Rare Earth Recycling and the Balance Problem

Koen Binnemans

KU Leuven - University of Leuven (Belgium)
Rare earths are critical raw materials

Source: report EU commission “Critical raw materials for the EU” (2010)
Supply risk of rare earths

Global reserves are large and well distributed.

Reserves are at least 110 M t REO when the world consumption is expected to be 150 kT REO in 2016.
Production is largely concentrated in China (monopoly position)

USA 6 to 8%

CIS 1 to 2%

China 90%

India 1 to 2%

No problem of resources, but a real supply problem
Applications of rare earths

Total volume in 2012: 110,000 tonnes of REO
## REE usage by application

<table>
<thead>
<tr>
<th>Application</th>
<th>La</th>
<th>Ce</th>
<th>Pr</th>
<th>Nd</th>
<th>Sm</th>
<th>Eu</th>
<th>Gd</th>
<th>Tb</th>
<th>Dy</th>
<th>Y</th>
<th>Other</th>
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<tbody>
<tr>
<td>Magnets</td>
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<td>23.4</td>
<td>69.4</td>
<td></td>
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<td>2</td>
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<tr>
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<td>10</td>
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<tr>
<td>Metallurgy</td>
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<tr>
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<td>2</td>
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<td>FCC</td>
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<td>Glass additives</td>
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<td>66</td>
<td>1</td>
<td>3</td>
<td></td>
<td></td>
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<td></td>
<td>2</td>
<td>4</td>
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<td>Phosphors</td>
<td>8.5</td>
<td>11</td>
<td>4.9</td>
<td>1.8</td>
<td>4.6</td>
<td>69.2</td>
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<td>Ceramics</td>
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<td>6</td>
<td>12</td>
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<td></td>
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<td>53</td>
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<tr>
<td>Others</td>
<td>19</td>
<td>39</td>
<td>4</td>
<td>15</td>
<td>2</td>
<td>1</td>
<td></td>
<td></td>
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<td>19</td>
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</tbody>
</table>

*(source: Lynas Corporation)*
Recycling = closing the materials loop

Primary Ores → Primary Residues & Recyclates → Concentrates → Metals, materials and alloys → (Intermediate) Products/Consumer goods

Enhanced Landfill Mining:
- Landfills (MSW, MWS/IW, IW)
- Urban Mining

Recycling:
- Residues (S/L)/Scrap

Inevitable losses due to 2nd law of Thermodynamics
Why recycling?

- Supply of critical raw materials
- Efficient use of natural resources
- Balance problem (specific for REEs)
- Extra advantage: no issues with radioactivity
Energy saving by recycling

Why Care about Recovering Materials?
Environmental Benefits of Recycling

• Recycled materials can save significant energy

Source: Diran Apelian, CR³ presentation Leuven, 2010
## Depletion of non-renawables

*Table 2.4: predicted peak and depletion of different fuels and metals, and main area of usage.*

*Source: ITRE, March 2009*

<table>
<thead>
<tr>
<th>Commodity</th>
<th>Peak</th>
<th>Depletion</th>
<th>Main area of usage</th>
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<tbody>
<tr>
<td>Oil</td>
<td>2006-2026</td>
<td>2055-2100</td>
<td>Energy generation</td>
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<td></td>
<td></td>
<td></td>
<td>Chemical industry and pharmaceuticals</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>Construction</td>
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<tr>
<td>Natural gaz</td>
<td>2010-2025</td>
<td>2075</td>
<td>Energy generation</td>
</tr>
<tr>
<td>Coal</td>
<td>2100</td>
<td>2160-2210</td>
<td>Energy generation</td>
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<tr>
<td>Antimony</td>
<td>-</td>
<td>2020-2035</td>
<td>Metal alloys</td>
</tr>
<tr>
<td>Copper</td>
<td>-</td>
<td>2040-2070</td>
<td>Energy transport</td>
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<td></td>
<td></td>
<td></td>
<td>Piping</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>Electronics</td>
</tr>
<tr>
<td>Gallium</td>
<td>may have passed</td>
<td>-</td>
<td>Electronics (mobile phones, solar cells)</td>
</tr>
<tr>
<td>Indium</td>
<td>-</td>
<td>2015-2020</td>
<td>Electronics (LCDs, solar cells)</td>
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<tr>
<td>Lead</td>
<td>Passed</td>
<td>2030</td>
<td>Automobile industry</td>
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<td></td>
<td></td>
<td></td>
<td>Chemical industry</td>
</tr>
<tr>
<td>Platinum</td>
<td>-</td>
<td>2020</td>
<td>Electronics (printer, etc)</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>Industry (plug, catalyster, glass production)</td>
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<td></td>
<td></td>
<td></td>
<td>Medicine (pooomaker)</td>
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<tr>
<td>Silver</td>
<td>-</td>
<td>2020-2030</td>
<td>Electronics</td>
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<td></td>
<td></td>
<td></td>
<td>Pharmaceuticals</td>
</tr>
<tr>
<td>Taftalum</td>
<td>-</td>
<td>2025-2035</td>
<td>Electronics (mobile phone, automobiles)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Pharmaceuticals</td>
</tr>
<tr>
<td></td>
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<td>Chemical industry</td>
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<tr>
<td>Uranium</td>
<td>-</td>
<td>2035-2045</td>
<td>Energy generation</td>
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<td>Zinc</td>
<td>-</td>
<td>2030</td>
<td>Anti-corrodees</td>
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<td></td>
<td></td>
<td></td>
<td>Energy storage</td>
</tr>
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</table>
**Balance problem**

- **Balance problem** = demand and supply of the individual rare-earth elements (REEs) have to be equal at any time
- Also called: *Balancing problem*
- Became an issue when applications shifted from the use of mixed rare earths to pure rare earths
- Of importance for REE manufacturers
- Concept introduced by P. Falconnet (Rhone-Poulenc)  
Early applications: mixed rare earths

- Mainly lanthanum and cerium
- **Catalyst industry**
  - Stabilization of zeolites for fluid cracking catalysts (FCC) during steam regeneration
- **Metallurgy (mischmetall)**
  - Graphite nodularization in cast iron
  - Ultimate desulfurization of steels
  - Lighter flints made of iron-mischmetall alloy
  - Grain growth inhibition in light metals
  - Battery alloys (NiMH)
- **Glass industry**
  - Polishing powder (CeO₂)
Modern applications: pure rare earths

• **Permanent magnets**
  - NdFeB (Nd,Pr,Dy)
  - SmCo (Sm) (< 2% of market)

• **Phosphors**
  - Phosphors for trichromatic fluorescent lamps (Y, Eu, Tb, La, Ce)
  - Phosphors for CRTs (color television, computer monitors (Eu,Y)
  - X-ray intensifying screens (Gd,La,Tb)

• **Glass industry**
  - Optical glass (La)
### REE content of selected minerals (%)

<table>
<thead>
<tr>
<th>REE</th>
<th>Bastnasite Mountain Pass, USA</th>
<th>Bastnasite Bayan Obo, China</th>
<th>Monazite Mt. Weld, Australia</th>
<th>Xenotime Lehat, Malaysia</th>
<th>High Y RE laterite Longnan, China</th>
<th>Low Y RE laterite Xunwu, China</th>
<th>Loparite Kola Peninsula, Russia</th>
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<tbody>
<tr>
<td>La</td>
<td>33.8</td>
<td>23.0</td>
<td>25.5</td>
<td>1.2</td>
<td>1.8</td>
<td>43.4</td>
<td>25.0</td>
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<td>Ce</td>
<td>49.6</td>
<td>50.0</td>
<td>46.7</td>
<td>3.1</td>
<td>0.4</td>
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<td>50.5</td>
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<tr>
<td>Pr</td>
<td>4.1</td>
<td>6.2</td>
<td>5.3</td>
<td>0.5</td>
<td>0.7</td>
<td>9.0</td>
<td>5.0</td>
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<tr>
<td>Nd</td>
<td>11.2</td>
<td>18.5</td>
<td>18.5</td>
<td>1.6</td>
<td>3.0</td>
<td>31.7</td>
<td>15.0</td>
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<td>Sm</td>
<td>0.9</td>
<td>0.8</td>
<td>2.3</td>
<td>1.1</td>
<td>2.8</td>
<td>3.9</td>
<td>0.7</td>
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<td>0.2</td>
<td>0.4</td>
<td>Trace</td>
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<td>0.1</td>
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<tr>
<td>Gd</td>
<td>0.2</td>
<td>0.7</td>
<td>&lt;0.1</td>
<td>3.5</td>
<td>6.9</td>
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<td>0.6</td>
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<tr>
<td>Tb</td>
<td>0.01</td>
<td>0.1</td>
<td>&lt;0.1</td>
<td>0.9</td>
<td>1.3</td>
<td>Trace</td>
<td>Trace</td>
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<td>Dy</td>
<td>0.03</td>
<td>0.1</td>
<td>0.1</td>
<td>8.3</td>
<td>6.7</td>
<td>Trace</td>
<td>0.6</td>
</tr>
<tr>
<td>Ho</td>
<td>0.01</td>
<td>Trace</td>
<td>Trace</td>
<td>2.0</td>
<td>1.6</td>
<td>Trace</td>
<td>0.7</td>
</tr>
<tr>
<td>Er</td>
<td>0.01</td>
<td>Trace</td>
<td>Trace</td>
<td>6.4</td>
<td>4.9</td>
<td>Trace</td>
<td>0.8</td>
</tr>
<tr>
<td>Tm</td>
<td>0.01</td>
<td>Trace</td>
<td>---</td>
<td>1.1</td>
<td>0.7</td>
<td>Trace</td>
<td>0.1</td>
</tr>
<tr>
<td>Yb</td>
<td>0.01</td>
<td>Trace</td>
<td>---</td>
<td>6.8</td>
<td>2.5</td>
<td>0.3</td>
<td>0.2</td>
</tr>
<tr>
<td>Lu</td>
<td>Trace</td>
<td>Trace</td>
<td>---</td>
<td>1.0</td>
<td>0.4</td>
<td>0.1</td>
<td>0.2</td>
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<tr>
<td>Y</td>
<td>0.1</td>
<td>Trace</td>
<td>&lt;0.1</td>
<td>61.0</td>
<td>65.0</td>
<td>8.0</td>
<td>1.3</td>
</tr>
</tbody>
</table>
Consequences of REE abundances

- To get 1 ton of Eu$_2$O$_3$ from bastnäsite, one needs to produce (and sell) the following amounts of REOs (tons):

<table>
<thead>
<tr>
<th>Compound</th>
<th>Amount (tons)</th>
</tr>
</thead>
<tbody>
<tr>
<td>La$_2$O$_3$</td>
<td>300</td>
</tr>
<tr>
<td>CeO$_2$</td>
<td>450</td>
</tr>
<tr>
<td>Pr$<em>6$O$</em>{11}$</td>
<td>38</td>
</tr>
<tr>
<td>Nd$_2$O$_3$</td>
<td>118</td>
</tr>
<tr>
<td>Sm$_2$O$_3$</td>
<td>7.3</td>
</tr>
<tr>
<td>Gd$_2$O$_3$</td>
<td>1.4</td>
</tr>
<tr>
<td>Y$_2$O$_3$</td>
<td>0.9</td>
</tr>
</tbody>
</table>

Ref.: P. Falconnet, Basic and Applied Aspects of Rare Earths, 1989 (Venice –Italy) p. 27
Balance problem

- Ideal situation: perfect balance between demand and production of all REE elements
- Market in balance corresponds to lowest price for any REE: production costs are shared by all elements
- Market in balance is very difficult to obtain, because of changes in demand by changes in applications
- Present light REE market is driven by demand for Nd for NdFeB magnets (about 25,000 tons in 2011)
- Sufficient quantities of REE ores have to be mined to produce at least 25,000 tons of Nd
- Ce, Pr, Sm are produced in excess (stockpiled)
- HREE market is smaller and mainly driven by Eu, Tb, Dy, Y
Recycling and the balance problem

- Recycling of NdFeB magnets to recover Nd and Dy means that less primary ores have to be mined to ensure supply of Nd and Dy
- Less mining means less over production of cerium, samarium, ....
- Recycling of Eu, Tb, Y from lamp phosphors helps to maintain the balance of HREE

Recycling of rare earths

• *Less than 1%* of the REEs were being recycled in 2011
  inefficient collection, technological issues, lack of incentives
• Main sources:
  – lamp phosphors (*Eu, Tb, Y, Gd, La, Ce*)
  – permanent magnets (*Nd, Pr, Tb, Dy*)
  – nickel metal hydride batteries (*La, Ce*) + Ni
Recycling of rare earths: a critical review

Koen Binnemans\textsuperscript{a,\ast}, Peter Tom Jones\textsuperscript{b}, Bart Blanpain\textsuperscript{b}, Tom Van Gerven\textsuperscript{c}, Yongxiang Yang\textsuperscript{d}, Allan Walton\textsuperscript{e}, Matthias Buchert\textsuperscript{f}

\textsuperscript{a} KU Leuven — University of Leuven, Department of Chemistry, Celestijnenlaan 200F, Box 2404, B-3001 Heverlee, Belgium
\textsuperscript{b} KU Leuven — University of Leuven, Centre for High Temperature Processes and Sustainable Materials Management, Department of Metallurgy and Materials Engineering (MTM), Kasteelpark Arenberg 44, Box 2450, B-3001 Heverlee, Belgium
\textsuperscript{c} KU Leuven — University of Leuven, Department of Chemical Engineering (CIT), Willem de Croylaan 46, Box 2423, B-3001 Heverlee, Belgium
\textsuperscript{d} TU Delft, Department of Materials Science and Engineering, Meikleweg 2, 2628 CD Delft, The Netherlands
\textsuperscript{e} University of Birmingham, School of Metallurgy and Materials, Edgbaston, Birmingham B15 2TT, UK
\textsuperscript{f} Öeko-Institut e.V., Infrastructure & Enterprises Division, Rheinstrasse 95, D-64295 Darmstadt, Germany
# Lamp phosphors

<table>
<thead>
<tr>
<th>Year</th>
<th>Phosphors</th>
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</thead>
<tbody>
<tr>
<td>1960</td>
<td>$\text{Ca}_5(\text{PO}_4)_3\text{Cl}:\text{Sb}^{3+},\text{Mn}^{2+}$ (white)</td>
</tr>
<tr>
<td>1974</td>
<td>$\text{BaMg}<em>2\text{Al}</em>{16}\text{O}<em>{27}:\text{Eu}^{2+}$, $\text{CeMgAl}</em>{10}\text{O}_{19}:\text{Tb}^{3+}$, $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$</td>
</tr>
<tr>
<td>1990</td>
<td>$\text{BaMgAl}<em>{10}\text{O}</em>{17}:\text{Eu}^{2+}$, $(\text{Sr},\text{Ca})_5(\text{PO}<em>4)<em>3\text{Cl}:\text{Eu}^{2+}$, $\text{CeMgAl}</em>{10}\text{O}</em>{19}:\text{Tb}^{3+}$, $(\text{Gd},\text{Ce})\text{MgB}<em>5\text{O}</em>{10}:\text{Tb}^{3+}$, $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$</td>
</tr>
<tr>
<td>2005</td>
<td>$\text{BaMgAl}<em>{10}\text{O}</em>{17}:\text{Eu}^{2+}$, $(\text{La},\text{Ce})\text{PO}_4:\text{Tb}^{3+}$, $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$</td>
</tr>
</tbody>
</table>
Different options for recycling of lamp phosphors

- Direct re-use
- Separation of phosphors in individual components
- Recovery of REE content
Direct re-use of lamp phosphors

- Advantages
  - Very simple method
  - No chemical processing required

- Disadvantages
  - Only applicable to one type of fluorescent lamps, because different lamps make use of different phosphor mixtures
  - Phosphors deteriorate over the lifetime of the lamp
Separation of phosphors in individual components

• Advantages:
  – Relatively simple process
  – No or limited amounts of chemicals are consumed

• Disadvantages:
  – Very difficult to obtain very pure phosphor fractions
  – Separation process may change the phosphor particle size
  – Phosphors deteriorate over the lifetime of the lamp

• Techniques:
  – Flotation
  – Pneumatic separation
  – Gravity separation in a dense medium
Recovery of REE content

• Advantages:
  – Generally applicable to all types of phosphor mixtures
  – Same processing steps as those used for extraction of rare earths from primary ores (acid and alkali attack of phosphors)
  – Gives very pure rare-earth oxides that can also be used for other applications

• Disadvantages:
  – Many process steps required before obtaining new lamp phosphors
  – Consumption of large amounts of chemicals
  – Generation of large amounts of waste water
Crushing and sieving of fluorescent lamps
Composition of waste lamp phosphor fraction

Lamp Composition
- Glass 88%
- Metals 5%
- Plastics 4%
- Powder 3%
- Hg 0.005%

Waste phosphor Powder Composition
- Halogen phosphate 45%
- Others 15%
- Rare Earths 10-20%
- Alumina 12%
- Fine glass 20%

Phosphor coating
- Mercury (U.V.)
- Electrode
- Plastic
- Metal

BAM
BaMgAl$_{10}$O$_{17}$:Eu$^{2+}$

YOX
Y$_2$O$_3$:Eu$^{3+}$

LAP
(\(La,Ce\))PO$_4$:Tb$^{3+}$

CAT
CeMgAl$_{10}$O$_{19}$:Tb$^{3+}$
Removal of mercury

• Mercury removal is an issue closely related to the recycling of REEs from lamp phosphors (environmental hazard)
• The mercury content of fluorescent lamps varies widely depending on type of lamp (linear tubes versus compact fluorescent lamps), the brand, the wattage and the year of manufacturing.
• General trend is a steady decrease in mercury content over the years due to more strict environmental laws
• Since 2012, compact fluorescent lamps with a power lower than 50 W are not allowed to contain more than 3.5 mg Hg/lamp, according to EU regulations
• For compact fluorescent lamps, more than 85% of Hg ends up in the phosphor layer when the lamp reaches the end of its lifetime
• Hg is a problem for recycling of REE from lamp phosphors, but Hg made efficient collection of end-of-life fluorescent lamps possible
Removal of mercury

• Thermal treatment of the phosphor mixture for several hours at 400-600 °C in vacuum removes larger part of Hg
• full removal of Hg is only possible by heating the phosphor powder to at least 800 °C
• very energy-consuming and cannot be used when wet sieving procedures are being applied to separate the phosphors from glass and metallic parts in crushed fluorescent lamps.
Rhodia Solvay's lamp recycling project

LOOP
The Rhodia recycling project

Phosphor powders in Level 1 landfill

< 10% final residue

Phosphate to valorization

Tons of REO
Y, La, Ce
Tb, Eu, Gd

ReturnType
Rhodia Solvay's lamp recycling project

A 3 steps process

1. Rare Earth extraction (La,Ce,Y,Eu,Tb,Gd)

2. Rare Earth Separations

3. Production of YOX (red) & LAP (green) & others

Recyclers

Patented Recycling Process

Waste phosphor powders

Phosphates

Final residue

Rare Earths concentrate

RE Separations

Pure La
Pure Ce
Pure Tb
Pure Gd
Pure Eu
Pure Y

Finitions

LAP
Others
YOx

Lamp makers
Recycling of REE from NdFeB magnets

- Main focus on NdFeB magnets (98-99% of the market) Topic of MC-ITN “European rare-earth magnet recycling network” (ERean)
- During pyrometallurgical recycling of metals from electronic scrap and used catalysts, they end up as oxides in slags. Concentration in oxide slags is too low for recycling
- In many applications, the amount of REE per item is low (a few grams), so that deep-level dismantling is recommended to recover REE-containing objects.
- Dismantling can be automated (e.g. Hitachi)
- REEs lost to oxide slags (low concentration)
- slags used as building material
Amounts of rare earths in electronic devices

Magnets: 2 wt.% of HDD
REE: 0.6 wt.% of HDD

Mobile phones: only 0.1 to 0.25 g of REEs

Source: Öko-Institut
Dismantling by Hitachi

Source: Hitachi
Flow sheet for recycling of magnets
Different options for recycling of magnets

- Direct re-use in current form/shape
- Reprocessing of alloys to magnets after hydrogen decrepitation
- Hydrometallurgical methods
- Pyrometallurgical methods
- Gas-phase extraction
Direct re-use in current form/shape

- Advantages:
  - Most economical way of recycling (low energy input, no consumption of chemicals)
  - No waste generated
- Disadvantages:
  - Only for large easily accessible magnets (wind turbines, large electric motors and generators in hybrid and electric vehicles)
  - Not available in large quantities in scrap today
Reprocessing of alloys to magnets after hydrogen decrepitation

• Advantages
  – Less energy input required than for hydrometallurgical and pyrometallurgical routes
  – No waste generated
  – Especially suited for hard disk drives (little compositional change over the years)

• Disadvantages
  – Not applicable to mixed scrap feed, which contains magnets with large compositional variations
  – Not applicable to oxidized magnets
Hydrometallurgical methods

• Advantages
  – Generally applicable to all types of magnet compositions
  – Applicable to non-oxidized and oxidized alloys
  – Same processing steps as those for extraction of rare earths from primary ores

• Disadvantages
  – Many process steps required before obtaining new magnets
  – Consumption of large amounts of chemicals
  – Generation of large amounts of waste water
Pyrometallurgical methods (liquid metal extraction)

- Advantages
  - Generally applicable to all types of magnet compositions
  - No generation of waste water
  - Fewer processing steps than hydrometallurgical methods
  - Direct melting allows obtaining master alloys
  - Liquid metal extraction allows obtaining REEs in metallic state

- Disadvantages
  - Larger energy input required
  - Direct smelting and liquid metal extraction cannot be applied to oxidized magnets
  - Electroslag refining and the glass slag method generate large amounts of solid waste
Gas-phase extraction

• Advantages
  – Generally applicable to all types of magnet compositions
  – Applicable to non-oxidized and oxidized alloys
  – No generation of waste water

• Disadvantages
  – Consumption of large amounts of chlorine gas
  – Aluminum chloride is very corrosive
Magnet recycling by SANTOKU

- SANTOKU Corporation has opened in 2012 a plant in Tsuruga (Japan) for recycling Nd and Dy from magnets
  - Motor magnets (air conditioners)
  - Magnet production scrap
- Demagnetization by heating (6 hours at 573 K)
- Milling under 75 micron by jaw crusher and pulverizer
- Oxidation by stirring for 12 hours in an alkaline solution
- Selective dissolution in HCl
- Magnet alloys prepared by molten salt electrolysis

Source: SANTOKU
Different options for recycling of NiMH batteries

- Hydrometallurgical routes
- Pyrometallurgical routes
Hydrometallurgical routes

- **Advantages**
  - Low investment costs
  - Recycling possible of different waste fractions (cathode and anode materials, metals from casing) that can be marketed separately

- **Disadvantages**
  - Many manual operations are required for dismantling of batteries and separating the different components
  - Large consumption of chemicals
Pyrometallurgical routes

• Advantages
  – Well-developed technology
  – Energy recovery from plastic casings and other organic components
  – Same processing steps used for extracting REEs from slags as from primary ores

• Disadvantages
  – High investment cost for furnace
  – REEs need to be extracted from slags
  – REEs are obtained as mixtures and further separation is required
Recycling process for NiMH/ Li-ion batteries @Umicore

Source: Maurits van Camp (Umicore)
Recycling process for NiMH/ Li-ion batteries
@Umicore

- Process developed for NiMH batteries
  - First industrial scale process
- Co-operation with Solvay Rhodia
  - Umicore produces REE-concentrate
  - Umicore separates REO from harmful elements
  - Solvay Rhodia refines REE concentrate
  - For EOL Portable NiMH batteries only
- Process of recovery of REE is not compatible with process of recovery PGM from exhaust catalysts
Conclusions

• Recycling cannot replace primary mining of REE ores, but complements mining

• Recycling of REEs is recommended for:
  – Efficient use of natural resources
  – Supply of critical raw materials
  – Balance problem

• Most interesting waste streams for REE recycling:
  – Lamp phosphors
  – NdFeB magnets
  – NiMH batteries

• REE recycling is technologically challenging, but not impossible
Thank you!

http://www.kuleuven.rare3.eu/