Scale-Up of Magnesium Production by Fully Stabilized Zirconia Electrolysis

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INFINIUM, Inc.
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Project ID: LM035

This presentation does not contain any proprietary, confidential, or otherwise restricted information.
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

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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
Increased Energy Security  Reduced Emissions

Reduced Dependence on Foreign Oil

Increased Fuel Efficiency

Lightweight Vehicles

Domestic, Clean, Cost-Effective Magnesium

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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

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## Approach

<table>
<thead>
<tr>
<th>Due</th>
<th>PHASE 1 MILESTONES</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nov 2012</td>
<td>Conduct electrolysis in alpha</td>
<td>Complete</td>
</tr>
<tr>
<td>Nov 2012</td>
<td>Demonstrate stable, $O_2$-producing anode assembly</td>
<td>Complete</td>
</tr>
<tr>
<td>Nov 2012</td>
<td>Calculate economically viable costs, energy use, &amp; emissions</td>
<td>Complete</td>
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<tr>
<td>Nov 2012</td>
<td>Achieve sufficient purity to meet Mg alloy specifications</td>
<td>Complete</td>
</tr>
<tr>
<td>Nov 2012</td>
<td>Identify potential plant site(s)</td>
<td>Complete</td>
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## Approach

<table>
<thead>
<tr>
<th>Due</th>
<th>PHASE 2 MILESTONES</th>
<th>Status</th>
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<tbody>
<tr>
<td>Nov 2013</td>
<td>Conduct electrolysis in beta</td>
<td>Extended to Jun 2014</td>
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<tr>
<td>Nov 2013</td>
<td>Produce sufficient anode assemblies for prototypes</td>
<td>Complete</td>
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<tr>
<td>Nov 2013</td>
<td>Provide sufficient Mg for tensile testing</td>
<td>Extended to Oct 2015</td>
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<tr>
<td>Nov 2013</td>
<td>Model plant site</td>
<td>Complete</td>
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</tbody>
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### Approach

<table>
<thead>
<tr>
<th>Due</th>
<th>PHASE 3 MILESTONES</th>
<th>Status</th>
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</thead>
<tbody>
<tr>
<td>Nov 2014</td>
<td>Achieve industry uptime standard for prototypes</td>
<td>Extended to Nov 2015</td>
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<tr>
<td>Nov 2014</td>
<td>Demonstrate scalable anode assembly manufacturing</td>
<td>Extended to Nov 2015</td>
</tr>
<tr>
<td>Nov 2014</td>
<td>Demonstrate satisfactory Mg performance in automotive parts</td>
<td>Extended to Nov 2015</td>
</tr>
<tr>
<td>Nov 2014</td>
<td>Model Mg process economics</td>
<td>On Schedule</td>
</tr>
</tbody>
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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

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Technical Accomplishments & Progress

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

Phase 2
- Design, build & test prototypes
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Technical Accomplishments & Progress

Phase 2
- Design, build & test prototypes
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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

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Technical Accomplishments & Progress

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

Phase 2
- Design, build & test prototypes
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- Produce Mg; make & test parts
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“Mg Credit Card”
Technical Accomplishments & Progress

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

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Technical Accomplishments & Progress

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Pure Oxygen Anode™ Assembly

Current Collector
Anode Liquid
Membrane
Flux
Cathode Cage

O₂⁻
Mg²⁺
e⁻
**Technical Accomplishments & Progress**

**Phase 2**
- Design, build & test prototypes
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**Diagram:**
- Flux Equilibrium
- Static Exposure
- Semi-Static Test
- LTAPe (vacuum) Electrolysis
- Atmospheric Electrolysis

**Vendor A tubes:**
2 powders, opt post-process

**Vendor B tubes:**
2 powders, opt post-process

**Vendor C tubes:**
2 powders, opt post-process

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Technical Accomplishments & Progress

Static Zirconia Tests
Samples soaked at 1150C

Phase 2
- Design, build & test prototypes
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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 intergranular porosity needs further study

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Technical Accomplishments & Progress

“Top Hat”

Phase 2
- Design, build & test prototypes
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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

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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**

**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

**Phase 2**

- Design, build & test prototypes
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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
• Powell, Adam “Systems Engineering for Scale-Up of the INFINIUM™ MagGen™ Primary Magnesium Production System,” Reactive Metal Workshop March 8, 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.

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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