CRADA 2012-04

Partner Name(s); KITECH and Ames Laboratory (DOE

# International Cooperation to Development of Strategy and R&D Collaboration for Substitution of Rare Earth Resources

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# 1. Executive Summary

A Memorandum of Understanding was signed between the Ames Laboratory and Korea Institute of Industrial Technology (KITECH) in April of 2011 to establish a close cooperative relationship of research on Rare Metals. The Ames Laboratory through its operating contractor, Iowa State University, and the KITECH, as one of the goals of a joint Memorandum of Understanding, made agreement on a Cooperative Research and Development Agreement (CRADA) for a personnel exchange in April of 2012. Ames Laboratory and KITECH would be jointly performing collaboration work in this cooperation as the Parties. The individual backgrounds and capabilities of the Parties are ideally suited to the successful execution of this work. The Ames Laboratory has a history spanning more than 60 years of pioneering work in rare-earth (RE) research. With its team of internationally recognized rare-earth experts, the Ames Laboratory is uniquely positioned to provide the knowledge, expertise and training necessary to help ensure a global leadership position for the United States in rare-earth research, development and applications. The KITECH was founded to promote technological capability of small and medium-sized enterprises in Korea and has a mission of supporting research on materials science and engineering in Korea. In 2009, Korea Institute for Rare Metals (KIRAM) at KITECH was established. KIRAM makes selection and funding decisions for technology projects for strategic rare metals, and provides oversight of R&D programs. The KIRAM is focusing on the commercialization and recycling of particular rare metals. In addition KIRAM will be coordinating government, local universities, and small to medium enterprises to facilitate R&D support for core technologies. KIRAM will be carrying them through to commercialization, and establishing new industry.

During the last two years both KITECH and Ames Laboratory successfully performed research collaboration on (1) International cooperation to development of strategy (2) R&D collaboration for substitution of rare earth resources. The objective of this work was to prepare and to investigate common response between Korea and the US on the rare metals issue including rare earths. The scope of research places rare metals in historical context, highlights factors in markets, and reviews policy recommendations made by various governments and studies including the academic research report. As a result of cooperation, both KITECH and Ames Laboratory are performing the R&D collaboration for the substitution of rare earth resources. Substitution means finding alternative rare earth resources to replace original raw supply resources, which is focused on the reclaiming the rare earth resource from the waste materials. For example, the recycling of rare earths from magnet scraps. The research team includes Ames Laboratory scientist with representatives from KITECH. The research group is developing and testing the technique in Ames Lab's Materials Preparation Center, with a suite of materials science tools supported by the DOE Office of Science. Scientists at the Ames Laboratory and KITECH are working to more effectively remove the neodymium, a rare earth element, from the mix of other materials in a magnet. Initial results show recycled materials maintain the properties that make rare-earth magnets useful. Indeed, we propose to use a rapid assessments approach using by 3D additive manufacturing system based on bulk combinatorial materials science to significantly expand our search area of phase space, in a fraction of the time needed compared to using conventional synthesis and characterization techniques. By using this approach we will identify a composition region of interest and more closely focus our combinatorial approach on this region to identify compositions with enhanced magnetocaloric properties.

As a result of the base of these all efforts, in September of 2013, the U.S. Department of Energy opened a new Energy Innovation Hub, to be known as the Critical Materials Institute (CMI). Based on the successful collaboration between KITECH and Ames Laboratory during the first period, it is the proper time to expand the next step of cooperation of both institutes through R&D collaboration in various subjects, as well as opening the Joint Research Laboratory (JRL).

# 2. Characteristics of Rare Earth (RE)

## 2.1. Background

In today's environment in an effort to ensure supply for renewable energy technologies, science decision makers are formulating policy to mitigate supply risk. Sometimes this formulation is being done without a complete picture of the complexity of the various factors that contribute to the demand. Although rare metals or rare earths are not traded through an open commodities market, public attention was drawn to rapid price increases for many of the rare earth elements, beginning in 2010. As prices rose to astronomical levels-eventually reaching more than 10 times their pre-2010 levels for some elements-concerns emerged concerning the needs for these elements, and the security and stability of their supply. Metals that had previously been the province of a few technical specialists began to be discussed in the popular media, and political attention soon followed.

Rare metals have been called "vitamins of industry" and their importance in industry has been recognized for some time. However, recently, the industry has become highly dependent on products that cannot be made without using rare metals. Rare metals are becoming "the lifeline of industry". Examples of rare metals are indium (In), chromium (Cr), tungsten (W), cobalt (Co), manganese (Mn), and molybdenum (Mo), vanadium (V) etc. These rare metals, especially in industrial products, are essential materials for high performance and high functionality of industrial products and are key resources for mainstream industries. Therefore, the rare metals become a key issue, since they are quite rare in the earth's crust and are difficult to extract from ores. Although supply constraints affect many elements, this study will focus on elements that have appeared in novel technologies. It will focus on the demands that would be created by the growing need for rare elements as a result of the widespread adoption of a new technology. Recent examples of this kind of event include the explosive growth of use and subsequent wild fluctuations in the price of indium and tellurium (Te) over the past decade. Some elements are needed only at relatively low intensity (e.g. platinum in catalysts or tellurium in photovoltaic films), but may be extremely rare in the earth's crust. Other elements, like indium or silver, may be slightly more common, but would be needed in much larger quantities. Still others, such as neodymium (Nd) or lithium (Li), may be relatively common, but rarely concentrated by mineralogical processes. Moreover, some elements are particularly abundant in one country, but are not found in economically viable deposits in another country, raising geopolitical issues should demand for them escalate. Others are only produced in very limited quantities as co-products with other extraction operations so that their supply would not necessarily respond to demand. The importance for securing rare metals is different with circumference dependent of each country. Depending on the valance between the supply and demand of each country, the number of rare metals is distinguished by economic and political circumferences of countries. The unusually broad scope of this issue requires the involvement of experts in legislation, policy, administration, industrial structure, geology, material science, mineralogy, mining engineering, economics, and other disciplines. Co-sponsorship of this study by the government organization and collaboration with other scientific societies will facilitate the broad approach needed for this topic.

## 2.2. Objective

The aim of this work was to prepare and investigate common response between Korea and the United States on the rare metals issue including rare earths. The effort described in the summary is especially appropriate in view of the unique areas of expertise possessed by the Ames Laboratory and KITECH. Due to this fact, each institute's expertise and approach will be used to reach a synonymous goal. In today’s environment with an effort to ensure supply for renewable energy technologies, science decision makers are formulating policy to mitigate supply risk. This is sometimes being done without a complete picture of the complexity of the various factors that contribute to the demand. We will review various reports, as well as responses by various governments to the rare metals supply risk. After defining rare metals, we will discuss some aspects of their supply chain and markets, US and Korean policy developments, factors specific to rare earth elements, critical materials lists from selected countries, and the recommendations of a policy study by the scientific societies on rare metals. The proposed work will enhance the Ames Laboratory's thrust in rare earth metals and rare earth recycling, a core competency of the Ames Laboratory. This will develop a close working relationship with the KITECH who has a commitment to rare earth recycling and who has funded such work at the Ames Laboratory's facility (i.e., Materials Preparation Center; MPC) under work for others.

## 2.3. Definition of RE

Rare metals are defined as metals which are rare in the earth crust and also are difficult to extract from ores. In addition to the rarity, the mal-distribution of natural resources is a critical point in global materials flow. Even though the difficulties in accessibility to natural resources are similar with fossil fuels, requirements for total amounts of rare metals have increased as the global economics grows. It has been said that we are now in rare metal age and through the steel age. The terminology, “rare metal”, is not an academically defined one, and there is no consensus on which elements it pertains. In Korea, the term is often used to refer to the 11 metals elements shown in Figure 1, according to the definition set by the KIRAM with Korea Ministry of Trade, Industry and Energy.


Figure 1: Classification of rare metals in Korea (KIRAM report)

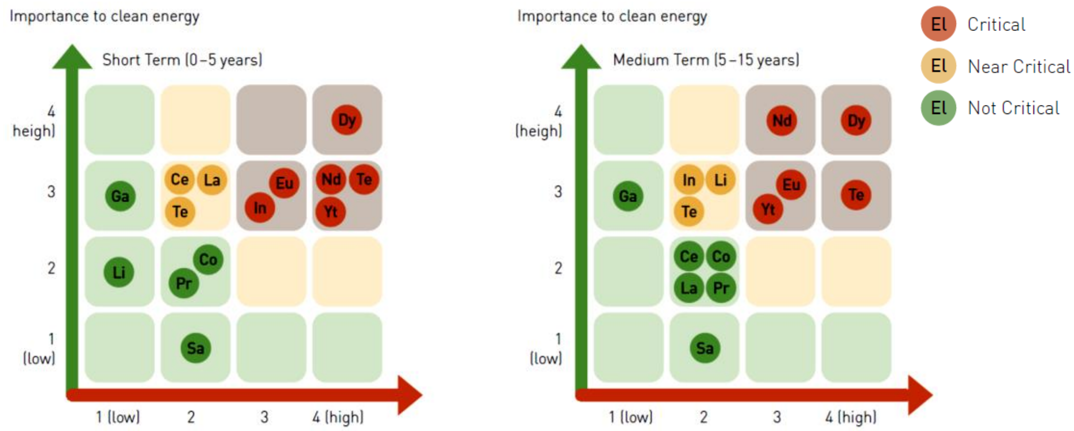



Figure 2: Critical levels of some elements (DOE report)

The definition of rare metals in the US is used to refer to the “Critical Materials” which are the 5 elements (neodymium, europium, terbium, dysprosium and yttrium) including near critical elements shown in Figure 2. This is the definition of rare metals according to the proposition by the Department of Energy’s Critical Materials Strategy. Sometimes, the 17 rare earth elements are counted as one kind. Usually there are a total of 56 existing elements with 35 species in the natural world. In order to select world common rare metals, it is important to understand that the definitions of rare metals are not exactly the same in each country. The following tables show rare metals tracked in different countries: The United States (Table 1), Japan (Table 2) and Korea (Table 3). Typically, some metals are defined as a rare metal in one country but not in another. However, there are common metals which are defined as rare metals in all countries listed below.

| Group | Elements |
|---|---|
| Alkaline earth metal | Li, Ce, Be, Sr, Ba |
| Metalloid | Ge, Bi, Se, Te, Si |
| Iron group | Co |
| Boron group | B, Ga, In, Tl, Cd |
| High fusion point metal | Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W, Mn, Re |
| Rare earth | La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Sc, Y |
| Platinum group | Ru, Rh, Pd, Os, Ir, Pt |
| Other group | Ca, Rb, Th, U, Pu |

Table 1: Rare metals in United States (ITU report)

| Group | Elements |
|---|---|
| Alkaline earth metal | Li, Ce, Be, Sr, Ba |
| Metalloid | Ge, As, Sb, Bi, Se, Te, Sn |
| Iron Group | Co, Ni |
| Boron Group | B, Ga, In, Tl |
| High fusion point metal | Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W, Mn, Re |
| Rare Earth | La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Sc, Y |
| Platinum Group | Ru, Rh, Pd, Os, Ir, Pt |

Table 2: Rare metals in Japan (ITU report)

| Group | Elements |
|---|---|
| Alkaline earth metal | Li, Mg, Ce, Be, Sr, Ba |
| Metalloid | Ge, P, As, Sb, Bi, Se, Te, Sn, Si |
| Iron group | Co, Ni |
| Boron group | B, Ga, In, Tl, Cd |
| High fusion point metal | Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W, Mn, Re |
| Rare earth | La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Sc, Y |
| Platinum group | Ru, Rh, Pd, Os, Ir, Pt |

Table 3: Rare metals in Korea (ITU report)

## 2.4. Analysis of demand

Aggregate demand for Rare earth elements (REEs) has grown significantly over the past decade, averaging increases of approximately 4.7% per annum from 2000 to 2010. Table 4 shows projected short-term growth of the REE group, indicating that demand already exceeds supply for some metals. Demand for metals from developed countries also stagnated as they began to emerge from the resource-intensive phase of their growth, contributing to lower prices. History suggests that a country's consumption of metals typically grows in line with per capita income until the country reaches a threshold of $15,000 to $20,000 income per capita (in PPP-adjusted dollars) at which time it goes through a period of industrialization and infrastructure building. At higher incomes, growth typically becomes more service-driven, and the per capita use of metals starts to stagnate.

There are two important factors to this general trend. First, countries have different development paths. For example, shown in Figure 3, China has experienced a much stronger urbanization shift than India, which has resulted in greater demand for steel for the construction of buildings and other urban infrastructure (at similar levels of per capita GDP to India's).

| Rare Earth Oxide Group | Demand: Tonnes REO | | Production: Tonnes REO | |
|---|---|---|---|---|
| | Global | ROW @ 35 % | Global | ROW |
| Light Rare Earths (La, Ce, Pr & Nd) | 145,000 t | 50,000 t | 165,000 t | 52,500 t<br>60,000 t |
| Medium Rare Earths (Sm, Eu & Gd) | 4,250 t | 1,500 t | 6,000 t | 1,000 t<br>1,350 t |
| Heavy Rare Earths (Tb, Dy, Er & Y) | 14,500 t | 5,000 t | 7,000 t | 300 t<br>750 t |

Forecast Global Rare Earths Demand in 2016 (t REO ± 15 %) (source: IMCOA and Rare Earth Industry Stakeholders)

| Application | China | Japan & NE Asia | USA | Others | Total | Market Share |
|---|---|---|---|---|---|---|
| Catalysts | 14,500 | 2,500 | 6,500 | 1,500 | 25,000 | 15 % |
| Glass | 6,000 | 1,000 | 1,000 | 1,000 | 9,000 | 6 % |
| Polishing | 19,000 | 2,000 | 3,000 | 1,000 | 25,000 | 15 % |
| Metal Alloys | 20,000 | 2,500 | 2,000 | 1,500 | 26,000 | 16 % |
| Magnets | 28,000 | 4,500 | 2,000 | 1,500 | 36,000 | 22 % |
| Phosphors (including Pigments) | 9,000 | 2,000 | 1,000 | 500 | 12,500 | 8 % |
| Ceramics | 4,000 | 2,000 | 2,000 | 1,000 | 9,000 | 6 % |
| Other | 6,500 | 3,500 | 8,000 | 2,000 | 20,000 | 12 % |
| Total | 107,000 | 20,000 | 25,500 | 10,000 | 162,500 | 100 % |
| Market Share | 66 % | 12 % | 16 % | 6 % | 100 % | |

Table 4: Estimated global REE demand in 2016, Red is committed and Blue is possible, ROW means rest of the world (UNEP report)

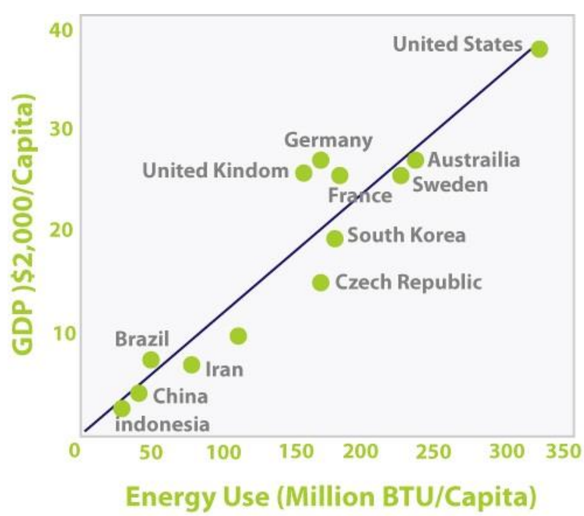



Figure 3: Estimated energy usage and GDP correlation (World Factbook)

Second, the mix of commodities that countries demand changes as they develop. Metals such as iron ore tend to attract stronger demand at a relatively low level of per capita GDP, which tends to be the case in the early stages of a country's shift out of agriculture and toward a more urban environment. Other metals such as platinum tend to be "late-cycle" commodities that attract demand at higher levels of per capita GDP.

Today, China is the world's largest producer and consumer of dozens of minerals and exports. The future supply of Rare earth oxide (REO) will mainly depend on the development of the total Chinese production and export quota. As shown in Figure 4, the supply forecasts for China in 2014 differ substantially. The estimate by Lynas of 114,000 tons, with 4,000 tons coming from recycling, is much less than the 160,000 to 170,000 tons. The projected demand for 2014 for the elements terbium, dysprosium, praseodymium and neodymium is higher than the estimated supply. This may lead to shortages for permanent magnets for wind turbines and hybrid and electric vehicles. Consequently, options for substitution of permanent magnets have to be considered. The potential shortage of neodymium and lanthanum may limit the production of NiMH batteries, necessitating substitution by Li-ion batteries. Shortages of europium and terbium affect the production of energy efficient lamps and displays and lack a substitution. Lanthanum is an indispensable element in catalysts for petroleum refining and processing for which there are no short term substitutions.

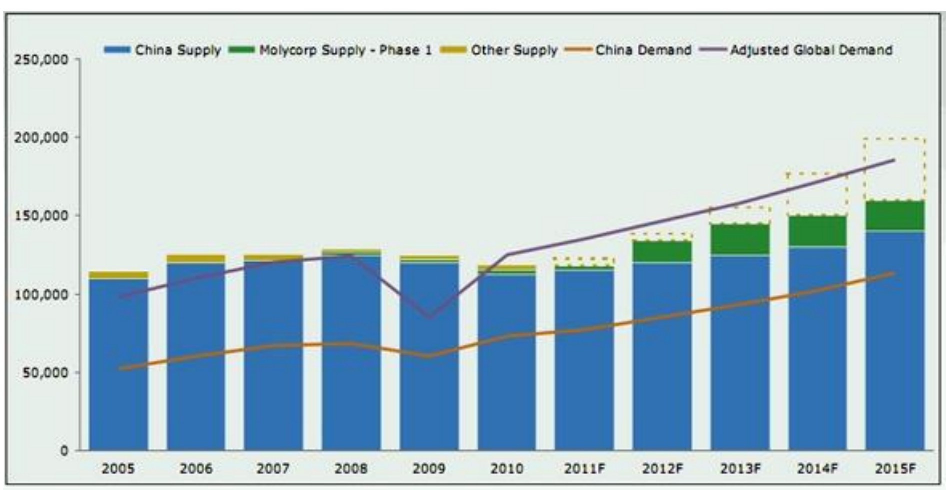



Figure 4: The global supply and demand of RE 2005-2015, mt, REO ±20% (IMCOA 2011)

### 2.4.1. Technological aspect

In the mining industry, the key inputs for the production of steel, iron ore, and coking, coal, currently account for around 30 percent of revenue. The importance of different metals has varied, especially REE produced as byproducts of dominant base metals, such as Cu, Al and Ni. (Figure 5) While steel, copper, and aluminum have always been dominant, other metals such as rare earths are today increasingly in demand (from a low base) due to their use in new consumer electronics, renewable energy, and military applications.

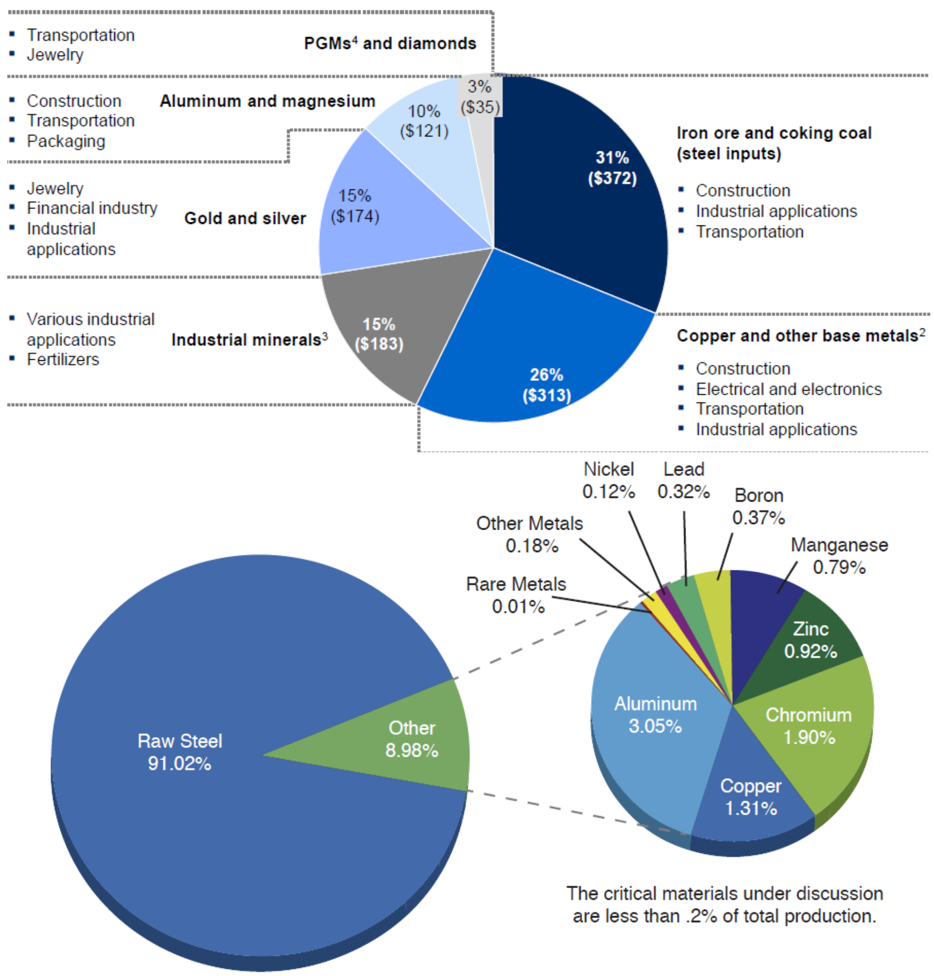



Figure 5: The spectrum of commodities in mining industry (100=US$1,198 bill.) and breakdown of metal production in 2009 (McKinsey and Resnick Institute reports)

In the United States, the government administration is making historic investments in clean energy. The American Recovery and Reinvestment Act was the largest one-time investment in clean energy - more than $80 billion. The DOE is investing $37 billion in Recovery funds in electric vehicles, batteries and advanced energy storage, smarter and more reliable electric grid, and wind and solar technologies, among many other areas. Through this investment, US will increase the renewable energy generation and manufacturing capacities. US will also deploy hundreds of thousands of electric vehicles and charging infrastructure to power them, weatherize at least half a million homes and expand their grid. The Chinese government is launching programs to deploy electric cars in 13 major cities, connecting urban centers with high-speed rail, building huge wind farms, ultra-supercritical advanced coal plants and ultra-high-voltage long-distance transmission lines with low line loss. India has launched an ambitious National Solar Mission, with the goal of reaching 20 gigawatts of installed solar capacity by 2020. In Europe, strong public policies are driving sustained investments in clean energy. Denmark is the world's leading producer of wind turbines, earning more than $4 billion each year in that industry. Germany and Spain are the world's top installers of solar photovoltaic panels, accounting for nearly three-quarters of a global market worth $37 billion last year. Other countries are also seizing this opportunity. Indeed, the market for clean energy technologies is growing rapidly all over the world. Around the world, investments in clean energy technologies are growing, helping create jobs, promote economic growth and fight climate change. These technologies will be a key part of the transition to a clean energy future. However, many of these technologies rely on the special properties of rare-earth metals. There's no reason to panic, but there's every reason to be smart and serious as we plan for growing global demand for products that contain rare earth metals and other strategic materials.

### 2.4.2. Industrial aspect (Trends by major demand sector)

Incremental world energy demand or emerging-market demand could increase by up to 15 percent depending on a range of plausible published projections of China's future growth rate and energy intensity (i.e., energy inputs per unit of economic output). It is projected that China's primary energy demand will grow by more than 2 percent per annum, accounting for more than 40 percent of incremental global energy demand by 2030, based on growth in China's real GDP of 6.8 percent per year.

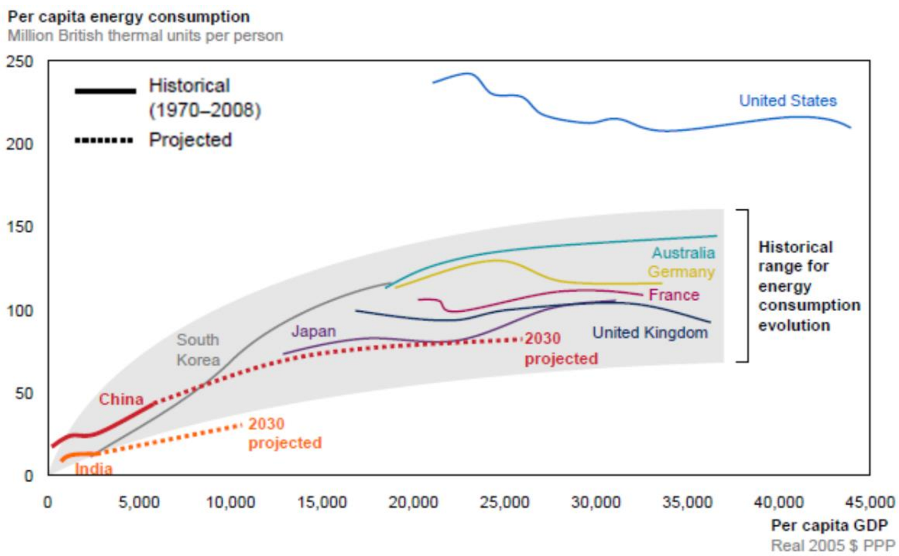



Figure 6: Resource demand compared to income rise (IHS Global Insight, McKinsey report)

In most developed countries, per capita energy consumption generally grows consistently until household income hits a threshold of $15,000 to $20,000 in purchasing power parity (PPP) terms. As shown in Figure 6, consumption then typically flattens as economies shift from energy-intensive industries, such as manufacturing, toward less energy-intensive service industries. China's current energy intensity is around the levels seen in South Korea and Singapore in the late 1980s. It can be assumed that, by 2030, China will reach per capita energy intensity around the level observed in South Korea and Singapore in the late 1990s.

### 2.4.2.1. Automotive industry;

Gasoline/Diesel engine vehicles, next-generation vehicles (HEV/PHEV/EV)

In the automotive industry, existing gasoline and diesel power vehicles are manufactured with a good deal of rare metals, as well as the next generation vehicles.

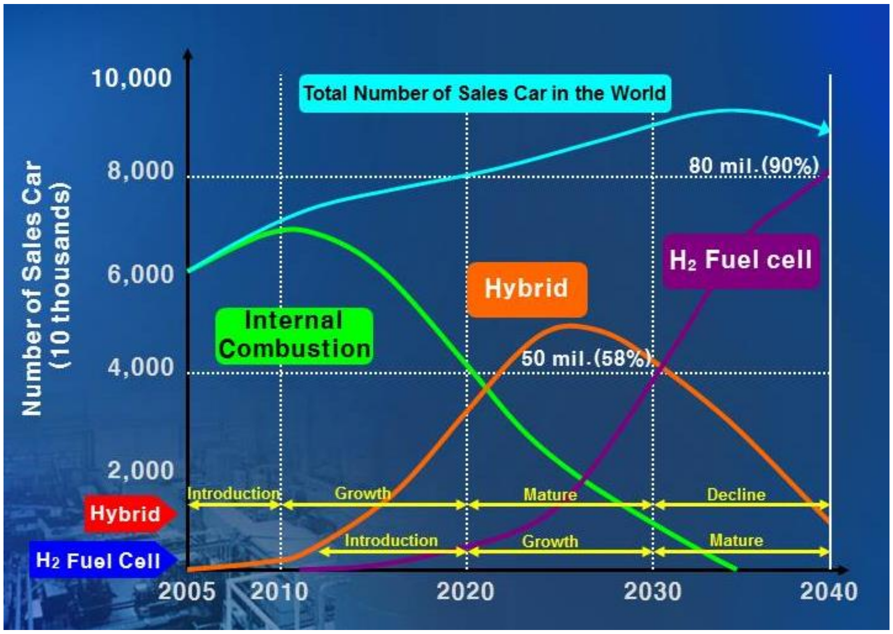



Figure 7: Estimated global automobile market trends (Automotive World Car Industry Forecast Report, Global Insight)

The demand for rare metals in this sector is focused on the additives for alloy metals. Cr, Mn and Ni, indispensable for standard and special steels account for the major portion of demand. Also, platinum groups used in three-way catalyst and oxygen sensor will remain important, though the volume is limited. In the meantime, the demand of Nd, Dy and Li for the use in next-generation vehicles appears to be in an increasing trend. The estimates on Figure 7 show that the proportion of demand breakdown by each kind of vehicle/ economy would be almost the same in 2015 as in 2010. China, among other parties, is estimated to continue to be the major consuming country in 2015 accounting for 60% of the world demand. The reason that China is in need of such a big amount implies that China is incentivized to increase the REE supply for itself. This would limit exportation to the rest of the world, and China supplies 40% of the total demand.

### 2.4.2.2. Energy-saving products;

Solar cell, lithium-ion battery, LED, fuel cell, wind power generation

Key energy-saving products such as solar cells, lithium-ion batteries, LEDs, fuel cells and wind power generation are reviewed in this research. The rare metal demand in 2009 was centered on the lithium-ion battery; especially Co, Mn, and Ni used in the positive electrode materials account for the major portion of demand. Currently Japanese positive electrode material manufacturers have overwhelming shares in the global market, while the domestic demand is increasing. In the future the demand for rare metals is expected to increase along with the full-scale commercialization and expansion of the new products such as fuel cells, solar cells and LED. During the next decade the demand for high performance rechargeable batteries will increase as a result of market evolution. This evolution includes the electrical and electronic portable equipment and electric vehicle market. In the markets for portable electronic equipment and other cordless equipment, the sustained demand for rechargeable batteries observed over the last ten years should continue.

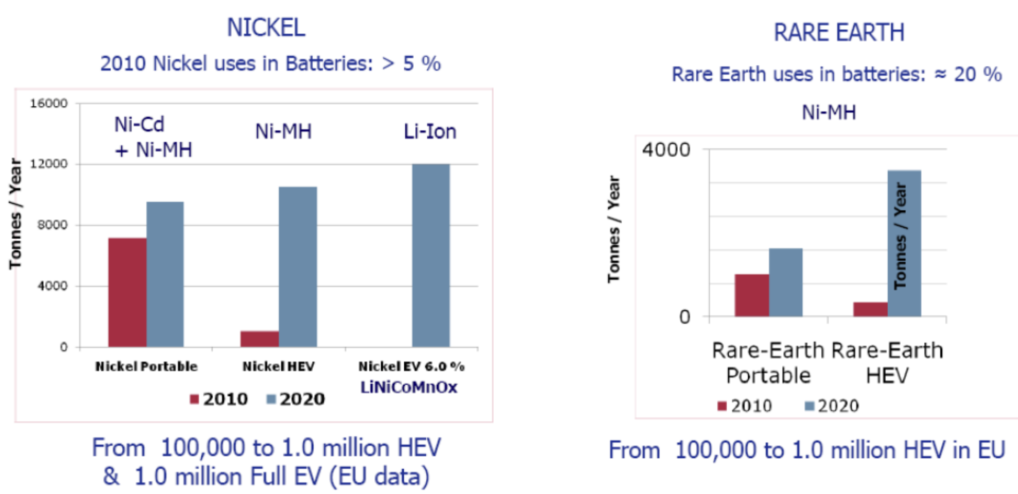



Figure 8: Trends the usage of battery materials in the EU (Ad-hoc Working Group report)

The growth rate in materials demand is thus expected to reach 5% per year over the next decade. This will require an increased use of rare earths, as well as Ni, Co and Li (notably nickel and cobalt-based specialty chemicals, and lithiated metallic oxides). Given the greater uncertainty associated with the development of the electric vehicle being more speculative, a conservative evolution has been assumed. The resulting demand for rare earths, nickel, cobalt and lithium is based on a production of 1.0 million electric or hybrid electric vehicles in 2020. Figures 8 displays the evolution of tonnes of rare earths, nickel, cobalt and lithium contained in portable batteries used in the EU between 2010 and 2020. Their tonnage in portable batteries might be multiplied by a factor of three to four for rare earths and nickel, up to about six for cobalt, and more than ten for lithium.

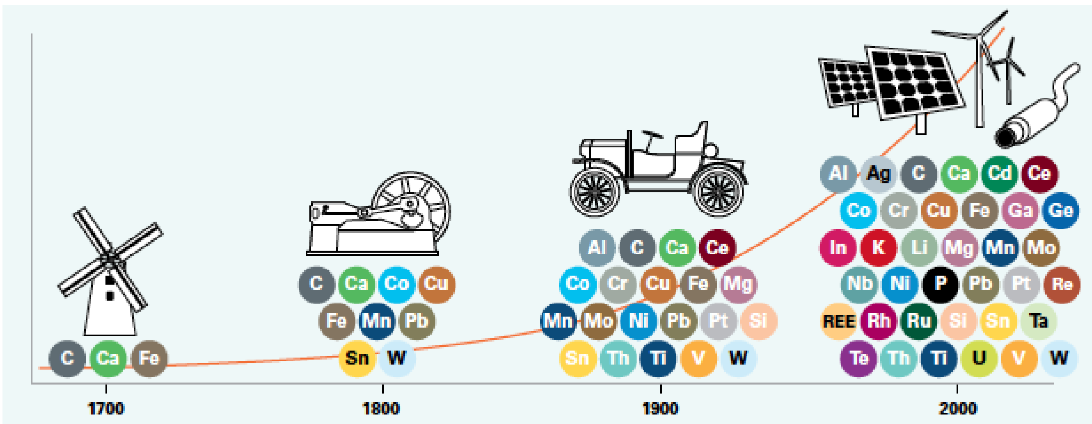



Figure 9: Diversity of elements used in advancing energy pathways (Materials critical to the energy industry, A. Reller)

Machines, devices and components which are designed to store and transport energy also need the rare elements. The role of such elements is important and crucial for the economy and ecology. All these new technologies require specific elements for their specific functionalities and the higher the technological innovation, the higher the number of elements used for the final technology as shown in Figure 9. To the contrary energy is necessary to obtain the required elements, process the raw materials to the final components and retrieve the elements from the end-product when it is no longer in use.

### 2.4.2.3. Home appliance electronics;

Mobile phone, digital camera, PC, TV, washing machine, refrigerator

Electronics products such as mobile phones, PCs, DSCs, TVs, washing machines, refrigerators, and air-conditioners are reviewed in this research. A great number of rare metals are being used in electronics products. The rare metals are used as additives in very small quantity for controlling the part quality and performance. The demand of rare metals for this sector in 2009 was centered on the capacitors, plating, batteries, motors and HDD used for mobile phones, digital cameras, PCs, TVs, washing machines, refrigerators and air conditioners. In the future the demand of Ni will increase centered on capacitors. The demand of indium, which is used for transparent electrode material, is likely to increase more than ever as applications such as touch panels and e-books become more popular. In 2002–2003 "indium depletion issues" were pointed out. As the possibility of running into indium shortage is undeniable, there are world-wide activities on the material composition change to avoid using indium, reduction of indium use volume by reducing the thickness of indium layer, and the development of alternative materials.

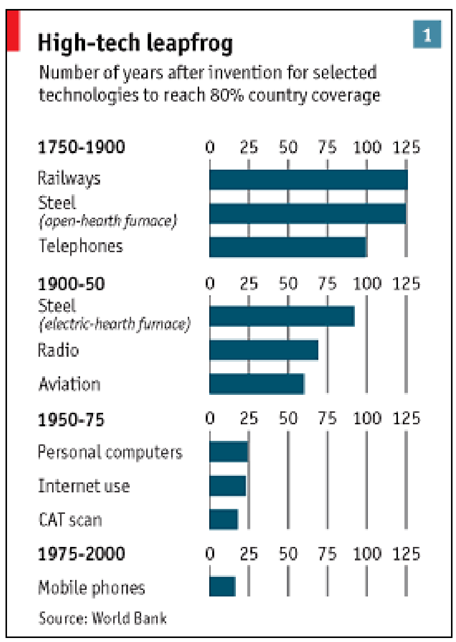



Figure 10: Technology adoption timeline in emerging market (The World Bank report)

The Figure 10 shows a technology adoption timeline from 1900 to 2005. The upshot is that technology is spreading to emerging markets faster than it has ever done anywhere. The study looked at how much time elapsed between the invention of a product and its widespread adoption (defined as when 80% of countries that use a technology first report it). For 19th-century technologies the gap was long: 120 years for trains and open-hearth steel furnaces, 100 years for the telephone. For aviation and radio, invented in the early 20th century, the lag was 60 years. But, for example, the gap of the PC was around 20 years and for mobile phones just 16 years. In almost all industrialized countries, once a technology is adopted it goes on to achieve mass-market scale, reaching 25% of the market for that particular device. In the World Bank's database, there are 28 examples of a new technology reaching 5% of the market in a rich country; of those, 23 went on to achieve over 50%. In other words, if something gets a foothold in a rich country, it usually spreads widely.

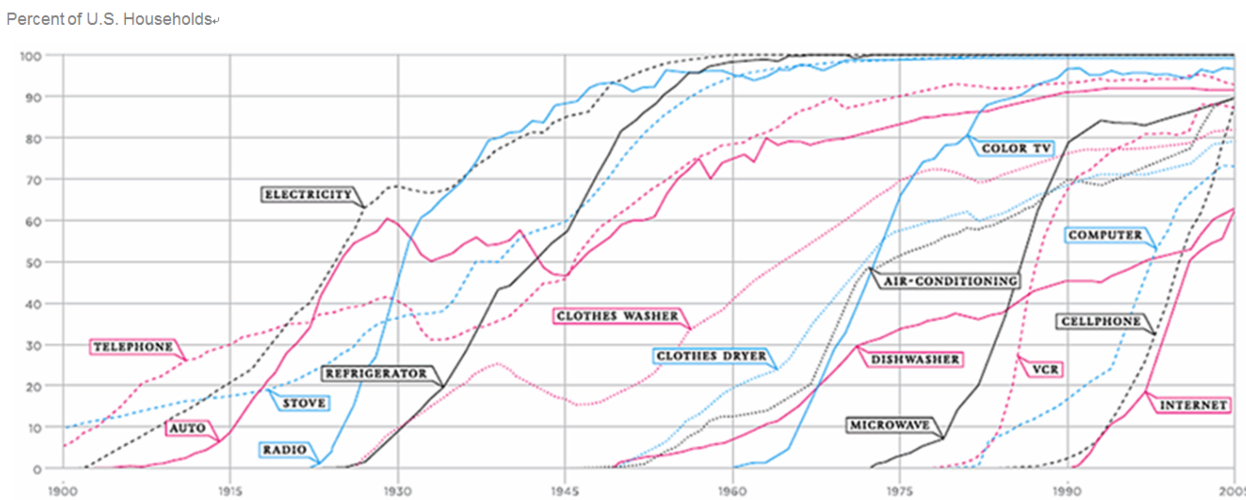



Figure 11: The consumption spreads rate of appliances and devices (NY Times)

The rate of adoption of consumer technologies since the beginning of the previous century has been rapidly increasing, so much so that today’s technology spreads at a rate never seen before. The following are U.S. stats that also describe similar trends in other Western countries and, to a large extent, a clear global trend. Electricity reached 90% penetration of U.S. households within 50 years; the refrigerator accomplished the same in 30 years, cell phones, in about 20 years. (Figure 11) Though these products would have been desirable in the past by most, to own such new innovative machines or devices (e.g. cars or phones) required appropriate funds.

These products were only affordable to businesses and the wealthiest of society, which played a role in constraining their proliferation. This granted society time to question, study and adapt to the tools which were about to become a part of their lives. This means the consumption or demand of rare metals for the new industry will explosively increase due to advancing technology.

### 2.4.3. Environmental aspect

The mining industry could face increasing pressure from regulators to pay for inputs such as carbon and water that are currently have no charge. A carbon price would have the most direct impact on coal producers (discussed in the energy section of this survey) but would also have an indirect impact on other operators through increases in the cost of energy inputs. Pricing water could have a dramatic impact on costs - and constrain output - given that 32 percent of copper mines and 39 percent of iron ore mines are in areas of moderate to high water scarcity. In water - scarce regions, some operators could face increased costs of up to 16 percent from the combined costs of water and carbon emissions. Especially in the US, there are numerous laws and regulations governing mining within the territory that create a complex and lengthy permitting process for any new mine. The potential number of combined environmental permits required by federal, state, and local laws varies between sixty to seventy permits. Table 5 provides a broad survey of environmental concerns, and the applicable laws that most mining companies must comply with before beginning operations. A number of federal environmental requirements have been fully or partially delegated to states to implement, such as permitting under the Clean Air Act and the Clean Water Act.132. In addition to federal permitting requirements, mines must satisfy a myriad of local and state requirements before operations may begin.

In the technological aspect, advances in 3D printing have had profound eco-friendly implications for the efficiency of mining and metals operations. Maintenance downtime is shorter because spare parts can be made on site, and time-saving approaches are multiplied for operations in remote regions. Cost savings are realized, and environmental benefits accrue. Use of 3D printing leads to lighter, smaller and more efficient designs that may last longer and work more efficiently, reducing the environmental impact of operations.

| Environmental Concern | Applicable Law or Permit(s) |
| --- | --- |
| Potential Environmental Impacts | National Environmental Policy Act of 1969[b] ("NEPA") – NEPA is prerequisite to numerous federal and state permit approvals and is applicable to any mining operation that occurs on federal lands or is considered to be a federal action.[c] |
| Air Quality | Clean Air Act[d] – Establishes requirements to obtain a Title V operating permit, which provides performance and emission standards for new hazardous air pollutants, and air toxic standards to prevent significant deterioration of clean air.[e] |
| Water Quality | Clean Water Act[f] – Establishes four surface water programs and applicable permits to regulate the discharge of pollutants to "restore and maintain the chemical, physical, and biological integrity of the Nation's waters"[g]:<br>(1) the National Pollutant Discharge Elimination System permit, known as the section 402 permit, regulates the point source discharge of pollutants, including storm water. Although this permit is generally granted through application to the state, often for rare earth mines that also contain uranium, the site is also monitored by the Nuclear Regulatory Commission;[h]<br>(2) the section 404 permit program, regulates placement of dredged or fill materials in water;<br>(3) the section 208 and 319 permits, regulate nonpoint sources of water pollution; and<br>(4) the section 311 program regulates discharges or spills of oil or hazardous substances.[i] |
| Hazardous and Solid Waste | Resource Conservation and Recovery Act (RCRA) – Governs the generation, transportation, treatment, storage, and ultimate disposal of hazardous waste.[j]<br><br>Comprehensive Environmental Response, Compensation, and Liability Act of 1980[k] ("CERCLA") – Establishes requirements concerning closed and abandoned hazardous waste sites and liability for releasing hazardous waste.[l] |
| Wildlife | Endangered Species Act of 1973[m] – Ensures threatened and endangered species are not destroyed, nor are their habitats adversely modified.[n] |
| Toxic Substances | Toxic Substance Control Act[o] – Regulates the production, important, use, and disposal, of certain substances that present risk to health or the environment.[p] |

a. Federal Environmental Laws That Govern U.S. Mining, National Mining Ass'n, http://www.nma.org/index.php/federal-environmental-laws-that-govern-u-s-mining (last visited March 31, 2013).
b. 42 U.S.C. §§ 4321 et seq. (2006).
c. *See* U.S. Envt'l Prot. Agency, EPA/530/R-95/043, Background for NEPA Reviewers: Non-Coal Mining Operations, 1-1–1-2 [hereinafter U.S. EPA, Background for NEPA Reviewers], *available at* http://www.epa.gov/compliance/resources/policies/nepa/non-coal-mining-background-pg.pdf.
d. 42 U.S.C. §§ 7401-7671q (2006).
e. *See id.* §§ 7401, 7661a, 7661c.
f. 33 U.S.C. §§ 1251–1368 (2006).
g. *See* U.S. EPA, Background for NEPA Reviewers, *supra* note 135, at 1–2.
h. *See* Tantom, *supra* note 127, at 7.
i. Summary of the Clean Water Act, EPA, http://www.epa.gov/lawsregs/laws/cwa.html (last visited Apr. 4, 2012).
j. Resource Conservation and Recovery Act, 42 U.S.C. §§ 6901-6992k (2006). History of RCRA, EPA, www.epa.gov/epawaste/laws-regs/rcrahistory.com (last visited June 7, 2013).
k. 42 U.S.C. §§ 9601–9675 (2006).
l. CERCLA Overview, EPA, http://www.epa.gov/superfund/policy/cercla.htm (last visited Apr. 4, 2012).
m. 16 U.S.C. §§ 1531–1544 (2006).
n. Digest of Federal Resource Laws of Interest to the U.S. Fish and Wildlife Service, U.S. Fish & Wildlife Serv., http://www.fws.gov/laws/lawsdigest/ESACT.HTML (last visited Apr. 4, 2012).
o. Toxic Substance Control Act, 15 U.S.C. §§ 2601–2695d (2006).
p. Summary of the Toxic Substances Control Act, EPA, http://www.epa.gov/lawsregs/laws/tsca.html (last visited Apr. 4, 2012).

Table 5: Major environmental laws affecting mine permission in US (N. Clagett, J. Energy & Environ. Law, 2013)

Broken-down or redundant equipment can be easily recycled for new parts. For terrestrial mines, 3D printing has helped to reduce costs by 50-80% compared with standard manufacturing methods. In the metals industry, specialist companies have applied 3D printing technology to print liquid metal that is conductive and can be printed at room temperature. Since this technology's introduction in the early 21st century, mining and metals companies have fully adopted it as initial barriers to its use have been rectified. In particular, safety and quality standards are unparalleled; the technology has a critical presence internationally and is supported with the appropriate skills; and the production cost of using 3D printing is lower than relying on external supply chains.

# 3. Analysis of Global Trends

## 3.1. United States

The growth of the clean energy industry in the US has stimulated concern about the availability of critical materials for energy. These materials include several rare-earth elements and are key components in many clean energy technologies such as wind turbines, electric vehicles, energy-efficient lighting, and solar panels. In U.S., the sixteen elements were assessed for criticality in wind turbines, EVs, PV cells and fluorescent lighting. The methodology used was adapted from one developed by the National Academy of Sciences. The criticality assessment was framed in two dimensions: importance to clean energy and supply risk. Five rare earth elements (REEs)-dysprosium, terbium, europium, neodymium and yttrium-were found to be critical in the short term (present-2015). These five REEs are used in magnets for wind turbines and electric vehicles or phosphors in energy-efficient lighting. Other elements-cerium, indium, lanthanum and tellurium-were found to be near-critical. Between the short term and the medium term (2015-2025), the importance to clean energy and supply risk shift for some materials

Recently, critical materials have been prominent within the U.S. government. The White House Office of Science and Technology Policy (OSTP) convene a working group that focuses on critical material prioritization, R&D and information. The U.S. Department of Energy (DOE) has made significant R&D investments, particularly in substitute technologies for batteries, photovoltaic cells, magnets, motors and generators. Further efforts have been made through the Department of Energy (DOE), which has supported research on a range of advanced materials and clean energy technologies. In January 2013, the DOE announced its latest Energy Innovation Hub, the Critical Materials Institute (CMI), which has been awarded $120 million to fund research for the next five years. There is also recognition of the importance of education and training to support the national knowledge base across the critical materials supply chain. Data and information continue to be important to inform understanding and support decision-making. DOE has pursued multilateral international collaboration on R&D and information sharing.

## 3.2. European Union

Although raw materials are essential for the EU economy, their availability is increasingly under pressure. Within the framework of the EU Raw Materials Initiative, it was decided to identify a list of critical raw materials at EU level, in close cooperation with Member States and stakeholders. In order to address these complex and interrelated challenges, the European Commission has launched an integrated strategy in November 2008: the EU Raw Materials Initiative. It encompasses measures in three areas to secure sustainable access from outside Europe, improving framework conditions for extracting minerals within Europe, and promoting the recycling and resource efficiency of such materials. One priority action of the Initiative is to identify a common list of critical non-energy raw materials at EU level, in close cooperation with Member States and stakeholders. Some countries have already carried out assessments with the aim of determining how critical some materials are to their economy, but up until now there has been no comprehensive study at the European level.

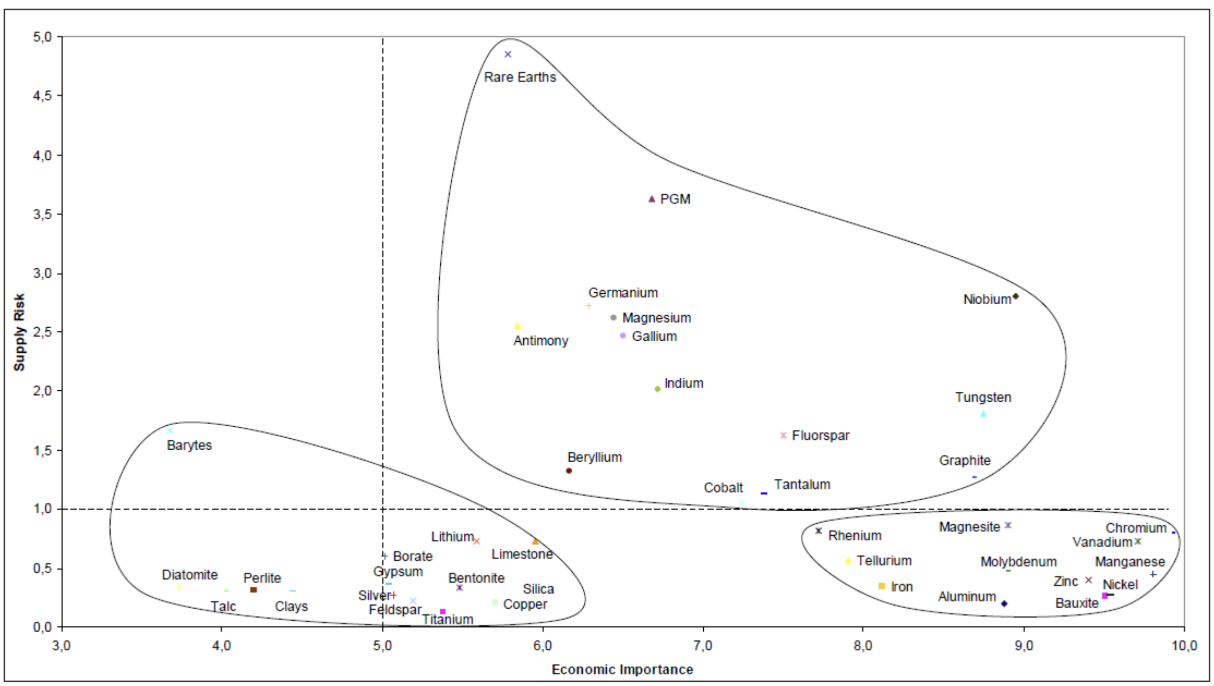



Figure 12: Criticality of minerals and metals in EU (Ad-hoc Working Group report)

EU analyses 41 minerals and metals by a relative concept of criticality, as shown in Figure 12. As noted, this is due to their high relative economic importance and to high relative supply risk. The 'environmental country risk' metric does not change this list of critical materials. The criticality of raw materials is classified by the risks of supply shortage and their impacts on the environment. Two types of risks are considered: a) the "supply risk" taking into account the political-economic stability of the producing countries, the level of concentration of production, the potential for substitution and the recycling rate; and b) the "environmental country risk" assessing the risks that measures might be taken by countries with weak environmental performance in order to protect the environment and, in doing so, endanger the supply of raw materials to the EU.

## 3.3. Japan

The demand of rare metals in Japan for automobile applications, energy-saving and electronic products was 198,800 tons. This tonnage was comprised of 88.4 percent for automobile applications, 7.8 percent for energy-saving products and 2.9 percent for key electronics products. Japan’s domestic rare metal demand is expected to grow considerably to 272,200 tons in 2015 and to 295,400 tons in 2020, about 1.5 times of the demand in 2009. About 90 percent of the domestic demand of rare metals in 2009 was made up of 5 species of rare metals, chromium, manganese, nickel, magnesium and cobalt. From now on, “energy-saving” products will increase the demand of rare metals, which include next-generation automobile (lithium-ion battery, motor, etc), LED, solar cell, fuel cell and wind-power generation. Especially fast demand-growing rare metals are nickel, tungsten, cobalt, vanadium, magnesium, lithium, palladium, rhodium, neodymium, dysprosium and indium, and the activities on the stable supply of these rare metals are becoming important. In June 2010, REEs were categorized as “strategic rare metals” together with minerals such as lithium and tungsten due to the expected rise in their demand. This was due to Japan’s high dependency on certain countries for supply, the high geographical concentration of REE reserves, and the high risk of supply shocks. The stated policy goal for

Japan is to achieve 50% self-sufficiency in these metals and minerals by 2030 through securing supplies from reserves in other countries, increased recycling and the development of substitute materials. For the securing mineral resources, stockpiles that amount to 60 days of consumption are accumulated by the Japan Oil, Gas and Metals National Corporation (JOGMEC) and private companies. The Japanese government is pouring a substantial amount of money into this strategy. For fiscal year 2011, Japan allocated $650 million to secure REEs and other rare minerals market by developing alternate REE sources overseas, recycling and developing substitutes.

## 3.4. China

China has been the world’s dominant supplier for REEs since 1994. However, its apparent unwillingness to continue to play this role by restricting the production and export of REEs has resulted in soaring and volatile REE prices, and has raised resource security issues for key consuming nations such as Japan, Korea, the United States and the European Union. Such developments have spurred companies and governments to seek alternative sources of supply. Although global REE reserves are widely distributed, current production of REEs is heavily concentrated in China. The China’s share in REE mine production has risen dramatically from a mere 27% to current levels of 97%. In spite of their relatively modest reserves, China has been the dominant worldwide producer since the early 1990s. China dominates the front end of the supply chain, from mining to the production of alloys and metals. China produces 97% of the world’s REE supply, 97% of the world’s oxide production, 90% of the world’s rare earth alloys or powder, and for roughly 60% of REE finished products such as permanent magnets. Refined metal is also available only exclusively from Chinese companies. As of 2008, the managing the rare earth industry function was placed under the Ministry of Industry and Information Technology (MIIT). Of its responsibilities, a key mandate of the Rare Earth Office within the MIIT is developing production plans for the country’s strategic commodities, including rare earths. The plans include determining overall production quotas, and distributing these quotas to the individual provinces. Chinese rare earth mines are also known to cause environmental damage. By installing new technologies and taking other measures, the government tries to reduce the environmental impact of the mines. Another goal of the Chinese government is to reduce illegal mining, which causes the most serious damage to the environment, but also attempts are made to increase the efficiency and treating the products with more care.

## 3.5. Korea

Korea has limited natural resources, limited mineral-supporting industry, and weak recycling. Korean government stressed the importance of a production stage intermediate between extraction and the production of finished products in which raw rare metals are converted into refined materials and alloys, called materialization. One of the principal goals of Korea's rare metals strategy is to design policies that will make Korea self-sufficient by recycling rare metals. The process began with the identification of 56 "rare" elements, subject to instability in supply and price fluctuations. These aspects are illustrated in Figure 13, the rarity is normalized to steel.

**Rarity**

| Element | Resource Rarity* |
|---|---|
| PGM | 23,000,000 |
| In | 3,800,000 |
| Se | 1,200,000 |
| Cd | 250,000 |
| Bi | 240,000 |
| Sb | 180,000 |
| Ta | 66,000 |
| W | 33,000 |
| Sn | 29,000 |
| Ge | 17,000 |

* Exhaustion rate of steel =1

**Unstable supply**

| Element | 1st Product Country | Share |
|---|---|---|
| REE | China | 97.1 |
| Nb | Brazil | 89.8 |
| W | China | 86.5 |
| Sb | China | 81.6 |
| Ta | Australia | 62 |
| B | Turkey | 58.7 |
| PGM | South Africa | 58.6 |
| Si | China | 57.7 |
| In | China | 54.8 |
| Bi | China | 52.5 |

**Unstable price**

| Element | $/ton (2002yr) | $/ton (2007yr) | Price Variation (%) |
|---|---|---|---|
| Se | 8267 | 72222 | 774 |
| Mo | 8840 | 70260 | 695 |
| In | 87140 | 680800 | 681 |
| Ni | 6772 | 37181 | 449 |
| Bi | 6658 | 31437 | 372 |
| W | 5400 | 24826 | 360 |
| V | 9662 | 43295 | 348 |
| Co | 15719 | 64440 | 310 |
| Cr | 717 | 2761 | 285 |
| Ti | 5980 | 22530 | 277 |

Figure 13: The correlation between rarity, supply instability, and unstable prices (KIRAM report)

The platinum group metals (PGMs), for example, are 23 million times rarer than steel. A group of 11 of the 56 elements (counting REEs and PGMs each as one) were identified as strategic critical elements. The core of Korean government's policies is overcome for limiting supply risk for these strategic critical elements. These policies cover four major areas: (1) securing natural resources; (2) enhancing R&D for materialization; (3) circulation and recycling; and (4) establishing infrastructure for development of the rare metal industry.

Korean government was attempting to secure supply both domestically and overseas. For overseas supplies, Korea was actively gathering information and dispatching investigation teams to explore potential resources, forming strategic alliances (such as KCMIC with China), participating and investing in mine developments, and modifying regulations that would encourage investments in foreign developments related to rare-metal resources. On the domestic front, Korea actively stockpiles 21 elements to cover 60 days of domestic demand. These policies range from the immediate short term (stockpiling) to the long term (exploration and development). The strategies have been adopted by the Korean government to establish infrastructure in support of their rare-metal industry. Three concrete organizational actions have been implemented. The first is the formation of a Rare Metal Industry Governing Committee that advises the government on policy matters and has members from industry, academia, and the Korea Ministry of Trade, Industry and Energy (MOTIE; former Korea Ministry of Knowledge & Economy). The second is the establishment of Korea Institute for Rare Metals (KIRAM) at KITECH that highlighted its role in coordinating government, local universities, and small and medium enterprises to facilitate R&D support for core technologies. KIRAM carries them through to commercialization, and establishing new industry, especially small and medium enterprises as well as makes selection and funding decisions for technology projects for strategic rare metals, and provides oversight of R&D programs. The ambitious goals set for Korea to secure its supply of critical rare metals, for 2018 relative to 2009: increasing self-sufficiency in materials from 12% to 80%, increasing their technical level from 60% to 95%, and increasing the number of specialized companies founded from 25 to 100.

# 4. International Collaboration

## 4.1. Development of Strategy

The strategies for securing rare metals are mostly composed by government leading multilateral policies. First, guarantee continuously supplement of natural resources in the future by investment and exploring of foreign resources with diplomatic relationship. Second, increase stockpiles of the strategic and economic essential rare metals to set up flexible execution. Third, reduce and replace the consumption of rare metals by the development of novel materials reduction and replacing technology. Fourth, recycle and reuse the disposed end of life industrial products which have rare metals to create alternative resources by urban mines. The main task of the collaboration is to support the determination of strategies and perspectives on rare resources. The strategical policy responses may include: (a) Advocating place the supervision of this problem in the portfolio of a specific government organization, (b) Establishing programs to encourage the development of new, secure rare resources where appropriate. This may include legislation of law, support of new, basic research, (c) Raising awareness in the international policy community of the importance of specific critical elements as they may affect relations with specific nations, (d) Advocating for awareness of the importance of research into substitutional physics and chemistry so that potentially rare or unavailable elements can be replaced by more common materials.

### 4.1.1. International Rare Metals Workshop

The International Workshop on Rare Metals (IWRM) was part of an ongoing discussion to secure current and future access to critical materials. These communications were crucial to exchange views and perspectives on the availability and supply challenges of critical materials. Emphasis has been on rare earth metals and other elements critical for future technology and use. It was the latest in a series of symposia, workshops and conferences of which the following can be considered as landmarks in promoting international cooperation on this subject:

1. Awareness of current status on rare resources
2. Identify the element, the emerging technology, and
    a. Identify the scientific/engineering reason for the critical role of the element.
    b. Estimate the intensity in key applications, and
    c. The requirements for scaling to widespread use.
3. Survey and assess the issues that determine the availability of the element including:
    a. Fundamental rarity, geological occurrence, mineralogy, known and projected reserves, geographical distribution of reserves and political concerns, likelihood of new discoveries.
    b. Extraction issues: environmental impact, toxicity, sustainability.
    c. Cost/availability relationships, including additional features due to co-product production.
    d. Secondary sources: potential for recycling and thresholds for salvaging rare elements as byproducts.
4. Make consensus among participate countries on the overcoming for the rarity of resources by R&D activities. The specific technical fields in which the cooperative activities are to take place shall primarily be those that are within the scope and expertise of members and they include the following:
    a. Research and development activities on rare metals including rare earths
    b. Advanced Technologies related with to Materialization, Extraction, Recycling, Replacement, Reduction and Purification of rare metals
    c. Technology transfer to perspective industrial applications, subject to the requirements under laws, regulations and directives of each countries

#### 4.1.1.1. The 1st International Workshop on Rare Metals

The importance of certain critical materials; Rare Metals, for national industry and economic prosperity has been recognized for many decades. The Korea Government endorses a proposal for a measure of the issues raised by the possible large-scale adoption of rare materials-related common wills that would require a dramatic increase in the availability of rare elements. In this reason, the 1st International Workshop on Rare Metals was held April 18-19, 2011 in Incheon at Korea organized by the Korea Institute for Rare Metals (KIRAM). The Workshop aims at providing a forum for researchers and practitioners working on the diverse issues of rare metals among worldwide countries. (Figure 14) Attendees included representatives from the Korea Resources Corporation, the Ames Laboratory, the Leibniz IFW Institute, the York University, the Nakaoga Tech. University, Ministry of Indonesia Economy, and other delegations. The Workshop will be undertaken in collaboration with the KITECH. Other scientific societies, notably the Korea Resources Corporation (KORES) and the Korea Ministry of Knowledge Economy (MKE) will provide input to the Workshop. The aim of this Workshop is to identify the range and nature of policy issues associated with the supply of critical rare elements and recommend mechanisms to track these evolving issues. Another main objective of Workshop is to develop international common R&D activities to overcome all difficulties related with recycling, reuse, replacement and reduction of rare materials.


Figure 14: The advertising of the 1st IWRM held in Incheon, Korea

### 4.1.1.2. The 2nd International Workshop on Rare Metals

The first International Workshop on Rare Metals took place in Incheon, South Korea, in April of 2011, and it was undertaken as collaboration with the Korea Institute and Industrial Technology (KITECH) and other scientific societies: notably, the Korea Resources Corporation (KORES) and the Korea Ministry of Knowledge Economy (MKE) provided input to the Workshop. As the extension bottom line of awareness of Criticality on REE, the 2nd International Workshop on Rare Metals was held in November 25th of 2012 at Boston, Massachusetts. Organized by The Ames Laboratory and the Korea Institute for Rare Metals (KIRAM), the Workshop aims to provide a forum for researchers and practitioners working on the diverse issues of rare metals, among countries worldwide, for the discussion of high-level issues and strategies to ensure secure supplies of these essential materials (Figure 15).

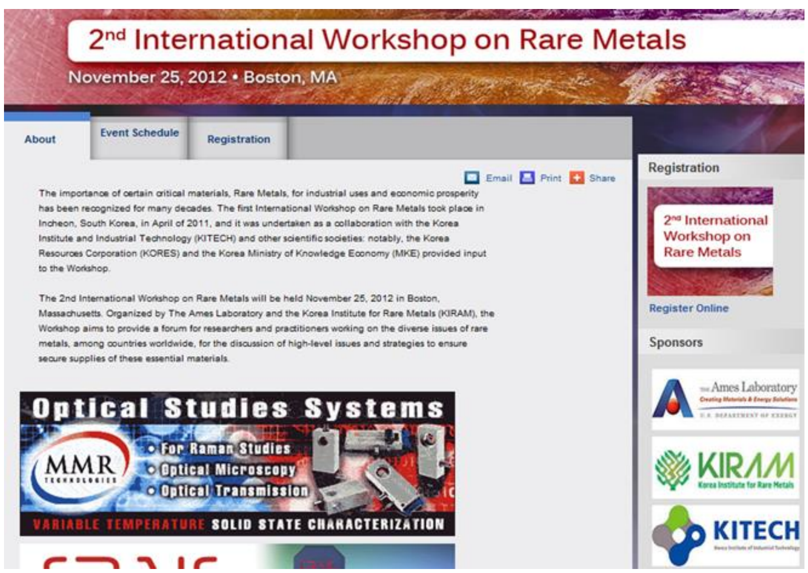



Figure 15: The advertising of the 2nd IWRM held in Boston, US

### 4.1.1.3. The 3rd International Workshop on Rare Metals

This third Rare Metals workshop at the Conference of Metallurgists, building on the success of the inaugural international workshops previously held in April 2010 and November 2012, provides a collaborative forum to share ideas and technical advances, as well as to discuss these critical and timely issues (Figure 16). The response to this new symposium has exceeded our expectations. The presentations in workshop contained fifty-one papers from academia, government and industry from 16 countries. Besides complementing talks from Canada and the United States, there were significant contributions from China, Germany, Japan, Korea, Belgium, the UK and elsewhere. The Symposium provides an opportunity for authors and delegates from around the world and across all aspects of the rare earth supply chain to meet their colleagues and discuss common interests and concerns.


Figure 16: The advertising of the 3rd IWRM held in Montreal, Canada

With the interest generated by the importance of the subject area, two international Rare Earth organizations would be holding meetings concurrently with the REE Symposium in Montreal. The eight-country member International Rare Metals Working Group, chaired by the Korea Institute of Industrial Technology, would be convening its annual meeting, and the Rare Earth

Technology Alliance, the US-based international member industry association under the auspices of the American Chemical Council would be holding its quarterly meeting. The presentations in the workshop have been laid out in six thematic categories: Rare Earth Element Industry Overview, Mineralogy and Beneficiation of Rare Earth Resources, Hydrometallurgy of Rare Earth Mineral Concentrates, Rare Earth Element Separation, Rare Earth Elements in Advanced Materials, and Recycling of Rare Earth Elements, reflecting the structure of this year's full three-and-a-half day Symposium. It is also important to understand the economic and environmental issues related to meeting the demand for rare earth-based materials. Success will be significantly facilitated through national and multinational collaboration.

## 4.2. Development of Technology

### 4.2.1. Substitution of RE resource by alternative resource (Nd, Dy recycling)

The increasing need for specific materials in advanced technologies, especially green energy technologies, has led to an imbalance in the supply-demand of certain materials. The instabilities in the supply and demand of specific materials leads them to be defined as critical materials. While the definition of what is a critical material is not universally defined by the US Department of Energy (Figure 2) shows their methodology for classifying critical materials. (Figure 2) From the chart, it is very clear the rare earth (RE) elements, especially Nd and Dy, are very high on the criticality list. Nd and Dy are important to advanced energy technologies due to their ubiquitous use is high-strength permanent magnets. Specifically, more than 50,000 tons of RE-Fe-B-type magnets are manufactured each year. The supply risk arises, however, from the fact the mining of the RE elements and the production of the RE magnets are localized to specific geographic locations. The localization of the supply and production of the RE elements and magnets, respectively, combined with the overall limited supplies has created an imbalance in the supply/demand of RE elements. To address this imbalance, alternative supplies of RE elements are being considered. While opening up new mines is an important component of this, an equally important approach is developing recycling technologies that can supplement the RE supplies.

To develop an advanced recycling technology for the recovery of RE elements from scrap magnet material, the Korea Institute of Industrial Technology (KITECH) and The Ames Laboratory (USA) partnered to develop an advanced liquid metal extraction technology that can be utilized to recover high-purity RE metals from various RE-type permanent magnet alloys. This project focused on utilizing liquid Mg to extract RE elements from magnet scrap, which is based on technology previously developed at Ames Laboratory. Our initial work focused on identifying the basic processing conditions necessary for the extraction of the RE elements since the initial work at Ames focused on the fundamental diffusion behavior in liquid Mg. For this work we started with a model alloy system containing only Nd-Fe-B (~33 wt. % Nd). All of our melting experiments were done via induction heating in order to assure adequate mixing of the molten liquid. The model alloy or scrap magnet material was placed in a stainless steel mesh box that was attached to a stopper rod. The stopper rod plugs the bottom hole on the stainless steel crucible for which the liquid Mg is melted in. After a specified melting time, the stopper rod is lifted up and the liquid Mg alloy is poured into a Cu mold. Figure 17 shows the furnace that was used for the liquid metal extraction experiments.

Figure 17: Induction furnace used for liquid metal extraction experiments

Since there are many process variables, we tried examine them independently. The first variable that we examined was the effects of the ratio of liquid Mg to scrap material (Nd-Fe-B). For these experiments, we sieved the scrap material to 0.6-2.0 mm in size and enclosed it in a stainless steel mesh box. The samples were all heated at 850 ℃ for 1 hour. The 3 Mg-to-scrap ratios that were utilized are shown below Table 6:

| Sample | Mass Nd-Fe-B (g) | Mass Mg (g) | $M_{Mg}/M_s$ | Mass Casting (g) |
|---|---|---|---|---|
| RTO-1-1 | 50 | 207 | 4 | 153.5 |
| RTO-1-3 | 80 | 200 | 2.5 | 141.1 |
| RTO-1-5 | 200 | 200 | 1 | 143.8 |

Table 6: Initial masses of scrap material and Mg metal along with mass of Mg-Nd casting

From these 3 ratios we measured the amount of Nd in the Mg alloy after casting and found that all three alloys closely matched the theoretical values for full extraction of Nd from the scrap material, sample RTO-1-5 (1:1 Mg:scrap ratio) showed the highest concentration of Nd in the Mg-Nd alloy, Table 7. Furthermore, all three alloys showed minimal Fe contamination during processing, which confirmed the effectiveness of the process.

| Element | RTO-1-1 | RTO-1-3 | RTO-1-5 |
|---|---|---|---|
| Mg | 92.3% | 87.9% | 72.6% |
| Nd | 7.46% | 11.87% | 26.99% |
| Fe | 0.12% | 0.13% | 0.16% |
| B | <0.001% | <0.001% | <0.001% |

Table 7: Chemical analysis of Mg-Nd castings

From our analysis of the scrap material in the mesh box after casting to evaluate effects of particle size on reactivity, we found that a significant amount of the Mg-Nd liquid was trapped between the particles during casting. The degree of this entrapment of the liquid Mg-Nd is likely dependent on the size of particles.

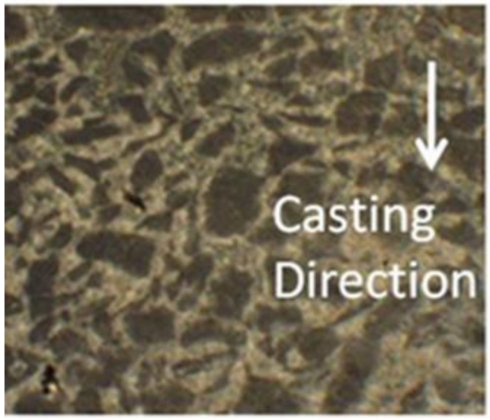



Figure 18: Optical micrograph of material left in mesh box after casting. Light colored phase in Mg-Nd liquid and dark is scrap material.

The smaller particles will pack together more densely and make it more difficult for the liquid to pour out during casting. Increasing particle size however, increases the diffusion length, which can require extremely long processing times for the extraction of the RE elements from the scrap material.

To examine the effects of the particle size on the extraction process, we utilized three different size ranges: 0.6 - 2.0, 2.0 - 4.0 and > 5 mm. Table 8 shows the amount of Nd and Fe present in the liquid Mg after the extraction experiment for two different particle size ranges. Our results showed that the extraction of the Nd into the Mg was approximately the same for both particle size ranges suggesting that the longer diffusion path length did not affect the extraction of the RE elements significantly. For particles sizes above 5 mm we found that the RE elements did not fully diffuse out in the processing time used, and thus, was not suitable for this process.

| Element | 0.6 – 2.0 mm | 2.0 – 4.0 mm |
|---|---|---|
| wt. % Nd in casting | 26.99% | 25.06% |
| wt. % Fe in casting | 0.16% | 0.19% |

Table 8: Measured Nd content in Mg-Nd alloy for different scrap particle size ranges

Once the processing conditions for extracting the RE elements into the liquid Mg were established, the next goal of the work was to develop a process for separating the RE elements from the Mg (enrichment). Achieving this can be done using vacuum distillation, which takes advantage of the relatively low melting temperature and high vapor pressure of Mg compared to the RE elements to distill the Mg out of the Mg-RE alloy. The major limitation of using this process on the Mg-RE alloys from the liquid metal extraction experiments is the RE elements are the minority component. As can be seen in Table 8, the RE content in the Mg-RE alloy after liquid metal extraction from the magnet scrap is less than 30 wt. %. This low amount of RE present in the alloy makes vacuum distillation considerably more difficult. To address this, we tried to increase the amount of RE in the Mg-RE alloy by utilizing a multi-step liquid metal extraction process, which is shown schematically in Figure 19. It should be noted that for this work we utilized actual permanent magnet alloys (N35 grade), which contained Nd, Pr and Dy. In this process, the Mg-RE alloy that is synthesized in the first step is used as the liquid extractant in the second step, instead of pure Mg, to increase the amount of RE in the final Mg-RE alloy. The effects of this two-step process on the RE content can be seen in Table 9.

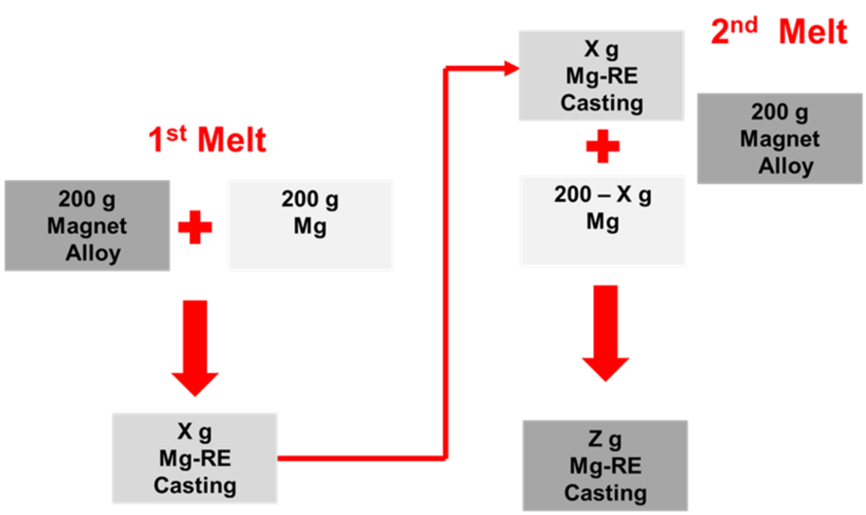



Figure 19: Schematic of two-step process for enriching RE content in Mg-RE alloys

From the results shown in Table 9, it can be seen that the two-step process does increase the amount of RE in the Mg-RE alloy. The RE elements are still the minority component in the alloy, but the enrichment makes vacuum distillation more suited for yielding a high-purity RE alloy.

| Element | $1^{st}$ Melting | $2^{nd}$ Melting |
|---|---|---|
| Mg | 75.8 % | 64.6% |
| Nd | 19.81 % | 27.08 % |
| Pr | 3.93 % | 7.50 % |
| Dy | 0.080 % | 0.12 % |
| Fe | 0.19 % | 0.22 % |
| Ni | 0.054 % | 0.34 % |
| B | < 0.001 % | < 0.001% |
| Total RE Metals | 23.82% | 34.70% |

Table 9: Effect of 2-step process on RE content in Mg-RE alloy

Our initial work examining the processing conditions for the recovery of the RE elements from the magnet scrap focused on relatively small batches of magnet scrap (200g). To examine the feasibility of scaling up the liquid metal extraction process of the RE metals to a larger scale, we examined the efficiency of RE recovery for larger batches of magnet scrap. For each process, the Mg/scrap ratio was 1:1, and the two-step process described above was utilized. The total amount of RE elements in the Mg-RE alloy after the two-step enrichment process for the different batch sizes are shown below in Table 10. From the results, we see the efficiency of the process is not degraded by scaling up to 2000 g of scrap. In fact, the total amount of RE elements actually increases a little. While preliminary, the results suggest that this process can be scaled up to effectively recover the RE elements from bulk batches of magnet scrap.

As discussed above, to separate the Mg from the RE elements we utilized vacuum distillation. The conditions for the distillation experiments were T = 700 °C for 48 and 96 hours. Table 11 shows the chemical analysis the RE material after the distillation of the Mg. From the results we find that the total RE purity after distillation is > 98% with very little Mg present. Furthermore, the results suggest that increasing the distillation time from 48 to 96 hours does not lead to a measurable increase in the RE purity in the alloy. It should be noted that the Mg recovered from the Mg distillation process was > 99.9 % pure.

| Element | 200 g scrap | 1000 g scrap | 2000 g scrap |
|---|---|---|---|
| Mg | 64.6% | 50.8% | 53.6% |
| Nd | 27.08 % | 36.42% | 33.09 % |
| Pr | 7.50 % | 11.74 % | 10.72 % |
| Dy | 0.12 % | 0.23% | 0.095 % |
| Fe | 0.22 % | 0.46 % | 0.47 % |
| Total RE Metals | 34.70% | 48.39% | 43.9% |

Table 10: Total amount of RE elements in Mg-RE alloy after 2-step enrichment process as a function of process scale

| Element \ Time | 48 hrs | 96 hrs |
|---|---|---|
| Mg | 0.062% | 0.029% |
| Nd | 73.9% | 73.8% |
| Pr | 23.88% | 23.46% |
| Dy | 0.24% | 0.29% |
| Fe | 0.17% | 0.19% |
| Ni | 0.11% | 0.20% |
| Total REM Purity | 98.02% | 97.55% |

Table 11: Chemical analysis of distilled Mg-RE alloy for two different times at 700 °C

To further investigate the effectiveness of the recycling process, we used the high-purity RE metal recovered from the distillation process to synthesize permanent magnet alloys. The recycled RE metals were arc melted with other elements to form a $RE_2Fe_{14}B$-type alloy, which was then melt spun and flash annealed. For comparison, an alloy of identical composition (without the impurity elements) was synthesized from non-recycled (pure) RE elements. For the alloys used in this analysis, the microstructures were not tailored for optimal extrinsic magnetic properties, but rather, we are only comparing the intrinsic magnetic properties.

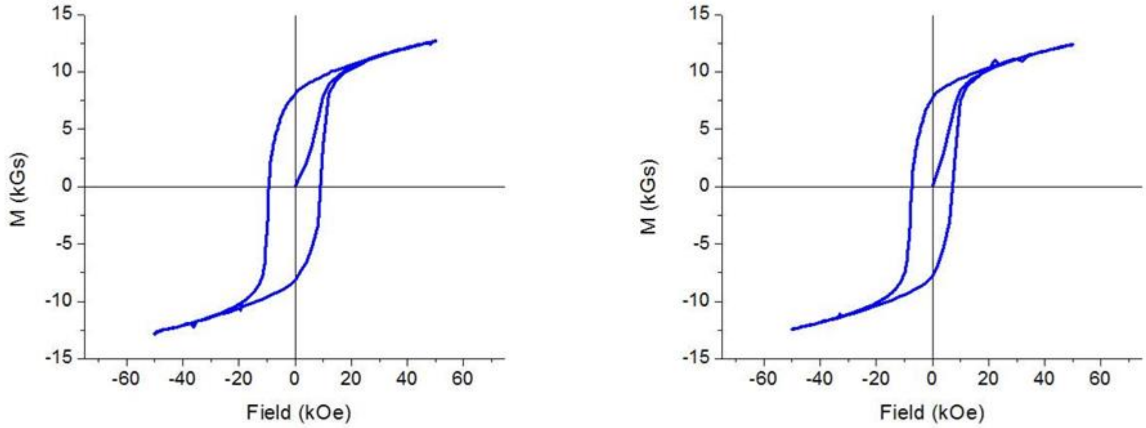



Figure 20: Hysteresis curves for permanent magnet alloys synthesized from RE metals synthesized from oxides (left) and recovered from magnet scrap (right)

Figure 20 and Table 12 show that the analysis results of permanent magnet alloy synthesized from the recycled RE metals showing quite similar intrinsic magnetic properties to that of the alloy synthesized from non-recycled RE metals. These results suggest the impurities present in the RE alloy after distillation do not appreciable degrade the subsequent permanent magnet alloy. It is important to note the magnet scrap utilized in these experiments were uncoated magnets, however, RE containing magnets are often coated with metals such as Ni. To examine the effects of the coating on the permanent magnet alloys synthesized from the recovered RE metals, we utilized Ni coated magnets in the liquid metal extraction experiments.

| Property | Recovered RE Metals | High-Purity RE Metals |
|---|---|---|
| Saturation Magnetization ($M_s$) | 13.6 kGs | 14.0 kGs |
| Anisotropy Field ($H_a$) | 68.6 kOe | 70.7 kOe |

Table 12: Intrinsic magnetic properties of permanent magnet alloys synthesized from RE metals recovered from magnet scrap and synthesized from RE oxides

| Element | $1^{st}$ Melting | $2^{nd}$ Melting |
|---|---|---|
| Mg | 75.6 % | 61.3 % |
| Nd | 16.35 % | 26.30 % |
| Pr | 4.76 % | 7.74 % |
| Dy | 0.081 % | 0.061 % |
| Fe | 0.18 % | 0.28 % |
| Ni | 0.39 % | 0.65 % |
| B | < 0.001 % | < 0.001% |

Table 13: Chemical analysis of Mg-RE alloy after liquid metal extraction experiments on Ni-coated magnet scrap

From the chemical analysis shown in Table 13, we see that the Ni content in the Mg-RE alloy does increase with the subsequent enrichment step. This incorporation of Ni into the Mg-RE lead to increased Ni impurities in the final RE metal after distillation. The effects of the increased Ni content in the recovered RE metals were investigated by synthesizing permanent magnet alloys from both recycled and non-recycled RE metals. Interestingly, the incorporation of the Ni from the scrap magnet coating does not appear to have a deleterious effect on the properties of the permanent magnet alloy synthesized from the recycled RE metals, Figure 21.

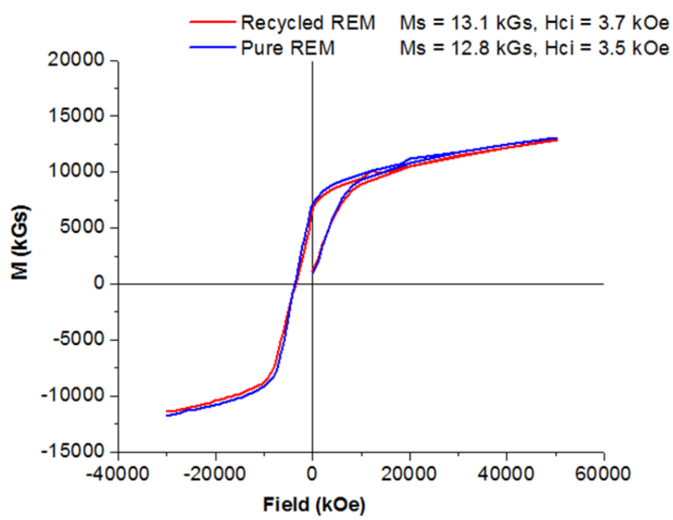



Figure 21: Magnetic properties of permanent magnets synthesized from rare earth metal (REM) recovered from Ni-coated magnet scrap and pure REM

It is well known that commercial permanent magnets utilize different RE elements to tailor the magnetic properties. For example, Dy is added to $RE_2Fe_{14}B$-type magnets to help maintain the coercivity at elevated temperatures. The large variation in compositions in commercially available permanent magnets highlights the importance of understanding the individual behavior of different RE elements during the liquid metal extraction process. To investigate this, we utilized a high-Dy containing magnet composition during liquid melting extraction experiments at three different temperatures. The actual amount of the individual RE elements present in the Mg-RE alloy as well as their theoretical composition based on the full extraction of them from the magnet scrap are show in Table 14. From the measured compositions of the RE elements in the Mg-RE alloy, it is quite clear the Nd and Pr can be effectively extracted from the magnet scrap alloy, however, the Dy is not. The efficiency of the Dy extraction does show a small increase with increasing temperature, but does not approach its theoretical maximum. Further evidence of Dy not effectively diffusing into the Mg during the liquid metal extraction step can be seen in Figure 22, which shows the solidified microstructure at the magnet/Mg-RE alloy interface.

| Element \ Temp. | 850 C | 900 C | 950 C | Full Extraction Wt. % |
|---|---|---|---|---|
| Dy | 0.96% | 1.55% | 1.52% | 3.47% |
| Nd | 13.11% | 14.02% | 14.38% | 14.03% |
| Pr | 4.32% | 4.55% | 4.78% | 4.68% |
| Mg | 80.9% | 77.8% | 76.9% | |
| Fe | 0.18% | 0.35% | 0.34% | |
| Ni | 0.050% | 0.042% | 0.039% | |
| Total REM | 18.4% | 20.1% | 20.7% | 22.2% |

Table 14: Measured compositions of elements in Mg-RE alloy processed at different temperatures. The theoretical composition of each RE element in the Mg-RE alloy based on full extraction from the magnet scrap is also shown

The interface region corresponds to a region depleted in Nd and Pr since they have diffused into the Mg-RE alloy and rich in Dy that has not diffused out. Since Dy is an extremely critical material, effectively recovering it is important. The fact that the light RE elements (Nd and Pr) diffuse into the Mg liquid, while the heavy RE Dy remains in the magnet alloy provides a process by which a Dy-enriched alloy can be recovered. More work needs to be done to optimize this process, but it does appear to be a promising method for recycling Dy-rich alloys.

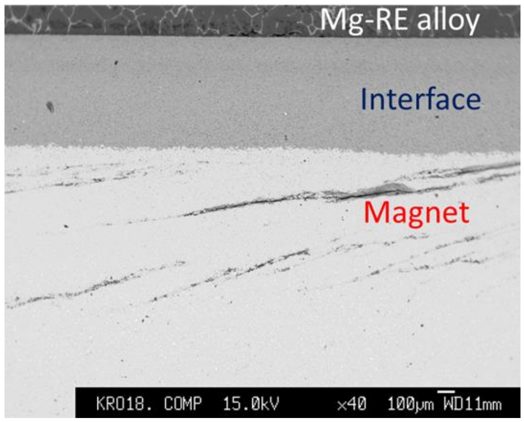



Figure 22: SEM micrograph of magnet/Mg-RE alloy interface showing region that is depleted in Nd and Pr an enriched in Dy

Since Dy is an extremely critical material, effectively recovering it is important. The fact that the light RE elements (Nd and Pr) diffuse into the Mg liquid, while the heavy RE Dy remains in the magnet alloy provides a process by which a Dy-enriched alloy can be recovered. More work needs to be done to optimize this process, but it does appear to be a promising method for recycling Dy-rich alloys. Moreover, this unique recycling technique (Ames process) can be applied to the recovering Co and Ni from the WC hard cutting-tool scraps using a molten-zinc (Zn) bath.

### 4.2.2. Substitution of RE by abundant metals (Magnetocaloric materials)

Around 15% of the total energy consumption worldwide is used for refrigeration purposes. Commercial and residential refrigeration and air-conditioner are a mature, relatively low capital cost but a high-energy demand industry. Because current Carnot cycle using systems are based on processes involving the use of compressed gasses throughout the system. Moreover, typical

Carnot cycle systems are not applicable to the extremely large-scale cooling capacity due to low energy efficiency, harmful vibration, noise etc. However new magnetocaloric materials are expected to be applied as large-scale air conditioners and heat pumps for the highly integrated computing facility (such as Data Center of Google, Facebook and VISA etc.).

Magnetic refrigeration (MR) is rapidly becoming competitive with conventional gas compression technology because it offers considerable operating cost savings by eliminating the most inefficient part of the refrigerator: the compressor. In addition to its energy savings potential, MR is an environmentally sound alternative to vapor-cycle refrigerators and air conditioners. These compressed gasses are known to be harmful to the o-zone layer. Some of the more commonly used blends of these gasses are Hydro-Chlorofluorocarbon (HCFC) and Hydro-fluorocarbon (HFC). MC (Magnetocaloric) refrigerators use a solid materials based refrigerant(s) and common heat transfer fluids (e.g. water, water-alcohol solution, air, or helium gas) with no ozone-depleting and/or global-warming effects. In 1997, Karl A. Gschneider Jr. at Ames Laboratory developed and first "proof of concept" in-room-temperature MCR process showing a thirty percent improvement in energy efficiency in comparison to previous works in the field. This feat was accomplished through the use of the rare earth metal Gadolinium (Gd) as the refrigerant. The metal, however, was alloyed with other elements such as silicon and germanium in order to produce the maximum amount of MCE possible. This was also mainly due to feasibility in mass production of the Gd alloy commercial grade compound which only required a small amount of the rare earth metal in the alloying process. This accomplishment also made use of a "permanent" magnet, a new feature which promises the continual use of magnets within the system for continual refrigeration system applications.

The problem with current MCR systems is the use of high purity REE (i.e., Gd, Tb, and Dy) or toxic materials (i.e., As, P and Sb) to maximize operational MCE and the use of liquid nitrogen to cool the solid material when it is passed through a magnetic field. The use of toxic metal renders such system of little use for domestic application due to the danger and high cost of these handlings. The cost of the raw materials is generally stated to be an advantage of Gd-based MCE material, because it is thought to be too expensive. The MCE properties of MnAsSb alloys are outstanding and these alloys are among the leading candidates as near room temperature magnetic refrigerant materials; however, the high vapor pressure of arsenic (As; boiling point 876 K) makes it difficult to prepare large quantities (tons) of MnAs in an economical way. A second problem is the fact that As is a governmentally regulated poison, which means special handling facilities would be required for preparing the MnAsSb material, and special environmental regulations will need to be met to place such cooling devices into commerce.

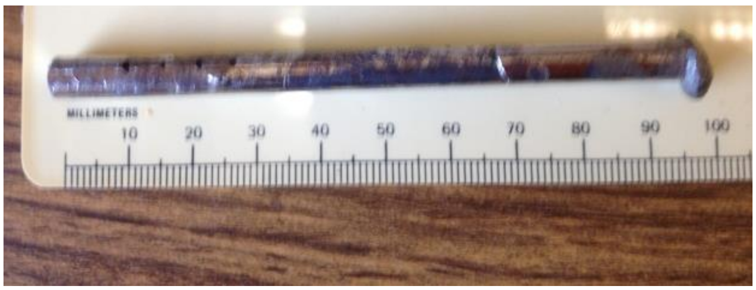



Figure 23: Optical image of the as-cast Ni-Mn-Cu-Sn MCE alloy ingot synthesized by Arc melting followed rapid cooling process

Figure 24: The general quenching specimen after annealing in vacuum

| No | ID | at.% | | | | | | | | Arc melting ID |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | MC-NiMnSn | 45.1 | Ni | 31.2 | Mn | | | 23.7 | Sn | AHI-134 ① |
| 2 | MC-NiMn0CuSn | 39.7 | Ni | 39.8 | Mn | | | 20.5 | Sn | AHI-134 ② |
| 3 | MC-NiMn2CuSn | 39.6 | Ni | 37.9 | Mn | 2.0 | Cu | 20.5 | Sn | AHI-134 ③ |
| 4 | MC-NiMn4.5CuSn | 39.5 | Ni | 35.6 | Mn | 4.5 | Cu | 20.4 | Sn | AHI-134 ④ |
| 5 | MC-NiMn7CuSn | 39.3 | Ni | 33.4 | Mn | 6.9 | Cu | 20.3 | Sn | AHI-134 ⑤ |
| 6 | MC-NiMn2CoSn | 39.7 | Ni | 38.0 | Mn | 1.9 | Co | 20.5 | Sn | AHI-134 ⑥ |

Table 15: Nominal compositions of elements in MCE alloys processed at different elemental compositions

The goal is to develop new MC materials replaced high purity REEs or toxic metals to common elements without degradation of MCE. The acquired data could be used to bring MCR to the domestic market as well as to areas of the world with limited access to energy resources. We focus on the change of magnetocaloric properties of series of Ni-Mn-Cu-Sn alloys by the changing of elemental compositions. Figure 23 and Figure 24 show one of the sample ingots prepared by Arc-melting process and quenched specimen after annealing in vacuum at Materials Preparation Center (MPC) of the Ames Laboratory, respectively. This material belongs to the family of NiMn-based metamagnetic Heusler shape-memory compounds. At an appropriate composition, with excess of Mn with respect to the 2-1-1 stoichiometry, they undergo a martensitic transition below the Curie temperature within the ferromagnetic state.

In the vicinity of this martensitic transition, these alloys display giant inverse magnetocaloric effect (IMCE) which means that a huge increase of entropy can be induced by isothermal application of a magnetic field, whereas cooling (decrease of temperature) occurs when the field is adiabatically applied. The nominal compositions of candidate 5 alloys based on NiMnSn alloy are shown in Table 15. It is well know that Cu has been shown to have an important effect on the properties of the Ni-Mn-Sn compound. It has been reported that replacement of Mn by a little amount of Cu (≤3%) in $Ni_{43}Mn_{46-x}Cu_xSn_{11}$ results in a considerable shift of the martensitic transition to a higher temperature while the transition slightly shifts to a lower temperature when Cu replaces Ni in $Ni_{46-x}Cu_yMn_{43}Sn_{11}$.

Figure 25: The MR measurement system in Ames Lab

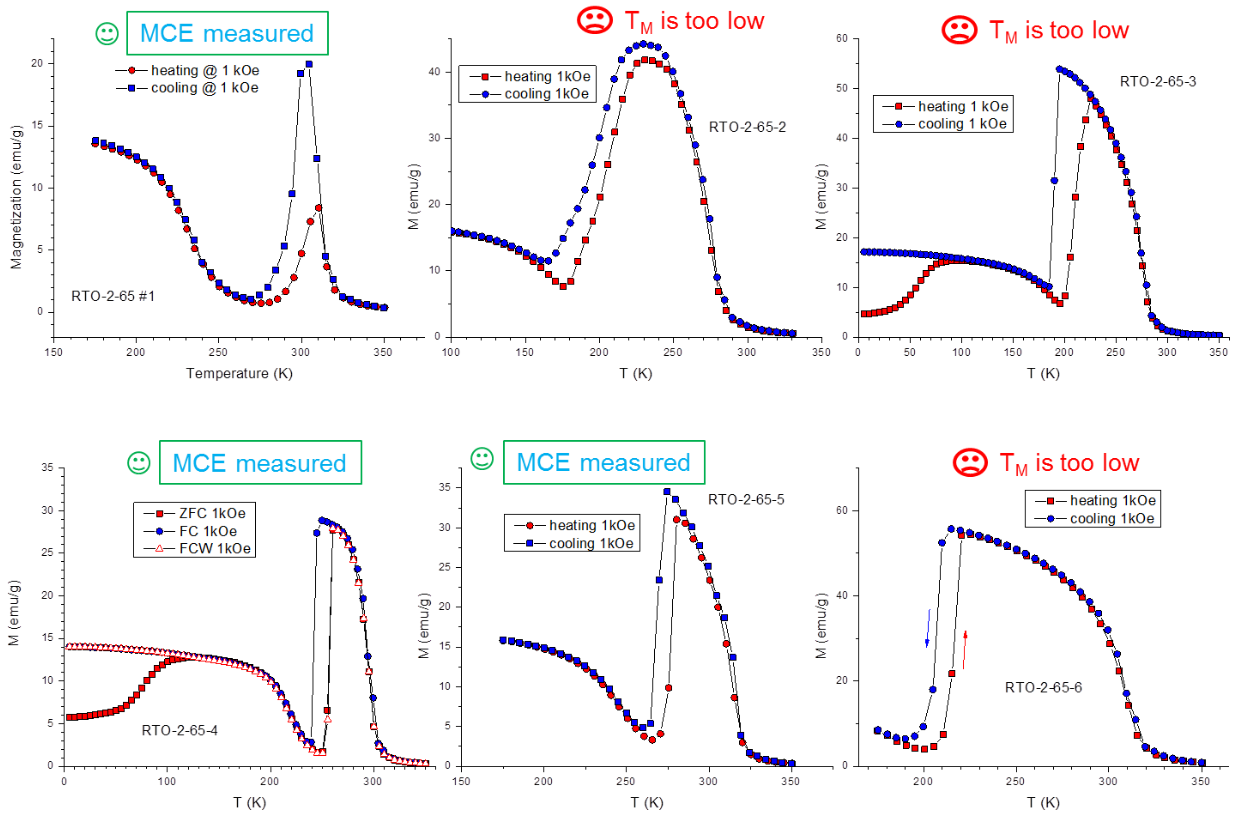



Figure 26: Magnetization versus temperature curves at selected alloys of the applied magnetic field at 1k Oe

A series of polycrystalline NiMnCuSnCu(Co) alloys were synthesized from pure elements by arc melting in an Ar atmosphere followed by a rapid cooling process in a water-cooled copper crucible. The ingot was encapsulated under vacuum in quartz glass, annealed at 1123K (850 $^{o}$C) during 24 h and quenched into an ice-water mixture. A specimen of size 4.6 x 3.2 x 3 mm$^3$ (mass, m≈0.35 g) for length change and calorimetric measurements was cut from the ingot by means of a low speed diamond saw. A smaller specimen of mass 32.6 mg was cut from the same ingot for magnetic measurements. Transition temperatures were determined by means of DSC (Perkin Elmer Pyris-1) measurements. The compositions of samples were determined by Energy-dispersive X-ray photoluminescence (EDX). Magnetic measurements were carried out using by Superconducting quantum interference device (SQUID; Quantum Design) and Physical Property

Measurement System (PPMS; Quantum Design) magnetometer. Figure 25 shows the MR property measurement system in Ames Laboratory.

The magnetocaloric effect can be measured from the measured magnetization or heat capacity, both as a function of temperature and magnetic field. The six compositions were characterized magnetically by the VSM technique. A temperature profile was made for each sample. At constant temperature M is measured as a function of the applied magnetic field, H. From the isothermal magnetization measurements, magnetization versus temperature can be plotted, as shown in Figure 26.

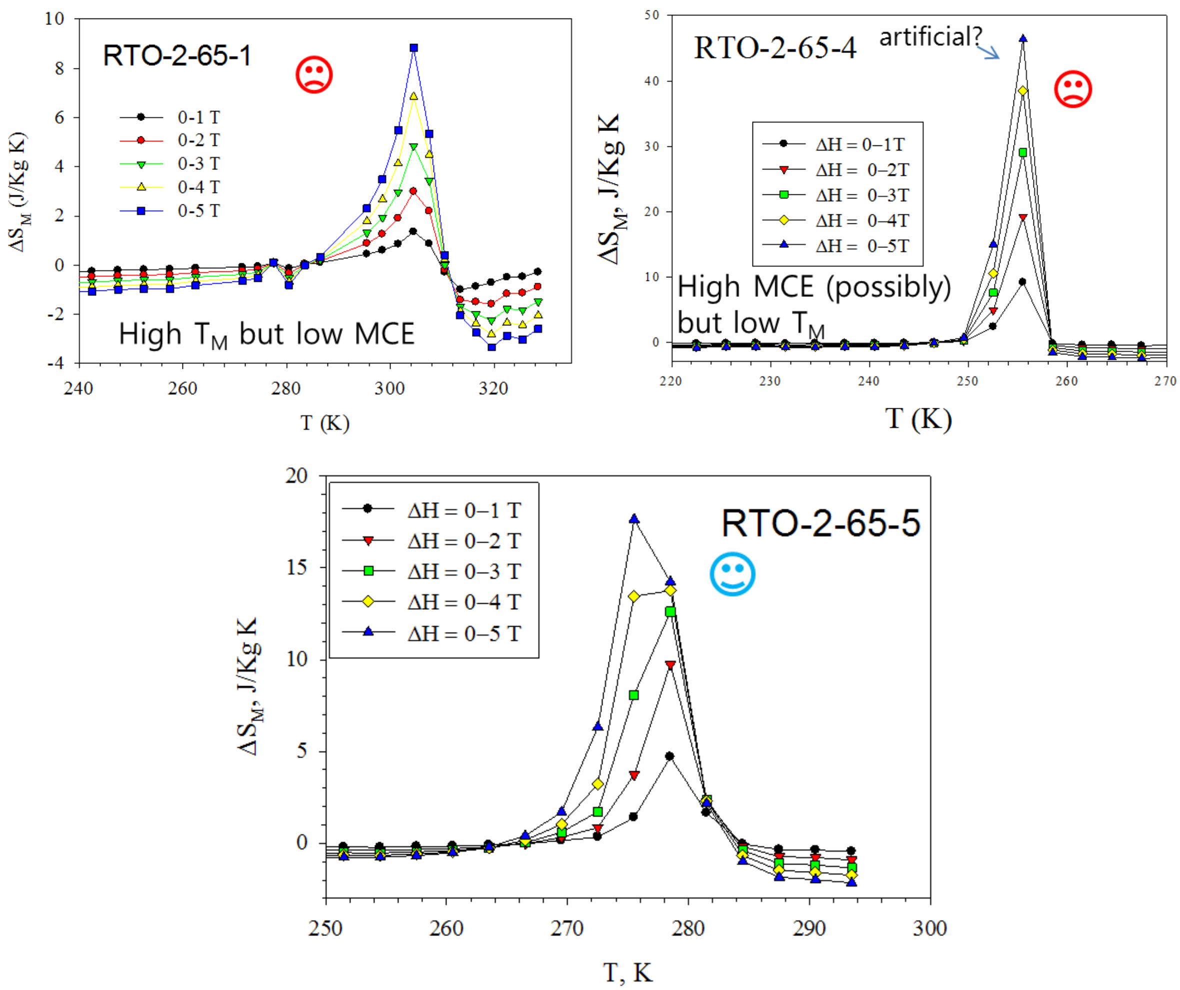



Figure 27: Magnetic field induced entropy changes as a function of temperature for selected composition of the applied field up to 0.5 T

The magnetization data confirms the ferro-magnetic nature of the samples at relatively high level containing of Cu. The transition temperature is lowered with decreased Cu amount or replaced by Co. However, the tunability of the transition with adjusting Cu containing level is a very advantageous property of magnetic refrigerants. The magnetization versus temperature curves in Figure 26 clearly demonstrates that the transition temperature varies continuously with amounts of Cu content. The fact that the change in transition temperature is continuous makes tuning of the transition very easy. However, we measured MCE for the three candidate samples to verify the level of magnetic field induced entropy change. Figure 27 shows the magnetic field induced entropy change measurement results of the selected alloys (RTO-2-65-1, RTO-2-65-4 and RTO-2-65-5 compositions) as a function of temperature for the different applied magnetic fields from 1 T to 5 T. In case of the RTO-2-65-1 sample which prepared reference sample for the new MCE materials shows very low MCE, even it has high transition temperature, $T_M$. On the contrary, the RTO-2-65-4 sample shows very high level of MCE, even it has low transition temperature. The RTO-2-65-5 sample which includes 6.9 at% Cu and 20.3 at% Sn shows the best properties in both MCE and $T_M$. These values are not high comparing excellent MCE materials but good enough to compare typical MCE materials.

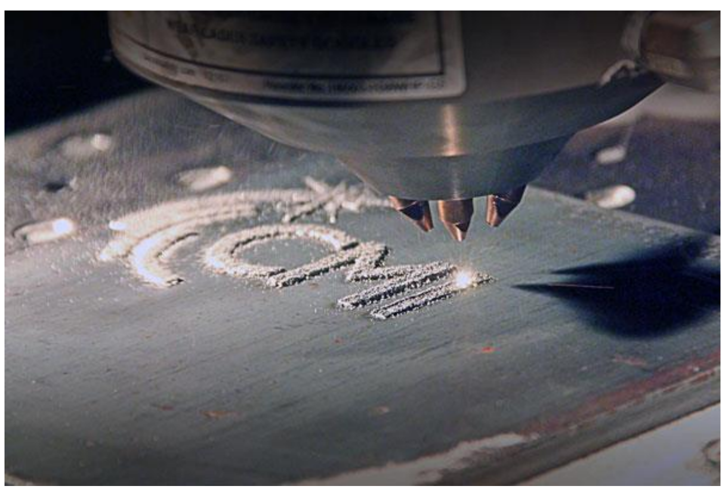


Figure 28: The Ames Laboratory/CMI LENS MR-7 3D additive manufacturing system to streamline the process of bulk combinatorial materials research, producing a large variety of alloys in a short amount of time

At this point, however, these results are preliminary results obtained from the initial step of research progress. Therefore we need more understanding on the relationship between chemical stoichiometric composition and MCE with the shifting of $T_M$.

Since most of the magnetic refrigerant materials are inorganic compounds or brittle intermetallic compounds, these materials will be difficult to fabricate in high efficiency forms-wires, screens or foils. Introducing the 3D additive manufacturing techniques recently appealed as one of strong points of the Ames Laboratory, shown in Figure 28, to produce efficiency form of MC materials combined with development of new economical MC materials are promise technologies for the industrial application of MCE. The initial work utilized arc melting and casting ingots of known composition for characterization of the magneto-thermal properties. Here we propose to use a rapid assessments approach based on bulk combinatorial materials science to significantly expand our search area of phase space, in a fraction of the time needed compared to using conventional synthesis and characterization techniques. To accomplish this, we will utilize a Laser Engineered Net Shaping (LENS) system which feeds powders into a melt pool created by a laser in order to fabricate 3D shapes. Unlike conventional 3D printers, the key advantage of the LENS systems is that it allows for the in situ tailoring of alloy compositions in bulk samples simply by adjusting the relative ratios of the powder feeders. This feature provides an extremely fast way to synthesize multiple compositions over a very short time frame. For example, recent experiments on ternary systems have shown that 36 unique composition libraries of bulk (minimum dimension ~2 mm) samples could be synthesized in less than 2 hours compared to what would take days to complete via conventional arc melting and casting. These capacities will allow us to produces a large array of compositions over a very short time period, which is critical for identifying improved materials for magnetic refrigeration applications.

To synthesize samples via laser deposition, we will start with arc melted ingots of the ternary Ni-Mn-Sn base alloy. This alloy will then be crushed and sieved into powders (+45/-125 μm in size) and placed in one of the powder hoppers of the LENS system. The other powder hoppers will be filled with pure powders of Cu, Co and other selected transition metals. By adjusting the relative feed rates of the powder hoppers, bulk alloys of the Ni-Mn-Sn ternary and selected transition metals can be deposited. The capability to change the material and relative feed rate in situ will provide for a rapid methodology to synthesize a large library of compositions. Since the LENS system allows for the printing of 3D geometries, we propose to synthesize the bulk alloys is rods for testing of their magneto-thermal properties. Following deposition of the samples, the rods will be heat treated to homogenize the structure and composition. These samples will then be run in the auto-sample VSM to measure the $T_m$. For samples that exhibit a sufficiently high transition temperature, the magnetocaloric effect will be measured by measuring the entropy of magnetization. By using this approach we will identify a composition region of interest and more closely focus our combinatorial approach on this region to identify compositions with enhanced magnetocaloric properties. Through this approach, our goal is to identify new and improved materials for magnetic refrigeration applications in a fraction of the time it would take using more conventional methodologies.

## 4.3. Development of Analysis Technique

### 4.3.1. Standardization

Due to insufficient supply for increasing demands of rare metals, many countries prepare various policies for stable supply of rare metals such as overseas resource development, recycling promotion, alternative material development, saving rare metals for emergency, export control policy, etc. Stable supply of rare metal materials is significant in the maintenance and strengthening of mainstream industry's international competitiveness. There is a large opportunity to curtail future demand for rare metals through technology that increases the efficiency with which we use rare metals, and through increased recycling. Even more impact on demand could be achieved through the adoption of the "circular economy" concept that aims to reduce, re-use, and recycle resources. A circular economy is an industrial system that is restorative or regenerative by intention and design. It replaces the "end-of-life" concept with restoration; shifts toward the use of renewable energy; eliminates the use of toxic chemicals, which impair re-use; and aims for the elimination of waste through the superior design of materials, products, systems, and within this, business models. Therefore, in order to prepare a rare metals exhaustion situation, it is urgently required for countries to standardize and conduct policy researches for collection and recycling of rare metals included in wasted products. Therefore, the followings should be studied:

a. Analysis of Rare Metals contents contained in each industrial product.
b. Analysis of supply chain of rare metals considering the mines and future demand.
c. Analysis of how to describe and recycle the rare metal contents on each industrial product for recycling convenience.

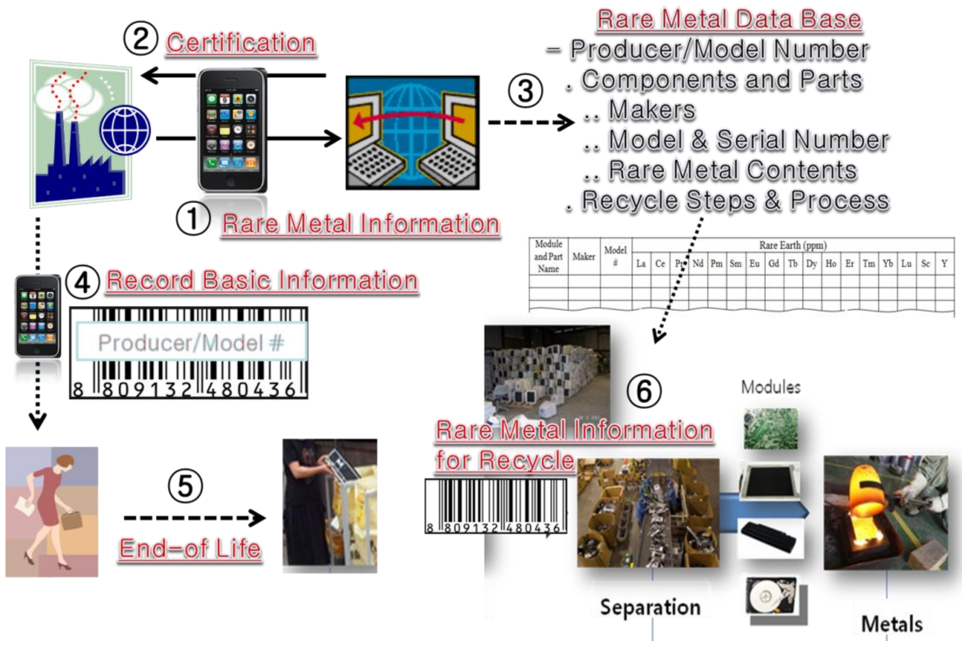



Figure 29: The example of procedure for the recycling of RE from ICT goods

Rare metals may have a key role in the development of ICT goods' further functionalities; however the amount of available rare metals is not sufficient to satisfy the industrial demand. In order to ensure the appropriate provision of rare metals to the ICT industry, recycling of rare metals becomes a crucial objective. Therefore, it is important to estimate which quantity of rare metals is used in each ICT good to the extent that this investigation is legally, technically and economically feasible, and provided that this is meaningful to recyclers. The example of standardization procedures of recycling REE from ICT goods shows in Figure 29, it also represents how to get exact information of REE contents in ICT goods through digitization of components and parts.

As a result of these efforts, the International Telecommunication Union (ITU) which is the United Nations specialized agency in the field of telecommunications, information and communication technologies (ICTs) recommended "the Recommendation ITU-T L.1100" as standard (Figure 30) to provide information on the recycling procedures of rare metals in information and communication technology (ICT) goods. It also defines a communication format for providing recycling information of rare metals contained in ICT goods. This ITU standardization focuses on the rare metals listed in Table 1 of this Recommendation. When relevant (e.g., a non-negligible amount), it is recommended that the amount of rare metals used in ICT goods be clearly provided to ensure an efficient recycling process.

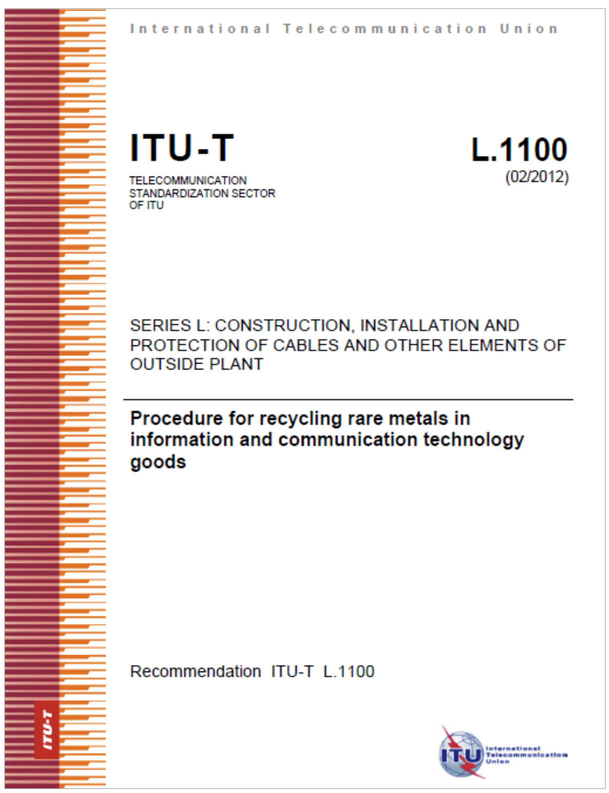
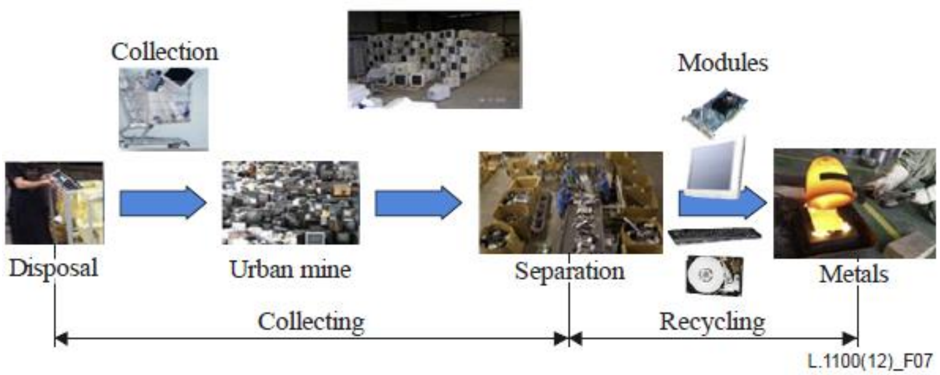



Figure 30: The Recommendation ITU-T L.1100 as standard to provide information on the recycling procedures of rare metals in information and communication technology (ICT) goods.

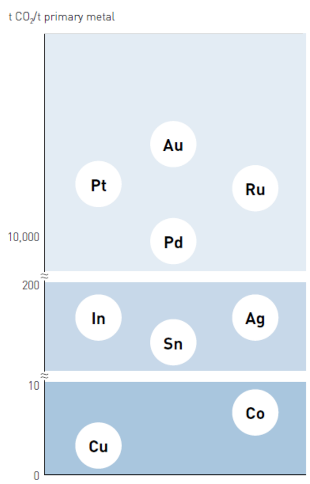
It is important to take into account all components and modules constituting ICT goods to facilitate the recycling process. Suppliers of components and modules are encouraged to provide the assembling company of the final ICT goods or intermediate modules with relevant information on the type and the amount of rare metal elements and their quantity embedded in these components and modules. For efficient recycling, it is beneficial that all ICT goods contain such rare metal information which is then finally provided to recycling industries. Collecting rare metal information can be done directly or indirectly. It is possible for example to store the information on rare metal on barcodes, vericodes, or by radio frequency identification (RFID) tags in the ICT good for example, so that this information is available. In this case, rare metal recycling industries can acquire rare metal information directly from the ICT goods. In an indirect way, rare metal information may be collected and managed by a designated authority from which recycling industries can acquire rare metal information.




| Module and part name | Maker | Model number | Rare earth (ppm) | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | La | Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu | Sc | Y |
| | | | | | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | |

Figure 31: The typical example of collecting format with recycling process

The ICT goods are easily dismantled by using acquired rare metal information and the dismantled parts are put into recycling processes to extract each rare metal element. Example of typical formats for collecting rare metal information and recycling process are shown in Figure 31 and may be defined by designated authorities, national policies, international standards or industry agreements. Recycling of purer metals often “simply” consists of remelting, generating a small fraction of the emissions shown in Figure 32. To remelt pure copper using methane gas and air creates ca. 0.1 t of $CO_2$ per tonne of Cu, compared to around 3 t of $CO_2$ per tonne of Cu from ore. Recycling of platinum group metals and many of the scarce/critical/valuable metals found in waste electrical and electronic equipment (WEEE) can lead to particularly large $CO_2$ savings, because initial $CO_2$ emissions per tonne of these metals can be very high. Figure 32 shows the average $CO_2$ tonnage that results from the primary production of metals (NB: in 2010, global GHG emissions were 33.6 Gt $CO_2$).




| Metals used in EEE | Primary production intensity t $CO_2$/t metal | Demand for EEE t/a | $CO_2$ emissions Mt/a |
|---|---|---|---|
| Au – Gold | 16,991 | 327 | 5.56 |
| Pt – Platinum | 13,954 | 8 | 0.11 |
| Ru – Ruthenium | 13,954 | 16 | 0.22 |
| Pd – Palladium | 9,380 | 30 | 0.28 |
| Au – Silver | 144 | 4,917 | 0.71 |
| In – Indium | 142 | 717 | 0.10 |
| Sn – Tin | 16 | 129,708 | 2.09 |
| Co – Cobalt | 8 | 16,470 | 0.13 |
| Cu – Copper | 3 | 7,174,000 | 24.39 |
| | | $CO_2$ total [Mt/a] | 33.58 |

Figure 32: Primary carbon footprint of elements in WEEE goods (UNEP report)

Considering the 33.58 Mt/a of $CO_2$ emitted during production of the base- and precious metals, 0.1 % of the world's $CO_2$ emissions can be attributed to these important metals in WEEE products. It is clear from the above example for pure copper that significant emissions savings are possible. However, the more important consideration is that these metals are all part of a future sustainability-enabling infrastructure and, through their high-tech use, will indirectly help decreasing society’s carbon footprint.

## 4.4. Development of Constitutional Network

### 4.4.1. Joint Research Laboratory

The Ames Laboratory has a history spanning more than 60 years of pioneering work in rare-earth (RE) research. With its team of internationally recognized rare-earth experts, the Ames Laboratory is uniquely positioned to provide the knowledge, expertise and training necessary to help ensure a global leadership position for the United States in rare-earth research, development and applications. The KITECH was founded to promote technological capability of small and medium-sized enterprises in Korea and has a mission of supporting research on materials science and engineering in Korea. The KIRAM is focusing on the commercialization and recycling of particular rare metals with highlighted its role in coordinating government, local universities, and small and medium enterprises to facilitate R&D support for core technologies, carrying them through to commercialization, and establishing new industry. These days, the KITECH actively reorganized its structure and diversified SME supporting channels to overcome and cope with an upheaval technology R&D world. Importance of the International cooperation in R&D is becoming more and more emphasized and a very essential part of its success. More complicated technology fusion, shorten technology lifespan and enlargement tendency of technology development scale has increased risk of working-alone type of R&D which requires more time and budget. Therefore, it is essential to establish international cooperative network and take advantage of advanced technology and manpower of the world.

As a result of cooperation, both KITECH and Ames Laboratory are performing the R&D collaboration for the substitution of rare earth resources which is focused on the reclaiming the rare earth resource from the waste materials. Substitution, in this context, means finding alternative rare earth resources to replace original raw supply resources. Based on the successful collaboration between KITECH and Ames Laboratory until now, we are working on an agreement for setting up the Joint Research Laboratory (JRL) to expand the next step of cooperation of both institutes through the organization of JRL, as shown in Figure 33. The research team of JRL includes Ames Laboratory scientists with representatives from KITECH.

The research group is developing and testing the technique in Ames Lab's Materials Preparation Center, with a suite of materials science tools supported by the DOE. Therefore, R&D collaboration on 'Rare Metals for Energy Technologies' for the future between KITECH and Ames Laboratory will be covering a wide range of research schemes from basic science to applied research for commercialization and will be synergizing the common research perspectives of both institutes.

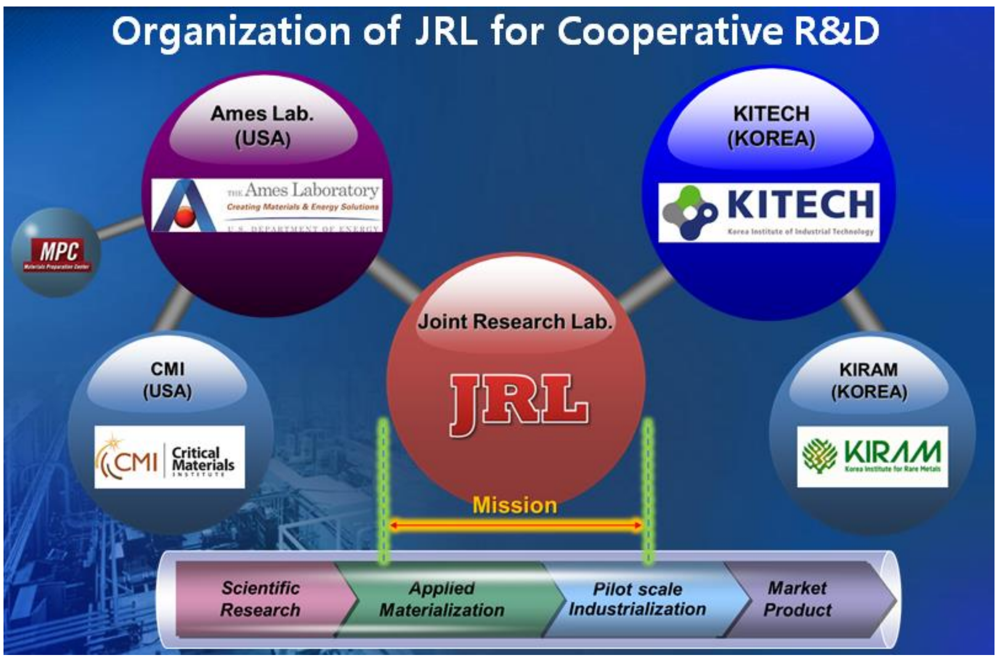
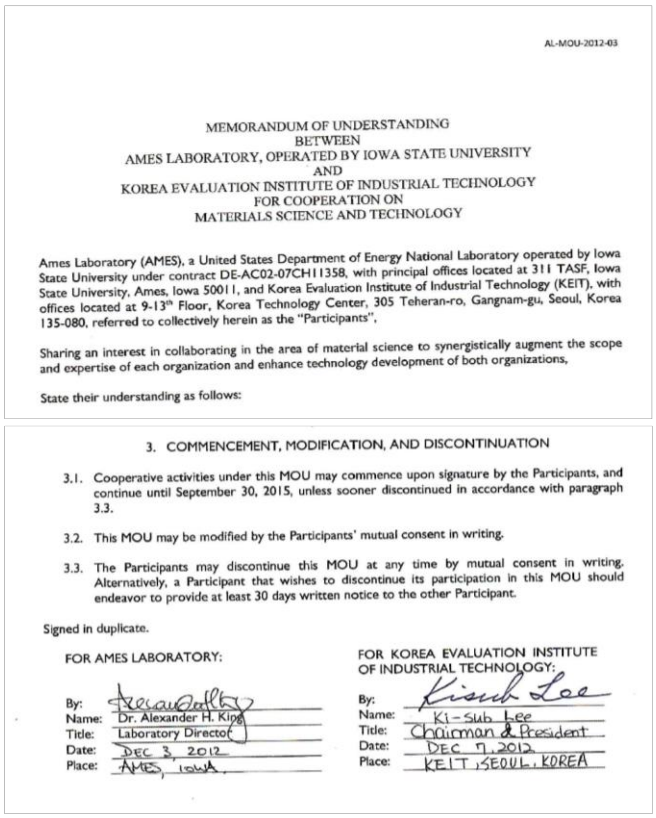



Figure 33: The example of international R&D collaboration through the organization of Joint Research Laboratory (KIRAM report)

### 4.4.2. Human Resource Exchange

To cope with the new technology frontier, low carbon sources, and green energy technology, establishment and activation of international network play a key role in the success of collaboration by exchanging outstanding personnel, as well as exchanging human and research resources. Cooperation with the qualified partners abroad includes a memorandum of understanding between the foreign research institute and the Korean collaboration partner, as well as exchanging talented research staff, research samples, and an excellent idea in science and technology. The international collaboration partners mostly R&D institutes of both countries, but sometimes, shown in Figure 34, it may involve the collaboration of administration institutes by the demand of cooperation missions, such as NSF, KEIT (Korea Evaluation Institute of Industrial Technology) and KIAT (Korea Institute for the Advancement of Technology).



AL-MOU-2012-03

MEMORANDUM OF UNDERSTANDING
BETWEEN
AMES LABORATORY, OPERATED BY IOWA STATE UNIVERSITY
AND
KOREA EVALUATION INSTITUTE OF INDUSTRIAL TECHNOLOGY
FOR COOPERATION ON
MATERIALS SCIENCE AND TECHNOLOGY

Ames Laboratory (AMES), a United States Department of Energy National Laboratory operated by Iowa State University under contract DE-AC02-07CH11358, with principal offices located at 311 TASF, Iowa State University, Ames, Iowa 50011, and Korea Evaluation Institute of Industrial Technology (KEIT), with offices located at 9-13th Floor, Korea Technology Center, 305 Teheran-ro, Gangnam-gu, Seoul, Korea 135-080, referred to collectively herein as the "Participants",

Sharing an interest in collaborating in the area of material science to synergistically augment the scope and expertise of each organization and enhance technology development of both organizations,

State their understanding as follows:

3. COMMENCEMENT, MODIFICATION, AND DISCONTINUATION

3.1. Cooperative activities under this MOU may commence upon signature by the Participants, and continue until September 30, 2015, unless sooner discontinued in accordance with paragraph 3.3.

3.2. This MOU may be modified by the Participants' mutual consent in writing.

3.3. The Participants may discontinue this MOU at any time by mutual consent in writing. Alternatively, a Participant that wishes to discontinue its participation in this MOU should endeavor to provide at least 30 days written notice to the other Participant.

Signed in duplicate.

| FOR AMES LABORATORY: | FOR KOREA EVALUATION INSTITUTE OF INDUSTRIAL TECHNOLOGY: |
|---|---|
| By: [signature] | By: [signature] |
| Name: Dr. Alexander H. King | Name: Ki-Sub Lee |
| Title: Laboratory Director | Title: Chairman & President |
| Date: DEC 3, 2012 | Date: DEC 7, 2012 |
| Place: AMES, IOWA | Place: KEIT, SEOUL, KOREA |

Figure 34: The memorandum of understanding between the Ames Laboratory and Korea Evaluation Institute of Industrial Technology (KEIT) through KITECH

One of the internationally advanced research institutions, the Ames Laboratory and KITECH, are collaborated together by exchanging scientist and delegation from both countries to promote research on Materials Science & Engineering including Rare Metals, and in educating and training Korean scientist by joining research activities. (Figure 35) However, sometimes the international collaboration between foreign institutes needs cooperative work with administrative department to resolve any incoherency of two different operating systems of each organization.

For example, as shown in Figure 36, KITECH should have to register US federal government Data Universal Numbering System (DUNS) for the collaboration project with Ames Laboratory.

THE Ames Laboratory
Creating Materials & Energy Solutions
U.S. DEPARTMENT OF ENERGY

**KITECH**
Wednesday, Dec. 5, 2012

| *Time* | *Location* | *Activity* |
|---|---|---|
| 9:30 – 10:00 a.m. | 111 TASF | Arrive & check in. Introductory tour. |
| 10 – 10:30 a.m. | 311 TASF | Meet with Mark Murphy, Ames Laboratory chief operations officer |
| 10:30 – 11 a.m. | 311 TASF | Meet with Deb Covey, Ames Laboratory associate director for sponsored research programming |
| 11 a.m. – noon | 121 MD | Tour the Materials Preparation Center |
| Noon – 1 p.m. | 311 TASF conference room | Lunch |
| 1 – 2:30 p.m. | G40 TASF | Meet with Environmental Health, Safety & Assurance staff |
| 2:30 – 3 p.m. | 311 TASF | Meet with Cynthia Jenks, Ames Laboratory division director of chemical and biological sciences and assistant director of scientific planning, and Tom Lograsso, Ames Laboratory interim deputy director and division director for materials sciences and engineering. |
| 3 – 3:30 p.m. | 311 TASF | Meet with Ames Laboratory director Alex King |

THE Ames Laboratory
Creating Materials & Energy Solutions
U.S. DEPARTMENT OF ENERGY

**Korean Visitors**
Thursday, March 21, 2013

| *Time* | *Location* | *Activity* |
|---|---|---|
| 10 :00 a.m.– noon | 121 MD | Meet with Larry Jones and Tour the Materials Preparation Center |
| Noon – 1:00 p.m | | Lunch |
| 1:00 p.m. – 2:30 p.m. | 208 MD | Meet with Min-Ha Lee |
| 2:30 p.m. – 3:30 p.m. | 311 TASF Conference room | Meet with Ames Laboratory director Alex King , Meet with Deb Covey, Ames Laboratory associate director for sponsored research programming, and Tom Lograsso, Ames Laboratory interim deputy director and division director for materials sciences and engineering. |
| 3:30 p.m. – 4:00 p.m. | 311 TASF Conference room | Meet with Karl Gschneidner Jr |
| 4:00 p.m. – 4:30 p.m. | 106 MD | Meet with Ryan Ott |

List of Korean visitors
1. Dr. Jin-Young LEE/ KIGAM (Korea Institute of Geoscience and Mineral Resources)
2. Dr. Sung-Wook CHO/ KIGAM (Korea Institute of Geoscience and Mineral Resources)
3. Dr. Ha-Kyun JUNG/ KRICT (Korea Research Institute of Chemical Technology)

Figure 35: The visiting of scientist, delegation from Korean national institutes to Ames Laboratory for collaboration

This effort stems from the R&D collaboration through joint-projects on common interesting areas performed by both institutes in the last several years. Moreover, the personnel of Ames Laboratory visited KITECH and KIRAM to discuss research progress. The director of Ames Laboratory was invited to become a member of the International Advisory Board of KITECH (Figure 37) which recently established strong inter-personal or inter-institutional cooperation networks in prominent research institutions, universities and agencies. This was done by conducting an international joint R&D, personnel and information exchanges required for the

R&D, as well as pursuing cooperation between four overseas operating locations: United States, China, Indonesia and Vietnam.

D&B
Decide with Confidence

D&B D-U-N-S Registered™ Member Since: Sep-12-2012

D&B D-U-N-S Registered™ Profile

Corporate Information
**Company Name:** Korea Institute of Industrial Technology
**Business Address:** Yangdaegiro-gil, Ipjang-myeon, seobuk-gu, 89, Cheonan, Chungnam, 331820, Republic of Korea
**Tel:** +82 (41)58982722
**Fax:** +82 (41)58982600
**Current Website:** http://www.dunsregistered.com
**Main Website:** http://www.kitech.re.kr
**Email Address:** ohk16@kitech.re.kr
**Employee Size (Range):** 1303

Registration Information
**Line of Business:** Scientific and Technical Services
**SIC Codes:**
87339904 - Scientific research agency
**Business Type:** Others
**Date of Registration:** 1989
**Registration Number:** Business Registration Number 1198201008, Corporation Number 1151220000210

D&B Information
**DUNS Number:** 688465665

Key Employees
**Chief Executive Officer :** Mr. Kyoung Hoan Na

Business Information
**Linkage Information:**
**parent:** 631128089 Ministry of Knowledge Economy
**Global Ultimate:** 688005487 Government of The Republic of Korea

Figure 36: The Data Universal Numbering System (DUNS) registration of KITECH for working with Ames Laboratory

KITECH

Taejin Hwang, Ph.D.

Korea Institute of Industrial Technology, KITECH
http://www.kitech.re.kr

89 Yangdaegiro-gil, Ipjang-myeon
Seobuk-gu, Cheonan-si
Chungcheongnam-do
331-822 Korea (Rep. of)
Tel. +82 41 5898 261
Fax. +82 41 5898 260
E-mail greathtj@kitech.re.kr

September 12th, 2013
Cheonan-si

**Invitation to the Intl. Advisory Board of Korea Institute of Industrial Technology**

Dear Dr. Alexander H. King,

Figure 37: The invitation of Ames Laboratory director to International Advisory Board of KITECH

Finally, valuable ideas from the scientists with insight and excellence are expected to help more effectively utilize resources needed for international cooperation, including basic and industrial technology, human resources, research facility and cooperation networks.

# 5. Conclusion

The addressed "The future will be defined by a creative divide" by Park Geun-hye, the President of the Republic of Korea in 2014 World Economic Forum Annual Meeting at Davos, Switzerland, emphasizes focus on promoting creativity and innovation to drive sustainable growth and improve economic resilience of world. For the next generation economy to reach its full potential, we must work together with creativeness to ensure stable supplies of the materials required. That means working together to research and development of diversify solution for overcoming global crisis, as well as investing into manufacturing, processing, substitutes and finding ways to recycle critical materials.

To proactively secure the availability of rare metals or rare earths and other strategic materials required for the next generation economy, we must take considering globalized supply chains for strategic materials. Doing so will improve our flexibility as we address the materials demands of the clean energy economy. This means investing in R&D to develop substitutional resources and other technologies that reduce our dependence on rare earths. However keeping in mind, there is no place to apply advanced technology without the consumer market; it means that the perspectives of R&D have to follow the megatrends of the final objective industry. Therefore, for the securing short term strategy, we will examine urgent issues related to supply chain of critical materials together with industrialized country which has strengthening in manufacturing of mainstream products by transforming advanced technology to commercialization technology, like South Korea. The United States has talent and innovative capacity in materials science and can be harnessed to create the next generation of rare earth sciences and competing technologies. However, it would be recommended collaboration with common interested countries to analyze and to be covering exactly weak points in the whole supply chain of main stream industry. The best long-term solution for a rare earth shortage would be the discovery of a viable substitute for rare earths or environmental friendly method to recycle rare earths. The first nation that develops a reliable substitute for critical materials will gain a significant advantage over other countries that have not developed an alternative to rare earths. And we must promote recycling, re-use and more efficient use of strategic materials, to get more economic value out of each ton of ore extracted and refined. As in the case of magnet recycling program performed by Ames Laboratory, re-use can help mitigate potential supply constraints. Widespread recycling and re-use could significantly lower world demand for rare earths and other strategic materials. Also, if recycling and re-use followed adequate procedures, i.e., international standard, this could also reduce the lifecycle environmental footprint of these materials. The opportunity to boost recycling is significant. Strong demand for metals over the past decade has led to high resource prices meaning that a significant amount of scrap metal is available.

As a part of this project, we have presented some examples of the international collaboration to develop a common response for the demand of RE including R&D works. This work has been presented at numerous international conferences and discussed with delegations from research institutes/universities. As a part of human resource exchange within this project, Ames Laboratory also hosted several visiting scientists/students for 3 months stays.

# 6. References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2. MIT Energy Initiative Workshop Report, "Critical elements for new energy technologies", 2010
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11. KIRAM Report, "Strategies and perspectives for securing rare metals in KOREA", 2011
12. Nichole Clagett, "A rare opportunity: Streaming permitting for rare earth materials within United States", J. Energy & Environ. Law, 2013
13. Royal Netherlands Chemical Society Report, "Scarcity of rare earth elements", 2012
14. World Economic Forum Public Report, "Top 10 emerging technologies 2014", 2014
15. World Economic Forum Scoping Paper, "Mining and metals in a sustainable world", 2014

# 7. Appendices

## 1. Introduction of KIRAM

KIRAM is

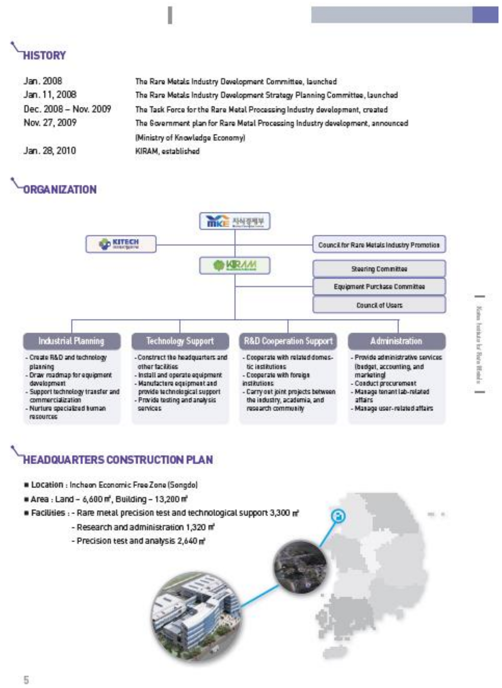
INTRODUCTION

*FUNDAMENTAL OF KIRAM*

The Korea Institute for Rare Metals (KIRAM) was founded on January 28, 2010 through the support of the Ministry of Knowledge Economy. Aiming to realize Korea's ambition for a global powerhouse of rare metals, KIRAM has since developed policies to promote the domestic rare metals industry and constructed the core infrastructure. As the central institution for the rare metals sector, KIRAM will play a leading role in forming an organic cooperative system between the industry, academia, and research community, laying the foundation for the stable supplies of rare metals, developing new technologies, and building the basic infrastructure for the rare metals industry.

PURPOSES OF KIRAM

To systematically promote the rare metals industry by expanding rare metal reserves, laying the foundation for joint research on material processing and circulation technology, and supporting the industry's technology development efforts.



HISTORY

| | |
|---|---|
| Jan. 2008 | The Rare Metals Industry Development Committee, launched |
| Jan. 11, 2008 | The Rare Metals Industry Development Strategy Planning Committee, launched |
| Dec. 2008 – Nov. 2009 | The Task Force for the Rare Metal Processing Industry development, created |
| Nov. 27, 2009 | The Government plan for Rare Metal Processing Industry development, announced (Ministry of Knowledge Economy) |
| Jan. 28, 2010 | KIRAM, established |

ORGANIZATION



HEADQUARTERS CONSTRUCTION PLAN

- Location : Incheon Economic Free Zone (Songdo)
- Area : Land – 6,600 ㎡, Building – 13,200 ㎡
- Facilities : - Rare metal precision test and technological support 3,300 ㎡
  - Research and administration 1,320 ㎡
  - Precision test and analysis 2,640 ㎡

5

Rare metals

*What are the rare metals in KOREA?*

Definition of rare metals in KOREA

35 species, 56 elements
Rare earth Element (REE) : 17 elements
Platinum group metal (PGM) : 6 elements

Positioning Trap between China and Japan

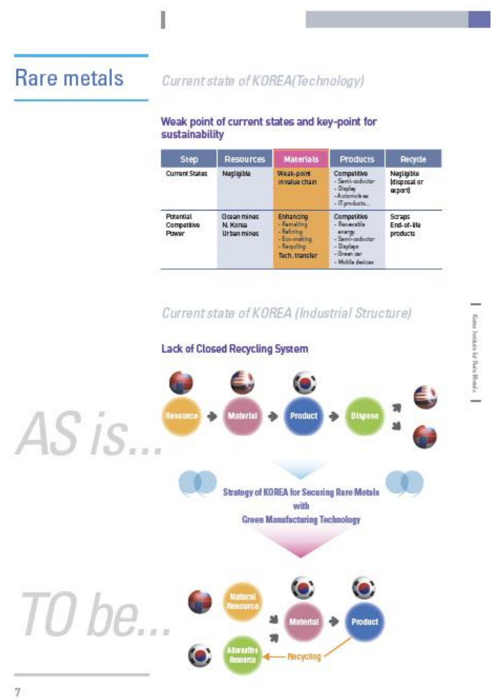
Resource Based Countries
Regulation of Export
Korea(Import base) / Lack of resource
Huge commercial industry / Weak supporting industry
Technology based Countries
Closed recycling system

*As Critical Resources*

Rarity + (Distribution × Demand) + Rate of popularity



6



Rare metals

*Current state of KOREA(Technology)*

Weak point of current states and key-point for sustainability

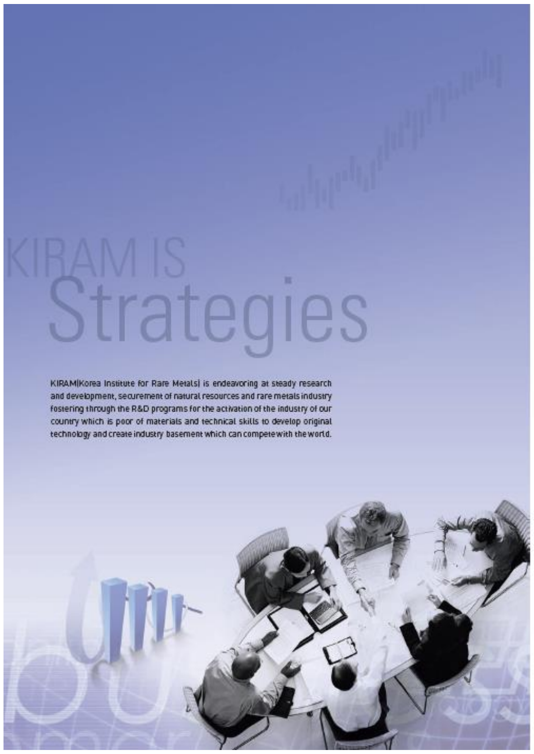
| Step | Resources | Materials | Products | Recycle |
|---|---|---|---|---|
| Current States | Negligible | Weak-point in value chain | Competitive - Semi-conductor - Display - Automotive - IT products... | Negligible (disposal or export) |
| Potential Competitive Power | Ocean mines N. Korea Urban mines | Enhancing - Remelting - Refining - Eco-making - Recycling Tech. transfer | Competitive - Renewable energy - Semi-conductor - Displays - Green car - Mobile devices | Scraps End-of-life products |

*Current state of KOREA (Industrial Structure)*

Lack of Closed Recycling System



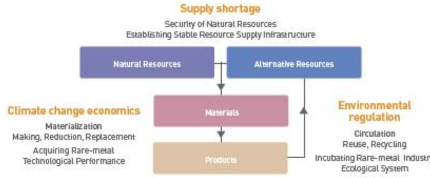
7




KIRAM(Korea Institute for Rare Metals) is endeavoring at steady research and development, securement of natural resources and rare metals industry fostering through the R&D programs for the activation of the industry of our country which is poor of materials and technical skills to develop original technology and create industry basement which can compete with the world.

## Strategies

*Perspectives for Rare Metals in KOREA*
*(Scope & Concept)*




*Strategies for Rare metals in KOREA*

**Securing Foreign/Oversea Natural Resources**

- Investment and exploration of foreign resources
- Diplomatic effort to Enforcing relationship(ODA*)

*ODA: Official Development Assistance

**Securing Domestic Natural Resources (Stockpiles)**

- Increase volume of strategic/economic stockpiles
- Set up flexible execution

**Focusing on R&D for Materialization (Reduction/Replacement)**

- Enhancing R&D activities for materials
- Technology for reduction in usage and replacement novel metals

**Circulation Technology and Infrastructuring (Recycle/ Reuse)**

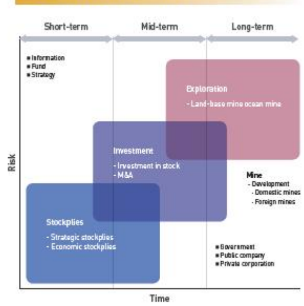
- Reuse and recycle of scraps and end-of-life products
- Urban mines

9

## Strategies

*Strategies for Rare Metals in KOREA*

**A. Securing Natural Resources**




**1. Securing Overseas Natural Resources**

**Enforcing Resource Politics**

- Dispatching investigation team : South Africa, Zimbabwe, Madagascar
- Establishing Korea-China collaboration : KCMIC(Korea-China Material Industry Committee)

**Increasing Foreign Mine Development**

- Prior Development & Production of 4 Minerals : Cr, Mn, Mo, W
- Prior Investigation of 2 Minerals : Li(Chile, Bolivia), RE

**Modifying Regime/Law to Enhancing Support System**

- Designation 6 Critical Strategic Elements(CSE) : Cr, Mn, Mo, W,Li, RE
- Endorsing Incentives to Develop Foreign Mineral Resources for CSEs

**2. Securing Domestic Natural Resources**

**Investigating Stockpiles & Reserves**

- Accurate Investigation & Estimating Economical Viability

**Domestic Mine Development**

- Re-estimating Reserves of 5 minerals for Securing 18 Mil. ton(~2015) : W, Mo, Ti, RE, Mg
- Prior to Redevelopment of 6 Old Mines(~2020): RE

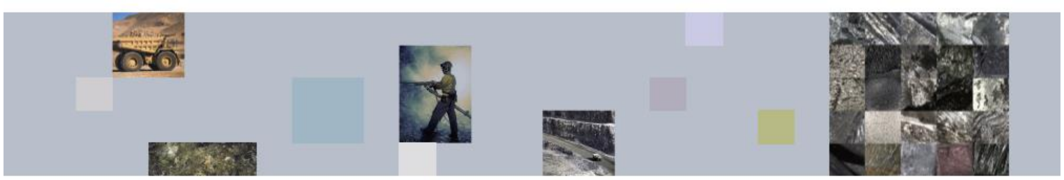
**3. Increasing Volume & Species of Domestic Stockpiles**

**Government Driving Plan**

- 60 days Consumption amount/ Each element for 21 Metals

**Industry Driving Plan**

- Supplying Governmental Stockpile System for Industries
- Endorsing Priority to SME for Purchasing of CSEs in Emergency Situation



10 11

## Strategies



### B. Enhancing R&D Activities for Materialization

**1. Designation Strategic Rare metal & Essential Core Technology**

- Strategic Rare metals(~2009) : 11 Elements, Enlarge to 20 Elements
- Essential Core Technology : 40 Technologies, Develop R&D Programs

Starting from R&D programs

- 11 strategic rare metals
- 40 target technologies

**2. Establishing Roadmap in Each Sections**

Prior Commercialization

- Demand Increasing Elements : In, PGM, MG
- R&D collaboration between Demander & Supplier

Cutting edge Industry

- Government Leading R&D : Li, RE, Ti
- Critical Elements in Mainstream Industry

**3. Supporting Rare metals Technology R&D Program**

Refining(Purifying), Smelting

- Develop Technology for Demand increase & Low technical level CREs

Processing, Treatment

- Develop Technology for Materialization of Rare metals

Replacing, Reducing

- Develop Technology for Maldistributed & shortly Exhausted 6 CREs

PRecycling(Circulation)

- Develop Technology for Recycling of each Rare metals

Development of 40 Essential Core Technologies(300 $US MiL/10yr)






### C. Circulation for Alternative Resources

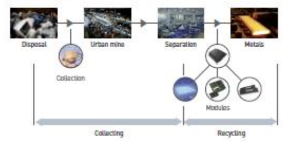
Goal of Circulation

- Increasing national efficiency of materials utilization

Target of Circulation

- Recycling of scraps during manufacturing
- Reuse and recycling of end-of-life products(e-wastes)

Urban Mining




### D. Activating Circulation of Rare metals

Regulating Recycling System

- Electronic Product : Expand Species(2010~)
- Automobile : Expand Recycling Component(2013~)
- Byproduct : Classifying as specific product(2011~)

Increasing Collect Efficiency

- Ordinary Collecting System of Scraps
- Appliances : Duty of Manufacturer for Separation & Discharging

Introducing Content Indication System

- "Rare metal Indication System" for the 6 CREs of IT product : Cell phone, Digital camera, PMP, MP3, Pocket game, Navigation

## Strategies

Strategies for Rare metals in KOREA

### E. Establishing Infrastructures for Rare metal Industry

Organizing Rare metals Industry Governing Committee

- Adjusting & Consulting for Government Rare-metals Policy
- Members: Government (MKE), Industries, Universities, Institutes

Organizing Korea Institute for Rare Metals(KIRAM)

- Deciding Technological Issues on Strategic Rare-metals
- Governing R&D programs

Designation Local Rare metals Commercialization Center

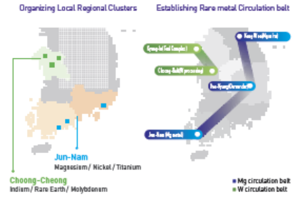
- Kang-won(Mg, Ti), Choong-cheong(In, Pt), Jun-nam(Mg, Ni)

Master plan of Korea Institute for Rare Metals (KIRAM)






**1. Organizing Rare metals Industry Clusters**




**2. Incubating Rare metals Specialized Company**

Support Investment Fund

Tax Exemption & Refund

Enlarging Government-Industry Investment

- KORES will invest 820 $US Mil. until 2013 to found Rare metals Specialized companies

Ex) Hwang-Yang FerroAlloy Co.(Mo) was founded 2007 by joint venture of KORES(GOV.) and KTC

Industry Investment

- Large Enterprises increase investment on Rare metals infrastructure

| | | |
|---|---|---|
| Refining, Smelting Total 520 $US Mil. | Co Sulfide 2010~2013 30 $US Mil. | COSMO |
| Processing, Treatment Total 90 $US Mil. | Ti Slab 2010~2012 30 $US Mil. | posco |
| Recycling (Circulation) Total 210 $US Mil. | RM recycling 2009~2015 210 $US Mil. | LS-Nikko |

## Strategies

*Strategies for Rare metals in KOREA*

### 3. Establishing Infrastructure for Rare metal Industry

**Setting up Materials Flow & Statistical DB**

- Analyzing materials flow for Critical Strategic Rare metals
- Updating HSK code, designate new HSK code for each rare metals
- Analyzing IP for CSR, provide IP information

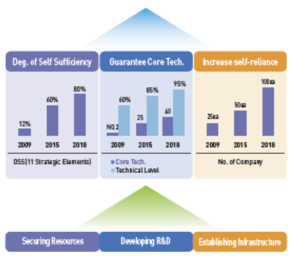
**Training Rare metal Specialist**

- Starting Rare metal Technology education Course, training Laborer
- Designating Rare metal Specialized Universities
  Kongju Natl. Univ., Soonchoen Univ., Hanyang Univ., Incheon Natl. Univ.

**Establishing International Collaboration**

- Prospecting International joint R&D, Seminar, Researcher Exchange
  Refining : China, Canada, Russia, Indonesia
  Processing : Japan(AIST), Germany(Leibniz), USA(LANL)
  Recycling : Japan(NIMS), USA(Ames Lab.), UK(York Univ)

**Realization of Strong Power Nation in Rare metal Industry (Resource-Industry-Chain of Value) - RICH KOREA 1040 -**






## The member of KIRAM


TAEK SOO KIM (Director)
Chief Operating


MIN HA LEE (Principal Researcher)
International cooperation in rare metals industry


HYO SOO LEE (Principal Researcher)
Rare Metals Industry Trend Analysis


HAN SHIN CHOI (Senior Researcher)
Utilization of Rare Metal Industry Development


YONG HWAN KIM (Senior Researcher)
Recycling of Rare Metals Industry Technology Development


BUM SUNG KIM (Senior Researcher)
Center for equipment purchases and operating


KYOUNG MOOK LIM (Senior Researcher)
Laying the groundwork for the establishment of centers


SOO YOUNG LEE (Researcher)
Planning Support


MI HYE LEE (Researcher)
Planning Support

2. The 1st International rare metals workshop program

## The 1st INTERNATIONAL WORKSHOP ON RARE METALS 제1회 희소금속 국제워크샵

**International Cooperation for Strategies and Perspective on Rare Metals** 희소금속의 전략 및 전망에 관한 국제협력

DATE 18(MON) - 19(TUE) APRIL, 2011 VENUE Sheraton Incheon 3F GRAND BALLROOM

ORGANIZED BY KIRAM KITECH Incheon Metropolitan City MKE Ministry of Knowledge Economy

### April 18(Mon.)

| Time | Program | | Speaker |
|---|---|---|---|
| 09:30~09:40 | Welcome & Overview<br>Opening Speech | | Cho Seok (Deputy minister for industry)<br>Ministry of Knowledge and Economy/ Korea<br>조 석 지식경제부 실장 |
| 09:40~09:46 | | | Young-Hwan Kim (Chairman)<br>National Assembly, Knowledge Economy Committee/ Korea<br>김영환 지식경제위원회 위원장 |
| 09:46~09:52 | | | Dong Keun Shin (Deputy mayor for political affairs)<br>Incheon Metropolitan City / Korea<br>신동근 인천광역시 정무부시장 |
| 09:52~09:58 | | | Il-Pyo Hong (Congressperson)<br>National Assembly / Korea<br>홍일표 국회의원 |
| 09:58~10:04 | | | Kyoung-Hoan Na (President)<br>KITECH / Korea<br>나경환 한국생산기술연구원 원장 |
| 10:04~10:10 | | | Taek-Soo Kim (Director)<br>KIRAM / Korea<br>김택수 희소금속산업기술센터 센터장 |
| 10:10~10:20 | Break | | 3F Lobby |
| 10:20~11:00 | Keynote Speech | National R&D Strategy of Korea<br>우리나라 국가 R&D 전략 | Jong-Man Park (Director)<br>KEIT / Korea<br>박종만 한국산업기술평가관리원 단장 |
| 11:00~11:45 | Session I | Rare Metals Related Issues in Korea<br>(Steel Industry)<br>우리나라 철강산업과 희소금속 현황 | Oh-Joon Kwon (CTO)<br>POSCO / Korea<br>권오준 포스코 부사장 |
| 11:45~12:30 | | Rare Metals Related Issues in Korea<br>(Mineral Resources)<br>우리나라 희소금속 광물 정책 | Seok Jin Song (Executive vice president, business development division)<br>KORES / Korea<br>송석진 광물자원공사 자원개발본부 본부장 |
| 12:30~14:00 | Lunch | | Grand Ballroom 1 |
| | International Committee establishing ceremony & Luncheon (VIP)<br>VIP 오찬 및 국제 희소금속 협의회 발족식 | | Lotus 3 |
| 14:00~15:30 | Session II | Rare Metals Related Issues in US<br>(Rare Earths)<br>미국의 희토류 현황 | Alex King (Director)<br>Ames Lab. / USA<br>알렉스 킹 미 국립에임즈 연구소 소장 |
| 15:30~15:45 | Coffee Break | | 3F Lobby |
| 15:45~16:30 | Session III | Rare Metals Related Issues in Japan<br>(Rare Earths)<br>일본의 희토류 연구현황 | Nobuhito Imanaka (Dean)<br>Osaka Univ. / Japan<br>노부히토 이마나가 일 오사카 대학 학장 |
| 16:30~17:15 | | Rare Metals Related Issues in Germany<br>(Strategy/industrial application)<br>독일의 희소금속 산업현황 및 전략 | Jurgen Eckert (Director)<br>Leibniz IFW Institute / Germany<br>위르겐 에커트 독 라이프니쯔 연구소 부소장 |
| 17:15~18:00 | | Rare Earth Metals Research at McGill University and Canada<br>캐나다의 희소금속 연구 현황 | Steve Yue (Chairman)<br>McGill Univ. / Canada<br>스티브 츄 캐나다 맥길대/코렘사 이사장/학장 |
| 18:00~20:00 | Dinner | | Grand Ballroom 1 |

### April 19(Tue.)

| Time | Program | | Speaker |
|---|---|---|---|
| 09:00~09:45 | Session IV | Rare Metals Related Issues in Korea<br>(R&D, Application)<br>우리나라 희소금속 연구개발 현황 | Kyung-Mok Cho (President)<br>KIMS / Korea<br>조경목 재료연구소 소장 |
| 09:45~10:30 | | Rare Metals Related Issues in Japan<br>(Rare Earths)<br>일본의 희소금속 효율적 활용 | Koichi Niihara (President)<br>Nagaoka Tech. Univ./ Japan<br>고이찌 니하라 일 나가오카대학 총장 |
| 10:30~10:45 | Coffee Break | | 3F Lobby |
| 10:45~12:15 | Session V | Rare Metals Related Issues in USA<br>(Resources Survey)<br>미국의 희소금속 광물 현황 | Pui-Kwan Tse (Chief)<br>USGS / USA<br>피관 체 미 지질자원조사국 수석 분석관 |
| 12:15~13:30 | Lunch | | Grand Ballroom 1 |
| 13:30~14:15 | Session VI | Rare Metals Related Issues in Indonesia<br>인도네시아의 희소금속 광물 현황 | Flory Dryanti (Head of sub director)<br>Ministry of Industry / Indonesia<br>플로리 다리안티 인도네시아 경제부 부국장 |
| 14:15~15:00 | | Rare Metals Related Issues in Japan<br>(Industry/Application)<br>일본의 희소금속 산업 및 재활용 현황 | Takashi Goto (Dean)<br>Tohoku Univ. / Japan<br>다카시 고토 일 동북대학 학장 |
| 15:00~15:15 | Coffee Break | | 3F Lobby |
| 15:15~16:00 | Session VII | Rare Metals Related Issues in China<br>중국의 희소금속 자원 현황 | Xujun Mi (Director)<br>GRINM / China<br>슈준 미 중 유색금속연구소 부소장 |
| 16:00~16:45 | | Rare Metals Related Issues in Germany<br>(Technological Application)<br>독일의 희소금속 연구개발 현황 | Bernd Szyszka (Director)<br>Fraunhofer IST Institute / Germany<br>버든 시스즈카 독 프라운호퍼 연구소 부소장 |
| 16:45~17:30 | | Rare Metals Related Issues in UK<br>(Technological Application)<br>영국의 희소금속 연구개발 현황 | Avtar Matharu (Dean)<br>Univ. of York / UK<br>아브타 마타루 영 요크대학 부학장 |
| 17:30~18:20 | Summary & Concluding Remarks<br>총론 및 맺음말 | | Taek-Soo Kim (Director)<br>KIRAM / Korea<br>김택수 희소금속센터 센터장 |
| 18:20~20:00 | Dinner | | Grand Ballroom 1 |

※ Presentation (35min) / Q&A(10min) per each person (각 35분 발표, 10분 질의응답 진행)

3. The 2nd International rare metals workshop program

**Event Schedule**

- **8:15 AM** - Welcome
  Alex King, The Ames Laboratory
- **8:30 - 8:50 AM** - GE Approaches to Assessing and Mitigating Rare Metal Risks
  Steve Duclos, GE Global Research
- **9:00 - 9:20 AM** - Industrial and Scientific R&D Trends in Rare Metal in Korea
  Taek-Soo Kim, KITECH
- **9:30 - 10:00 AM** - New Stage of Resource Issues and Strategic Elements Initiative in Japan
  Kohmei Halada, National Institute of Materials Science
- **10:00 AM** - BREAK
- **10:30 - 10:50 AM** - What's Ahead for the Rare Earths? Chaos? Calm? or Both?
  Karl Gschneidner, The Ames Laboratory
- **11:00 - 11:20 AM** - Rare Metal Issues and Strategies in Canada
  William Mercer, Avalon Rare Metals, Inc.
- **11:30 - 11:50 AM** - Rare Metals: New Resources, Opportunities and Challenges in Brazil
  Kirstin Elaine Myers, Sparx3
- **Noon** - LUNCH
- **1:30 - 1:50 PM** - Critical and Conflict Materials Important to Global Security
  Alan Hurd, Franklin Fellow, U.S. Department of State
- **2:00 - 2:20 PM** - PHYTOCAT: Catalysing the Growth in Metal Recovery
  Andrew Hunt, The University of York
- **2:30 - 2:50 PM** - Durability Assessment and Recycling Strategies for Rare Earth Magnets
  Annett Gebert, IFW Dresden
- **3 PM** - BREAK
- **3:30 - 3:50 PM** - Current Research and Development Rare Earth Elements in Canada
  In-Ho Jung, McGill University
- **4:00 - 4:20 PM** - Material Efficient Design for Sustainable Energy Applications
  Jürgen Eckert, IFW Dresden
- **4:30 - 4:50 PM** - Atom-Probe Tomography and the Science of New Classes of High-Temperature Al-Sc-RE Based Alloys
  David Seidman, Northwestern University

## 4. The 3rd International rare metals workshop program

### RARE EARTH ELEMENT INDUSTRY OVERVIEW



### MINERALOGY AND BENEFICIATION OF RARE EARTH RESOURCES



### HYDROMETALLURGY OF RARE EARTH MINERAL CONCENTRATES



### RARE EARTH ELEMENT SEPARATION



### RECYCLING OF RARE EARTH ELEMENTS



### RARE EARTH ELEMENTS IN ADVANCED MATERIALS

5. Bilateral international workshop program organized by KIRAM



PROGRAM

| 시간 | | 프로그램 | 발표자 |
|---|---|---|---|
| 09:30-10:00 | | 등록 및 자료 배포 | |
| 10:00-10:10 | | 개회 및 축사 | |
| 10:10-10:40 | Plenary Lecture | 다양한 분쇄 및 물리적 분리 기술을 이용한 E-스크랩으로부터의 희소금속 추출 | 와세다 대학교 소우지 오와다 교수 |
| 10:40-11:10 | | 용융 마그네슘을 이용한 폐 Nd-Fe-B 자석으로부터 Nd의 선택적 추출 | 한국생산기술연구원 김택수 박사 |
| 11:10-11:20 | | Coffee break | |
| 11:20-11:40 | 세션 I (제품) | 폐 LCD의 재활용 공정 개발 | 고등기술연구원 홍현선 박사 |
| 11:40-12:00 | | 자동차에서의 희토류 재활용 전략 | ㈜오토에코 원종수 대표이사 |
| 12:00-13:00 | | 중 식 | |
| 13:00-13:20 | 세션 II (부품) | 폐 탄탈륨 콘덴서로부터 정제된 산화 탄탈륨의 회수 | 동북 대학교 이츠수로 시바타 교수 |
| 13:20-13:40 | | 폐 리튬이온 배터리로부터 귀금속을 회수하기 위한 건식공정 | 산업기술종합연구소 시게키 코야나가 박사 |
| 13:40-14:00 | | $FeCl_3$용액을 이용한 폐 무연솔더로부터 Sn, Cu, Ag의 회수 | 한국해양대학교 유경근 교수 |
| 14:00-14:20 | | 희소금속이 함유된 인쇄 회로 기판의 분쇄 방법 | 와세다 대학교 치하루 토코로 박사 |
| 14:20-14:40 | | Coffee break | |
| 14:40-15:00 | 세션 III (공정) | 폐촉매로부터의 유가금속 회수 | 한국생산기술연구원 김홍대 박사 |
| 15:00-15:20 | | 폐전자기기로부터 귀금속 재활용 | 한국지질자원연구원 이재천 박사 |
| 15:20-15:40 | | 백금 그룹 금속 재활용을 위한 친환경 공정 개발 | 동경 대학교 카츄시로 노세 박사 |
| 15:40-16:00 | | 습식 순환공정을 이용한 전략 금속 회수 | 산업기술종합연구소 미키야 타나카 박사 |
| 16:00-16:30 | | 폐회 및 맺음말 | |



Program

프로그램

| 시간 | | 프로그램 | 비고 |
|---|---|---|---|
| 09:30 ~ 09:50 | | 등록 및 자료 배포 | |
| 09:50 ~ 10:00 | | 개회 및 축사 | |
| 10:00 ~ 10:30 | 세션 I | 한국과 아시아의 희소금속 정책현황과 KITECH 소개 및 연구개발활동 | 한국생산기술연구원 김택수 센터장 |
| 10:30 ~ 11:00 | | 1) Rare metal policy and strategy in Europe 2) Introduction of IFW Dresden | IFW Dresden Dr. Jürgen Eckert Director |
| 11:00 ~ 11:20 | | 한국 합금철 산업 및 R&D 동향 | 연세대학교 민동준 교수 |
| 11:20 ~ 11:40 | 세션 II | 희성금속(주) 사업 현황 및 방향 | 희성금속(주) 윤원규 상무이사 |
| 11:40 ~ 12:40 | | 중 식(제공) | |
| 12:40 ~ 13:00 | 세션 II | Material Efficient Design for Sutainable Energy Applications | IFW Dresden Dr. Sergio Scudino |
| 13:00 ~ 13:20 | 세션 III | 희소금속 리싸이클링 현황 및 사례 | 고등기술연구원 홍현선 센터장 |
| 13:20 ~ 13:40 | | Stability and recyclability of rare earth magnets | IFW Dresden Dr. Annett Gebert |
| 13:40 ~ 14:00 | | 희소금속류의 생물학적 회수 | 한국생산기술연구원 김상용 박사 |
| 14:00 ~ 14:20 | | Coffee break | |
| 14:20 ~ 14:40 | 세션 IV | Superconducting materials for energy and magnet applications with reduce rare metal content | IFW Dresden Dr. Kazumasa Iida |
| 14:40 ~ 15:00 | | 희토류 영구자석에서 중희토류 저감 기술 | 재료 연구소 이정구 박사 |
| 15:00 ~ 15:20 | | The role of rare metals for magnetocaloric cooling | IFW Dresden Dr. Anja Waske |
| 15:20 ~ 15:30 | | 폐회 및 맺음말 | |




모시는 글

희소금속은 첨단산업의 경쟁력을 뒷받침하는 산업의 비타민으로, 정부는 국가 주력산업의 글로벌 경쟁력 및 취약한 희소금속 산업의 경쟁력을 높이고자 희소금속산업 발전 종합계획을 수립하고 범국가적 육성 정책을 추진해 오고 있습니다.

이에 한국생산기술연구원 희소금속산업기술센터에서는 희소금속 자원 강국인 중국과 함께 최신 기술 동향 및 발전 전략을 살펴보는 「**제4회 국제 워크샵(韓-中)**」을 개최합니다. 이와 더불어 센터 내에 입주한 희소금속 전문기업들을 챔피언 기업으로 육성하겠다는 의지와 비전을 담아 「**한국희소금속산업기술센터 입주기업(챔스기업) 출범식**」을 함께 진행하고자 합니다.

희소금속 산업 분야의 한·중 네트워크를 강화하고, 국내 희소금속 관련 기업인들의 의욕을 고취할 목적으로 마련한 자리인 만큼 모쪼록 참석하시어 행사를 빛내주시고 힘을 실어주시기 바랍니다.

2013년 4월

한국생산기술연구원장 나경환

프로그램

## WORKSHOP

워크샵

| Time | Program | Speaker |
|---|---|---|
| 09:00~10:00 | 등록 및 자료 배포 | |
| 10:00~10:10 | 인사 말씀 | 김택수 본부장 (한국생산기술연구원) |
| 10:10~10:30 | 중국의 희토류 산업 및 정책<br>Situation and Development Trend of China's Rare Earth Industry | Zhao ZengQi 부총공정사 (보강그룹) |
| 10:30~10:50 | 한중 과학기술협력 동향과 전망<br>The Trend and Future of Korea-China S&T Cooperation | 이춘근 센터장 (한중과학기술 협력센터) |
| 10:50~11:00 | Coffee Break | |
| 11:00~11:20 | 수소저장 전극 재료의 연구 현황<br>Research Status of Hydrogen Storage Electrodes Materials | Ma ZhiHong 부원장 (희토연구원) |
| 11:20~11:40 | 희토류 형광체 대체 및 저감<br>Replacement and Reduction of REE Phosphors | 김범성 박사 (한국생산기술연구원) |
| 11:40~12:00 | 국내 모나자이트광에서 희토류 회수 기술<br>Rare Earths Recovery from Korean Monazite Ore | 이진영 박사 (한국지질자원연구원) |
| 12:00~13:00 | Luncheon (미추홀타워 21F, 피에스타) | |
| 13:00~13:20 | 희토 분리추출 기술<br>Ionic Liquids-based Rare Earth Green Separation and Clean Technique | Chen Ji 교수 (장춘응용화학연구원) |
| 13:20~13:40 | 미세조직 제어에 의한 Dy-free Nd계 영구자석 소재 개발<br>Development of High-performance Dy-free Nd-Fe-B Base Permanent Magnets By Micro-structural Control | 이정구 박사 (재료연구소) |
| 13:40~14:00 | 다기능 발광 나노 재료의 특성 제어 및 생의학적 적용<br>Multifunctional Luminescent Nanomaterials: Controlled Fabrication, Properties and Biomedical Applications | Lin Jun 교수 (장춘응용화학연구원) |
| 14:00~14:20 | 한국의 형광체와 희토류 원소<br>Phosphor and Heavy REE in Korea | 김정곤 교수 (인천대학교) |
| 14:20~14:30 | 출범식장 이동 | |

# 6. Scientific papers

APPLIED PHYSICS LETTERS **101**, 124103 (2012)

## Effect of tungsten metal particle sizes on the solubility of molten alloy melt: Experimental observation of Gibbs-Thomson effect in nanocomposites

M. H. Lee,[1,a)] J. Das,[2] D. J. Sordelet,[3] J. Eckert,[4] and A. J. Hurd[5]
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[3]*Caterpillar Advanced Materials Technology Group, Mossville, Illinois 61552, USA*
[4]*IFW Dresden, Institute for Complex Materials, P.O. Box 27 01 16, D-01171 Dresden, Germany*
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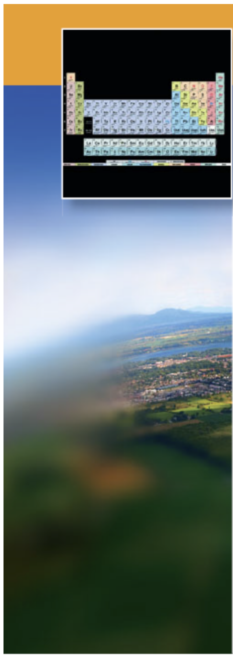
We investigated the effect of tungsten particle sizes on the thermal stability and reactivity of uniformly dispersed W particles in molten Hf-based alloy melt at elevated temperature (1673 K). The solubility of particles less than 100 nm in radius is significantly enhanced. In case of fine W particles with 20 nm diameter, their solubility increases remarkably around 700% compared to that of coarse micrometer-scale particles. The mechanisms and kinetics of this dynamic growth of particle are discussed as well as techniques developed to obtain frozen microstructure of particle-reinforced composites by rapid solidification. 
[http://dx.doi.org/10.1063/1.4754546]

Tungsten and tungsten alloys are the common refractory materials showing superior properties such as high density, hardness, very high melting temperature, relatively high radiation opacity, and good thermal conductivity combined with very low thermal expansion.[1] One of the important applications of tungsten metals is machining tools, in carbide phase, due to high rigidity.[2] Moreover, ultrafine-grained (UFG) particles in molten metallic melts at elevated temperature (1673 K).

$Hf_{44.5}Cu_{27}Ni_{13.5}Nb_5Al_{10}$ (at. %) metallic alloy ingots, prepared by arc melting under Ar atmosphere, and W powders (>99.9% purity, Perkin Elmer/Metco, SPI12430, Osram) of 10 vol. % were used as starting materials. For this study, the diameters of W particles were selected to be under





## Energy-critical elements for sustainable development

Alan J. Hurd, Ronald L. Kelley, Roderick G. Eggert, and Min-Ha Lee

Energy-critical elements (ECEs) are chemical and isotopic species that are required for emerging sustainable energy sources and that might encounter supply disruptions. An oft-cited example is the rare-earth element neodymium used in high-strength magnets, but elements other than rare earths, for example, helium, are also considered ECEs. The relationships among abundance, markets, and geopolitics that constrain supply are at least as complex as the electronic and nuclear attributes that make ECEs valuable. In an effort to ensure supply for renewable-energy technologies, science decision makers are formulating policies to mitigate supply risk, sometimes without full view of the complexity of important factors, such as unanticipated market responses to policy, society's needs for these elements in the course of basic research, and a lack of substitutes for utterly unique physical properties. This article places ECEs in historical context, highlights relevant market factors, and reviews policy recommendations made by various studies and governments. Actions taken by the United States and other countries are also described. Although availability and scarcity are related, many ECEs are relatively common yet their supply is at risk. Sustainable development requires informed action and cooperation between governments, industries, and researchers.

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**SOW 1 Title:** International Cooperation to Development of Strategy and R &D Collaboration for Substitution of Rare Earth Resources

**SOW 2 Title:** Development of Magnetocaloric Alloys without Critical Elements

## 1. Background

The La-Fe-Co-Si compounds with an NaZn13-type structure (hereafter referred to as 1:13) are one of the most promising candidates for magnetic refrigeration applications, due to their giant magnetocaloric effect (MCE) combined and minimal amount of critical materials. The MCE properties of the La-Fe-Co-Si system are directly dependent on the volume fraction of the 1:13; however, this phase is difficult to obtain, due to the incompleteness of the peritectic reaction between La and Fe during cooling. In order to obtain the 1:13 single phase, the cast ingots must be annealed under vacuum at high temperature for 10-50 days. This complicated processing scheme, raises the need to develop an efficient preparation method for these alloys. Laser engineering net shaping (LENS) is a novel method to synthesize various materials directly from powders. It is an alternative and efficient route for preparing desired phases with a significantly reduced fabrication time. With this motivation a series of the magnetocaloric $La(Fe,Co,Si)_{13}$ compounds have been prepared through the LENS process. The effects of different experimental conditions on formation behavior of the 1:13 phases in La-Fe-Co-Si alloys are investigated. The phase, microstructure and magnetic properties of these samples have been characterized.

## 2. Experimental

The La $(Fe,Co,Si)_{13}$ master alloys were prepared by arc melting in an argon (Ar) atmosphere. The composition of these alloys is determined with a lower Si and a higher Co amount for the applications nearby room temperature, as shown in table 1. The as-cast samples were crushed and sieved to a size fraction of 45-150 µm for the LENS printing.

**Table. 1. Compositions and element weight percentages of starting powders.**

| Sample No. | Composition | x(La)/wt.% | x(Fe)/wt.% | x(Co)/wt.% | x(Si)/wt.% |
|---|---|---|---|---|---|
| Powder A | $LaFe_{11.05}Co_{0.90}Si_{1.05}$ | 16.56 | 73.57 | 6.39 | 3.48 |
| Powder B | $LaFe_{10.70}Co_{1.30}Si_{1.00}$ | 16.49 | 71.01 | 9.17 | 3.33 |

The powders were fed into a melt pool created by a Yb laser (1 kW) to build rod shape geometries. The printing was done in a low oxygen (< 5 ppm) Ar environment inside a glove box. Printed rods were annealed at 1,050˚C for 15 days under inert conditions, followed by water quenching. The microstructure was observed in backscattered electron (BSE) mode by SEM. The crystal structure was studied using X-ray diffraction (XRD) with Cu Kα irradiation. Finally, magnetic measurements were performed with maximum magnetic field ranged from 1-5 T.

## 3. Results and Discussion

### 3.1. Preparation of La-Fe-Co-Si compounds by LENS method

The initial work was focused on determining if LENS could be used to synthesize La-Fe-Co-Si alloys, and, if the composition and properties of these alloys could be tailored. Fig. 1 shows the XRD patterns of the initial powders crushed from the master alloys (Table. 1.). For both powders, the phases can be indexed as α-Fe with a small amount of LaFeSi phase.

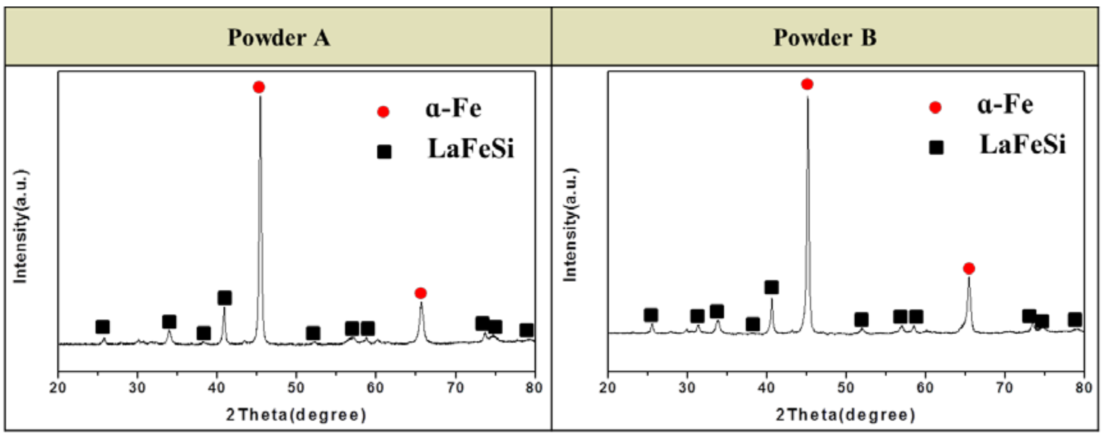


Fig. 1. XRD patterns of initial powders

Samples were prepared by LENS by in situ mixing powder A and B in different ratios. All the samples were fabricated in a rod shapes. The composition of the rods varied from A: $LaFe_{11.05}Co_{0.90}Si_{1.05}$ to B: $LaFe_{10.70}Co_{1.30}Si_{1.00}$. By analyzing the X-ray diffraction patterns of the different rods, it can be found that the small amount of the 1:13 phase was formed after LENS process. In general, the 1:13 phase appears to be extremely difficult to obtain with low Si amount and without annealing. However, this result indicates that the LENS process does promote the direct formation of the 1:13 phase during solidification.

| Sample No. | Powder A | Powder B |
|---|---|---|
| #1 | 4.0 | 0 |
| #2 | 3.5 | 0.5 |
| #3 | 4.2 | 0.8 |
| #4 | 3.6 | 1.4 |
| #5 | 3.0 | 2.0 |
| #6 | 2.0 | 3.0 |
| #7 | 1.2 | 3.8 |
| #8 | 1.0 | 4.0 |
| #9 | 0.8 | 4.2 |
| #10 | 0 | 5.0 |

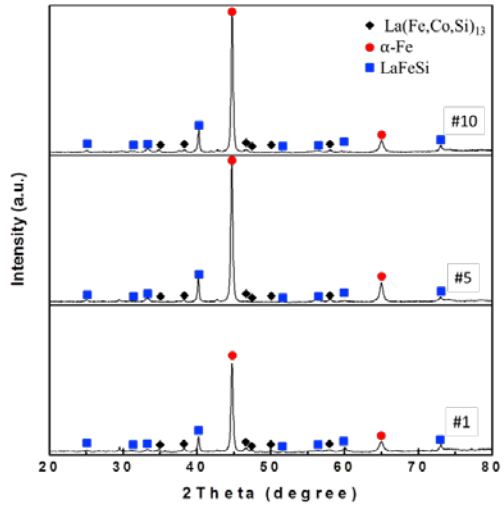


Fig. 2. Experimental conditions and XRD patterns of the LENS samples

### 3.2. Annealing Effects

As discussed above, extended annealing times is the generally used to obtain a high volume fraction of the 1:13 phase in the La-Fe-Co-Si cast alloys. We utilized annealing for the as printed LENS samples as well as the initial master alloy from which the powders were made from. The annealing of the LENS samples (shown in section 3.1) was performed at 1050˚C for 10 days, however, there was no obvious phase changes observed compared to the as printed samples. For further investigation, the annealing of the master alloy at 1050˚C for 15 days was also performed. Fig. 3 shows the XRD patterns of the as-cast sample, powder annealed at 1050˚C for 15 day and LENS sample fabricated from annealed powder. For the annealed powder, a significant amount of the 1:13 phase is formed, but it almost all disappears after the LENS printing process. These results indicate that the annealing is less effective method for the formation of the 1:13 phase of La-Fe-Co-Si prepared by LENS process. The reasons for this need to be investigated further.

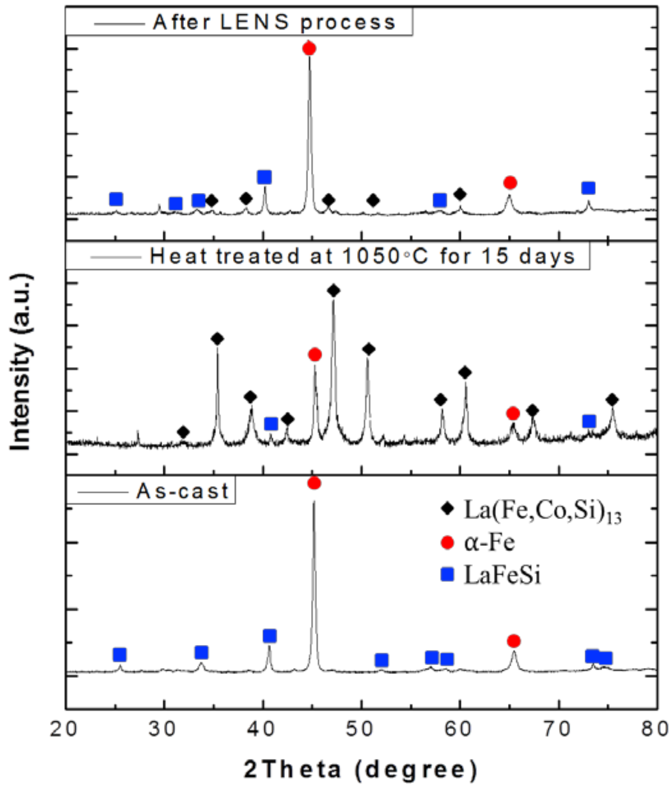


Fig. 3. XRD patterns of La-Fe-Co-Si samples prepared by LENS process.

### 3.3. Direct Synthesis from La-Si, Fe and Co powders

The results discussed above have focused on the LENS samples that were prepared from La-Fe-Co-Si master alloy powders. However, our results have shown that these conditions were not effective for forming a significant amount of the 1:13 phase. Therefore, we utilized a direct synthesis of the La-Fe-Co-Si compounds using La-Si, α-Fe and α-Co powders. Initially, the $La_{45}Si_{55}$ alloy powder was prepared to prevent segregation and to make it easy to form the 1:13 phase. Then the La-Fe-Co-Si compound was directly synthesized from these powders by LENS printing. Interestingly, we found that a large amount of 1:13 phase was identified with significantly reduced α-Fe and LaFeSi phase, as shown in Fig. 4. Up until now; there have been no literature reports on the 1:13 phase that can be directly obtained from powders without preparation of a master alloy and any annealing process as well.

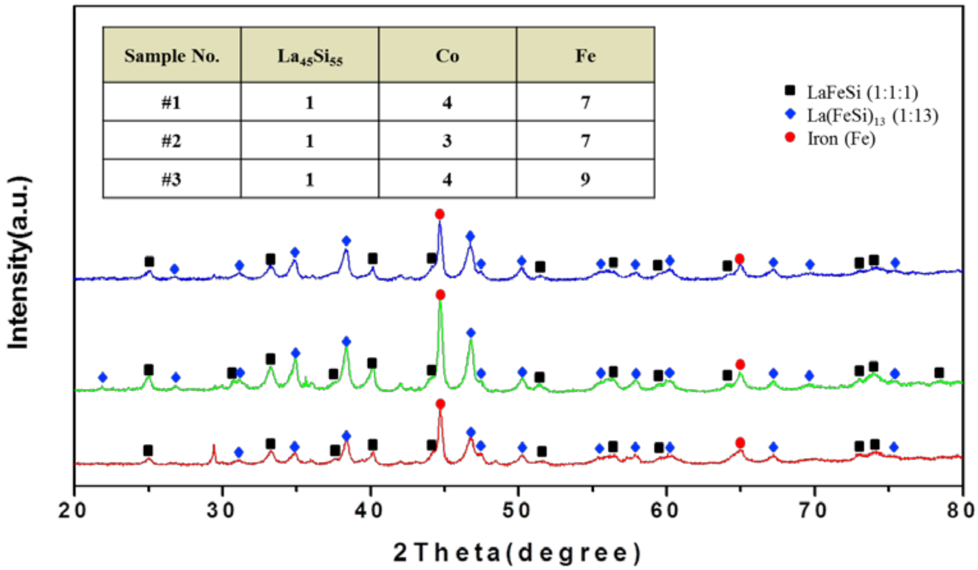


Fig. 4. XRD patterns of the LENS samples directly synthesized method.

In order to better understand the microstructure, the backscattering electron (BSE) mode is taken for analysis. It can be seen from Fig. 5. that there exists three phases for the LENS compound. According to the result of the EDS analysis of the LENS sample, shown in table 2, the composition of the three phases are very close to 1:13, α-Fe and LaFeSi, respectively. It can be seen that a large amount of the 1:13 phase is homogeneously distributed in the sample.

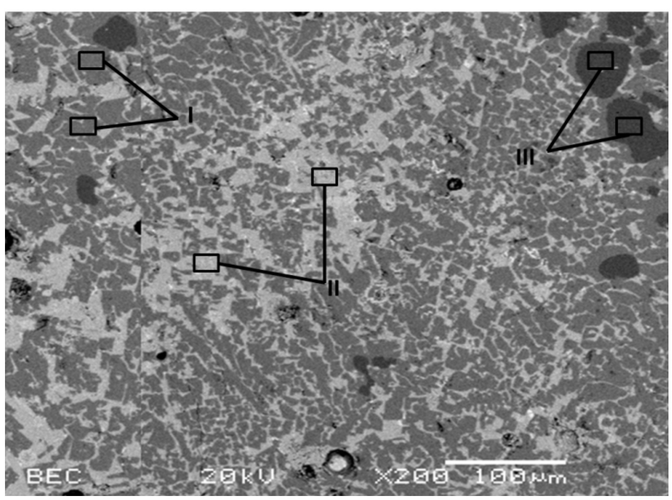


Fig. 5. BSE image of the LENS sample

Table. 2. EDS analysis of different positions in Fig. x.

| Position | x(La)/% | x(Fe)/% | x(Co)/% | x(Si)/% | Phase |
|---|---|---|---|---|---|
| I | 68.31 | 13.43 | 12.32 | 5.94 | 1:13 phase |
| II | 55.74 | 14.31 | 17.06 | 12.89 | LaFeSi |
| III | 0.39 | 87.73 | 9.94 | 1.95 | α-Fe |

### 3.5. Magnetocaloric Properties

The magnetocaloric properties of the LENS sample measured, as shown in fig. 6. The $T_c$ of the LENS sample was around 325 K, which is above room temperature. The maximum magnetic entropy change of the LENS sample was around 2.5J/kgK. at 0-5T. This value is lower than other reported results; however, as seen from the XRD and SEM, the phase fraction of the 1:13 phase is not optimized in the as printed rods. It important to note, that the LENS method does present the possibility of synthesizing magnetocaloric materials by a simple process in a relatively short time. Post printing heat treatment can perhaps be utilized to further improve the magnetocaloric performance of the alloys.

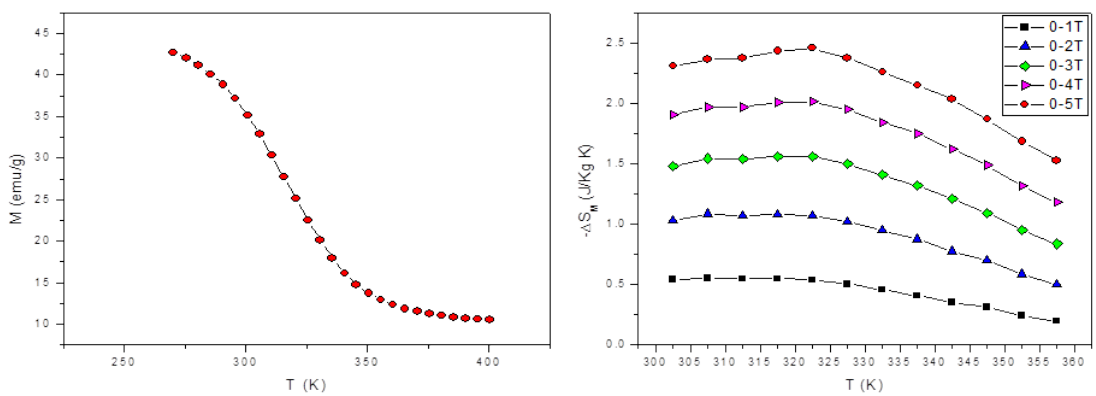


Fig. 6. Temperature dependent magnetization, M and ΔS for the LENS samples printed from the individual powders of La-Si, Fe and Co.

### 4. Summary

Laser Engineered Net Shaping (LENS) has been used to print magnetocaloric alloys based on the LaCoFeSi system into rod geometries. Samples were printed using both pre-alloyed powders of the final LaCoFeSi composition along with individual powders of La-Si, Fe and Co. The samples printed from the pre-alloyed powders showed no significant formation of the 1:13 phase, which only appeared after post-printing annealing. For the samples printed from the individual powders, the 1:13 phase could be formed in the as-printed structure. Measurements of the magnetocaloric properties are lower than those of fully annealed samples, however, the formation of the 1:13 during printing does open up the possibility of printing complicated geometries for which the performance can be improved with further heat treatments.