CMI

Accelerating
Energy
Innovations

Critical Materials Institute

AN ENERGY INNOVATION HUB

Alex King, Ames Laboratory
2015 AMO Peer Review – May 28, 2015

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CYTEC Delivering Technology Beyond Our Customers' Imagination™
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U.S. DEPARTMENT OF
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Materials criticality is affecting us *today*

- The target date for transition to high-output T5 fluorescent lamps has been delayed by two years because manufacturers claim that there is a shortage of Eu and Tb for the phosphors.

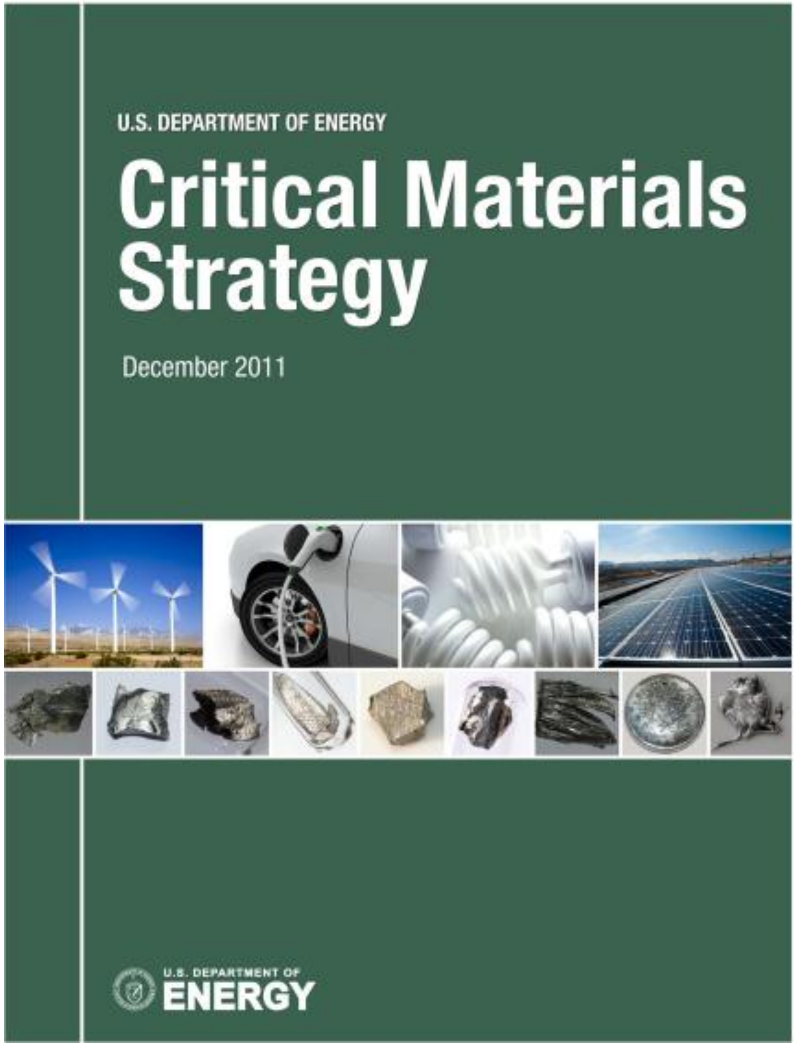
- Utility-scale wind turbine installations are overwhelmingly gearbox-driven units, despite the high failure-rate of the gearboxes, because of the cost and unavailability of Nd and Dy required for direct-drive units.

A three-pillared research strategy

Find ways to:

- diversify our sources;
- provide alternatives to the existing materials;
- make better use of the existing supplies through recycling and re-use.

Some of these approaches work better than others for specific materials.

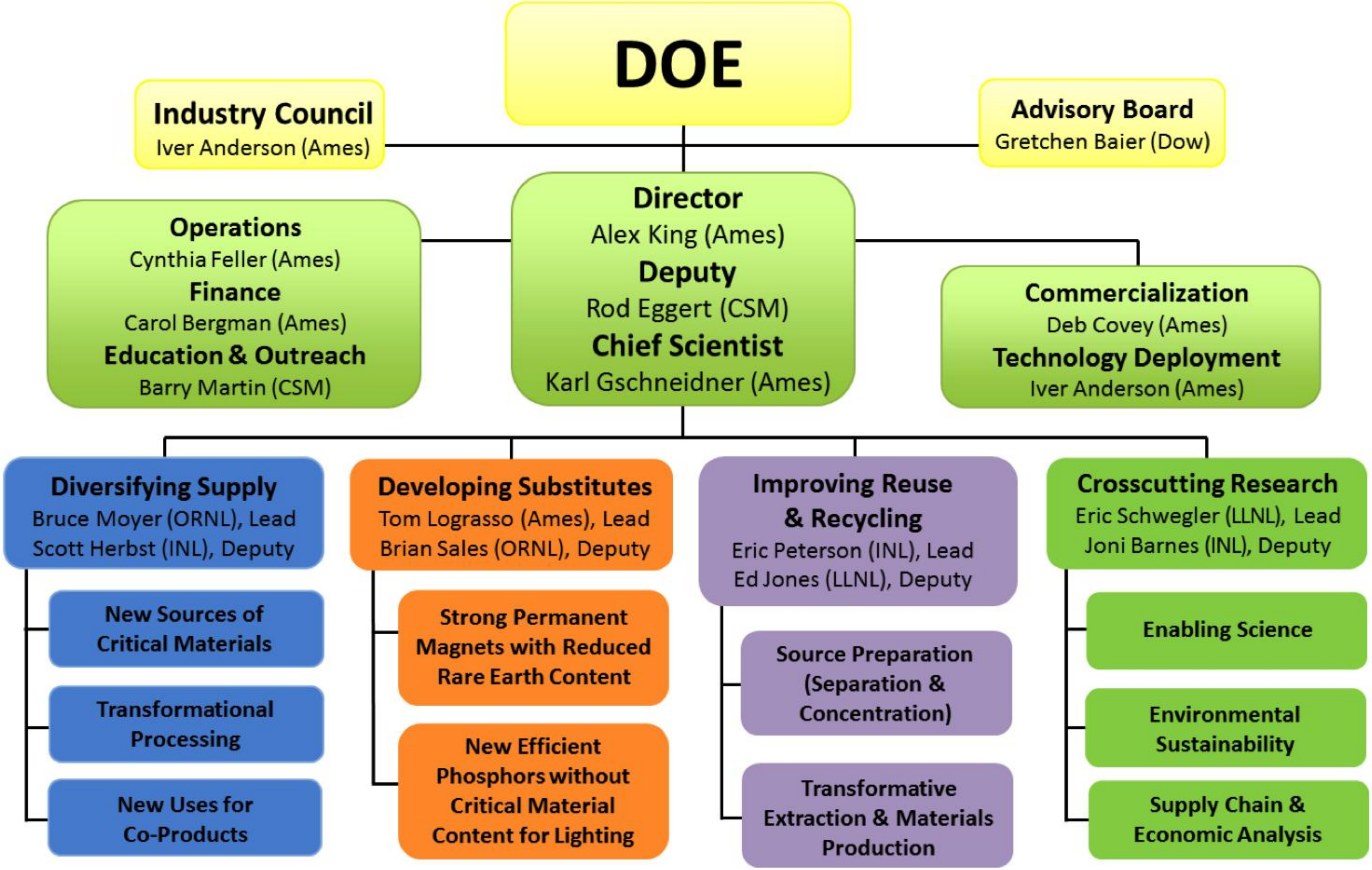


U.S. DEPARTMENT OF ENERGY

Critical Materials Strategy

December 2011

U.S. DEPARTMENT OF ENERGY



DOE

Industry Council
Iver Anderson (Ames)

Advisory Board
Gretchen Baier (Dow)

Operations
Cynthia Feller (Ames)
Finance
Carol Bergman (Ames)
Education & Outreach
Barry Martin (CSM)

Director
Alex King (Ames)
Deputy
Rod Eggert (CSM)
Chief Scientist
Karl Gschneidner (Ames)

Commercialization
Deb Covey (Ames)
Technology Deployment
Iver Anderson (Ames)

Diversifying Supply
Bruce Moyer (ORNL), Lead
Scott Herbst (INL), Deputy

- New Sources of Critical Materials
- Transformational Processing
- New Uses for Co-Products

Developing Substitutes
Tom Lograsso (Ames), Lead
Brian Sales (ORNL), Deputy

- Strong Permanent Magnets with Reduced Rare Earth Content
- New Efficient Phosphors without Critical Material Content for Lighting

Improving Reuse & Recycling
Eric Peterson (INL), Lead
Ed Jones (LLNL), Deputy

- Source Preparation (Separation & Concentration)
- Transformative Extraction & Materials Production

Crosscutting Research
Eric Schwegler (LLNL), Lead
Joni Barnes (INL), Deputy

- Enabling Science
- Environmental Sustainability
- Supply Chain & Economic Analysis

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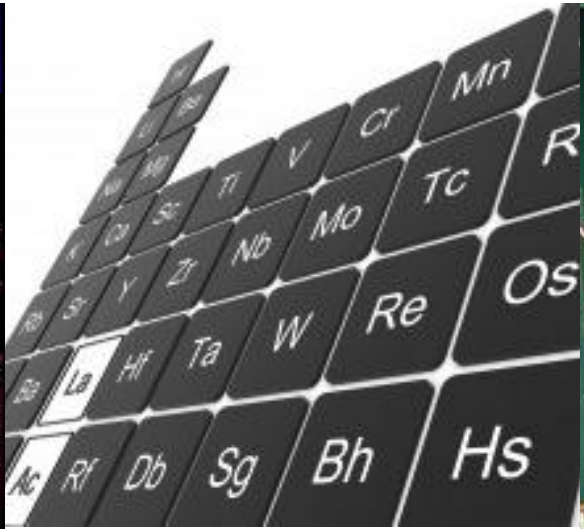
Five-Year Goals

Within its first five years, CMI will develop at least one technology, adopted by U.S. companies, in each of three areas:

Diversifying & expanding production

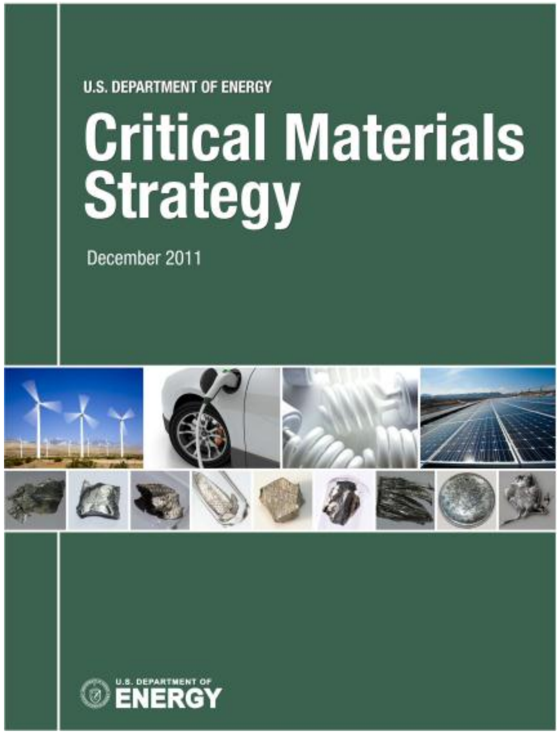


Developing substitutes

Reducing wastes

Criticality Analysis

- Identifies the *propensity* for supply-chain problems to occur. It does not predict that they *will*.
- Identifies the need for *appropriate attention*, not panic.



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Critical Materials Strategy

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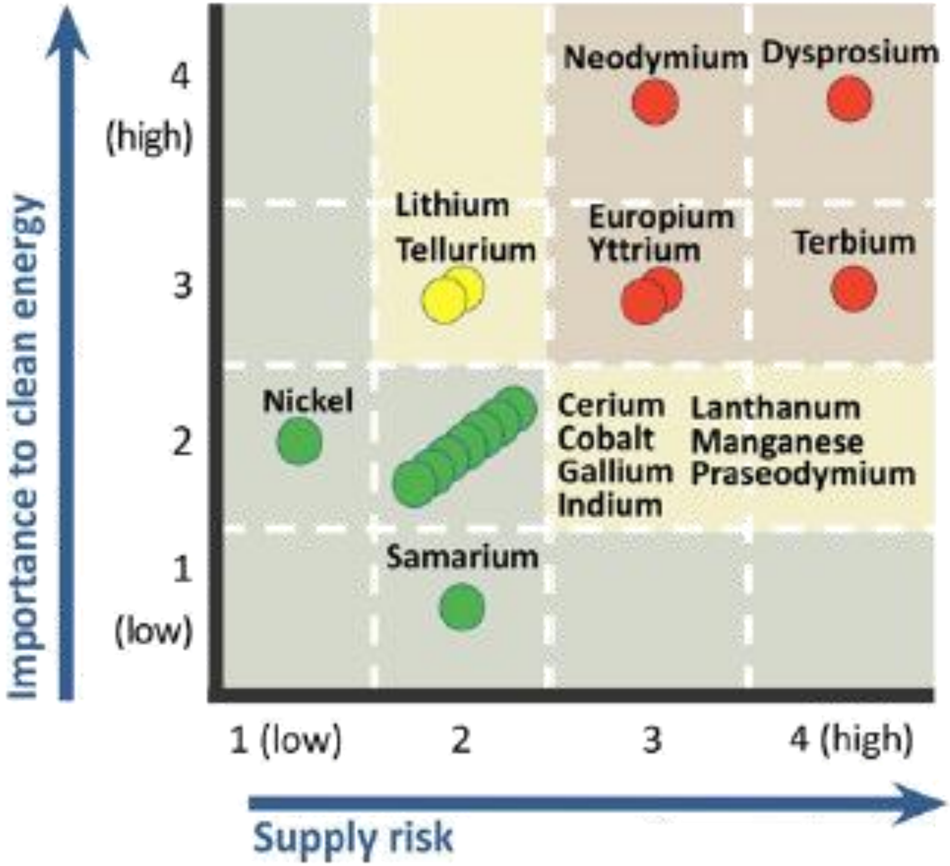

DOE Medium Term Outlooks: *2015 – 2025*

Strategy

- Determine what industry needs in order to meet US energy goals

- Develop technologies that have potential to meet those needs
  - Lower capex and/or opex for materials producers: *increased supply*
  - Materials substitution and/or manufacturing efficiency: *reduced demand*

- Link fundamental & applied research to industry needs
  - Obtain industry input early and often
  - Establish necessary tools and expertise
  - Link CMI efforts to national technology roadmaps
  - Link CMI project roadmaps internally

Salient facts

- Started operations on June 1, 2013
- 318 researchers & staff on payroll (83.2 FTEs)
- Several new facilities established
  - Improved criticality assessment capacity
  - Bulk combinatoric library production facility
  - Thin-film combinatoric library production facility
  - High-throughput analysis (with JCAP and JCESR)
  - Solvent exchange (SX) pilot scale test facility
  - Electrophoretic deposition capability
  - Filtration test facility
  - Toxicology test capability
  - Thermal analysis in high magnetic fields
  - Rapid magnetic property assessment
  - Rapid thermodynamic property assessment
  - Micro-x-ray fluorescence analysis capability
  - Metal reduction capabilities
  - Robotic high-throughput catalyst development system
- Extensive industrial outreach
- 35 invention disclosures, 4 patent applications
- 30+ technical publications, 140+ presentations

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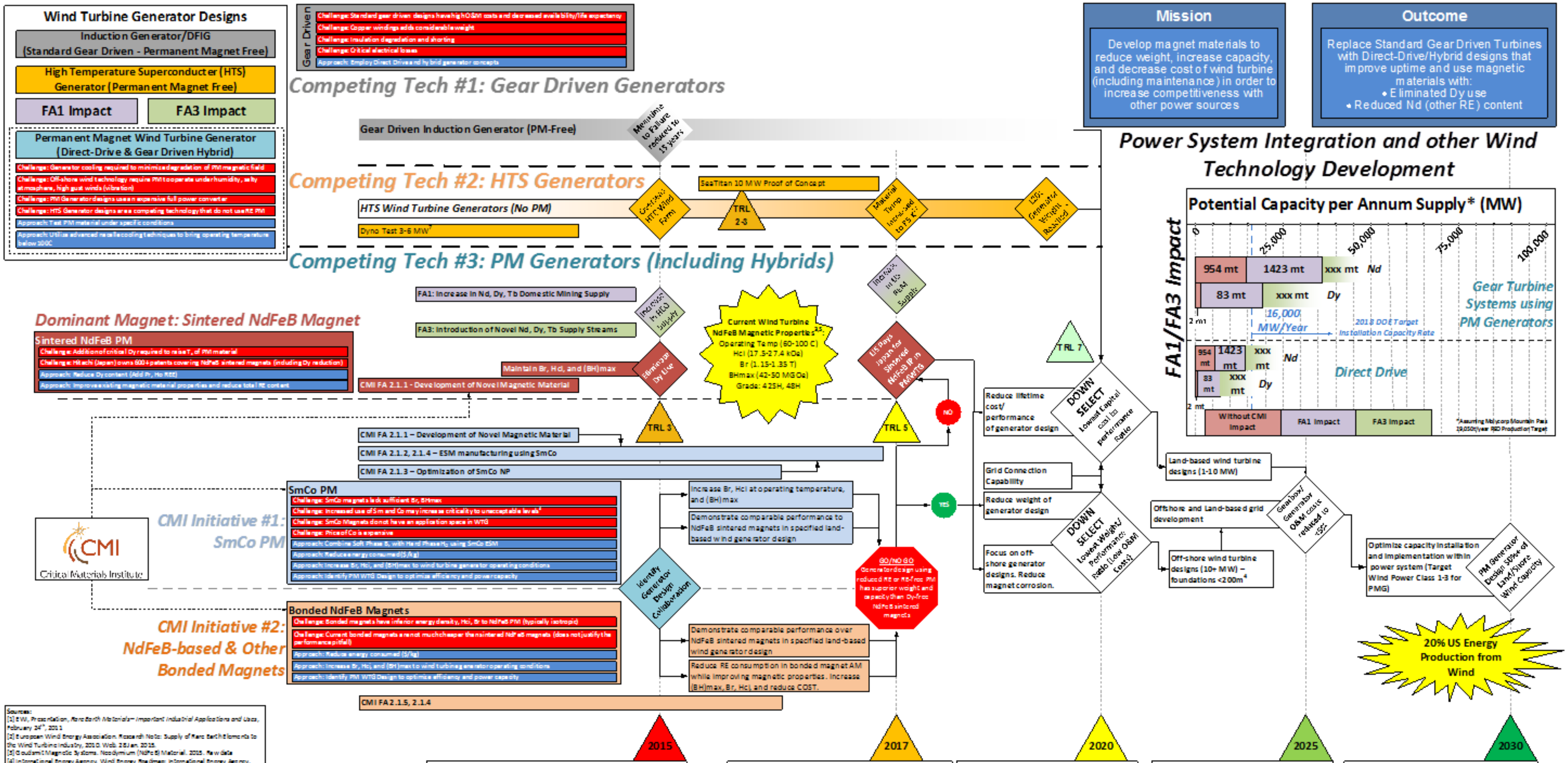
Critical Materials Institute - Wind Turbine Technology – Permanent Magnet Technology Development



Mission

Develop magnet materials to reduce weight, increase capacity, and decrease cost of wind turbine (including maintenance) in order to increase competitiveness with other power sources

Outcome

Replace Standard Gear Driven Turbines with Direct-Drive/Hybrid designs that improve uptime and use magnetic materials with:

- Eliminated Dy use
- Reduced Nd (other RE) content

2015

Success Metrics:

US Wind Technology:

- 0.7 cents/KWh lifecycle O&M costs[11]
- **1.4 MW/tonne PM (Jim. H)[5]**
  - 0.23-0.25 tonne Nd2O3/MW (Direct Drive)[1, 2]
  - 0.023 tonne Nd2O3/MW (Gear Driven Hybrid)[2]
- PMG Weight approx. 300 t[12]

Current Bonded Magnet Properties (% of Average NdFeB Sintered PM Material)[3]

- Br: 0.65 T (50% of Sintered NdFeB)
- Hci: 15 kOe (85% of Sintered NdFeB)
- BHmax: 12.5 MGOe (30% of Sintered NdFeB)

Current Average SmCo Magnet Properties (% of Average NdFeB Sintered PM Material)[3]

- Br: 0.95 T (75% of Sintered NdFeB)
- Hci: 25 kOe (140% of Sintered NdFeB)
- BHmax: 25.1 MGOe (60% of Sintered NdFeB)

2017

Success Metrics:

US Wind Technology Milestones:

- 1.75 MW/tonne PM[5]
- US installation rate of 7,000 turbines/year[10]
- PM Weight approx. <170t

Bonded Magnet Property Increase[3]

- Br – Improve by 50%
- Hci – Improve by 15%
- (BH)max – Improve by 230%

SmCo Magnet Property Increase[3]

- Br – Improve by 25%
- Hci - Maintain
- (BH)max – Improve by 70%

Possible GO/NO GO Metrics[5]:

- Br > 13 kG (1.3 T), (BH)max 42 MGOe
- Hci > 17.5 kOe
- <$60/kg of PM Material (2014,NdFeB)[6]

2020

Success Metrics:

Offshore Testing Environment[9]:

240h @ 130C and 95% Relative Humidity. Prove mass reduction no greater than 2 mg/cm² for uncoated magnet 10 days after exposure (VAC)

US Wind Technnology Milestones:

- 20% Wind Energy LCOE reduction[6]
- 2-3 MW/tonne PM[5,12]
- 140 GW installed wind capacity (Wind Capacity Installation Rate: 16 GW/year)[10]
- Offshore Market: Primarily 6-8 MW DD turbines[12]

2025

Success Metrics:

US Wind Technology Milestones:

- 3-4 MW/tonne PM[5,12]
- 220 GW installed wind capacity (Wind Capacity Installation Rate: 16 GW/year)[10]
- REE PM WTG Shares: 15-75% Land-Based, 25-75% Offshore[12]

2030

Success Metrics:

US Wind Technnology Milestones:

- 30% LCOE reduction[9]
- 5 MW/tonne PM[5,12]
- PM Weight approx. <130t
- 825 Million tonnes of CO2 displaced[10]
- 300 GW installed capacity (250 GW land-based, 50 GW off-shore)[10]
- Land-Based Capacity Factor: 38-53%[10]
- Shallow Off-Shore Capacity Factor: 40-55%[10]

Sources:

[1] EWI, Presentation, *Rare Earth Materials – Important Industrial Applications and Uses*, February 24th, 2011
[2] European Wind Energy Association. Research Note: Supply of Rare Earth Elements to the Wind Turbine Industry, 2010. Web. 28 Jan. 2015.
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[4] International Energy Agency. Wind Energy Roadmap: International Energy Agency, 2010. Web.
[5] Herchenroeder, Jim. "Magnetic Properties vs. Application." Message to the author. 21 Jan. 2015. E-mail.
[6] Lantz, E., M. Hand, and R. Wiser. Past and Future Cost of Wind Energy: Preprint (2012): 1. National Renewable Energy Laboratory. Aug. 2012. Web. 28 Jan. 2015.
[7] Maples, B., M. Hand, and W. Musial. Comparative Assessment of Direct Drive High Temperature Superconducting Generators in Multi-Megawatt Class Wind Turbines. Tech. no. NREL/TP-5000-49086. National Renewable Energy Laboratory, Oct. 2010. Web. 28 Jan. 2015
[8] Henriksen, Matthew, Technical University of Denmark. Comparison of MW-Class PM Wind Turbine Generator Concepts. DONG Energy. 2012
[9] Rollik, Doris, Bernard Schlaede, and Vacuumschmelze GmbH & Co. NdFeB Magnets Are Going Offshore in Wind Mill Generators. Hanau: Bodo's Power Systems, 2007. Print.
[10] US Department of Energy. 20% Wind Energy by 2030: Increasing Wind Energy's Contribution to U.S. Electricity Supply. Rep. no. DOE/GO-102008-2567, 2008. Web. Jan. 2015
[11] U.S. Department of Energy: Efficiency and Renewable Energy. 20% Wind Energy - Diversifying Our Energy Portfolio and Addressing Climate Change. Washington, D.C.: U.S. Department of Energy, 2008. Web. 28 Jan 2015
[12] US Department of Energy (2010). Critical Materials Strategy. Technical Report. REE content: US Department of Energy
[13] Jansson et. Al, Strategic Energy Technology Plan: Scientific Assessment in Support of the Materials Roadmap enabling Low Carbon Energy Technologies, JRC European Commission, EUR 25197 EN, 2012

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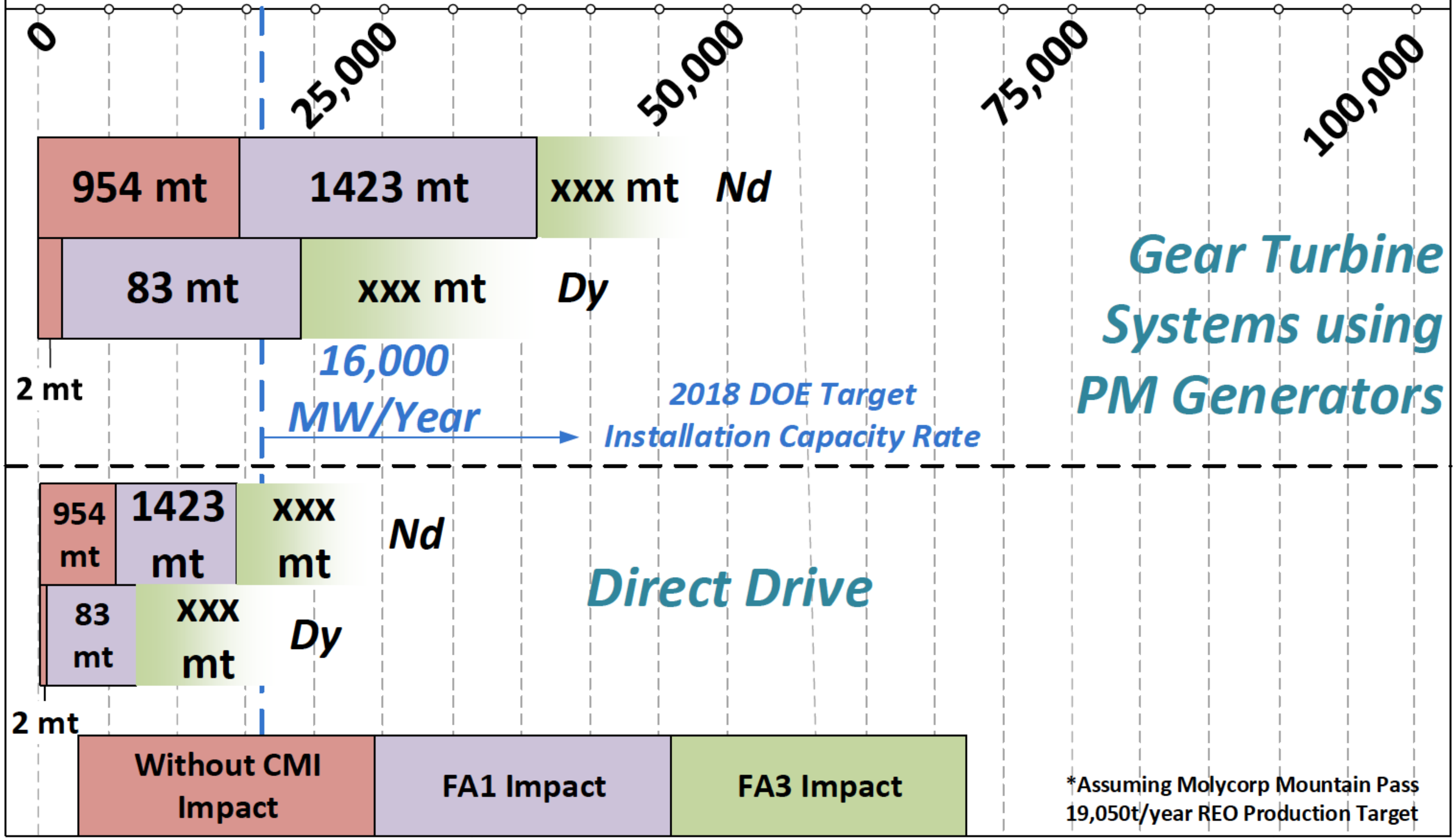
Revision	-- 5--
Date	28JAN2015
Charged by:	Devin Imholte

Potential Capacity per Annum Supply* (MW)



*Assuming Molycorp Mountain Pass 19,050t/year REO Production Target

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Focus Area 1

Diversifying Sources

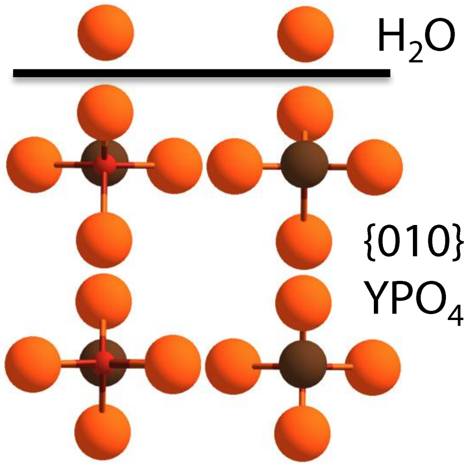
- Reducing capex and/or opex for traditional and non-traditional materials producers.

Advanced Beneficiation Techniques

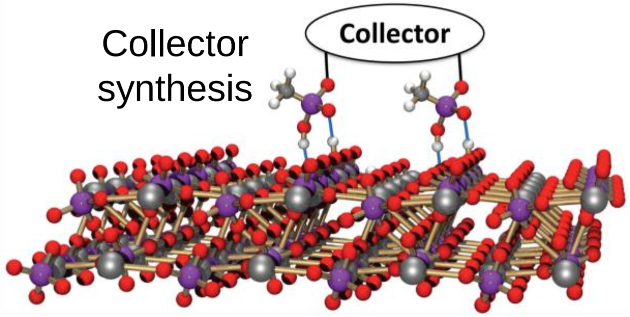
Colorado School of Mines, Oak Ridge National Laboratory
Partners: Molycorp, Cytec, Rare Element Resources

- The goal is to develop new or improved beneficiation techniques that will enable improved efficiency for processing of a range of rare earth ores.
- Improved supply of rare earths per ton of ore mined: potential for 10k tons or more REO per year at Molycorp Mountain Pass site alone. Other ores can be enabled, potentially equivalent to Mtn Pass production rate.
- Status: determining which fundamental processes (mineral surface charge, collector selectivity, etc.) are most responsible for REE mineral flotation; promising flotation observed (e.g., ancylite); useful gravity and magnetic effects observed; X-ray reflectivity measured for two minerals, interface structures determined.
- Utilize lessons learned from current work to design, test and improve new collector & suppressor molecules.
- TRL 6 (Pilot scale) by 2018 (see road map)

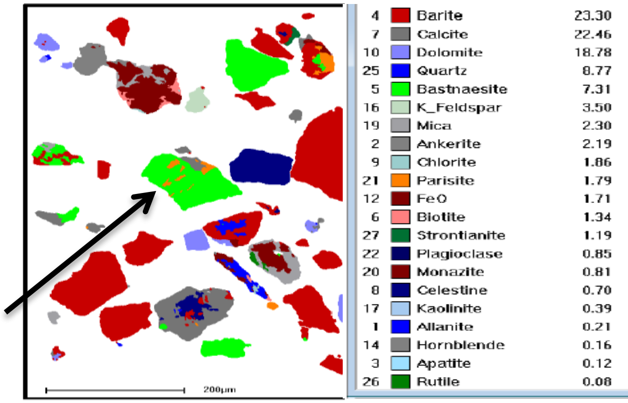
Atomic mineral structure



Collector synthesis



Ore mineral characterization



4	🟥	Barite	23.30
7	⬛	Calcite	22.46
10	🟪	Dolomite	18.78
25	🟦	Quartz	8.77
5	🟩	Bastnaesite	7.31
16	🟩	K_Feldspar	3.50
19	⬜	Mica	2.30
2	⬛	Ankerite	2.19
9	🟦	Chlorite	1.86
21	🟧	Parisite	1.79
12	🟫	FeO	1.71
6	🟥	Biotite	1.34
27	🟩	Strontianite	1.19
22	🟦	Plagioclase	0.85
20	🟫	Monazite	0.81
8	🟦	Celestine	0.70
17	🟦	Kaolinite	0.39
1	🟦	Allanite	0.21
14	⬛	Hornblende	0.16
3	🟦	Apatite	0.12
26	🟩	Rutile	0.08

Green = REE mineral

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Critical Materials Institute – Diversifying Supply – New Sources of Critical Materials – Advanced Beneficiation Techniques (Project 1.1.1)

Task Breakdown

- **Task 1 – Mineral Characterization and Testing**
- Task 2 – Mineral Surface Characterization
- Task 3 – Design and Synthesis of New Collectors
- Task 4 – Demonstration, Scale-up, and Engineering

Challenges

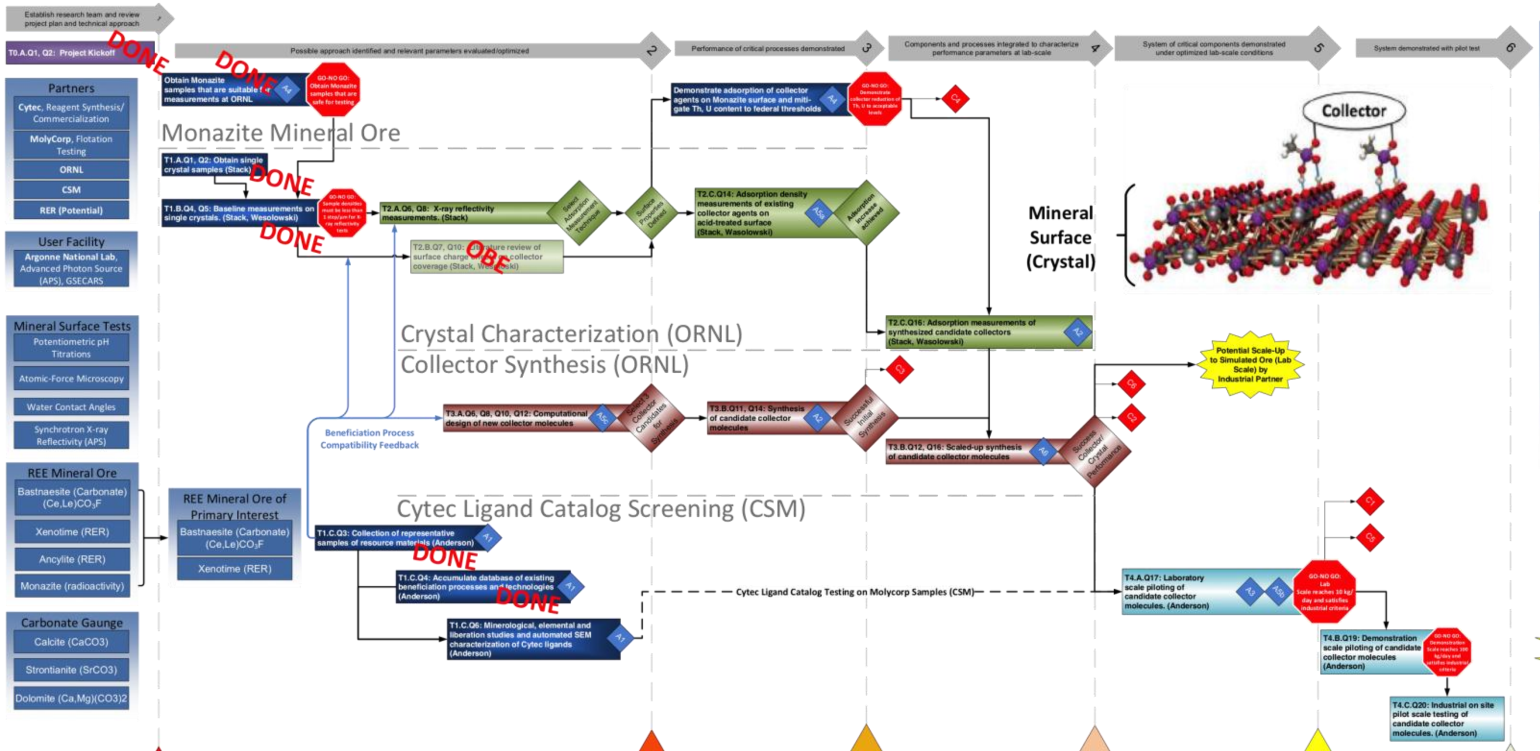
- C1: There is little information on the performance and interaction of a novel crystal/collector combination for bastnaesite and other mineral beneficiation
- C2: Froth flotation beneficiation of RE minerals occur in many types of ore along with gaunge that are often similar in composition
- C3: Current beneficiation recovery rates for bastnaesite are inefficient and send up to 35% of bastnaesite ore to tailing heap
- C4: Monazite contains Th, U (high radioactivity)
- C5: Recovery performance of novel synthesized novel collector with optimal crystal surface is unknown
- C6: It takes 5-7 years for a new molecule to go to market (Cytec)

Approach

- A1: Consult with industrial partners and CSM for information on current beneficiation process capabilities and RE-bearing minerals
- A2: Understand and control surface phenomena and develop flotation reagents that provides better selectivity for REE-ores
- A3: Increase recovery at froth flotation stage to dramatically increase beneficiation yield to more economic and efficient levels
- A4: Conduct materials balance to determine U, Th concentrations and develop beneficiation process to reduce concentrations to federal thresholds
- A5a: Pair commercial flotation agents (Cytec) with complementary mineral surface structure
- A5b: Demonstrate promising beneficiation methodology on real mineral feeds (Cyec, MolyCorp)
- A5c: Employ molecular-design techniques to define absorbent structures
- A6: Use Molycorp process to vet beneficiation process and ligand integration

Technology Readiness Levels

1	2	3	4	5	6	7	8	9
Basic Principle	Application Formulated	Proof of Concept	Integrated Components	Demonstrated with High Fidelity	Demonstrated in Relevant Environment	Final Configuration	Integrated Readiness	Fully Operational
		Bench Scale	Bench/Lab Scale	Lab Scale	Pilot Scale	Prototype Scale	First of a Kind (FOAK)	Production Scale



Establish research team and review project plan and technical approach 1

T0.A.Q1, Q2: Project Kickoff DONE

Possible approach identified and relevant parameters evaluated/optimized 2

Performance of critical processes demonstrated 3

Components and processes integrated to characterize performance parameters at lab-scale 4

System of critical components demonstrated under optimized lab-scale conditions 5

System demonstrated with pilot test 6

Partners

- **Cytec**, Reagent Synthesis/ Commercialization
- **MolyCorp**, Flotation Testing
- **ORNL**
- **CSM**
- **RER (Potential)**

User Facility

- **Argonne National Lab**, Advanced Photon Source (APS), GSECARS

Mineral Surface Tests

- Potentiometric pH Titrations
- Atomic-Force Microscopy
- Water Contact Angles
- Synchrotron X-ray Reflectivity (APS)

REE Mineral Ore

- Bastnaesite (Carbonate) (Ce,Le)CO_3F
- Xenotime (RER)
- Ancylite (RER)
- Monazite (radioactivity)

REE Mineral Ore of Primary Interest

- Bastnaesite (Carbonate) (Ce,Le)CO_3F
- Xenotime (RER)

Carbonate Gaunge

- Calcite (CaCO3)
- Strontianite (SrCO3)
- Dolomite (Ca,Mg)(CO3)2

Monazite Mineral Ore

Obtain Monazite samples that are suitable for measurements at ORNL A4 DONE

GO-NO GO: Obtain Monazite samples that are safe for testing

Demonstrate adsorption of collector agents on Monazite surface and mitigate Th, U content to federal thresholds A4

GO-NO GO: Demonstrate collector reduction of Th, U to acceptable levels

C4

T1.A.Q1, Q2: Obtain single crystal samples (Stack) DONE

T1.B.Q4, Q5: Baseline measurements on single crystals. (Stack, Wesolowski) DONE

GO-NO GO: Sample densities must be less than 1 step/µm for X-ray reflectivity tests

T2.A.Q6, Q8: X-ray reflectivity measurements. (Stack)

Select Adsorption Measurement Technique

T2.B.Q7, Q10: Literature review of surface charge effects on collector coverage (Stack, Wesolowski) OBE

Surface Properties Defined

T2.C.Q14: Adsorption density measurements of existing collector agents on acid-treated surface (Stack, Wasolowski) A5a

Adsorption increase achieved

T2.C.Q16: Adsorption measurements of synthesized candidate collectors (Stack, Wasolowski) A2

Mineral Surface (Crystal)

Collector

Crystal Characterization (ORNL)

Collector Synthesis (ORNL)

Beneficiation Process Compatibility Feedback

T3.A.Q6, Q8, Q10, Q12: Computational design of new collector molecules A5c

Select 3 Collector Candidates for Synthesis

T3.B.Q11, Q14: Synthesis of candidate collector molecules A2

Successful Initial Synthesis

C3

T3.B.Q12, Q16: Scaled-up synthesis of candidate collector molecules A6

Success Collector/ Crystal Performance

C6

C2

Potential Scale-Up to Simulated Ore (Lab Scale) by Industrial Partner

Cytec Ligand Catalog Screening (CSM)

T1.C.Q3: Collection of representative samples of resource materials (Anderson) A1 DONE

T1.C.Q4: Accumulate database of existing beneficiation processes and technologies (Anderson) A1 DONE

T1.C.Q6: Minerological, elemental and liberation studies and automated SEM characterization of Cytec ligands (Anderson) A1

Cytec Ligand Catalog Testing on Molycorp Samples (CSM)

T4.A.Q17: Laboratory scale piloting of candidate collector molecules. (Anderson) A3 A5b

GO-NO GO: Lab Scale reaches 10 kg/ day and satisfies industrial criteria

C1

C5

T4.B.Q19: Demonstration scale piloting of candidate collector molecules (Anderson)

GO-NO GO: Demonstration Scale reaches 100 kg/day and satisfies industrial criteria

T4.C.Q20: Industrial on site pilot scale testing of candidate collector molecules. (Anderson)

Opportunity

Existing REE beneficiation techniques are costly and inefficient for bastaesite and xenotime – sending a large portion of these ores to tailings waste. This project has the potential to improve beneficiation techniques across a wide range of rare earth ores – thereby improving process efficiency and diversifying US supply.

Market Impact and Penetration

Currently, Molycorp's primary mined RE-bearing mineral is Bastaesite and their recovery is approximately 60% (~50% being Ce_2O_3). Successful deployment of a novel froth flotation process would increase bastnaesite recovery to 75% and also establish additional processes for efficient xenotime and monazite recovery.

This equates to a ~5,000 MT TREO increase in US annual production (20,000 MT REO/year projected 2015 Molycorp production).

ID

Prototype beneficiation process deployed by industry

TRL1

Basic Principles

TRL2

Application Formulated

Mineral Characterization and Testing

- Single-crystal crystalline samples that demonstrate clear cleavage plane
- Sample impurities <10% are acceptable
- If first two sample step densities > 1 step/micrometer, alternate samples will be obtained
- Surface analysis: 2 samples analyzed above will be measured with 3 collector agents
- Bulk measurements: 2 samples and 3 collector agents will be measured
- >= 6 representative samples of resource materials collected

Mineral Surface Characterization

- GSECARS access procured
- 3 surface potential curves (0.1, 0.01, 0.001M)

Design and Synthesis of New Collectors

- Identify top 5 collectors from Host Designer software
- Identify 2 additional collector candidates for synthesis

TRL3

Proof of Concept

Mineral Surface Characterization

- Detect if acid-treatment enhances known collector adsorption coverage by 2x or greater above that of pristine mineral surfaces
- Demonstrate 2x or greater adsorption coverage of a putative collector agent into a REE-phosphate above that of the known collector agents

Candidate Collector Molecule Synthesis

- 1g feed stock batch process
- >50% yield target (Will be improved with scale-up if target not met)
- >95% purity target (Will be improved with scale-up if target not met)

TRL4

Integrated Components

- **Candidate Collector Molecule Synthesis Scale-Up Metrics**
- 50 g of compound
- Target >50% yield
- Target >95% purity

TRL5

Lab Scale in Simulated Environment

Lab Scale

- 10 kg feed stock Batch Lab Scale

TRL6

Demonstrated in Relevant Environment

Initial Pilot Scale

- 100 kg/day scale using optimization and locked cycle testing

On-site Pilot

- 1 tonne/day of resource material produced

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Idaho National Laboratory

Revision:	-- 8 --
Date:	16DEC2014
Changed by:	Devin Imholte

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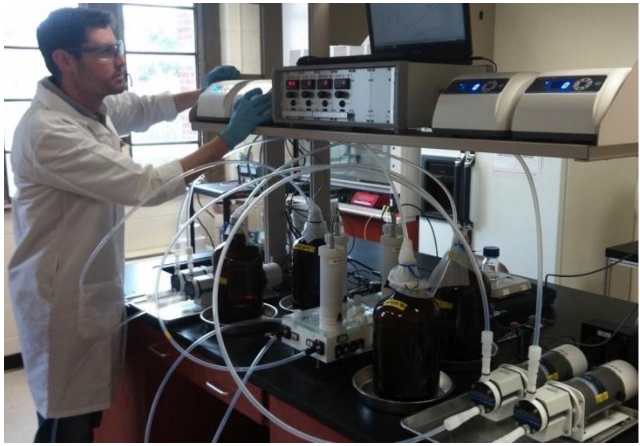
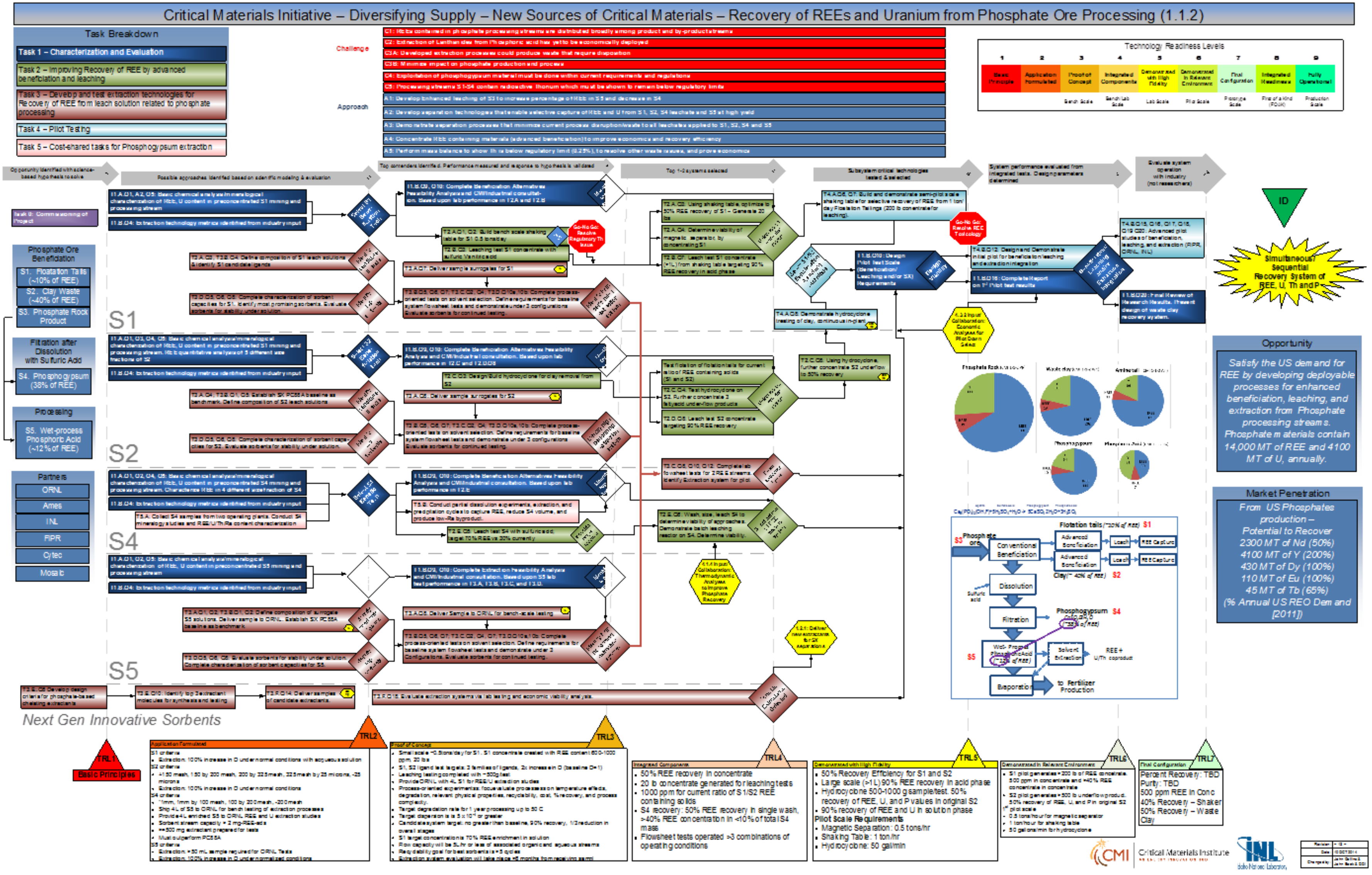
Critical Materials from Phosphate Ore Processing

Oak Ridge National Laboratory; Florida Industrial and Phosphate Research Institute; Ames Lab; Idaho National Laboratory; Mosaic Company; Cytec; Rutgers Univ.; UC-Davis; OLI Systems

- Goal: Establish alternative critical materials source from large-scale US phosphate production.
- Existing US phosphate operations could supply large quantities of energy-critical REE, including twice the current global supply of Y; 1.3 times the supply of Dy, and 40% of global Nd production
- Bench-scale testing on industrial samples has identified attractive source materials and concentration/extraction technologies
- Future activities include bench and pilot testing with actual materials in conjunction with engaged industry, including Mosaic and Cytec.
- Commercialization involves demonstration of economic REE recovery from a relevant stream by 2018.



Critical Materials Initiative – Diversifying Supply – New Sources of Critical Materials – Recovery of REEs and Uranium from Phosphate Ore Processing (1.1.2)



Task Breakdown

- Task 1 – Characterization and Evaluation
- Task 2 – Improving Recovery of REE by advanced beneficiation and leaching
- Task 3 – Develop and test extraction technologies for Recovery of REE from leach solution related to phosphate processing
- Task 4 – Pilot Testing
- Task 5 – Cost-shared tasks for Phosphogypsum extraction

Challenge

- C1: REEs contained in phosphate processing streams are distributed broadly among product and by-product streams
- C2: Extraction of Lanthanides from Phosphoric acid has yet to be economically deployed
- C3A: Developed extraction processes could produce waste that require disposition
- C3B: Minimize impact on phosphate production and process
- C4: Exploitation of phosphogypsum material must be done within current requirements and regulations
- C5: Processing streams S1-S4 contain radioactive thorium which must be shown to remain below regulatory limits

Approach

- A1: Develop Enhanced leaching of S3 to increase percentage of REE in S5 and decrease in S4
- A2: Develop separation technologies that enable selective capture of REE and U from S1, S2, S4 leachate and S5 at high yield
- A3: Demonstrate separation processes that minimize current process disruption/waste to all leachates applied to S1, S2, S4 and S5
- A4: Concentrate REE containing materials (advanced beneficiation) to improve economics and recovery efficiency
- A5: Perform mass balance to show Th is below regulatory limit (0.25%), to resolve other waste issues, and prove economics

Technology Readiness Levels

1	2	3	4	5	6	7	8	9
Basic Principle	Application Formulated	Proof of Concept	Integrated Components	Demonstrated with High Fidelity	Demonstrated in Relevant Environment	Final Configuration	Integrated Readiness	Fully Operational
		Bench Scale	Bench/Lab Scale	Lab Scale	Pilot Scale	Prototype Scale	First of a Kind (FOAK)	Production Scale

Opportunity

Satisfy the US demand for REE by developing deployable processes for enhanced beneficiation, leaching, and extraction from Phosphate processing streams. Phosphate materials contain 14,000 MT of REE and 4100 MT of U, annually.

Market Penetration

From US Phosphates production –
Potential to Recover
2300 MT of Nd (50%)
4100 MT of Y (200%)
430 MT of Dy (100%)
110 MT of Eu (100%)
45 MT of Tb (65%)
(% Annual US REO Demand [2011])

TRL4 – Integrated Components

- 50% REE recovery in concentrate
- 20 lb concentrate generated for leaching tests
- 1000 ppm for current ratio of S1/S2 REE containing solids
- S4 recovery: 50% REE recovery in single wash, >40% REE concentration in <10% of total S4 mass
- Flowsheet tests operated >3 combinations of operating conditions

TRL5 – Demonstrated with High Fidelity

- 50% Recovery Efficiency for S1 and S2
- Large scale (>1 L) 90% REE recovery in acid phase
- Hydrocyclone 500-1000 g sample/test. 50% recovery of REE, U, and P values in original S2
- 90% recovery of REE and U in solution phase

Pilot Scale Requirements

- Magnetic Separation: 0.5 tons/hr
- Shaking Table: 1 ton/hr
- Hydrocyclone: 50 gal/min

TRL6 – Demonstrated in Relevant Environment

- S1 pilot generates >200 lb of REE concentrate. 500 ppm in concentrate and >40% REE concentrate in concentrate
- S2 pilot generates >500 lb underflow product. 50% recovery of REE, U, and P in original S2 1st pilot scale
- 0.5 tons/hour for magnetic separator
- 1 ton/hour for shaking table
- 50 gallons/min for hydrocyclone

TRL7 – Final Configuration

Percent Recovery: TBD
Purity: TBD
500 ppm REE in Conc
40% Recovery – Shaker
50% Recovery – Waste Clay

Focus Area 2

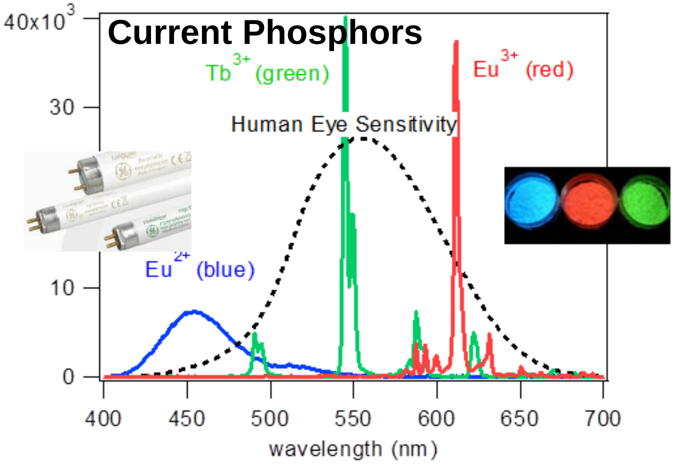
Materials Substitution

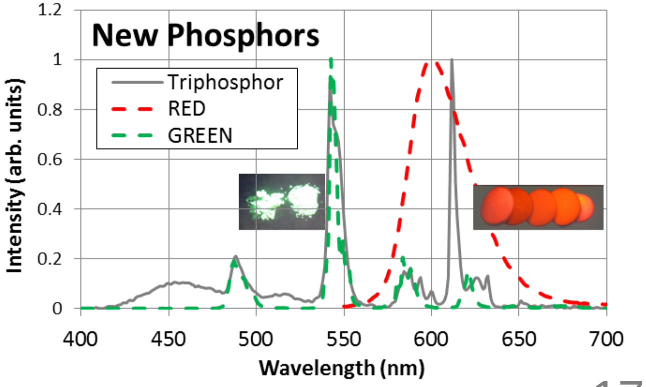
- Plug-in substitute materials for minimal process disruption
- Lower-cost materials for cost savings
- Meeting (not exceeding) performance requirements
- Linking materials development & product design

Fluorescent Lamp Phosphors with Low Rare Earth Content

- Goal: Replacements for the GREEN and RED phosphors using 90% less critical material content
- Impact: Fluorescent lighting will no longer be subject to market spikes in rare earth costs

- Status: Two leading candidates, $Zn_2P_2O_7$:Ce,Tb and AlN:Mn. Several candidates serve as backups, including zinc borates, garnets, and others.
- Next steps: Improvements in light yield:
  - $Zn_2P_2O_7$:Ce,Tb (15%) and AlN:Mn by 2.5x.
  - The issues appear to lie within the synthetic methods.
- We are making good progress in understanding the nature of energy transfer mechanism, and expect to have these issues resolved within a year.





Critical Materials Institute – Developing Substitutes – New Efficient Phosphors Without Critical Material Content For Lighting – Replacements of Eu^{3+} and Tb^{3+} by Non-Critical Phosphors (2.2.1 & 2.2.2)

Task Breakdown

- Task 1 – Identification
- Task 2 – Initial Synthesis and Characterization
- Task 3 – Phosphor Loss Mechanisms and Fundamental Studies
- Task 4 - Phosphor Optimization
- Task 5 - Lamp Performance Tests
- Task 6 – Full Optimization & Lamp Demonstration

Challenges

- C1: Emerging LED technology could outpace the need for phosphor lighting
- C2: Mn^{4+}, Sm^{3+}, and Pr^{3+} provide low emission efficiency and reduces the field of candidates
- C2: Tb^{3+} is currently not utilized to the most efficient extent
- C3: Fundamental understanding of reactivity mechanisms of phosphors with mercury in fluorescent lamps is lacking
- C4: Some phosphors undergo serious damage in fluorescent lamps during use and storage (mercury ion bombardment)

Approach

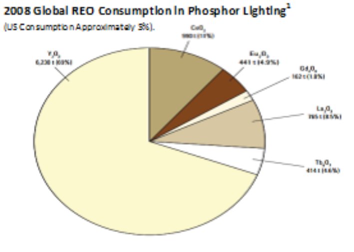
- A1: Surpass 85% survival rate of current phosphor technology to at least 90% to compete with LED
- A2: Research and utilize sensitizers to provide adequate absorption
- A3: Utilize chemical mechanism modeling of cross-cutting CMI project (*Unidentified Collaboration*)
- A4: Develop protective coatings using GE-established methods

Technology Readiness Levels

1	2	3	4	5	6	7	8	9
Basic Principle	Application Formulated	Proof of Concept	Integrated Components	Demonstrated with High Fidelity	Demonstrated in Relevant Environment	Final Configuration	Integrated Readiness	Fully Operational
		Bench Scale	Bench/Lab Scale	Lab Scale	Pilot Scale	Prototype Scale	First of a Kind (FOAK)	Production Scale

2008 Global REO Consumption in Phosphor Lighting[1]

(US Consumption Approximately 3%).



[1] Goonan, T.G., 2011, Rare earth elements—End use and recyclability: U.S. Geological Survey Scientific Investigations Report 2011–5094, 15 p. available only at http://pubs.usgs.gov/sir/2011/5094/.

Opportunity

This project targets fluorescent lamp technology that currently uses critical REEs Eu^{3+} and Tb^{3+} and aims to replace their use with Sm^{3+}/Mn^{4+} and Mn^{2+}, respectively. Additionally, the emission performance of Tb^{3+} will be optimized for more effective use.

Market Impact and Penetration

Approximately 1 billion linear fluorescent bulbs each contain a few grams of critical REEs and successful replacement of Eu and Tb from fluorescent lamps will have a tremendous impact on both lighting and REE supply for other Tb, Eu and Y technologies.

REO content in imported US fluorescent bulb technology 2010[2] (Years of 2013 US REO Production[3])

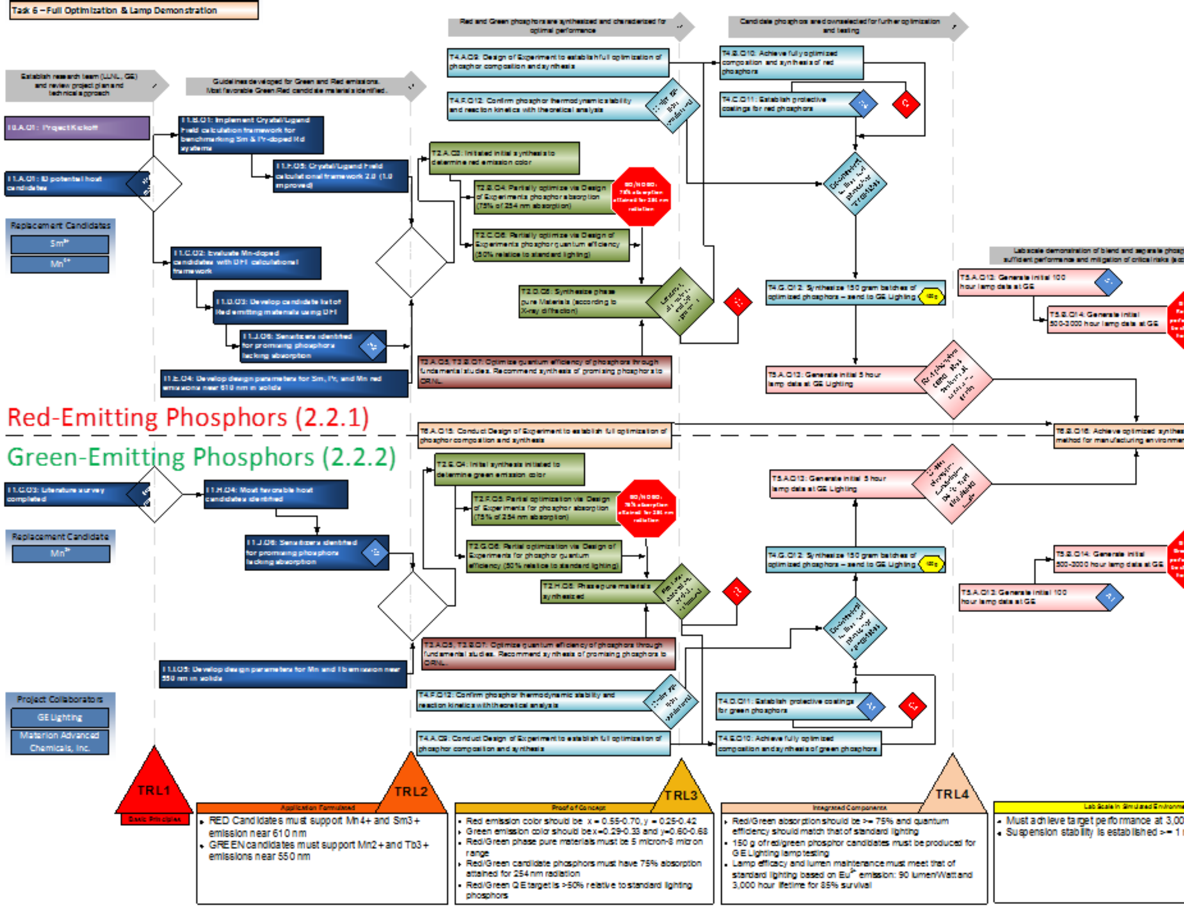
Eu_2O_3, 50 t (8)

Tb_4O_7, 56 t (180)

Y_2O_3, 740 t (30)

[2] Bleiwas, D.I., and Gambogi, Joseph, 2013, Preliminary estimates of the quantities of rare-earth elements contained in selected products and in imports of semimanufactured products to the United States, 2010: U.S. Geological Survey Open-File Report 2013–1072, 10 p., http://pubs.usgs.gov/of/2013/1072/.

[3] Molycorp. Molycorp Annual Report 2013. Rep. no 001-34827. Molycorp, Inc., 3 Mar. 2014. Web. 30 Nov. 2014.



TRL1 – Basic Principles

TRL2 – Application Formulated

- RED Candidates must support Mn4+ and Sm3+ emission near 610 nm
- GREEN candidates must support Mn2+ and Tb3+ emissions near 550 nm

TRL3 – Proof of Concept

- Red emission color should be x = 0.55-0.70, y = 0.25-0.42
- Green emission color should be x=0.29-0.33 and y=0.60-0.68
- Red/Green phase pure materials must be 5 micron-8 micron range
- Red/Green candidate phosphors must have 75% absorption attained for 254 nm radiation
- Red/Green QE target is >50% relative to standard lighting phosphors

TRL4 – Integrated Components

- Red/Green absorption should be >= 75% and quantum efficiency should match that of standard lighting
- 150 g of red/green phosphor candidates must be produced for GE Lighting lamp testing
- Lamp efficacy and lumen maintenance must meet that of standard lighting based on Eu^{3+} emission: 90 lumen/Watt and 3,000 hour lifetime for 85% survival

TRL5 – Lab Scale in Simulated Environment

- Must achieve target performance at 3,000 hours
- Suspension stability is established >= 1 month

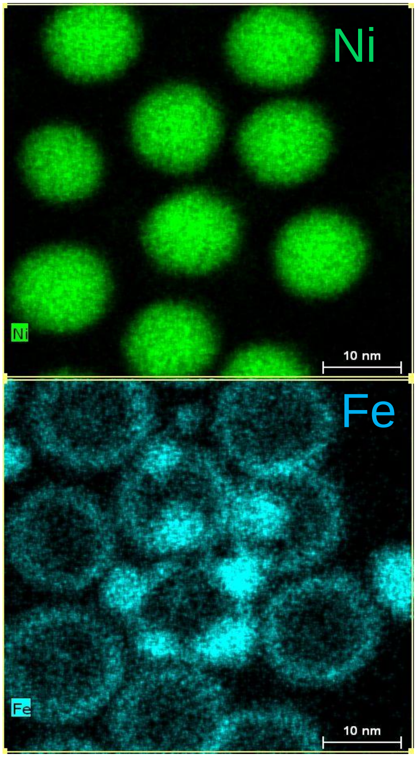
TRL6 – Demonstrated in Realistic Environment

- Survival rate and lumen maintenance greater than 90% at 3,000 hours

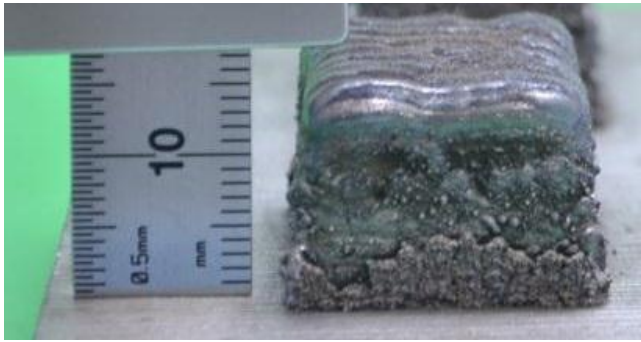
Revision	[illegible]
Date	[illegible]
Changed by	[illegible]

Advanced Magnet Manufacturing

- Goal: Exploit advanced manufacturing to reduce rare earth content in high performance magnets
- Impact: Significant reduction in Dy demand, reduction in waste, improved performance (higher operating temperature, higher energy density)
- Status: Producing feedstock material, developing AM techniques to produce magnets
- Refine parameters to improve magnet performance
- Initial use in high end motor/actuators (2018)



Ni core ferrite shell NPs



Initial laser additive trial of isotropic $Nd_2Fe_{14}B$ powder

Critical Materials Initiative – Developing Substitutes – Strong Permanent Magnets with Reduced Rare Earth Content – Advanced Manufacturing of Exchange Spring Magnets (Project 2.1.2), Optimization of Grain Boundaries and Interfaces in Fine Particle Magnets (2.1.3)

Task Breakdown

- Task 1 – Nanoparticle production and development research and tests
- Task 2 – Electrophoretic deposition (EPD) lab demonstration with NP plating and ESM applications
- Task 3: Process integration with other 2.1 projects
- 2.1.3 Task 1 – Mechanical Synthesis of Permanent Magnet (PM) Nanoparticles (NP)
- 2.1.3 Task 2 – Chemical Synthesis of Permanent Magnet (PM) Nanoparticles (NP)
- 2.1.3 Task 3 – Materials Chracterization

Challenges

- C1: High performance ESM magnets using NP in EPD processing have not been produced at any size scale
- C2: ESM fabrication by EPD requires control from nm to mm scale
- C3: Cost effective replacement of Nd2Fe14B magnets is the target that has yet to be proven
- C4: Current research outside CMI (Toyota NA) examining ESM fabrication using core-shell particles
- C5: Uniform distribution and optimal dispersion of NP difficult with EPD processing
- C6: PVD deposition of NP is slow and NP baseline properties must be improved before EPD is performed
- C7: Particles oxidize and deteriorate under unknown settings during processing
- C8: Current REE magnet NP properties are insufficient (high defects concentrations) for producing nanocomposite magnets that can compete with commercially available magnets (2.1.3)
- C9: Unidentified potentially critical non-magnetic properties (e.g. corrosiveness, brittleness, interdiffusion)

Approach

- A1: Fabricate ESMs with EPD using hard and soft NPs to form magnets replacing Nd2Fe14B in specific applications AND with post-deposition annealing/processing
- A2: Synthesize hard and soft magnet NPs using different processing routes to obtain required magnetic properties, particles shapes, and surface characteristics for EPD
- A3: Utilize temperature-independent routes to control nucleation and growth as alternative to PVD
- A4: Utilize protective coatings, such as CaO, on hydroxide SmCo NPs to reduce surfaces/remove oxide
- A5: Keep lines of communication open with Toyota NA within IP limits
- A6: Collaborate with CMI modeling teams to assess interdiffusion, micromagnetic behavior, particle size effects
- A7: Control oxidation of magnetic NP during processing
- A8: Quantify structural disorder and map interfaces within NP using high resolution STEM and APFIM techniques

Technology Readiness Levels

1	2	3	4	5	6	7	8	9
Basic Principle	Application Formulated	Proof of Concept	Integrated Components	Demonstrated with High Fidelity	Demonstrated in Relevant Environment	Final Configuration	Integrated Readiness	Fully Operational
		Bench Scale	Bench/Lab Scale	Lab Scale	Pilot Scale	Prototype Scale	First of a Kind (FOAK)	Production Scale

Task 0: Project Kickoff

Hard Phase NP Synthesis

- Chemical Synthesis (Brown)
- PVD of unagglomerated NP (LLNL)

Hard Magnet Phases

- SmCo$_5$
- Sm$_2$Co$_{17}$

Soft Phase NP Synthesis

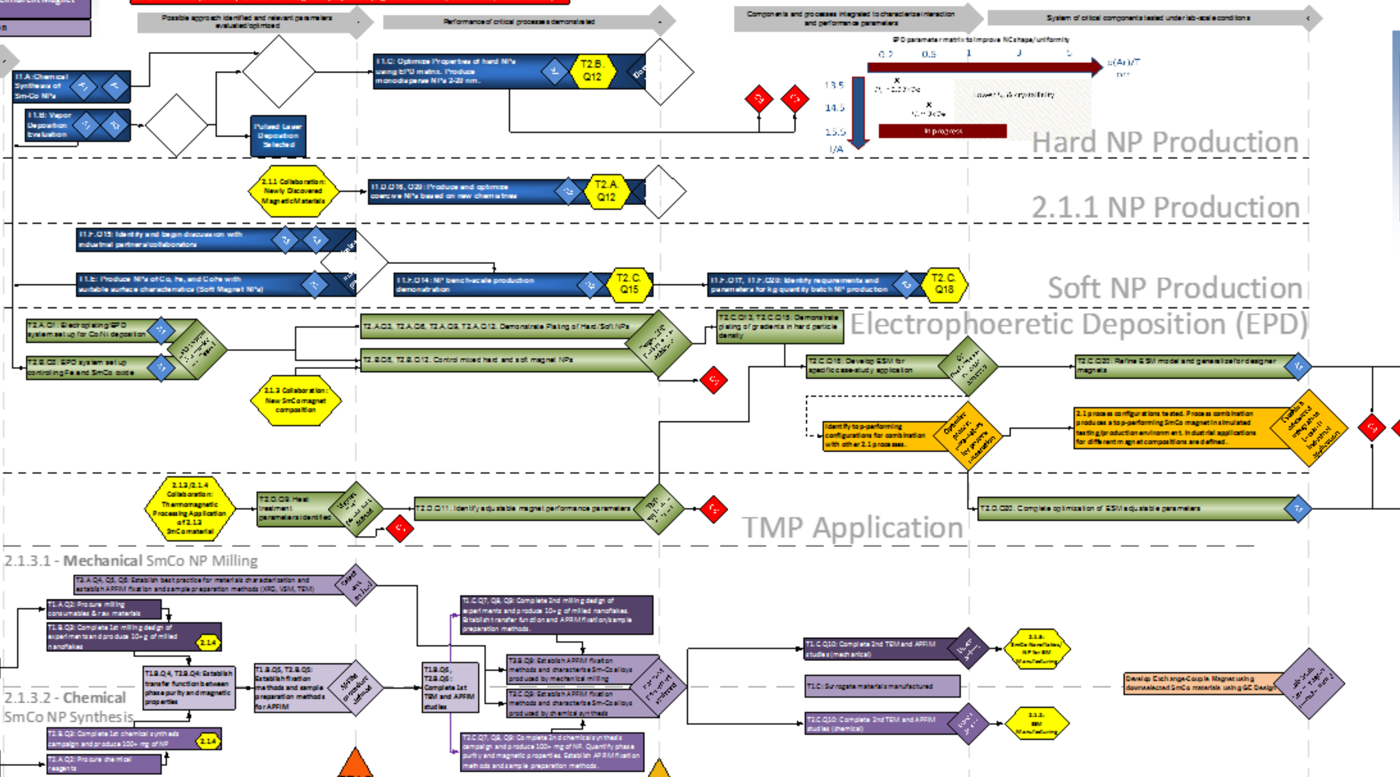
- Chemical Synthesis (Brown)

Soft Magnet Phases

- Fe
- Co

Collaboration

- CMI 4.3.2 – Identify Promising Markets
- CMI 2.1.3 - EPD done at different length scale
- 2.1.4 – Optimize magnetic characteristics of EPD magnets
- 2.1.1 – Replace Sm-Co NP for long term CMI goals)
- Toyota NA – Core-Shell Particle Research
- Magnequench (2.1.5)
- Electron Energy Corp.
- Arnold Magnetic Technologies



ID

Potential Pilot Scale Deployment and Technology Transition

Opportunity

The goal of this project is to develop additive manufacturing (AM) industrial processes that a) reduce the required fraction of REE (e.g. Nd, Dy) by fabricating high-performance Exchange Spring Magnets (ESM) to replace Nd2Fe14B magnets.

Market Impact

The U.S. is 100% reliant on foreign manufactured REO PMs.

A PM with a 50% or greater reduction in REO content with superior performance would greatly reduce or eliminate U.S. foreign reliance on PM REOs (i.e. Nd, Dy). This would also establish the U.S. as a dominant PM producer in the REO global supply chain.

TRL1

Basic Principles

- Initial benchmark of energy density >Sm-Co with 50% reduction in Sm for high T applications
- Long range target is to replace Nd2Fe14B with higher energy density, operating temp increased by >100 K, and 50% RE cost reduction

TRL2

Application Formulated

2.1.2

- Produce 0.1 gram quantities of SmCo5/Sm2Co17
- Measure/Calculate NP energy speed, deposition and yield
- SmCo$_5$, Sm$_2$Co$_{17}$ NPs with Hc(300K) > 15 kOe, monodisperse (+/- 25%)
- Ms (magnetic mass)> 50% Ms(bulk)
- Properties: H_c (Coercivity), M_s (Saturation Magnetization), and M_r (Remnant Magnetization) vs T, NP stability vs. oxidation defined for obtaining competitive energy density with Nd$_2$Fe$_{14}$B

2.1.3

- 1st Mechanical Milling Experiment: 10+ g of milled nanoflakes
- 1st Chemical Synthesis: 100+ mg of chem. synthesized NP
- Materials Characterization: Demonstrate crystalline volume fraction/texture 75%+ (?)

TRL3

Proof of Concept

2.1.2

NP fabrication

- For textured NPs, Mr (parallel) 50% > Mr (perp) or Ms (NP) >0.75Ms(bulk), Hc>15kOe

EPD

- Controlled distribution (+/-25%), coupling >30%
- 20% or better increase in Mr over bare NPs with path for further improvement
- Coat SmCo NP with 2nd metal (2 nm thickness)

2.1.3

Mechanical Milling:

- Demonstrate crystalline volume fraction/texture and remnant magnetization (M) > 75% of intrinsic magnetization of PM compound
- Complete 2nd milling experiment producing 10+ g of milled nanoflakes

Chemical Synthesis

- Demonstrate phase purity of >10% and M_r >50% of the intrinsic magnetization of the PM compound

Complete 2nd chemical synthesis experiment and produce 100+ mg of chemically synthesized NPs

TRL4

Integrated Components

2.1.2

- ESM 10% gain in energy density or 10% reduction in REE content
- Optimum NP Egn - (Hc)(material $) > Hc(Sm-Co)(Sm-Co $)
- NP produced according to Hc == Hc(Sm-Co/Fe) and Ms ==Ms(Sm-Co/Fe)

2.1.3

Mechanical Milling:

- Demonstrate crystalline volume fraction/texture and M_r > 90% of the intrinsic magnetization of the PM compound

Chemical Synthesis:

- Demonstrate phase purity > 35% and M_r > 50% of the intrinsic magnetization of the PM compound

TRL5

Lab Scale in Simulated Environment

2.1.2

- ESM with 300K energy density >= 350 kJ/m^3
- Max operating temp > 200 C
- Requiring 25% REE content as compared to Nd$_2$Fe$_{14}$B

2.1.3

Critical Materials Institute
AN ENERGY INNOVATION HUB

Focus Area 3

Reduce, Re-use & Recycle

- Reducing capex and/or opex for recyclers to improve supply.
- Improving manufacturing efficiency to reduce demand.

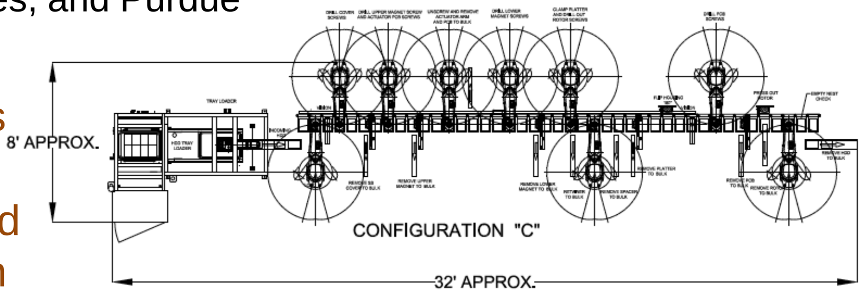
Rare earth Magnet Recovery & Reuse

Participating Institution – ORNL

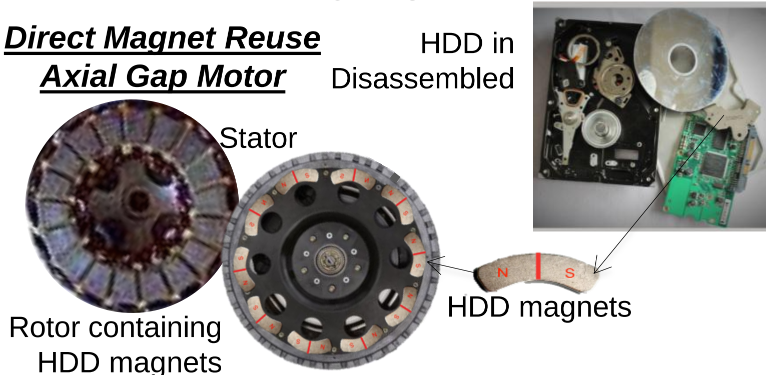
Co-participating Institutions – LLNL, Ames, CO School of Mines, and Purdue

- Project goal - Economically recover rare earth magnet material from end-of-life consumer products and magnet manufacturing.
- Impact - 1000s of metric tons of RE metal, oxide and intact magnets are made available for reintroduction into the marketplace ***(6 Invention Disclosures filed)***
- Current status – High throughput magnet recovery sub-processes validated on HDDs; magnet manufacturing swarfs efficiently separated for recovery; optimization method developed for improved manufacturing and dismantlement of magnet containing products
- Next steps – Build magnet recovery demonstration test bed; refine swarf separation processes; optimize software advisor to assist with design for manufacture
- Path to commercialization/target date – Formalize/enhance industry team to demonstrate economics of magnet and swarf recovery processes; implement software advisor in manufacturing/recovery process. ***(Technology Licensing)***

Automated Magnet Recovery

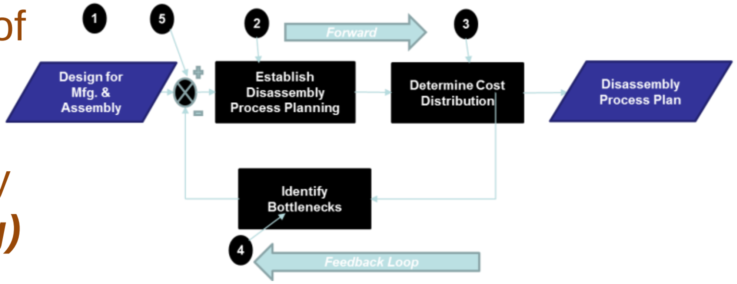


Direct Magnet Reuse
Axial Gap Motor



Manufacturing Design Advisor

Design-for Manufacturing & Assembly + Design-for-Disassembly



Swarf Recycling

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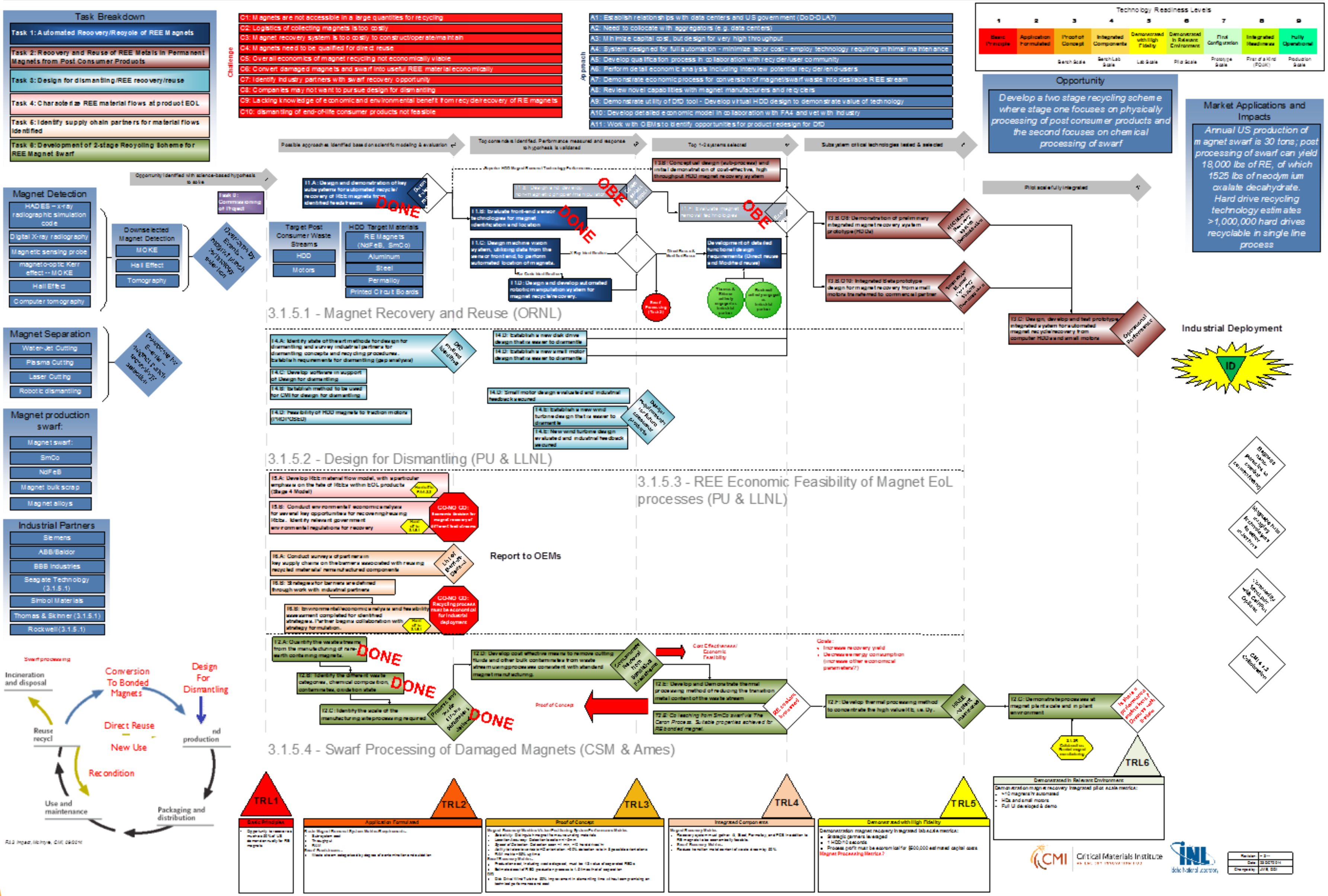
Critical Materials Institute – Improving Reuse & Recycling – Source Preparation (Separation and Concentration) – Transforming Rare Earth Magnet Reuse (Project 3.1.5)



Task Breakdown

- Task 1: Automated Recovery/Recycle of REE Magnets
- Task 2: Recovery and Reuse of REE Metals in Permanent Magnets from Post Consumer Products
- Task 3: Design for dismantling/REE recovery/reuse
- Task 4: Characterize REE material flows at product EOL
- Task 5: Identify supply chain partners for material flows identified
- Task 6: Development of 2-stage Recycling Scheme for REE Magnet Swarf

Challenge

- C1: Magnets are not accessible in a large quantities for recycling
- C2: Logistics of collecting magnets is too costly
- C3: Magnet recovery system is too costly to construct/operate/maintain
- C4: Magnets need to be qualified for direct reuse
- C5: Overall economics of magnet recycling not economically viable
- C6: Convert damaged magnets and swarf into useful REE material economically
- C7: Identify industry partners with swarf recovery opportunity
- C8: Companies may not want to pursue design for dismantling
- C9: Lacking knowledge of economic and environmental benefit from recycle/recovery of RE magnets
- C10: dismantling of end-of-life consumer products not feasible

Approach

- A1: Establish relationships with data centers and US government (DoD-DLA?)
- A2: Need to collocate with aggregators (e.g. data centers)
- A3: Minimize capital cost, but design for very high throughput
- A4: System designed for full automation - minimize labor cost - employ technology requiring minimal maintenance
- A5: Develop qualification process in collaboration with recycler/user community
- A6: Perform detail economic analysis including interview potential recycler/end-users
- A7: Demonstrate economic process for conversion of magnet/swarf waste into desirable REE stream
- A8: Review novel capabilities with magnet manufacturers and recyclers
- A9: Demonstrate utility of DfD tool - Develop virtual HDD design to demonstrate value of technology
- A10: Develop detailed economic model in collaboration with FA4 and vet with industry
- A11: Work with OEMs to identify opportunities for product redesign for DfD

Technology Readiness Levels

1	2	3	4	5	6	7	8	9
Basic Principle	Application Formulated	Proof of Concept	Integrated Components	Demonstrated with High Fidelity	Demonstrated in Relevant Environment	Final Configuration	Integrated Readiness	Fully Operational
		Bench Scale	Bench/Lab Scale	Lab Scale	Pilot Scale	Prototype Scale	First of a Kind (FOAK)	Production Scale

Opportunity

Develop a two stage recycling scheme where stage one focuses on physically processing of post consumer products and the second focuses on chemical processing of swarf

Market Applications and Impacts

Annual US production of magnet swarf is 30 tons; post processing of swarf can yield 18,000 lbs of RE, of which 1525 lbs of neodymium oxalate decahydrate. Hard drive recycling technology estimates >1,000,000 hard drives recyclable in single line process

Opportunity identified with science-based hypothesis to solve

Possible approaches identified based on scientific modeling & evaluation

Top contenders identified. Performance measured and response to hypothesis is validated

Top 1-2 systems selected

Subsystem critical technologies tested & selected

Pilot scale fully integrated

Magnet Detection

- HADES – x-ray radiographic simulation code
- Digital X-ray radiography
- Magnetic sensing probe
- magneto-optic Kerr effect -- MOKE
- Hall Effect
- Computer tomography

Downselected Magnet Detection

- MOKE
- Hall Effect
- Tomography

Magnet Separation

- Water-Jet Cutting
- Plasma Cutting
- Laser Cutting
- Robotic dismantling

Magnet production swarf:

- Magnet swarf:
- SmCo
- NdFeB
- Magnet bulk scrap
- Magnet alloys

Industrial Partners

- Siemens
- ABB/Baldor
- BBB Industries
- Seagate Technology (3.1.5.1)
- Simbol Materials
- Thomas & Skinner (3.1.5.1)
- Rockwell (3.1.5.1)

Task 0: Commissioning of Project

Target Post Consumer Waste Streams

- HDD
- Motors

HDD Target Materials

- RE Magnets (NdFeB, SmCo)
- Aluminum
- Steel
- Permalloy
- Printed Circuit Boards

3.1.5.1 - Magnet Recovery and Reuse (ORNL)

- T1.A: Design and demonstration of key subsystems for automated recycle/recovery of REE magnets from identified feedstreams — DONE
- T1.B: Evaluate front-end sensor technologies for magnet identification and location — DONE
- T1.E: Design and develop non-magnetic gripper/manipulator — OBE
- T1.F: Evaluate magnet removal technologies — OBE
- T1.C: Design machine vision system, utilizing data from the sensor front end, to perform automated location of magnets.
- T1.D: Design and develop automated robotic manipulation system for magnet recycle/recovery.
- Development of detailed functional design requirements (Direct reuse and Modified reuse)
- T3.B: Conceptual design (sub-process) and initial demonstration of cost-effective, high throughput HDD magnet recovery system
- T3.B.O8: Demonstration of preliminary integrated magnet recovery system prototype (HDDs)
- T3.B.O10: Integrated beta prototype design for magnet recovery from small motors transferred to commercial partner
- T3.C: Design, develop and test prototype integrated system for automated magnet recycle/recovery from computer HDDs and small motors

Operational Performance

3.1.5.2 - Design for Dismantling (PU & LLNL)

- T4.A: Identify state of the art methods for design for dismantling and survey industrial partners for dismantling concepts and recycling procedures. Establish requirements for dismantling (gap analysis)
- T4.C: Develop software in support of Design for dismantling
- T4.B: Establish method to be used for CMI for design for dismantling
- T4.D: Feasibility of HDD magnets to traction motors (PROPOSED)
- T4.D: Establish a new disk drive design that is easier to dismantle
- T4.D: Establish a new small motor design that is easier to dismantle
- T4.D: Small motor design evaluated and industrial feedback secured
- T4.E: Establish a new wind turbine design that is easier to dismantle
- T4.E: New wind turbine design evaluated and industrial feedback secured

3.1.5.3 - REE Economic Feasibility of Magnet EoL processes (PU & LLNL)

- T5.A: Develop REE material flow model, with a particular emphasis on the fate of REEs within EOL products (Stage 4 Model)
- T5.B: Conduct environmental/ economic analyses for several key opportunities for recovering/reusing REEs. Identify relevant government environmental regulations for recovery
- GO-NO GO: Economic decision for magnet recovery of different feed streams
- T6.A: Conduct surveys of partners on key supply chains on the barriers associated with reusing recycled materials/ remanufactured components
- T6.B: Strategies for barriers are defined through work with industrial partners
- T6.B: Environmental/economic analysis and feasibility assessment completed for identified strategies. Partner begins collaboration with strategy formulation.
- GO-NO GO: Recycling process must be economical for industrial deployment

Report to OEMs

3.1.5.4 - Swarf Processing of Damaged Magnets (CSM & Ames)

- T2.A: Quantify the waste streams from the manufacturing of rare-earth containing magnets. — DONE
- T2.B: Identify the different waste categories, chemical composition, contaminates, oxidation state — DONE
- T2.C: Identify the scale of the manufacturing site processing required — DONE
- T2.D: Develop cost effective means to remove cutting fluids and other bulk contaminates from waste stream using processes consistent with standard magnet manufacturing.
- T2.E: Develop and Demonstrate thermal processing method of reducing the transition metal content of the waste stream
- T2.E: Co leaching from SmCo swarf via The Caron Process. Suitable properties achieved for RE bonded magnet.
- T2.F: Develop thermal processing method to concentrate the high value REs, i.e. Dy.
- T2.G: Demonstrate processes at magnet plant scale and in plant environment

Cost Effectiveness/ Economic Feasibility

Proof of Concept

Goals:

- Increase recovery yield
- Decrease energy consumption (increase other economical parameters?)

Industrial Deployment

ID

CMI 4.1.2 Collaboration

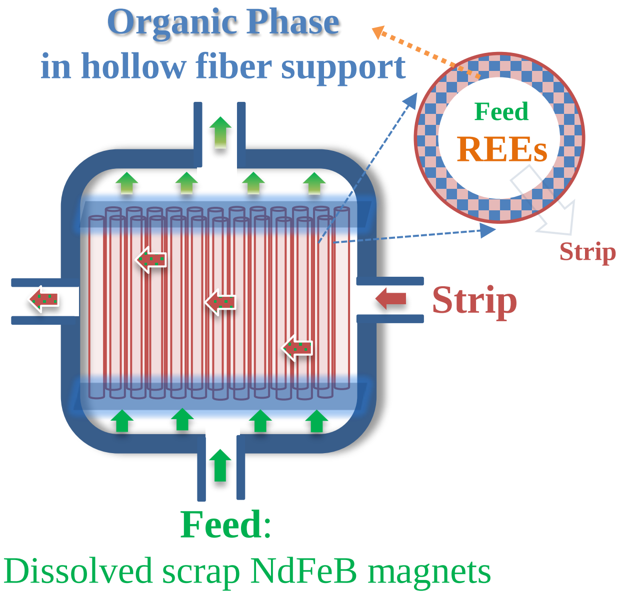
TRL	Stage	Metrics
TRL1	Basic Principles	[illegible]
TRL2	Application Formulated	[illegible]
TRL3	Proof of Concept	[illegible]
TRL4	Integrated Components	[illegible]
TRL5	Demonstrated with High Fidelity	Demonstration magnet recovery integrated lab scale metrics: Strategic partners leveraged 1 HDD/10 seconds Process profit must be economical for $500,000 estimated capital costs Magnet Processing Metrics?
TRL6	Demonstrated in Relevant Environment	Demonstration magnet recovery integrated pilot scale metrics: >10 magnets/hr automated HDs and small motors Full UI developed & demo

Critical Materials Institute

Membrane Solvent Extraction for REE Recovery

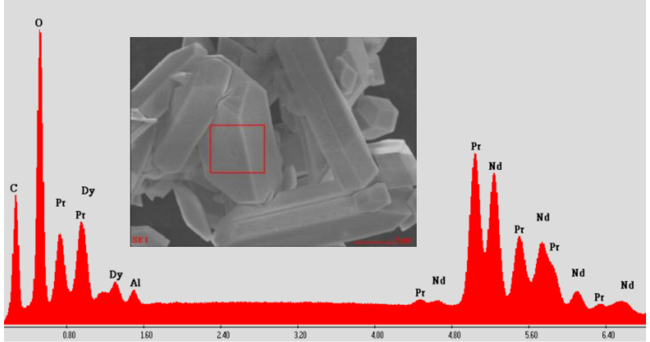
Oak Ridge National Laboratory
Idaho National Laboratory

- **Project goal**: recover high purity REEs suitable for reuse and recycle from scrap magnets dissolved in strong acids with highly selective extractants.
- **Impact if successful**: 1) major impact on process economics as the recovered REEs can be directly recycled without additional chemical/physical processing. 2) greatly minimizes hazardous waste compared to traditional separation technologies.
- **Current status**: successfully demonstrated the recovery of high purity REEs from several scrap magnet samples provided by industrial partners.
- **Next steps**: complete lab-scale extended duration tests (~1000 hours) using larger area modules.
- **Path to commercialization, and target date**: work closely with Molycorp and potential industry partners, and prepare for field demonstration by June 2017.



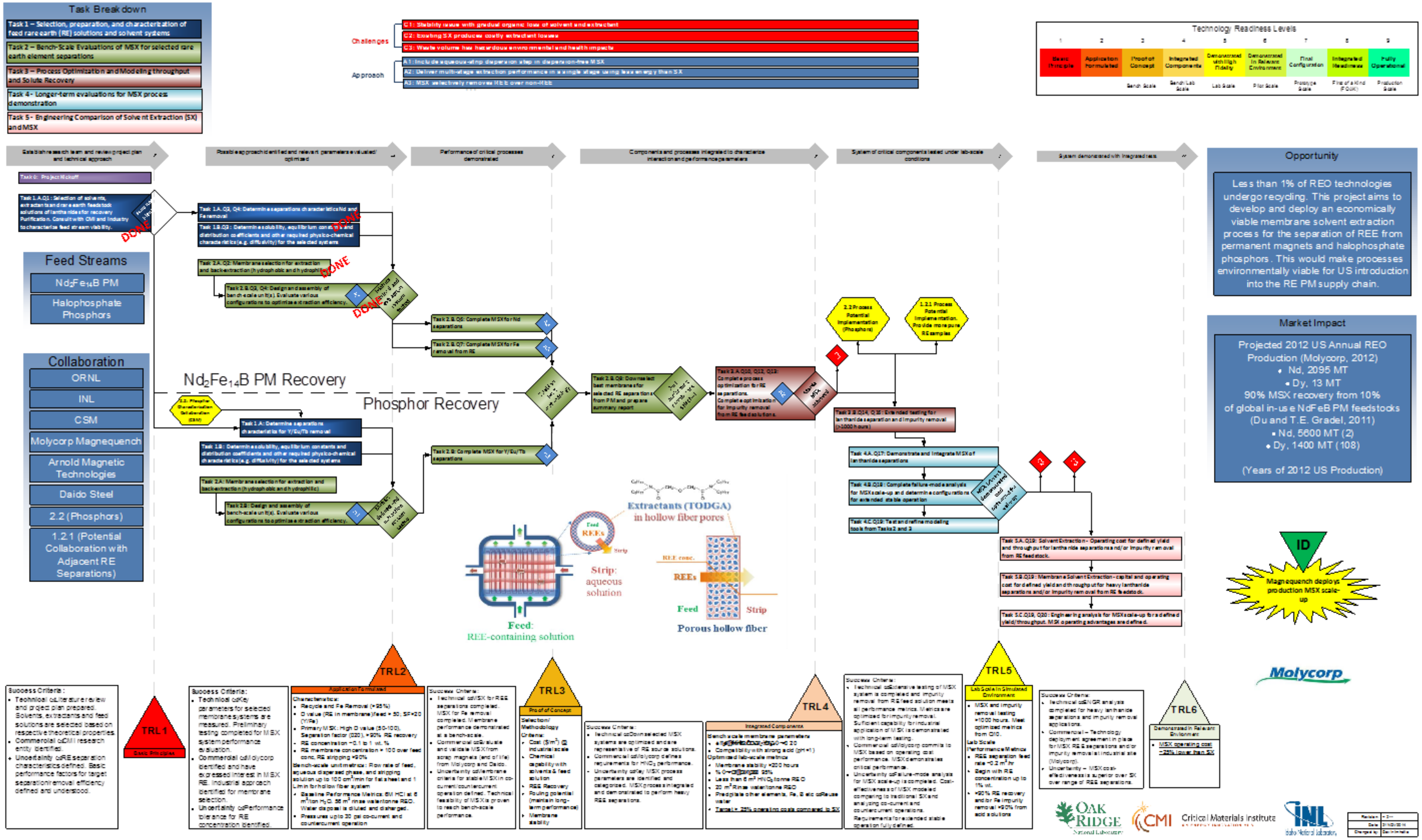
Organic Phase
in hollow fiber support

Feed:
Dissolved scrap NdFeB magnets



No co-extraction of non-REEs

Critical Materials Institute → Improving Reuse & Recycling → Transformative Extraction & Materials Production → Membrane Solvent Extraction for Rare Earth Separations (Project 3.2.2)



Task Breakdown

- Task 1 – Selection, preparation, and characterization of feed rare earth (RE) solutions and solvent systems
- Task 2 – Bench-Scale Evaluations of MSX for selected rare earth element separations
- Task 3 – Process Optimization and Modeling throughput and Solute Recovery
- Task 4 - Longer-term evaluations for MSX process demonstration
- Task 5 - Engineering Comparison of Solvent Extraction (SX) and MSX

Challenges
- C1: Stability issue with gradual organic loss of solvent and extractant
- C2: Existing SX produces costly extractant losses
- C3: Waste volume has hazardous environmental and health impacts

Approach
- A1: Include aqueous-strip dispersion step in dispersion-free MSX
- A2: Deliver multi-stage extraction performance in a single stage using less energy than SX
- A3: MSX selectively removes REE over non-REE

Technology Readiness Levels

1	2	3	4	5	6	7	8	9
Basic Principle	Application Formulated	Proof of Concept	Integrated Components	Demonstrated with High Fidelity	Demonstrated in Relevant Environment	Final Configuration	Integrated Readiness	Fully Operational
		Bench Scale	Bench/Lab Scale	Lab Scale	Pilot Scale	Prototype Scale	First of a Kind (FOAK)	Production Scale

- Task 1.A.Q1: Selection of solvents, extractants and rare earth feedstock solutions of lanthanides for recovery Purification. Consult with CMI and Industry to characterize feed stream viability.
- Task 1.A.Q3, Q4: Determine separations characteristics Nd and Fe removal
- Task 1.B.Q3: Determine solubility, equilibrium constants and distribution coefficients and other required physico-chemical characteristics (e.g. diffusivity) for the selected systems
- Task 2.A.Q2: Membrane selection for extraction and back-extraction (hydrophobic and hydrophilic)
- Task 2.B.Q3, Q4: Design and assembly of bench-scale unit(s). Evaluate various configurations to optimize extraction efficiency.
- Task 2.B.Q5: Complete MSX for Nd separations
- Task 2.B.Q7: Complete MSX for Fe removal from RE
- Task 1.A: Determine separations characteristics for Y/Eu/Tb removal
- Task 1.B: Determine solubility, equilibrium constants and distribution coefficients and other required physico-chemical characteristics (e.g. diffusivity) for the selected systems
- Task 2.A: Membrane selection for extraction and back-extraction (hydrophobic and hydrophilic)
- Task 2.B: Design and assembly of bench-scale unit(s). Evaluate various configurations to optimize extraction efficiency.
- Task 2.B: Complete MSX for Y/Eu/Tb separations
- Task 2.B.Q8: Downselect best membranes for selected RE separations from PM and prepare summary report
- Task 3.A.Q10, Q12, Q13: Complete process optimization for RE separations. Complete optimization for impurity removal from RE feed solutions.
- Task 3.B.Q14, Q15: Extended testing for lanthanide separation and impurity removal (>1000 hours)
- Task 4.A.Q17: Demonstrate and Integrate MSX of lanthanide separations
- Task 4.B.Q18: Complete failure-mode analysis for MSX scale-up and determine configurations for extended stable operation
- Task 4.C.Q19: Test and refine modeling tools from Tasks 2 and 3
- Task 5.A.Q19: Solvent Extraction – Operating cost for defined yield and throughput for lanthanide separations and/or impurity removal from RE feedstock.
- Task 5.B.Q19: Membrane Solvent Extraction - capital and operating cost for defined yield and throughput for heavy lanthanide separations and/or impurity removal from RE feedstock.
- Task 5.C.Q19, Q20: Engineering analysis for MSX scale-up for a defined yield/throughput. MSX operating advantages are defined.

Feed Streams

- $Nd_2Fe_{14}B$ PM
- Halophosphate Phosphors

Collaboration

- ORNL
- INL
- CSM
- Molycorp Magnequench
- Arnold Magnetic Technologies
- Daido Steel
- 2.2 (Phosphors)
- 1.2.1 (Potential Collaboration with Adjacent RE Separations)

Opportunity

Less than 1% of REO technologies undergo recycling. This project aims to develop and deploy an economically viable membrane solvent extraction process for the separation of REE from permanent magnets and halophosphate phosphors. This would make processes environmentally viable for US introduction into the RE PM supply chain.

Market Impact

Projected 2012 US Annual REO Production (Molycorp, 2012)
- Nd, 2095 MT
- Dy, 13 MT

90% MSX recovery from 10% of global in-use NdFeB PM feedstocks (Du and T.E. Graedel, 2011)
- Nd, 5600 MT (2)
- Dy, 1400 MT (108)

(Years of 2012 US Production)

Magnequench deploys production MSX scale-up

TRL1 – Success Criteria:
- Technical → Literature review and project plan prepared. Solvents, extractants and feed solutions are selected based on respective theoretical properties.
- Commercial → CMI research entity identified.
- Uncertainty → RE separation characteristics defined. Basic performance factors for target separation/removal efficiency defined and understood.

TRL2 – Success Criteria:
- Technical → Key parameters for selected membrane systems are measured. Preliminary testing completed for MSX system performance evaluation.
- Commercial → Molycorp identified and have expressed interest in MSX RE. Industrial approach identified for membrane selection.
- Uncertainty → Performance tolerance for RE concentration identified.

Application Formulated – Characteristics:
- Recycle and Fe Removal (>95%)
- D value (RE in membrane)/feed > 50; SF>20 (Y/Fe)
- Primary MSX: High D value (50-100), Separation factor (≥20), >90% RE recovery
- RE concentration ~0.1 to 1 wt. %
- RE membrane concentration > 100 over feed conc, RE stripping >90%

Bench-scale unit metrics: Flow rate of feed, aqueous dispersed phase, and stripping solution up to 100 cm³/min for flat sheet and 1 L/min for hollow fiber system
- Baseline Performance Metrics: 6M HCl at 6 m³/ton H_2O. 56 m³ rinse water/tonne REO. Water disposal is diluted and discharged.
- Pressures up to 30 psi co-current and countercurrent operation

TRL3 – Success Criteria:
- Technical → MSX for REE separations completed. MSX for Fe removal completed. Membrane performance demonstrated at a bench-scale.
- Commercial → Evaluate and validate MSX from scrap magnets (end of life) from Molycorp and Daido.
- Uncertainty → Membrane criteria for stable MSX in co-current/countercurrent operation defined. Technical feasibility of MSX is proven to reach bench-scale performance.

Proof of Concept – Selection/ Methodology Criteria:
- Cost (\$/m²) @ industrial scale
- Chemical capability with solvents & feed solution
- REE Recovery
- Fouling potential (maintain long-term performance)
- Membrane stability

TRL4 – Success Criteria:
- Technical → Down selected MSX systems are optimized and are representative of RE source solutions.
- Commercial → Molycorp defines requirements for HNO_3 performance.
- Uncertainty → Key MSX process parameters are identified and categorized. MSX process integrated and demonstrated to perform heavy REE separations.

Integrated Components – Bench scale membrane parameters:
- [illegible] 20
- Compatibility with strong acid (pH <1)

Optimized lab-scale metrics
- Membrane stability >200 hours
- % [illegible] 95%
- Less than 6 m³ HNO_3/tonne REO
- 20 m³ Rinse water/tonne REO
- Precipitate other elements, Fe, B etc → Reuse water
- Target > 25% operating costs compared to SX

TRL5 – Success Criteria:
- Technical → Extensive testing of MSX system is completed and impurity removal from RE feed solution meets all performance metrics. Metrics are optimized for impurity removal. Sufficient capability for industrial application of MSX is demonstrated with long-term testing.
- Commercial → Molycorp commits to MSX based on operating cost performance. MSX demonstrates critical performance.
- Uncertainty → Failure-mode analysis for MSX scale-up is completed. Cost-effectiveness of MSX modeled comparing to traditional SX and analyzing co-current and countercurrent operations. Requirements for extended stable operation fully defined.

Lab Scale in Simulated Environment:
- MSX and impurity removal testing >1000 hours. Meet optimized metrics from Q10.

Lab Scale Performance Metrics
- REE separation feed rate ~0.2 m³/hr
- Begin with RE concentration up to 1% wt.
- >90% RE recovery and/or Fe impurity removal >90% from acid solutions

TRL6 – Success Criteria:
- Technical → ENGR analysis completed for heavy lanthanide separations and impurity removal applications.
- Commercial – Technology deployment agreement in place for MSX REE separations and/or impurity removal at industrial site (Molycorp).
- Uncertainty – MSX cost-effectiveness is superior over SX over range of REE separations.

Demonstrated in Relevant Environment:
- MSX operating cost ~25% lower than SX

Focus Area 4

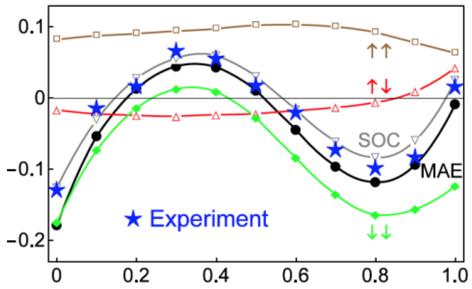
Crosscutting Research

- Providing tools and knowledge to support the other focus areas.

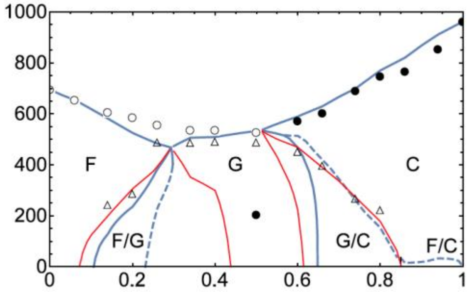
Multiscale modeling of permanent magnets

Ames Laboratory, Ames, IA
Livermore National laboratory, Livermore, CA

- Formulation of a multiscale method for the description of hysteretic phenomena in magnets
- Impact: Improving our understanding of known magnets with an opportunity to predict new magnets
- Current status: Created and applied two computational tools to describe magnetic phase transition and anisotropy in alloys as a function of temperature.
- Next steps: Experimental verification of predictions. Creating a link with nanoscale at finite temperature.



Anisotropy in $(Fe_xCo_{1-x})_2B$

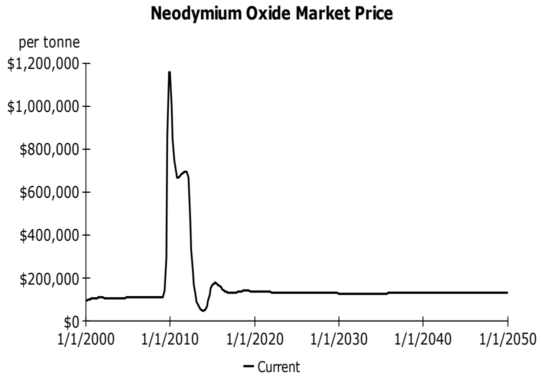


Magnetic phase diagram of $Fe_xMn_{1-x}Pt$ alloys

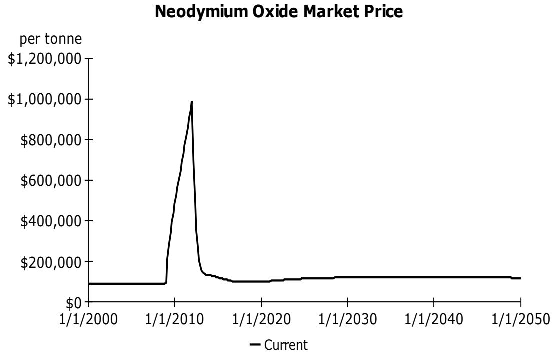
Economic Analysis of Global Materials Supply Chains

Idaho National Laboratory, Colorado School of Mines
Purdue University

- Build a better understanding of global material supply chains including CMI's impact on critical material economics
- Impact: Models will help CMI identify areas of importance and understand the global supply chain interaction.
- Current status: on schedule and budget
- Continue developing tier 2, expanded model. Convey and demonstrate results of tier 1 model.

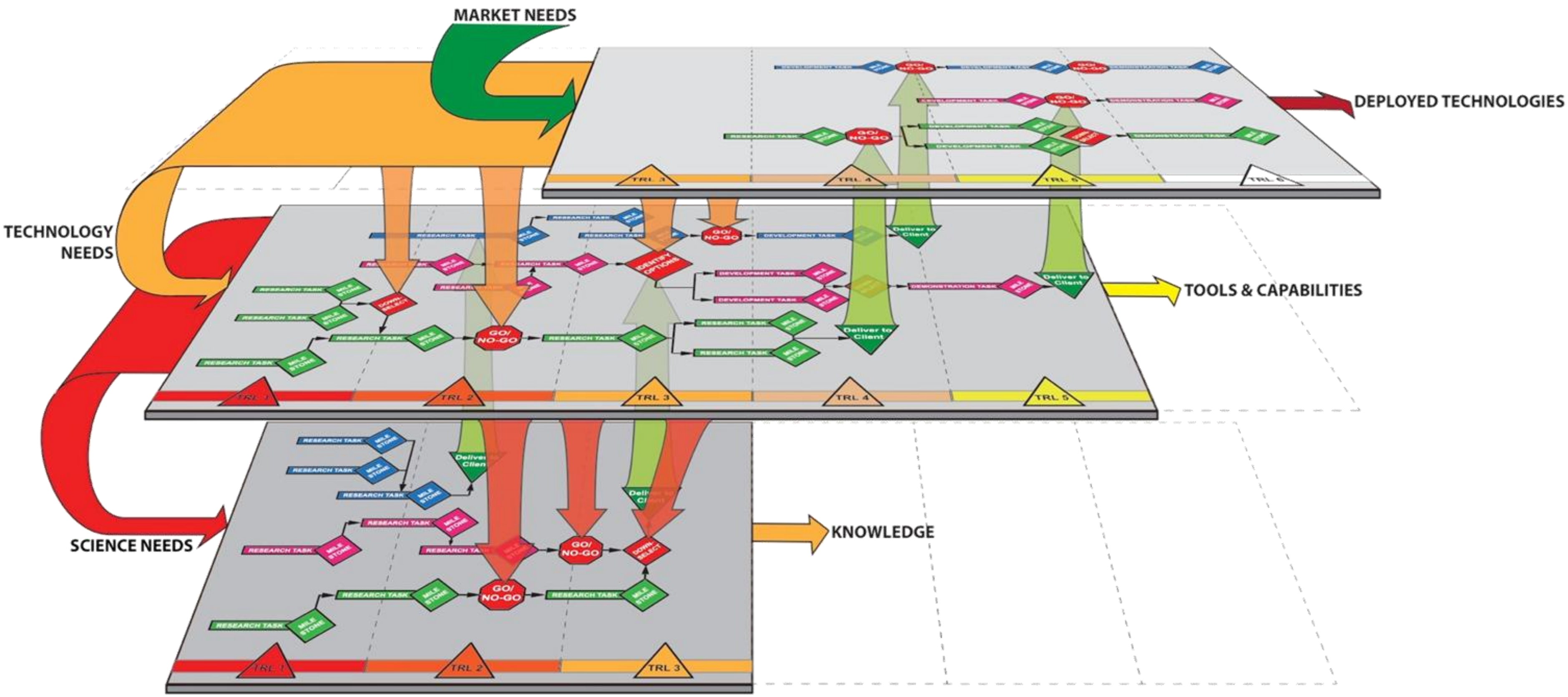


Generic system dynamics commodity market method



Dynamic supply and demand

Strategy



- Link fundamental research to product needs
  - Obtain industry input early and often
  - Link project roadmaps together
  - Link CMI-wide roadmaps to the outside world

Critical Materials Institute
AN ENERGY INNOVATION HUB

Thank You!

Achievements, to Date

The Critical Materials Institute has:

- Built an outstandingly capable research team of over 300 scientists, engineers and support staff
- Built a strong and energetic leadership team
- Established robust management capabilities
- Completed all major equipment acquisitions, providing unique and focused tools for the Hub’s work
- Established broad networks of industrial collaborations
- Established an exceptional level of global visibility
- Demonstrated the ability to adjust our program in response to emerging needs
- Proven that we can significantly accelerate the process of bringing innovations to the marketplace

Invention Disclosures

1. Extraction of rare earth elements from phosphoric acid streams
2. Extraction of rare earths from fly ash
3. Recovery of neodymium from neodymium iron boride magnets
4. Membrane solvent extraction for rare earth separations ✪
5. Selective composite membranes for lithium extraction from geothermal brines
6. Methods of separating lithium-chloride from geothermal brine solutions
7. Recovery of Dy-enriched Fe alloy from magnet scrap alloy via selective separation of rare earth elements ✪
8. Aluminum nitride phosphors for fluorescent lighting ✪
9. Novel surface coatings to improve the functional properties of permanent magnets
10. Additive manufacturing of bonded permanent magnets using a novel polymer matrix

✪ *Patent Applied For*

Invention Disclosures

11. Ceria-based catalyst for selective phenol hydrogenation under mild reaction conditions
12. Recycling and conversion of samarium cobalt magnet waste into useful magnet
13. Catalysts for styrene production
14. Task specific ionic liquids extractive metallurgy or rare earth minerals
15. Separation of neodymium from praseodymium
16. High throughput cost effective rare earth magnets recycling system
17. Recycle of Fe Nd B Machine Swarf and Magnets ✪
18. Directly Printing Rare Earth Bonded Magnets
19. Procedure for Concentrating Rare-earth Elements in Neodymium Iron Boron-based Permanent Magnets for Efficient Recycling/Recovery
20. Enhancing Consumer Product Recycling via Rapid Fastener Eradication

✪ *Patent Applied For*

Invention Disclosures

21. Automated Printed Circuit Board Disassembly by Rapid Heating
22. Electrochemistry Enabled Recovery of Value Metals from Electronics
23. Synthesis of High Surface Area Mesoporous Ceria
24. Self-Assembly of Low Surface Colloidal Nanoparticles into High Surface Area Networks
25. Selective Chemical Separation of Rare-Earth Oxalates (CSEREOX)
26. Carbothermic Preparation of $SmCo_x$ (x=5 to 8.5) Permanent Magnets Directly from Sm_2O_3
27. A One Step Process for the Removal of Nickel/Nickel Copper Surface Coating from the $Nd_2Fe_{14}B$ (neo) Permanent Magnets
28. Engineering Caulobacter Surface Protein for Rare Earth Element Absorption
29. Chemical Separation of Terbium Oxide (SEPTER)
30. Novel Methods towards Selective Surface Modification of $Nd_2Fe_{14}B$ Magnets to Achieve High Performance Permanent Magnets

✪ *Patent Applied For*

Invention Disclosures

31. Mesoporous Carbon and Methods of Use
32. Castable High-Temperature Ce-Modified Al Alloys
33. High Command Fidelity Electromagnetically Driven Calorimeter (High-CoFi EleDriCal)
34. 3D Printable Liquid Crystalline Elastomers with Tunable Shape Memory Behaviors and Bio-derived Renditions
35. Direct Write Additive Manufacturing Method for Heat Exchanger Production using Al-Ce-X Alloys

✪ *Patent Applied For*