Key & Critical Material Recycling
Advanced Batteries, lithium to lanthanoids
EPA Region 8 Rare Earth Elements Workshop, May 10, 2012

Steven E. Sloop
OnTo Technology LLC

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Overview, Introductions

• OnTo Technology Company
• Nickel Metal Hydride
  – Chemistry
  – Recycling Approaches
• Advanced Battery Recycling Developments
  – NiMH
  – Lithium-ion
    • Co, Mn, Fe based chemistries with critical and key elements
OnTo Technology Company

Advanced Battery Innovations and Materials Recycling

• Licensing and royalties business model
• The spectrum of technologies
  – Decommission for Safety
  – Disassembly processes
  – Direct rejuvenation of recycled materials
  – In-situ rejuvenation of whole batteries
• Alkaline
  – Single use and rechargeable
  – Nickel Metal Hydride: HEV, Plug-HEV, stationary storage
• Lithium
  – Small format: rechargeable & single use
  – Large format: HEV, EV, E-Bike, stationary storage
  – Repairing off spec. material
## OnTo Technology Business

*Widely covered patent pending positions for license*

*Contract research and development*

### Activity/Area

<table>
<thead>
<tr>
<th>Activity/Area</th>
<th>Li-ion cells</th>
<th>Li-Primary cells</th>
<th>Off-Spec-Cathode material</th>
<th>Metal Hydride</th>
<th>Alkaline</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Battery Decommission</strong></td>
<td>Available for Full and hybrid E vehicle, bike, grid</td>
<td>Available for Industrial staves, remote power</td>
<td>(N/A)</td>
<td>Available for Hybrid E vehicle, bike, grid</td>
<td>Available for Consumer / municipal sourced</td>
</tr>
<tr>
<td><strong>Material Rejuvenation</strong></td>
<td>Available 8/12</td>
<td>Available 8/12</td>
<td>Available</td>
<td>Available</td>
<td>-</td>
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<tr>
<td><strong>Battery Rejuvenation</strong></td>
<td>Available for Full and hybrid E vehicle, bike, grid</td>
<td>-</td>
<td>(N/A)</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

### Customers:
- Vehicle Recycling Partnership
- Environmental Protection Agency
- National Science Foundation
- US Department of Energy
- California Center for Sustainability
- Sumitomo Corporation of America
- InvenTek
- EcoBat
- Oregon Nanoscience and Microtechnologies Initiative
- LG Chem / Apple
- Tesla
Nickel Metal Hydride Battery

What is AB5? An overview on nano-scopic scale

β-Ni(OH)$_2$ & Co(OH)$_2$ proton host

- Positive electrode composed of nickel oxyhydroxide, and 5-10% cobalt oxyhydride

AB5 metal alloy, hydride host

A=La, Ce, Nd, Pr...
B= Ni, Co, Mn, Al

- Negative electrode, AB5
- Problem 1: Rare Earth Elements become expensive 2010
- Problem 2: REE recycling

M(OH)$_2$ + OH$^-$ $\rightleftharpoons$ MO(OH) + H$_2$O + e$^-$
(Charge cycle)

M + H$_2$O + e$^-$ $\rightleftharpoons$ MH + OH$^-$
(Charge cycle)

Blue : A type, Red, Green: B type

Tetrahedra: H Storage Sites

KOH / H$_2$O electrolyte

Proton to hydride charge cycle

NiO$_2^{2-}$

H$^+$
Metal Hydride Low Cost Development

Manufacturing Response to High Priced Rare Earth Elements

High Prices, 2011
- Neodymium: $250/kg
- Praseodymium: $250/kg
- Cerium: $140/kg
- Lanthanum: $140/kg

Situation: Mining of REE’s is 95% in China: now exports are severely limited.

Recent History: inexpensive REE’s with good availability from China.

Conventional AB5 alloy $82/kg
AB5 without Pr, Nd $59/kg
AB2 $19/kg

Elimination of Pr, Nd reduces AB5 formula 13%, AB2 alloy reduces costs 72% and increases capacity 26%

M. Fetcenko, 29th International Battery Seminar (2012)
Rare Earths, Lanthanides

*light, Group I, lanthanides are used in AB5 alloys*

Groups according to MP, BP, VP & radioactivity*

<table>
<thead>
<tr>
<th>Group I</th>
<th>Group II</th>
<th>Group III</th>
<th>Group IV</th>
</tr>
</thead>
<tbody>
<tr>
<td>La</td>
<td>Gd</td>
<td>Dy</td>
<td>Sm</td>
</tr>
<tr>
<td>Ce</td>
<td>Tb</td>
<td>Ho</td>
<td>Er</td>
</tr>
<tr>
<td>Pr</td>
<td>Sc (REE)</td>
<td>Er</td>
<td>Yb</td>
</tr>
<tr>
<td>Nd</td>
<td>Y (REE)</td>
<td>Sc (REE)</td>
<td>Tm</td>
</tr>
<tr>
<td></td>
<td>Lu</td>
<td>Group V</td>
<td>Actinides</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Pm*</td>
<td>Ac*, Th*, U*</td>
</tr>
</tbody>
</table>

**AB5 metallic alloy**

*A=La, Ce, Nd, Pr (group I, low MP, high BP)*

USGS Fact Sheet 87-02
Rare Earth, Lanthanide processing

general overview, how are RE metals separated from minerals?
Chemically similar, all are easily oxidized

Acid opened \( \text{Ln}_2(\text{SO}_4)_3 \) or Base opened \( \text{Ln}_2\text{O}_3 \) Followed by thorium removal

NH\(_4\)OH

Th salts ppt

HCl

Radioactive Th removal

Th\(_{\text{O}_2}\) ppt

Medallion Resources (2012)
Oxide to metal to AB5 alloy

Two pathways to light Ln, Group I metals used in AB5

Heavy and Light Ln’s

Light Ln Oxides
La, Ce, Pr, Nd

Heavy Ln’s

Fractional Crystallization: mixed light / mixed heavy
Ion exchange system: pure light / heavy

Fused Salt Electrolysis:
LnCl$_3$ + 3e$^- \rightarrow$ Ln
Mixed light Ln’s produce ‘mischmetal’

Use HCl to form LnCl$_3$
“Michmetal Chloride LnCl$_3$”

Use HF to form LnF$_3$

Induction melt with Ni, Co, Al
Solidify, crush, heat, grind, sieve

AB5 Alloy Supply for NiMH Manufacture

Metallothermic reduction:
LnF$_3$ + Ca $\rightarrow$ Ln + CaF$_2$
Applicable to Group I, II, III
NiMH life limitations for (-) electrode

Alloy Pulverization & component oxidation

- Note cracks in the SEM of the used electrode
  - (a) Disconnected material
    - Removed from the circuit
  - (b) Phase changes
    - A less-active material, slow H diffusion
  - (c) Surface oxidation
    - Coats the working material, decreases efficiency

H₂O

Current Collector

AB5

AB5

With stuck H

ABx

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NiMH Life Limitations for (+) electrode

- **Beta phase** provides the best performance.

  - **Beta phase** provides the best performance.

- **Phase transformation** hinders performance, consumes electrolyte.

  - Overcharge
    - $\beta$-NiO(OH)
    - $\gamma$-NiO$_x$(OH)$_{c<1}$

  - $\alpha$-Ni(OH)$_2$

Charge

Discharge
HEV WORLDMWIDE IN 2011
LESS THAN 0.9M HEV SOLD

HEV sold per year, M units, worldwide, 2000 - 2011
Penetration of hybrids in the global sales, 2000-2011

HEV Packs ready for Recycling WW

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AB5 value projection in HEV pack recycling

<table>
<thead>
<tr>
<th>AB5 comp.</th>
<th>Weight %</th>
<th>component price/kg</th>
<th>historic component price/kg</th>
<th>Embargo Era Value</th>
<th>Historic value</th>
</tr>
</thead>
<tbody>
<tr>
<td>La</td>
<td>12.50%</td>
<td>140</td>
<td>30</td>
<td>$17.50</td>
<td>$3.75</td>
</tr>
<tr>
<td>Ce</td>
<td>3.20%</td>
<td>140</td>
<td>30</td>
<td>$4.48</td>
<td>$0.96</td>
</tr>
<tr>
<td>Pr</td>
<td>1.50%</td>
<td>250</td>
<td>30</td>
<td>$3.75</td>
<td>$0.45</td>
</tr>
<tr>
<td>Nd</td>
<td>14.90%</td>
<td>250</td>
<td>30</td>
<td>$37.25</td>
<td>$4.47</td>
</tr>
<tr>
<td>Ni</td>
<td>50.20%</td>
<td>30</td>
<td>30</td>
<td>$15.06</td>
<td>$15.06</td>
</tr>
<tr>
<td>Co</td>
<td>10.40%</td>
<td>35</td>
<td>35</td>
<td>$3.64</td>
<td>$3.64</td>
</tr>
<tr>
<td>Mn</td>
<td>5.30%</td>
<td>10</td>
<td>10</td>
<td>$0.53</td>
<td>$0.53</td>
</tr>
<tr>
<td>Al</td>
<td>2.00%</td>
<td>10</td>
<td>10</td>
<td>$0.20</td>
<td>$0.20</td>
</tr>
</tbody>
</table>

100.00%  $82.41 $29.06

With Embargo, High $ REE, there is ‘feasibility’ for recycling

With historic values, nickel is the economic and technical driver
- produce stainless steel
- Possible REO side product

How to maximize the value potential of the metals in the end-of-life batteries?

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Honda & Japan Metals & Chemicals Announcement for Rare Earth Recycle

- Operational April 2012
- Source is their HEV fleet of ~800,000 vehicles and other sources.
  - Near future throughput?
- Japan is the world's largest RE importer.
- Used electronics disposed of in JP: 650,000 tons.
  - Contains 280,000 tons of RE and other metals: $1.03B
Metal Hydride Rare Earth Recovery

OnTo Approach

**Process**
- Decommission
- Disassemble
- Separate (+) and (-)
- Separate (-) oxides and metals
- Reintroduce metals to alloy manufacture
- Make metallic feedstock

**Intellectual Property Position**
- Patent pending methods for decommission, disassembly and separation
- Conversion and reduction dedicated towards metal hydride battery using classical approaches and trade secret methods
Decommission, Disassembly and Recycling Process Overview, NiMH

CO2 decommission

High RE content material From (-) electrode

Components

Article ready for machine work

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Metal Hydride Recycling Safety

OnTo Solution for flammability of negative electrode, US Patent & PCT Pending

Current processing:
Fire results

OnTo Processing:
Dry, No fire, No solvent

No flames after opening CO₂ rinsed cells.

- “Discharged” NiMH cell catches fire
- LnH + O₂ → LnOₓ + H₂O
- LnH + O₂ → LnOₓ + H₂O

- How? converts KOH to KHCO₃, drop in pH quenches hydrides
- Pierce-point shows carbonate build up
- Cell was charged before CO₂ rinse.
- Environmentally benign
- Rapid process, simple, low cost, patent pending:
Features and benefits of OnTo’s process

**Feature**

- Safe decommission reduces the hazards associated with large format storage batteries
- Flexible to various alkaline chemistries and lithium chemistries
- Low energy input process capability (low temp)

**Benefit**

- Saves $ by avoiding infrastructure for battery hazards.
- Saves $ by avoiding hazards and improved safety.
- Saves $ on high temperature process units.
- Creates a salable product.

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Lithium-ion battery recycling results: Manufacturing with recycled material: Co

- LiCoO$_2$ containing 100% recycled key and critical elements
- Manufacturing Qualification progress
  - Low trace metal (<100ppm)
  - High capacity (150mAh/g)
  - Coating on Al tape
  - Long life cycle full cells
- Low cost process
- Patent pending position

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EV sourced Li-iron phosphate: Recycled and refurbished for a very low cost: Fe

Harvested from a severely faded, abused, large format cell. Original specific capacity of the material is \(~130\) mAh/g
Lithium-ion EV Battery Recycling
Manufacturing with rejuvenated material: Mn

Recycling Process development results

Prototype Cell from Rejuvenated material harvested from Nissan EV
OnTo process developments for cradle to cradle critical materials

- Nickel
- Manganese
- Lithium-Ion
- Cobalt
- Iron
- Safe Decommission
- Critical Elements
- Material Rejuvenation
- Ni Metal Hydride
- Advanced Materials
Acknowledgements for Funding and Materials for Developmental Work

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- EPA # EP-D-12-0009
- ONAMI Gap Award
- Nissan North America
- Sumitomo Corporation of America
- InvenTek Corporation
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