Scale-Up of Magnesium Production by Fully Stabilized Zirconia Electrolysis

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INFINIUM, Inc.
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Project ID: LM035

Overview

TIMELINE
Project start date: 10/1/2011
Project end date: 12/31/2015
Percent complete: 65%

BUDGET
Total project funding: $12M
- $6M DoE
- $6M INFINIUM

Budget Period 1
- $2,000,000 DoE
- $2,027,924 INFINIUM

Budget Period 2
- $2M DoE
- $2M INFINIUM

Budget Period 3
- $1M DoE
- $1M INFINIUM

BARRIERS
Clean & cost-effective magnesium production

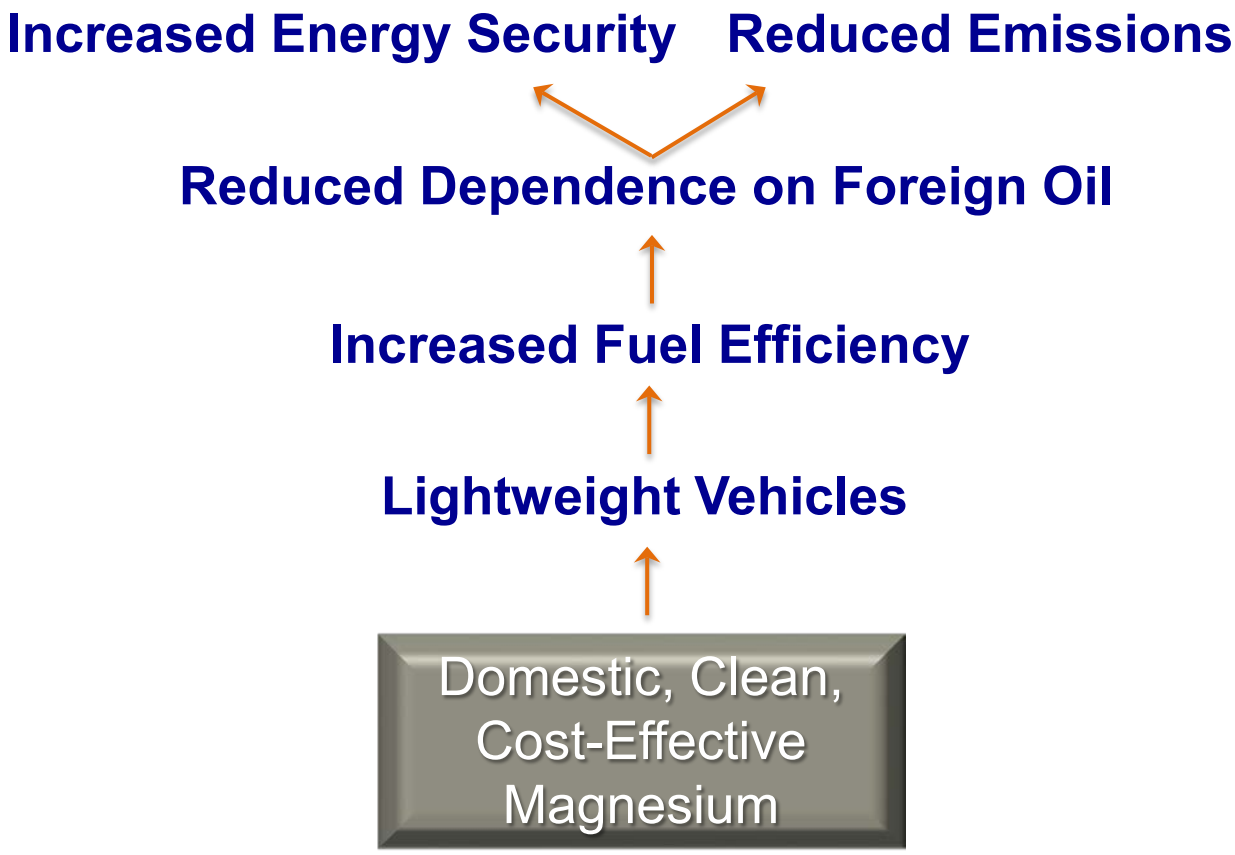
PARTNERS
Praxair, Inc.
Kingston Process Metallurgy
Boston University
Exothermics, Inc.
Spartan Light Metal
Cosma International, Automotive Partnerships Canada

Relevance

Objectives

- Scale up INFINIUM's primary magnesium production from laboratory demonstration to pre-production pilot plant
- Budget Period 2
  - Design, build, & test beta prototype
  - Achieve prototype-scale anode manufacturing
  - Produce magnesium; make & test parts
  - Model plant costs, energy use, & emissions

Relevance



Approach

Phase 1: Alpha Prototype

- Design, build, & test alpha prototype
- Optimize anode design
- Calculate costs, energy use, & emissions
- Produce & test magnesium
- Initiate plant design

Phase 2: Beta Prototype

- Design, build, & test beta prototype
- Achieve prototype-scale anode manufacturing
- Produce magnesium; make & test parts
- Model plant costs, energy use, & emissions

Phase 3: Prototype Operation & Plant Design

- Develop automated processes for alpha & beta prototypes
- Prepare for plant-scale anode manufacturing
- Produce & test magnesium automotive parts
- Model full lifecycle costs, energy use, & emissions

Approach

Due	PHASE 1 MILESTONES	Status
Nov 2012	Conduct electrolysis in alpha	Complete
Nov 2012	Demonstrate stable, O_2-producing anode assembly	Complete
Nov 2012	Calculate economically viable costs, energy use, & emissions	Complete
Nov 2012	Achieve sufficient purity to meet Mg alloy specifications	Complete
Nov 2012	Identify potential plant site(s)	Complete

Approach

Due	PHASE 2 MILESTONES	Status
Nov 2013	Conduct electrolysis in beta	Extended to Jun 2014
Nov 2013	Produce sufficient anode assemblies for prototypes	Complete
Nov 2013	Provide sufficient Mg for tensile testing	Extended to Oct 2015
Nov 2013	Model plant site	Complete

Approach

Due	PHASE 3 MILESTONES	Status
Nov 2014	Achieve industry uptime standard for prototypes	Extended to Nov 2015
Nov 2014	Demonstrate scalable anode assembly manufacturing	Extended to Nov 2015
Nov 2014	Demonstrate satisfactory Mg performance in automotive parts	Extended to Nov 2015
Nov 2014	Model Mg process economics	On Schedule

Technical Accomplishments & Progress

Phase 2

- **Design, build & test prototypes**
- Anode optimization & manufacturing
- Produce Mg; make & test parts
- Model plant costs, energy use, emissions

Alpha 2.0 Specifics

- 1 Electrolysis Site
- Continuous Mg condenser w/ successful pours
- Longest continuous uptime 672 hrs

Technical Accomplishments & Progress

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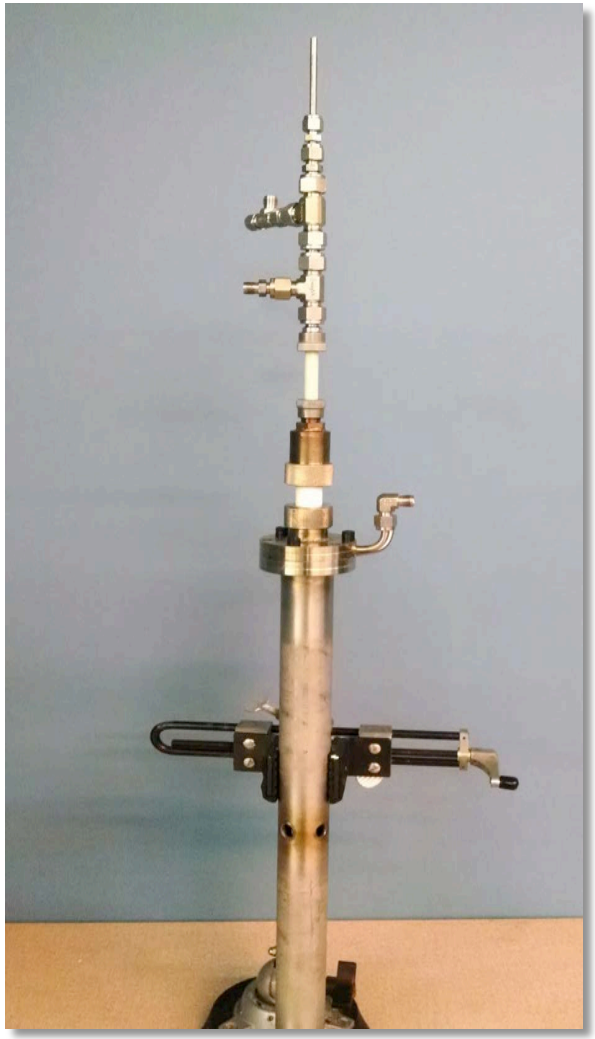
Integrated Anode-Cathode

- Low anode-cathode distance
- Integrates argon bubble curtain
- Promotes fast oxide ion mass transfer to anode
- Facilitates tube hot-swapping



Technical Accomplishments & Progress

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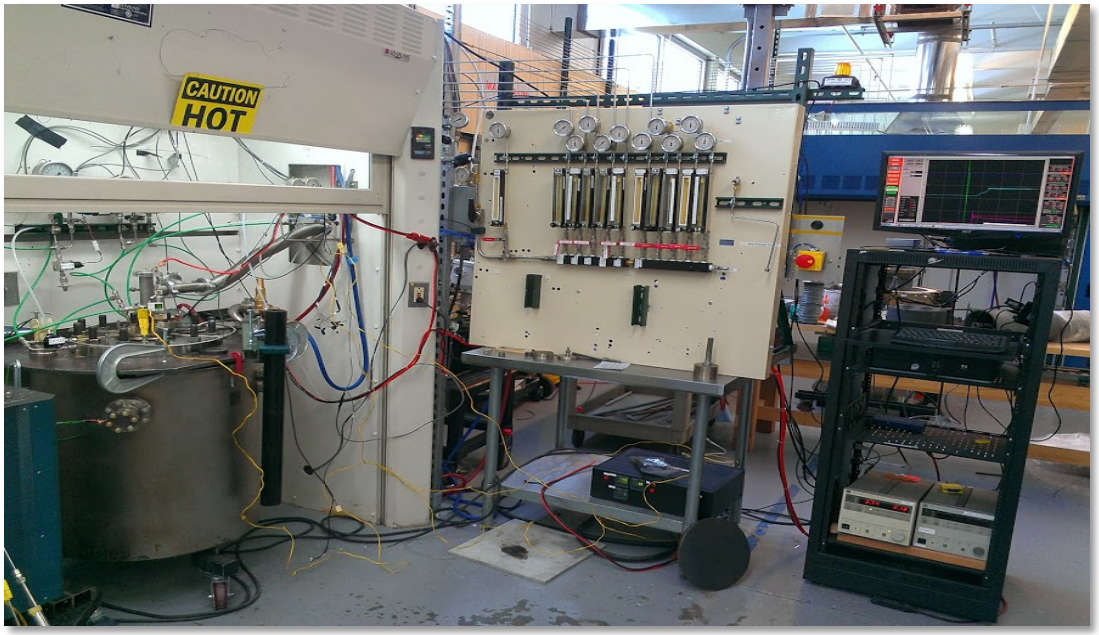

Alpha 3.0 Specifics

- 2 anode-cathode assembly Sites
- Reconfigured condenser for easier casting
- New casting system with controlled cooling
- Longest continuous uptime 2448 hrs

Technical Accomplishments & Progress

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Alpha 3.0 Accomplishments

- Uptime Platform
- Furnace ran continuously for 2448 hrs
- Used same flux for 3168 hrs
- Reduced anode-cathode assembly loading time
- Produced & diecast condensed Mg into solid

"Mg Credit Card"

Technical Accomplishments & Progress

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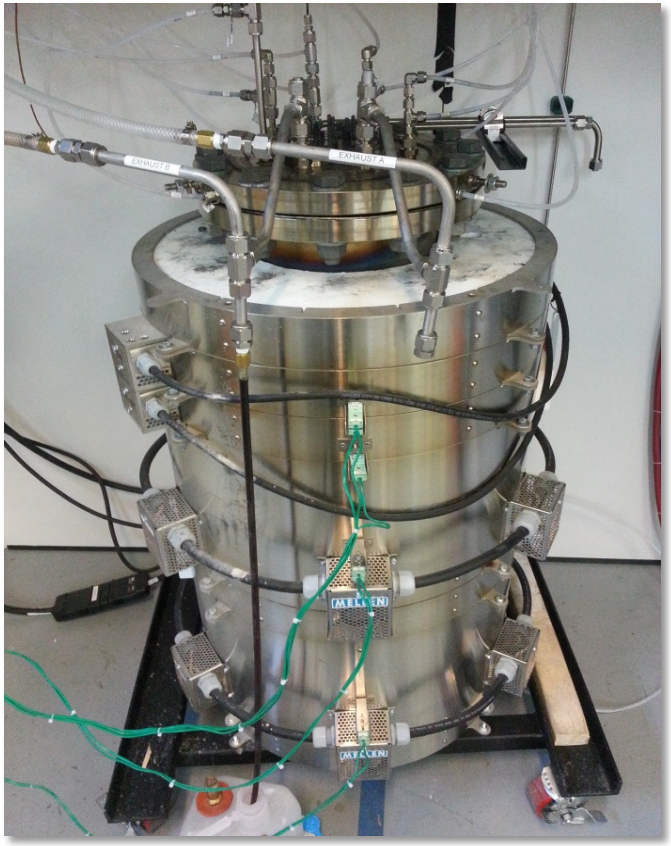
Beta 1.0



Beta 1.0 – NOV 2013

- Scale-up platform
- 4 anode-cathode assembly sites
  - Furnace can hold 10-20 sites
- Increased immersion depth to 12”
- Expanded batch condenser
- Programmable Logic Controller

Technical Accomplishments & Progress

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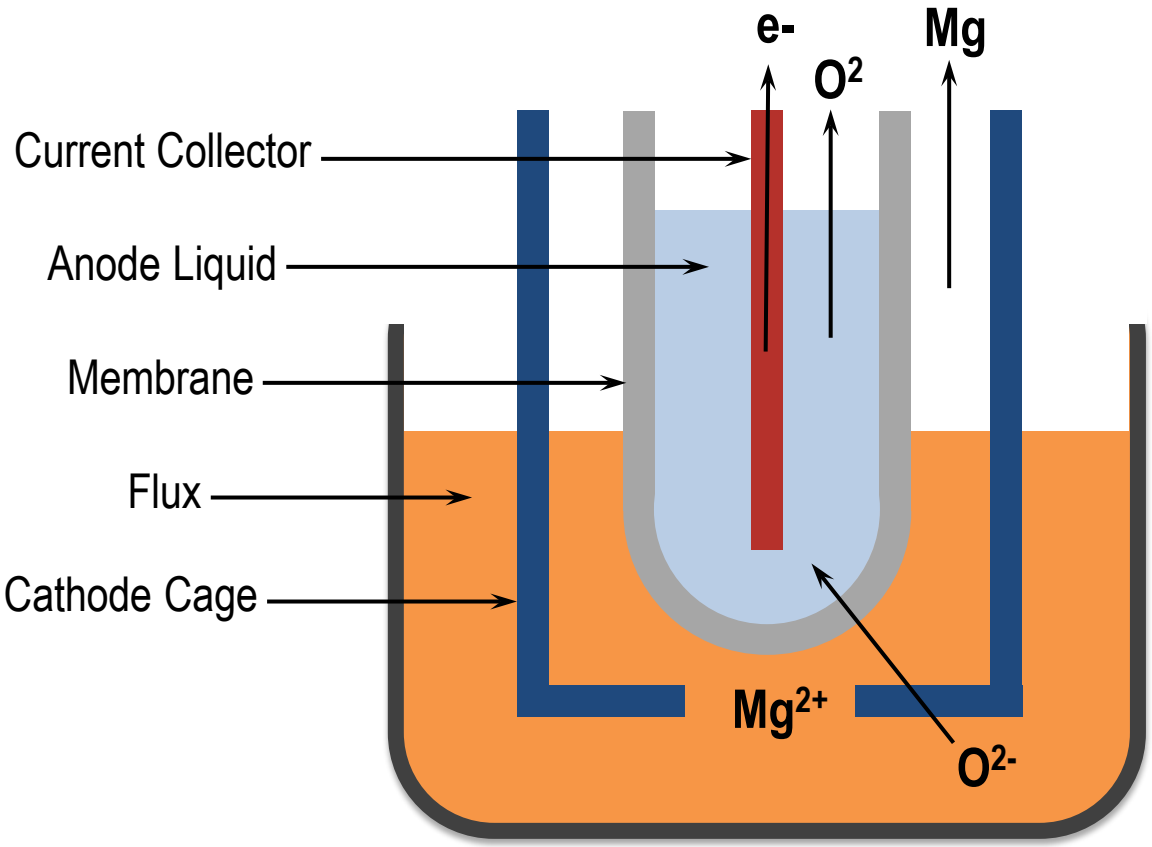
Pure Oxygen Anode™ Assembly



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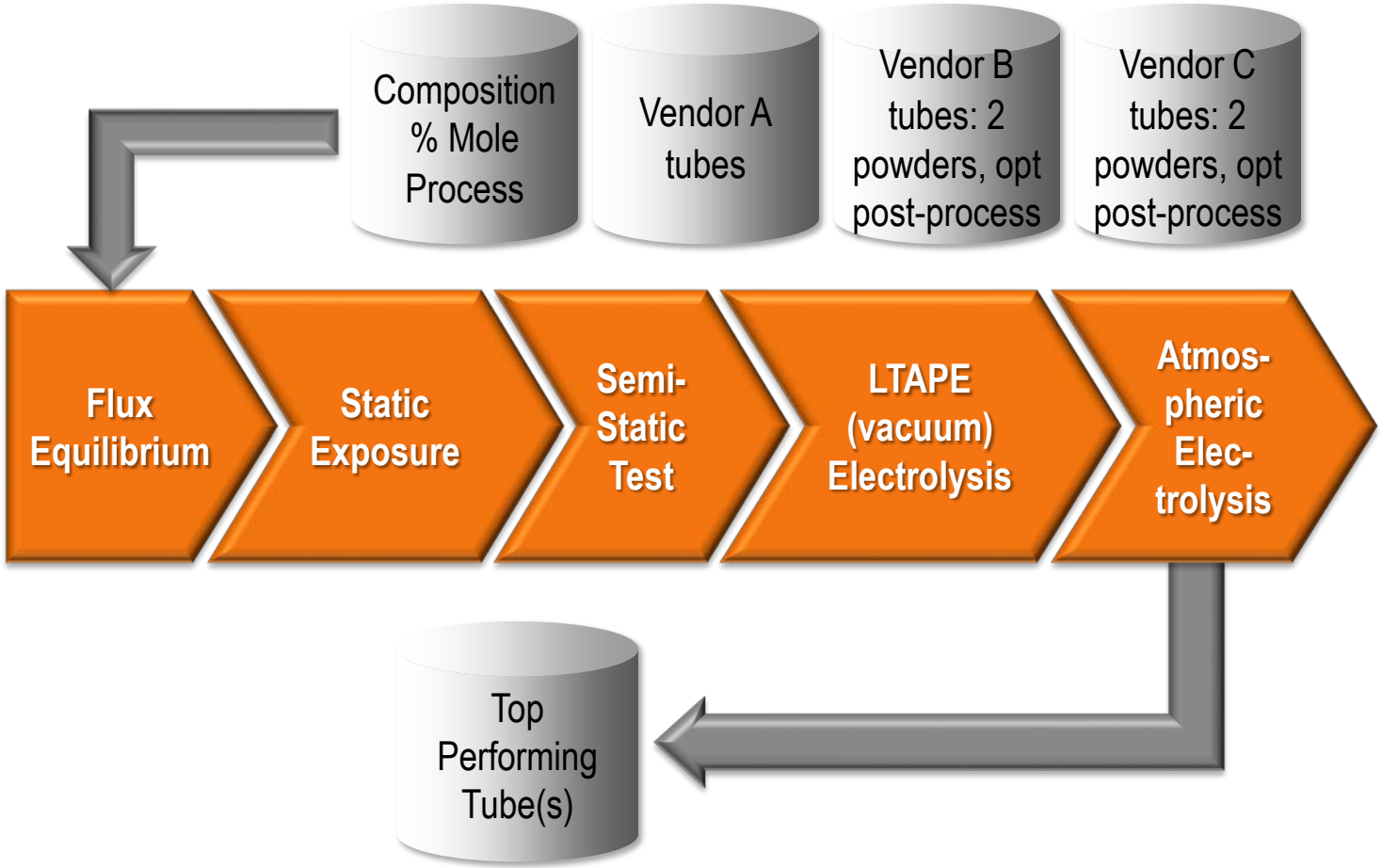

Technical Accomplishments & Progress

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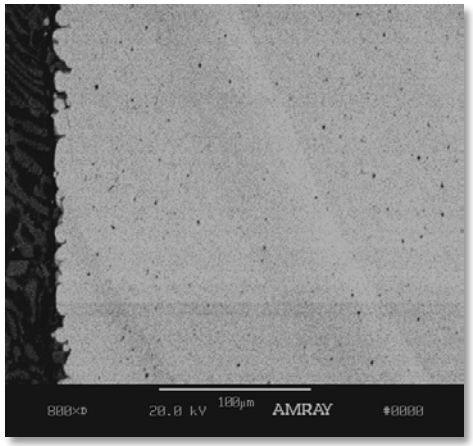
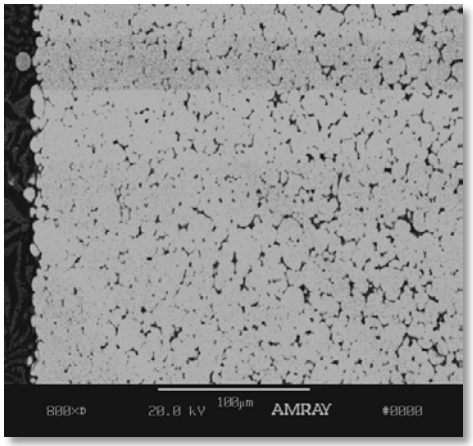
Static Zirconia Tests
Samples soaked at 1150C



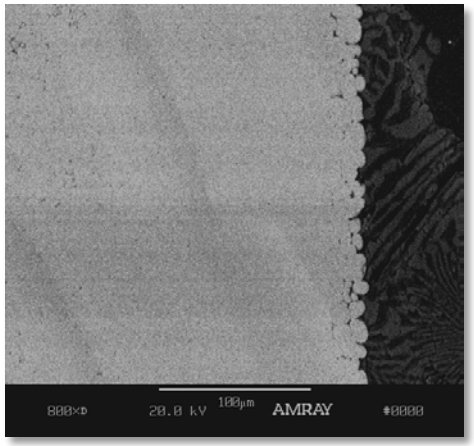
Vendor A

- Porosity does not appear to have increased significantly
- Minimal surface degradation



Vendor B

- Severe grain boundary attack throughout entire sample
- Salt appears to have penetrated entire sample



Vendor C

- Lowest internal porosity
- Salt does not appear to have penetrated sample
- Band of slight inter-granular porosity needs further study

Technical Accomplishments & Progress

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"Top Hat"

Semi-Static Testing Prototype

- 6 different full-length tubes & flux combinations simultaneously
- Correlate tube electrochemical performance to changes in salt composition before, during, & after soaking

Technical Accomplishments & Progress

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Zirconia Manufacturing

- In-house tubes 99.5% dense, exceed best COTS tubes at 97%
- Acquired sintering furnace onsite w/ 100% yield
- Maintaining inventory of COTS tubes, plus tubes manufactured in-house from 3 powder compositions

Technical Accomplishments & Progress

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Inert Anode Current Collector

- Fabricated hybrid LSM/nickel current collectors capable of running for extended periods of time in an oxygen atmosphere
- Mass spectrometry detected negligible impurities in oxygen gas from anodes

Technical Accomplishments & Progress

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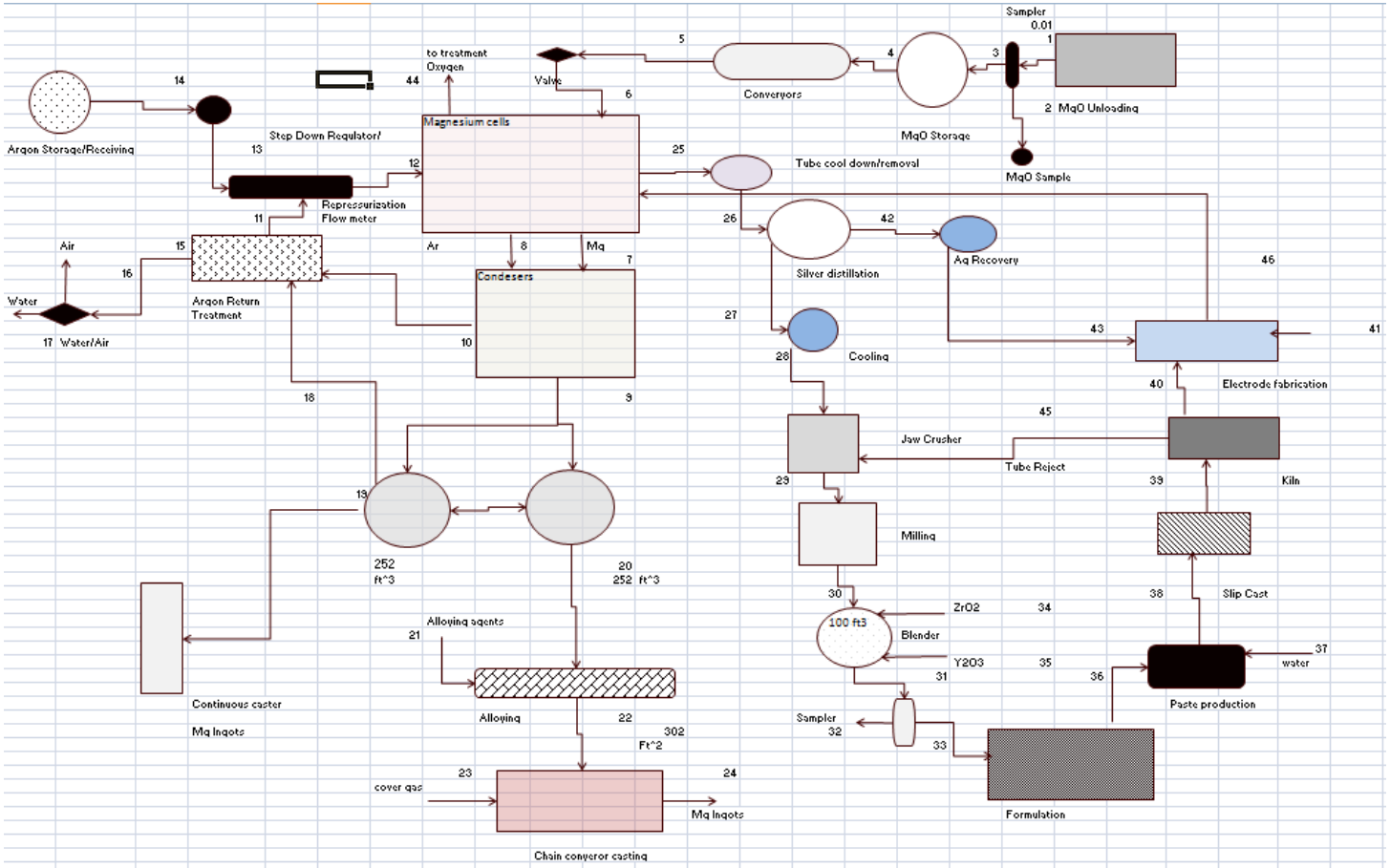
Full Process Flow Diagram



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Technical Accomplishments & Progress

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Cost Modeling

- Silver evaporation model shows silver losses ~0.76¢/kg Mg product, silver can be recaptured
- Plant Narrative describes cell layout, power supplies, cast house, etc. for detailed heat balance and cost model

Also in progress:

- Detailed capital cost model, coming in well below earlier estimates
- Detailed heat balance
- Sensitivity analysis

Using cost modeling to drive R&D

Collaboration & Coordination w/Other Institutions

- **Kingston Process Metallurgy**: contract R&D including transparent crucible electrolysis, salt recycling
- **Boston University**: contract R&D including current collector, salt-metal interactions, current efficiency improvements
- **Praxair**: process gases, argon recycling R&D, thermal modeling
- **Exothermics**: zirconia production/analysis, current collector R&D
- **Spartan Light Metals**: product testing by die-casting tensile specimens and other parts
- **Vehma**: product testing including die-casting vehicle components and testing those components in vehicle structures

Proposed Future Work

Phase III Tasks

- Develop automated processes for Alpha & Beta prototypes
- Prepare for plant-scale anode manufacturing
- Produce & test magnesium automotive parts
- Model full lifecycle costs, energy use, & emissions

Publications/ Presentations/Patents

- Powell, A. “Magnesium in Transportation: Unlocking Limitless Potential Through Primary Production and Recycling,” ARPA-E A-TEME (later METALS) Workshop January 31, 2013.
- Powell, A. “Modeling Electrodeposition in Materials Process Operations,” TMS Annual Meeting Short Course March 3, 2013.
- Pal, Uday “Electrolytic Production of Metals from Oxides Dissolved in Molten Salts,” TMS Annual Meeting High Temperature Electrochemistry Symposium March 4, 2013.
- Powell, Adam “Systems Engineering for Scale-Up of the INFINIUM™ MagGen™ Primary Magnesium Production System,” Reactive Metal Workshop March 8, 2013.
- Pal, Uday “Low Carbon Footprint Process for Metals Production,” Reactive Metal Workshop March 8, 2013.
- Milshtein, J. et al. “Yttria Stabilized Zirconia Membrane Stability in Molten Fluoride Fluxes for Low-Carbon Magnesium Production by the SOM Process,” J. Min. Metall. Sect. B 49(2):183-190, 2013.
- Guan, X. et al. “An Environmentally Friendly Process Involving Refining and Membrane-Based Electrolysis for Magnesium Recovery from Partially Oxidized Scrap Alloy,” JOM 65(10):1285-1292 October 2013.
- Guan, X. et al. “LSM (La0.8Sr0.2MnO3-δ)-Inconel Inert Anode Current Collector for Solid Oxide Membrane (SOM) Electrolysis” J. Electrochem Soc. 160(11):F1179-F1186, 2013.

Summary

- Significant progress: prototype engineering, anode-cathode assembly, anode fabrication
- Focus on longer term Anode operation
- Larger-scale operation and production in plans for 2014-2015