

Scale-Up of Magnesium Production by Fully Stabilized Zirconia Electrolysis

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Overview



TIMELINE

Project start date: 10/1/2011 Project end date: 12/31/2015 Percent complete: 83%

BUDGET

Total project funding: \$12M

- \$6M DoE
- \$6.03M INFINIUM
 Funding Received in FY14
 - \$1M DoE
 - \$1M INFINIUM

Funding for FY15

- \$1M DoE
- \$1M INFINIUM

BARRIERS

Magnesium supply base: Inexpensive and clean domestic source of magnesium

PARTNERS

INFINIUM, Inc. – Project Lead Praxair, Inc. Kingston Process Metallurgy Boston University Exothermics, Inc. Spartan Light Metal Cosma International, Automotive Partnerships Canada MagPro, LLC

Relevance



Objectives

- Scale up INFINIUM's primary magnesium production from laboratory demonstration to pre-production pilot plant
- Budget Period 3
 - Achieve industry standard uptime for prototypes
 - Prepare for plant-scale anode manufacturing
 - Produce and test magnesium automotive parts
 - Model full life cycle costs, energy use & emissions







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

- Achieve industry standard uptime for prototypes
- Prepare for plant-scale anode manufacturing
- Produce & test magnesium automotive parts
- Model full lifecycle costs, energy use & emissions





Phase, Due	Project MILESTONES	Status
1 Nov 2012	Conduct electrolysis in alpha	Complete
1 Nov 2012	Demonstrate stable, O ₂ -producing anode assembly	Complete
1 Nov 2012	Calculate economically viable costs, energy use, & emissions	Complete
1 Nov 2012	Achieve sufficient purity to meet Mg alloy specifications	Complete
1 Nov 2012	Identify potential plant site(s)	Complete
2 Nov 2013	Conduct electrolysis in beta	Extended to 6/2015
2 Nov 2013	Produce sufficient anode assemblies for prototypes	Complete
2 Nov 2013	Provide sufficient Mg for tensile testing	Extended to 10/2015
2 Nov 2013	Model plant site	Complete
3 Nov 2014	Achieve industry uptime standard for prototypes	Extended to 11/2015
3 Nov 2014	Prepare for plant scale anode manufacturing	Extended to 11/2015
3 Nov 2014	Produce and test magnesium automotive parts	Extended to 11/2015
3 Nov 2014	Model full life cycle costs, energy use, and emissions	On Schedule

New Approach



- Shift in project approach: primary production of magnesium for Mg-Nd master alloy
 - Necessary for AE44, WE43, ZEK100, other alloys
 - Can't be reliably sourced in US today
 - Direct electrolytic primary production from low-cost oxides simplifies alloying
 - Best first-product for INFINIUM Mg
 - Primary magnesium remains on the radar
- New project goal: produce 500 lbs of primary magnesium and Mg-Nd master alloy for WE43 alloy die casting trial (650 lbs total alloy)
- Technical advantage: simpler cell
 - Make liquid Mg-Nd master alloy at cathode
 - Focus on electrolysis cell development: bath, electrodes, etc.
 - No need for simultaneous coupled condenser development





Phase 3

- Achieve industry standard uptime for prototypes
- Prepare for plant scale anode manufacturing
- Produce and test magnesium automotive parts
- Model life cycle costs, energy use, emissions



Alpha Magnesium Furnace

- Version 2.0 2012: one electrolysis site, continuous condenser with successful pours
- Version 3.0 2013: two electrolysis sites, integrated anode-cathode assembly with hot swapping
- Version 4.0 2014: three ports, longest anode run time



Phase 3

Achieve
industry

standard uptime for prototypes

- Prepare for plant scale anode manufacturing
- Produce and test magnesium automotive parts
- Model life cycle costs, energy use, emissions

- Magnesium Master Alloy
 - Finalized master alloy process flow sheet
 - Finalized molten salt bath chemistry after testing four composition classes
 - Demonstrated first alpha-scale master alloy production
 - Preliminary beta-scale design, equipment sizing, 500 lbs production timeline





Phase 3

Achieve industry standard uptime for prototypes

- Prepare for plant scale anode manufacturing
- Produce and test magnesium automotive parts
- Model life cycle costs, energy use, emissions

Gamma prototype

- Neodymium production cell for master alloy
- Production of 650 lbs WE43 alloy will require approx. 150 lbs of Nd
 - 50 lbs in process on-site
 - 50 lbs in process off-site
 - 50 lbs in transit





- Production of larger anode tubes
- Phase 3
- Achieve industry standard uptime for prototypes
- Prepare for plant scale anode manufacture
- Produce and test magnesium automotive parts
- Model life cycle costs, energy use, emissions

- Larger tube diameter facilitates higher oxygen flow rate and larger current collector
- Cast tubes with 1" and 1.25" outer diameter (cf. older 0.75" tubes)
- Maintained very low porosity (0.5% cf. best COTS >2%), 100% process yield











High-current anode assembly design optimization Modeling goals:

- Optimize lead geometry for minimal energy use (resistance & heat)
- Displacing solid design
- Thermal stress, oxygen flow

manufacture New model:

Phase 3

Achieve

industry

standard

uptime for

prototypes

anode

parts

Produce and

Model life

cycle costs.

energy use,

emissions

test magnesium automotive

 Prepare for plant scale

- 200-400 A industrial tube design
- Axisymmetric, very fast
- Temperature-dependent properties
- Parameterized geometry

Result: optimal electrical-thermal resistance balance



Phase 3

- Achieve industry standard uptime for prototypes
- Prepare for plant scale anode manufacture
- Produce and test magnesium automotive parts
- Model life cycle costs, energy use, emissions

- Temperature-dependent
 - properties
 - Cell and electrode geometry

Embedded in full cost model

Results: global cost vs.

- Temperature
- Tube material type, thickness, diameter
- Current density
- Anode and current collector geometry
- Optimize parameters over entire system





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Response to 2014 Reviewers' Comments

- Is anode lifetime the rate-limiting step? Anode lifetime is an important factor determining production cost. Anode size is the rate-limitation in terms of kg/hr.
- Extended timeline is delaying return on investment, there are many opportunities for pure Mg
 We've made great progress on a technology challenge, and will continue to advance it toward timely deployment
- Can this be integrated with downstream processes e.g. rolling, extrusion?
 Typically the most practical integration is to transport liquid metal from reduction cells directly to the downstream process

- How does anode life here compare with other electrolysis processes?
 Other processes e.g. Hall-Héroult aluminum use a consumable anode with lifetime in hours cf. months for our technology
- What alloys can be produced by this method?
 Potentially many others depending on properties.
- Where is this project headed next? Production of 650 lbs WE43 alloy, testing in automotive parts, feasibility study for master alloy production plant



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, saltmetal 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
- **MagPro**: large batch alloy melting/blending, other processing

Proposed Future Work



Complete Phase III Tasks

- Conduct electrolysis in reconfigured beta system
- Produce 500 lbs primary Mg from MgO
- Produce 650 lbs WE43 alloy for casting
- Make & test magnesium automotive parts
- Model full lifecycle costs, energy use, & emissions





- Shift to magnesium-neodymium master alloy
- Extended cell operation time
- New larger higher-current anodes
- Focus on longer term Anode operation
- Larger-scale operation and production in plans for 2014-2015

Publications/ Presentations/Patents



- Powell, A. et al., "Apparatus and method for condensing metal vapor," U.S. Patents 8,617,457 and 8,926,727 issued December 31, 2013 and January 6, 2015.
- Powell, A. et al., "Pure Oxygen Anodes™ for Low- or Zero-Carbon Energy Efficient Metal Oxide Reduction," Presented at TMS Annual Meeting Energy and Carbon in Metal Production Symposium, February 18, 2014.
- Guan, X. et al., "Electrochemical Characterization and Modeling of a Solid Oxide Membrane-Based Electrolyzer for Production of Magnesium," Presented at TMS Annual Meeting Celebrating the Megascale, February 19, 2014.
- Powell, A. et al., "Pure Oxygen Anodes for Efficient Clean Production of Reactive Metals," Presented at Reactive Metal Workshop February 21-22, 2014.
- Gratz, E. et al., "<u>Mitigating Electronic Current in Molten Flux for the Magnesium SOM</u> <u>Process</u>," Metall. Mater. Trans. 45B(4):1325-1336 2014.
- Guan, X. et al., "<u>Energy-Efficient and Environmentally Friendly Solid Oxide Membrane</u> <u>Electrolysis Process for Magnesium Oxide Reduction</u>," Metall. Mater. Trans. 1E(2):132-144 2014.
- Guan, X. et al. "<u>Periodic Shorting of SOM Cell to Remove Soluble Magnesium in Molten</u> <u>Flux and Improve Faradaic Efficiency</u>," Metall. Mater. Trans. 45B(6):2138-2144 2014.