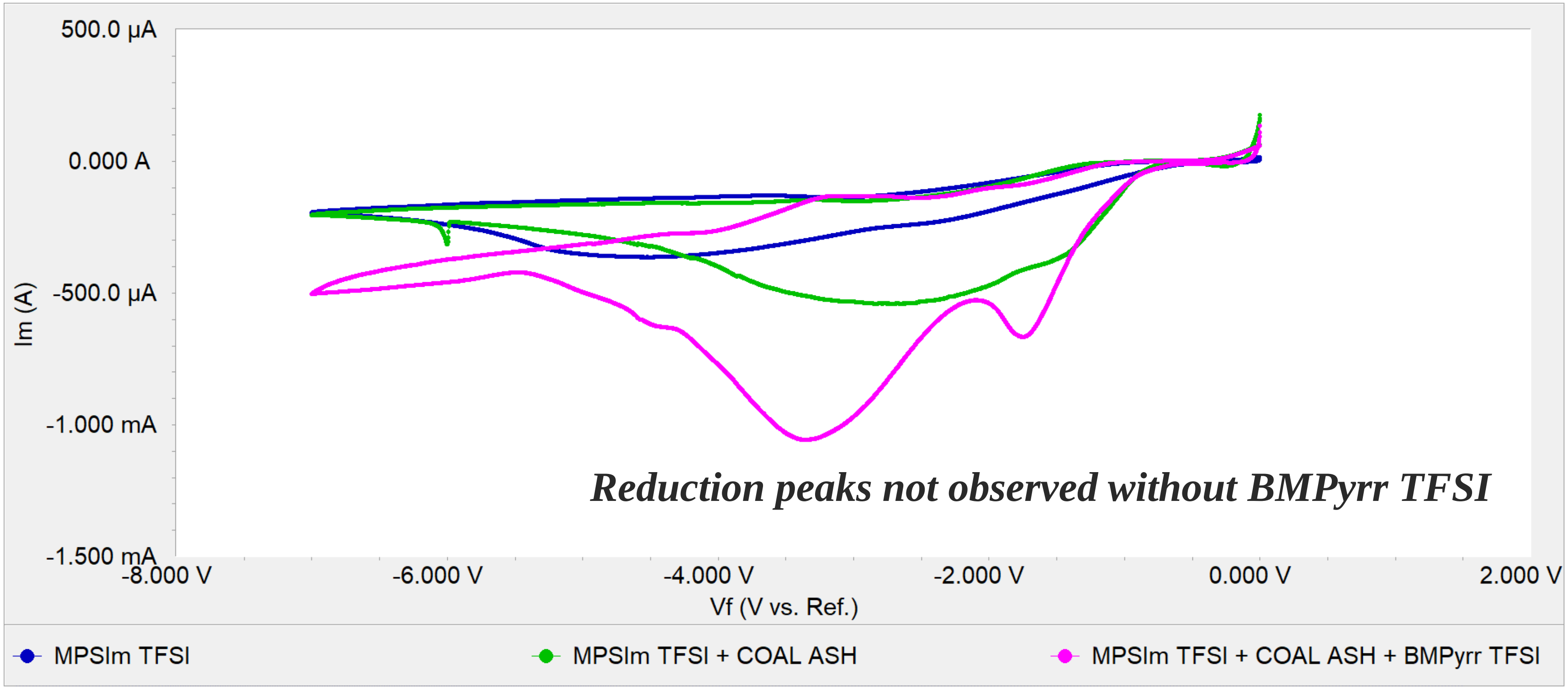
REE ElectroRecovery: Coal Ash Solutions

Using the data generated for model solutions, Faraday began to explore electrolyte formulation and conditions needed for REE recovery from coal ash (NIST Standard Reference Material® 2691, purchased from Sigma Aldrich)



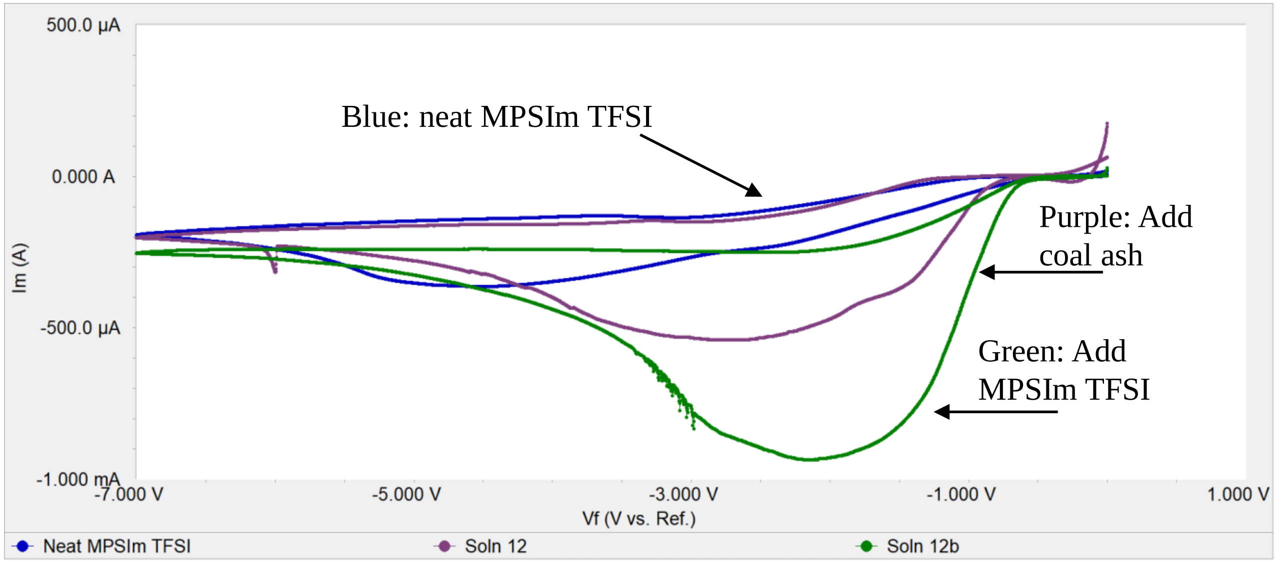
Reduction peaks not observed without BMPyrr TFSI

REE ElectroRecovery: Coal Ash Solutions

Deposition from coal ash solutions explored

- MPSIm TFSI and BMPyrr TFSI, and blends of these IL
- Constant voltage and pulse voltage conditions
- Room temperature and up to 100 °C
- No deposition was observed for REE or any other species in these studies

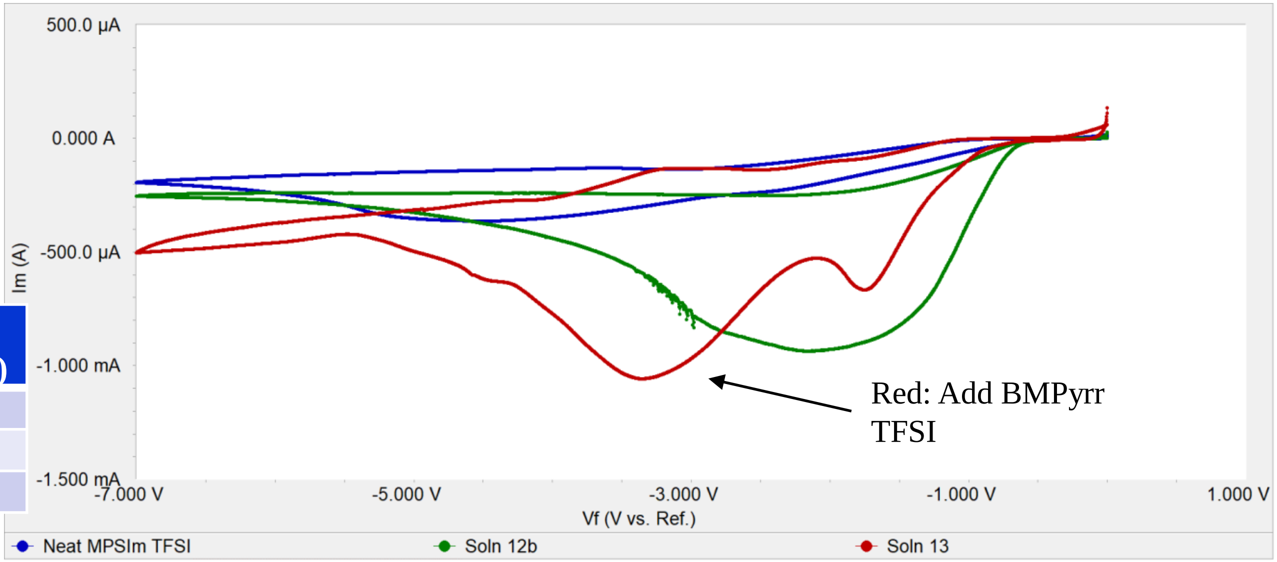
Soln ID	MPSIm TFSI (g)	BMPyrr TFSI (g)	Coal Ash (g)
A12	1.13		0.09
A12b	1.90		0.09
A13	1.90	1.17	0.09



Blue: neat MPSIm TFSI

Purple: Add coal ash

Green: Add MPSIm TFSI



Red: Add BMPyrr TFSI

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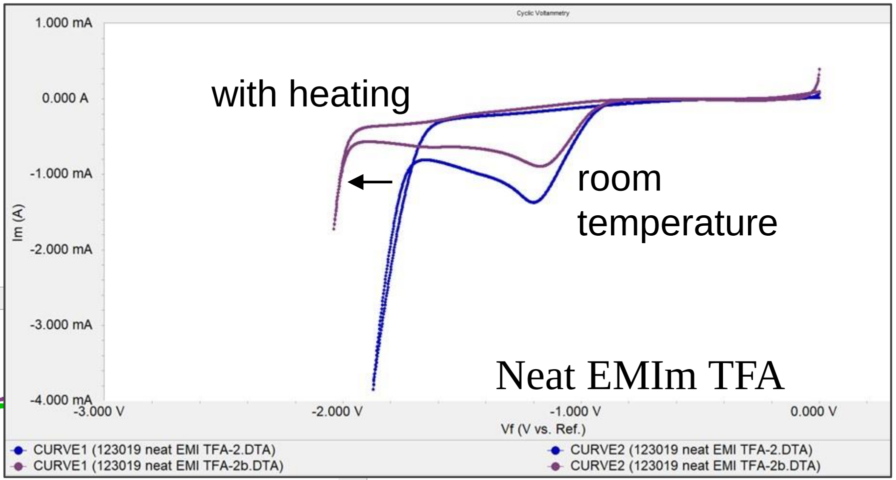
REE ElectroRecovery: Coal Ash Solutions

Faraday also explored recovery of REE using EMIm TFA/coal ash solutions

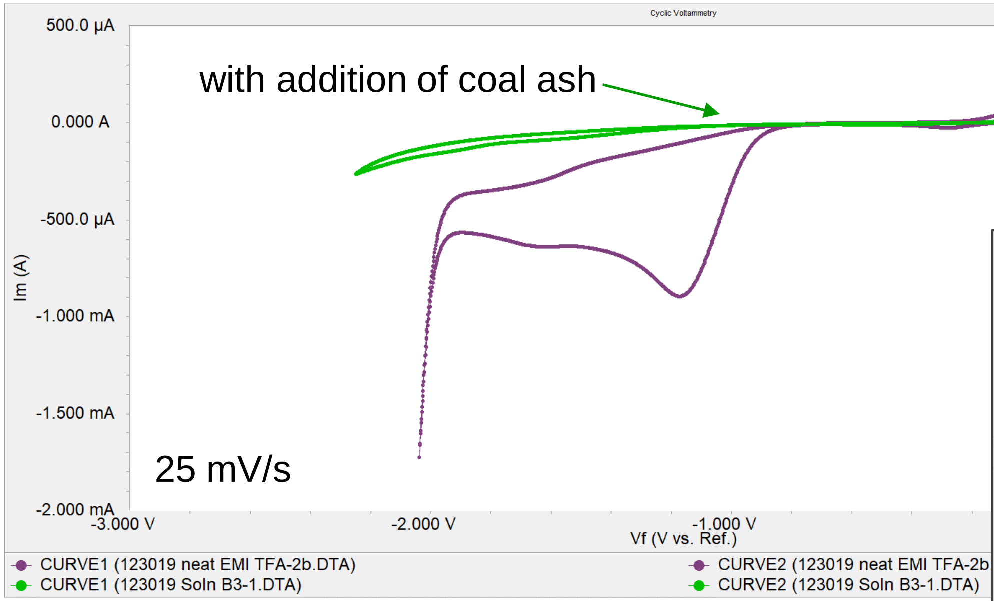
- Experiments conducted at 85-100 °C
- No deposition was observed from these solutions



with heating

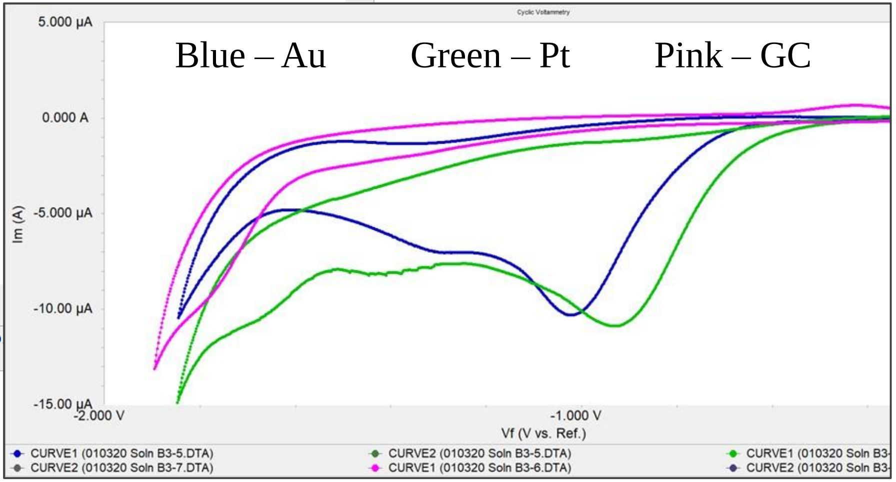
room temperature

Neat EMIm TFA



with addition of coal ash

25 mV/s



Blue – Au Green – Pt Pink – GC

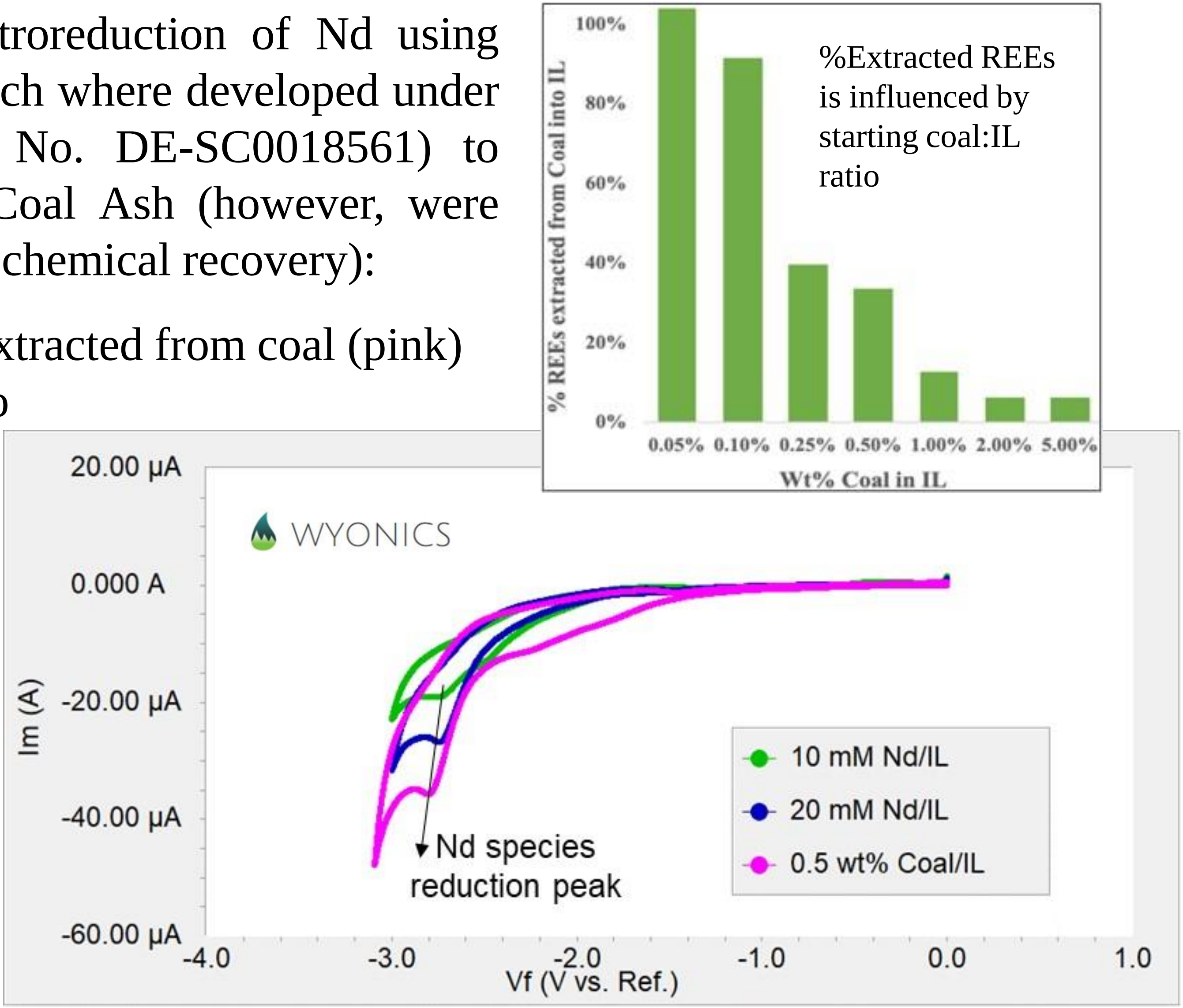
Key Findings

- Neodymium was recovered via FARADAYIC® ElectroRecovery experiments from simple ionic liquid electrolytes using oxide solutions; recovery was only observed with FARADAYIC® Waveforms. Constant voltage processes did not result in reduction under any conditions tested.
- Use of MPSIm TFSI, BMPyrr TFSI, EMIm TFA and blends of the ILs did not result in recovery/electroreduction (of REE or metals) when using coal ash solutions. It was speculated that these ILs did not provide proper solubilization.
- Pretreatment methods such as acid digestion did not improve electroreduction in the IL solutions tested.
- Faraday sought the partnership of Wyonics, who have proven technology for extracting REEs from coal and coal ash into IL matrix. Faraday ran preliminary tests on samples of their REE/IL solutions. Electrochemical detection of Nd is apparent, but these IL were not designed for electrochemical extraction. This challenge will be the focus for Phase II.

Electrolyte Characterization (Coal Ash Extracted Soln)

Faraday explored electroreduction of Nd using Wyonics' solutions which where developed under DOE funding (Grant No. DE-SC0018561) to solubilize REE from Coal Ash (however, were not designed for electrochemical recovery):

1. REE present in IL extracted from coal (pink) in 0.5% coal/IL ratio
2. Isolated Nd after from coal extraction after separation using IX resins (two concentrations, 10 and 20 mM, green and blue)

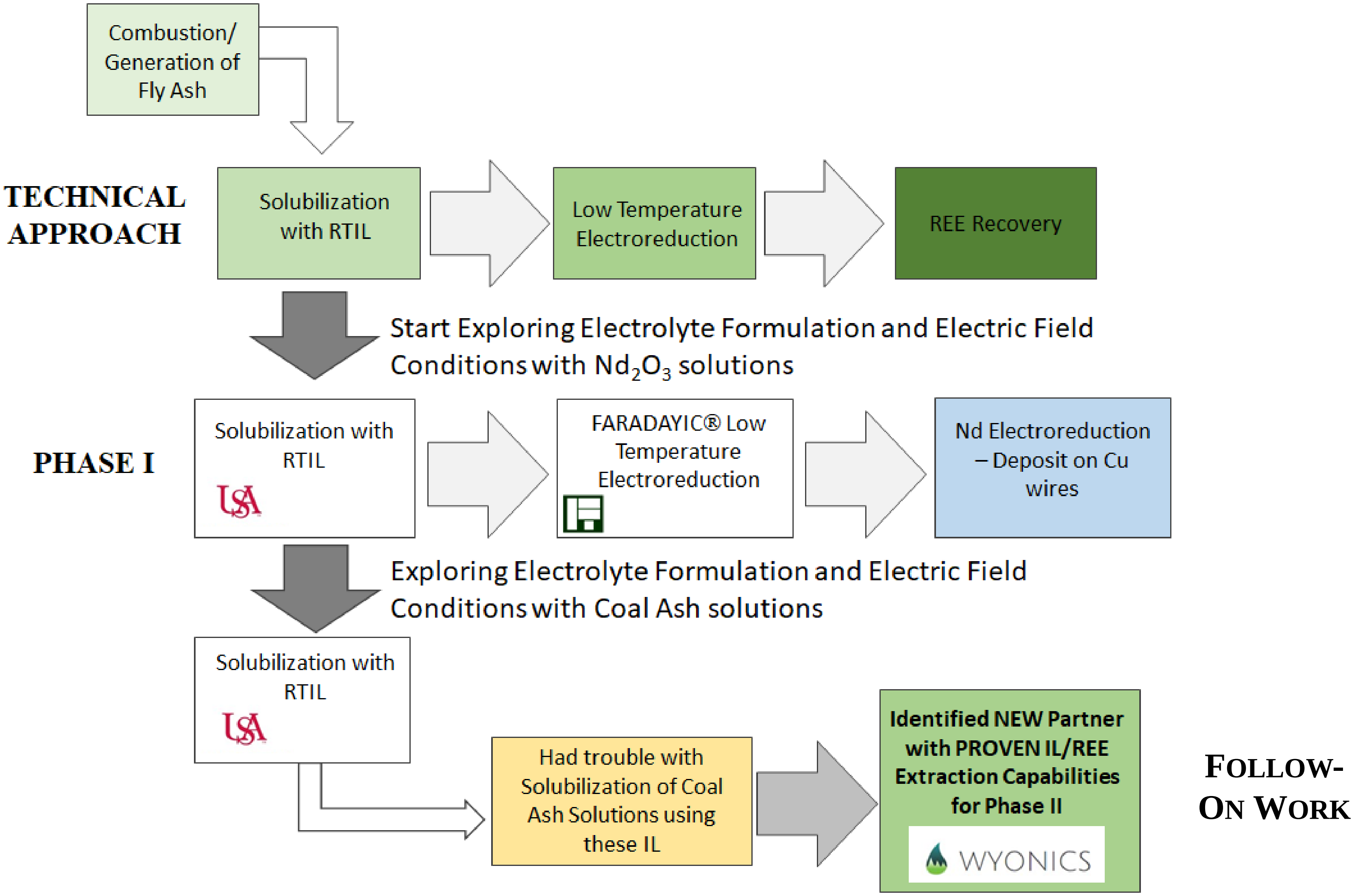

%Extracted REEs is influenced by starting coal:IL ratio

Nd species reduction peak

Follow-On Work

- Faraday has demonstrated the ability to reduce Nd from model IL/Nd oxide solutions using the FARADAYIC® Process
- Partner with proven extraction technology, such as Wyonics' proprietary process, proven to extract REEs from coal-based resources (they've demonstrated their extraction process for coal, coal ash and shale feedstocks)
- Combination of Faraday's novel low-temperature reduction and Wyonics' proprietary extraction → low temperature method to recover rare earth metals from coal ash using novel ionic liquid solutions and simple electrochemical methods

Technical Approach



Combustion/ Generation of Fly Ash

TECHNICAL APPROACH

Solubilization with RTIL

Low Temperature Electroreduction

REE Recovery

Start Exploring Electrolyte Formulation and Electric Field Conditions with Nd_2O_3 solutions

PHASE I

Solubilization with RTIL

FARADAYIC® Low Temperature Electroreduction

Nd Electroreduction – Deposit on Cu wires

Exploring Electrolyte Formulation and Electric Field Conditions with Coal Ash solutions

Solubilization with RTIL

Had trouble with Solubilization of Coal Ash Solutions using these IL

Identified NEW Partner with PROVEN IL/REE Extraction Capabilities for Phase II

WYONICS

FOLLOW-ON WORK

Patent Landscape

Preliminary assessment of the **intellectual property landscape** associated with the FARADAYIC® REE ElectroRecovery in combination with Wyonics REE Extraction **method** and **apparatus**