



*Mini review*

## **Recent advances in acid-free dissolution and separation of rare earth elements from the magnet waste**

**Grace Inman, Denis Prodius and Ikenna C. Nlebedim\***

Ames Laboratory, US Department of Energy and Critical Materials Institute, Ames, Iowa 50011-3020, USA

\* **Correspondence:** Email: [nlebedim@ameslab.gov](mailto:nlebedim@ameslab.gov); Tel: 5152948916.

**Abstract:** The availability of REEs is limiting the successful deployment of some environmentally friendly and energy-efficient technologies. In 2019, the U.S. generated more than 15.25 billion pounds of e-waste. Only ~15% of it was handled, leaving ~13 billion pounds of e-waste as potential pollutants. Of the 15% collected, the lack of robust technology limited REE recovery for re-use. Key factors that drive the recycling of permanent magnets based on rare earth elements (REEs) and the results of our research on magnet recycling will be discussed, with emphasis on neodymium and samarium-based rare earth permanent magnets.

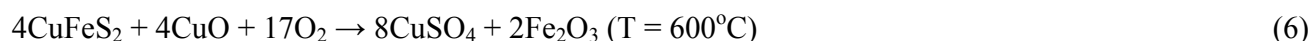
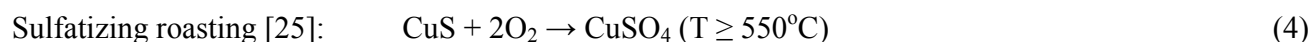
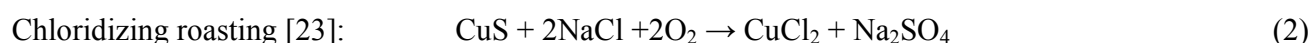
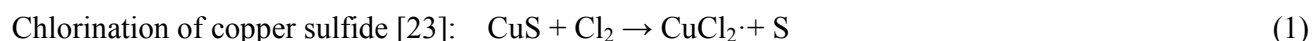
**Keywords:** acid-free dissolution; rare earth recovery; magnet waste recycling

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### **1. Introduction**

The unique physical and chemical properties of rare earth elements (REEs) drive their increasing demands in electronics, health care, aerospace, transportation, and defense applications. Technologies that produce substantially lower amounts of carbon dioxide emissions such as wind power and electric vehicles depend critically on neodymium and dysprosium for powerful magnets. The U.S. Department of Energy and the European Commission have considered REEs as critical materials, due to their importance in the clean energy economy and the possibility of disruption in their supplies [1–3]. Approaching large-scale deployment of the above-mentioned technologies will increase the demands for neodymium and dysprosium [4,5]. It is projected that the demand for dysprosium and neodymium alone could increase by a factor of 62–72 in the next 10 years [6]. The addition of expensive dysprosium

permits the use of Nd-Fe-B magnets to higher temperatures, therefore some amounts of critical materials are required to increase the performance of magnets. Nevertheless, the natural source for dysprosium (clays), currently mined mainly in southern China [7], is geographically unfavorable to many countries, including the USA. Moreover, the existing recycling rate of REE is about one percent, which is tremendously small and unsatisfactory [8–10]. A technological challenge is the low concentration of REEs in end-of-life devices and associated materials, which makes the extraction process complicated and cost-intensive [11]. Another fundamental challenge in the field is the similarity in the chemical properties of REEs, which makes their separation as individual elements difficult [12–16]. In general, the REE-containing materials can be recycled by direct reuse, reprocessing the materials before reuse, or via recovery of the chemical elements. The chemical recovery methods for REEs generally include either pyrometallurgical (PMG) or hydrometallurgical (HMG) methodologies [17–22] or a combination of them. In the PMG route, the REEs recovery efficiency is reduced by slag formation, due to the high affinity of the REEs with oxygen. PMG processes are energy-intensive and generate large amounts of solid wastes, although they can have the benefit of recovering the REEs in the form of metals, instead of oxides. HMG approaches allow better recovery efficiency of REEs, especially as oxides or in other non-metallic forms. However, most HMG methods need substantial amounts of hazardous chemicals, especially strong mineral acids such as sulfuric, nitric, and hydrochloric acids. Large amounts of wastes, including residual strong mineral acids, present recognizable environmental problems. Investments to contain both the acids and their contaminated wastes add to the cost of the HMG processes. Therefore, there is an obvious need for a cost-effective, environment-friendly and energy-efficient method for recycling REEs-containing materials. Aqueous solution of copper(II) salts can selectively dissolve (oxidize) magnetic alloy and efficiently transfer the relevant metals into the solution. Such an approach helps to avoid strong mineral acid use, hence eliminating the associated harsh reaction conditions. The recycling process allows for the copper content of the copper(II) salts to be recovered and reinserted into the value chain. Alternatively, copper salts can be prepared without any use of mineral acids as demonstrated by some selected examples below (Eqs 1–7):

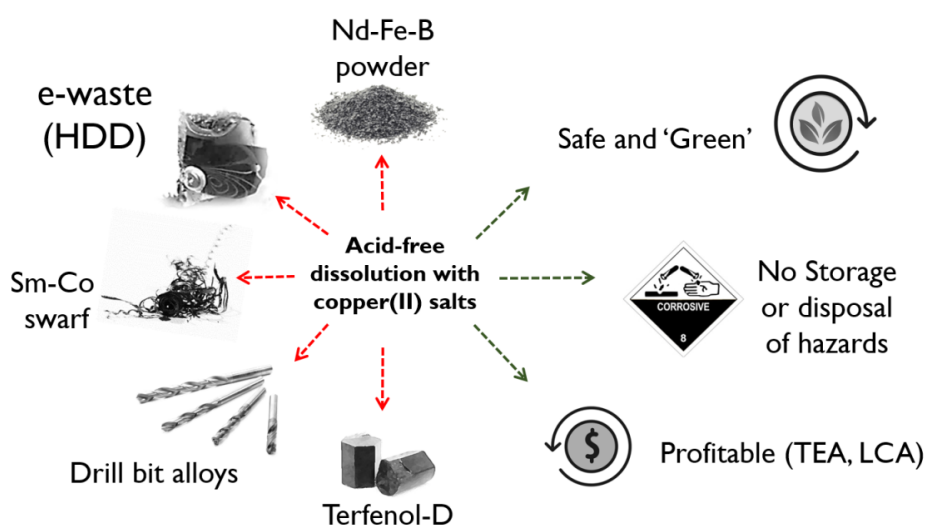


From copper via anhydrous copper(II) nitrate [26]:



Interestingly, a significant amount of global copper supply is obtained with the aid of microorganisms via bioleaching [27–29]. The recycling process by using copper(II) salts was developed

considering the application of green chemistry principles (acid-free dissolution of magnets and some alloys through the redox-dissolution) (Figure 1, Table 1). It was also developed because of potential commercial adoption such that the recovered REEs and other recycling co-products are suitable for reinsertion into the supply chain. Moreover, to maximize profit in the application, the process development included recovery and reinsertion of some of the chemicals back into the recycling process. The chemical dissolution method comprises contacting the REEs-containing material and an aqueous solution of a copper(II) salt to dissolve magnet materials. The REEs are then precipitated from the aqueous solution, which then can be calcined to produce REE-oxides or other REE compounds. This process also enables selective leaching of the REEs contained in magnet swarfs and waste magnets contained in e-waste products such as shredded hard disk drives (HDDs), decrepitated alloys, and crushed electric motors. Swarfs are typically oxidized metal powders generated from the post-manufacturing processing (cutting, grinding, etc.) and are normally contaminated by grinding media (including lubricants).

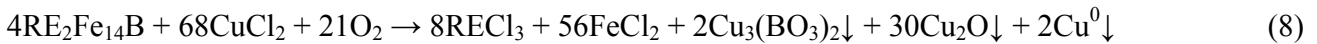


**Figure 1.** General overview of acid-free dissolution approach. TEA = techno-economic analysis; LCA = Life-cycle analysis; HDD = hard disk drives; Terfenol-D = magnetostrictive alloy  $Tb_xDy_{1-x}Fe_2$  ( $x \approx 0.3$ ).

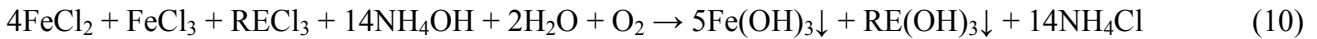
## 2. Discussion

**Materials and Experimental Method:** The recycling feedstock materials described in this work are (a) Nd-Fe-B and Sm-Co grinding swarfs from U.S. magnet plants, and (b) Nd-Fe-B contained in crushed electric motors [30]. All chemicals for recycling were purchased from commercial sources (Sigma-Aldrich, ACS reagent grade) and were used without further purifications. The acid-free leaching solutions were prepared by straightforward dissolution of Cu(II) salts in water.

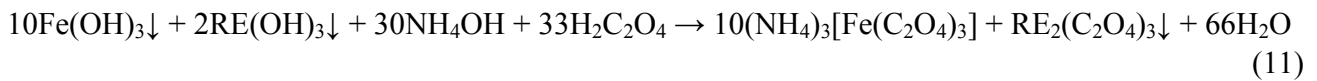
Complete oxidative dissolution at room temperature was accomplished for (neodymium, praseodymium)-iron-boron (RE-Fe-B) magnets according to Eqs 8 and 9 with copper(II) chloride salt as an example:



For recovering REE from the filtrate, a two-step approach was applied to eliminate the presence of iron in the final REE oxides. In the first step, the filtrate was stirred together with an aqueous ammonia solution at 60°C for 2 hrs which resulted in the precipitation of Fe(III) and RE(III) hydroxides (Eq 10):



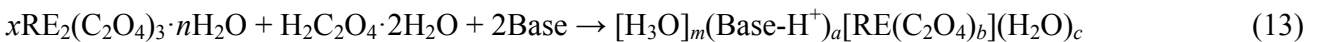
Then, a slight excess of solid oxalic acid was added, followed by heating to 80°C and stirring until insoluble REE oxalate precipitated and highly soluble double salt of iron-ammonium oxalate formed (Eq 11):



The REE oxalates were separated by filtration and washing in hot water, and were calcined in air at 800°C to obtain RE<sub>2</sub>O<sub>3</sub> (>98% yield; Eq 12):



Nd-Fe-B magnets usually contain Nd and Pr (a mixture of both elements is frequently named didymium). Dysprosium (Dy), which is considered even more critical, is added at lower a concentration for the higher temperature grades. Extraction of such a low concentration of Dy from a mixture of REEs is a challenging problem, from both technical and economic perspectives, even with some established separation procedures [41–48] (Table 2). For complex REE oxalates, an additional one-step separation process, which comprises water-based leaching of dysprosium with organic base/oxalic acid mixture, can be applied (Eq 13) [39]:



where  $x = 0.01 \div 1.6$ ;  $n = 10$  (RE = La to Er) and  $6$  (RE = Er to Lu);  $m = 0$  or  $1$ ;  $a = 1, 3, 4, 5, 8$ ;  $b = 2, 3, 4, 7$ ;  $c = 1, 1.5, 2$  and  $10$ ; Base = 1-methylimidazole, 1-ethylimidazole, N-methylpyrrolidine.

So this straightforward and environmentally benign chemical separation of heavy rare earth element (Dy) results in efficient extraction (>68%) even at low initial concentrations of metal (<5% of Dy) from processed magnet wastes. Recently, Schelter et al. reported the separation of neodymium and dysprosium by selective precipitation in benzene, diethyl ether (Et<sub>2</sub>O), or aqueous HCl of complexes with tripodal nitroxide ligand [46–48].

**Table 1.** Some selected hydrometallurgical approaches for leaching rare earth metals from magnets.

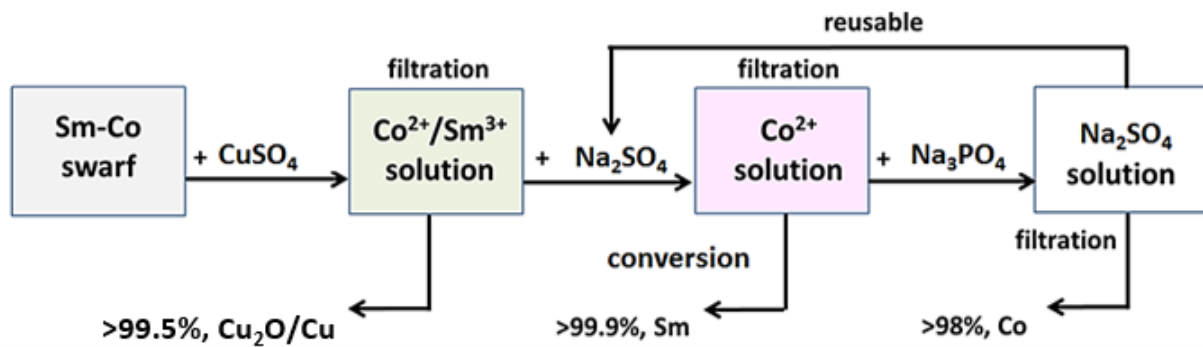
Year	Leaching reactants	Processes	Results	Reference
2015	H <sub>2</sub> SO <sub>4</sub>	Disk and ball-milling; roasting; thermodynamic stability and solubility of products of the considered metal sulfates (REE, Fe).	12–16 M acid solutions; drying at 110°C for 6–24 hrs; selective roasting at 650–850°C for 15–120 min; 98 wt% purity of REE product; SO <sub>2</sub> /SO <sub>3</sub> gas is a by-product.	[31]
2016	HCl	Heat treatment (1 hr, 450°C) followed by room temperature processing; dissolution for bonded and non-bonded NdFeB magnets; leaching time ~ 4 hrs.	Selective dissolution of NdFeB magnet; generates acidic waste; a recovery rate of >80%.	[32]
2016	CH <sub>3</sub> COOH	Pulverization; sieving; leaching with dilute acetic acid; leaching of shrinking-sphere model.	Extraction of Nd and Fe are quantitative using dilute CH <sub>3</sub> COOH (0.4 M) solution; leaching time of ~ 4 hrs, leaching kinetics study.	[33]
2017	HCl	Pulverization; corrosion in 3% NaCl (~ one week); additional chemicals used (NH <sub>4</sub> Cl, Cyphos® IL101); leaching time: 1–2 hrs.	Closed-loop hydrochloric acid (HCl)-based process; triple extraction; about 30% of B separation.	[34]
2017	HCl, H <sub>2</sub> SO <sub>4</sub>	Crushing and grinding; solvent extraction with NaCyanex302; pH = 0.5–1.2; leaching with HCl.	Separation of Dy vs. Nd; Stripping efficiency of loaded organic followed the order: HCl<HNO <sub>3</sub> <H <sub>2</sub> SO <sub>4</sub> .	[35]
2018	HNO <sub>3</sub>	Demagnetization; crushing; Hollow Fibre Membrane (HFM) operation in non-dispersive solvent extraction (NDSX) mode.	Dy separated from NdFeB magnetic scrap with >97% purity and 94% recovery; Separation behavior of Nd, Dy, and Pr under different hydrodynamic conditions.	[36]
2019	Organic acids	Hydrogen decrepitated starting material; roasting, sieving; solvent extraction (TBP, D2EHPA, Cyanex 272, and 923).	95% extraction at 70°C for REEs for ascorbic acid case; glycolic and maleic acids were more efficient as lixivants (time ≥ 6.5 hrs) than ascorbic acid; results are comparable to mineral acids leaching activity.	[37]
2020	Cu salts	Acid-free dissolution of NdFeB (swarf, e-waste) and Sm-Co magnets through the redox reactions at RT; partial separation of oxidized materials (iron oxides); leaching of magnets (≥4 hrs).	Selective acid-free leaching of rare-earth elements from magnet-containing electronic wastes, such as end-of-life (EoL) hard disk drives and electric motors; it excludes some energy-consuming steps.	[38]
2020	CuCl <sub>2</sub>	Acid-free dissolution of NdFeB magnet from the motor; chemical separation of rare earth oxalates within two subgroups (light REEs vs. heavy REEs).	Selective dissolution and separation of RE oxalates into an aqueous phase; successful extraction of dysprosium (extraction efficiency > 68%) from the low Dy-containing material.	[39]
2020	[PyH]Cl	Crushing (hydraulic press), ball-milling, solvent extraction (Cyanex 923, D2EHPA, and PC-88A).	Leaching in pure pyridinium chloride at 165°C avoided the consumption of other solvents; Nd and Dy were extracted at different concentrations of PC-88A.	[40]

**Table 2.** Some selected hydrometallurgical approaches for the separation of individual rare earth metals (Dy/Nd).

Year	Extractants	Solvents/Mineral acids used	Processes/Results	Separation factor	Reference
1957	HDEHP	Toluene-H <sub>2</sub> O/HCl, H <sub>2</sub> SO <sub>4</sub>	Bis-(2-ethylhexyl) phosphoric acid: industrially applied process.	41.5 (Dy/Nd)	[41,42]
1995	Cyanex 302	Heptane-H <sub>2</sub> O/HNO <sub>3</sub>	Bis-(2,4,4-trimethylpentyl)monothiophosphinic acid: Solvent extraction of Ln(III) (except for Pm) was studied at various aqueous pH values, extractant concentrations, and different temperatures (°C).	239.3 (Dy/Nd)	[43]
2015	TriNOx	THF-Et <sub>2</sub> O-Arenes-CHCl <sub>3</sub> -CH <sub>2</sub> Cl <sub>2</sub> /No acid	Rare-earth-metal coordination compounds with a tripodal nitroxide ligand (TriNOx <sup>3-</sup> ) undergo a self-association (dimerization) equilibrium based on cation size, which enables the separation of light and heavy rare earth metals.	359 (Dy/Nd)	[46–48]
2016	Ionic Liquid [MAIL]Tf <sub>2</sub> N [MAIL]Tf <sub>2</sub> N	[MAIL]Tf <sub>2</sub> N- [P <sub>666(14)</sub> ]Tf <sub>2</sub> N- H <sub>2</sub> O/HCl	A method for extracting a rare earth metal from a mixture of one or more rare earth metals in acidic aqueous solution with help of hydrophobic ionic liquid (phosphonium-based) and designed imidazolium-based ionic liquid ligand (MAIL).	1085 (Dy/Nd)	[20]
2020	Cyanex 572/TODGA	Isopar L (normal alkanes, isoalkanes, and cycloalkanes)/HNO <sub>3</sub>	The recovery and separation of rare earth elements (REEs) using supported membrane solvent extraction are provided. The immobilized organic phase includes a solvent and an extractant. The organic phase can include an isoparaffinic hydrocarbon solvent and a phosphorous-based chelating extractant and with a ratio by volume of between 1:1 and 3:1 or any other combination. The feed solution can include a pH maintained between 0 and 2.0, and optionally between 1.0 and 1.5.	400 (Dy/Nd)	[44]
2020	Organic base/ oxalic acid	H <sub>2</sub> O/No acid	The approach (CSEREOX) allows for selective solubilization of water-insoluble oxalates of rare earth elements even at low initial concentrations (<5%, Dy) from processed magnet wastes.	38 (Dy/Nd)	[39]
2020	Cyanex 923	PEG 200-alkanes-1-decanol/HCl, LiCl	Cyanex 923 is a commercial mixture of trialkyl phosphine oxides, with C <sub>6</sub> and C <sub>8</sub> chains. It has the advantage over trioctylphosphine oxide (TOPO) that it is a liquid at room temperature, and it is a stronger extractant than tri-n-butyl phosphate (TBP). It was demonstrated that nonaqueous solvent extraction can be integrated in conventional hydrometallurgical flow sheets to provide a sustainable process for the separation of Nd and Dy.	69 (Dy/Nd)	[45]

Despite outstanding separation factors in Table 2, some of these approaches were not considered yet as commercially viable. Some research groups (Nockemann, Binnemans) successfully applied ionic liquids [49] intending to enhance the separation selectivity of rare earth metals [50]. However, there is still a need in creating an environmentally friendly and commercially viable alternative for the standard liquid-liquid extraction (solvent extraction) of rare earth metals.

Samarium-cobalt (Sm-Co) magnets excel the high-performance Nd-Fe-B magnets when high temperature (>150°C) applications are required. The acid-free leaching process was adapted to Sm-Co swarfs with some specific modifications depending on the nature of copper salt (sulfate) (Figure 2).

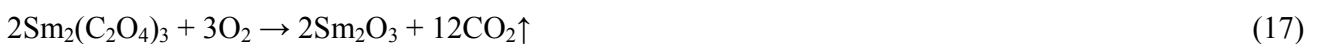
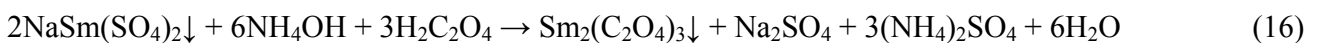


**Figure 2.** The overall strategy for recycling samarium and cobalt from Sm-Co swarf.

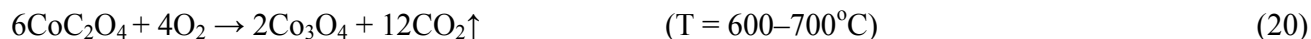
Most of the known recycling processes for Sm-Co magnets use an excess of hazardous sulfuric acid for leaching (a) and shifting of the solubility equilibrium (b) to the target insoluble (double salt) product [51]. The mechanism of redox dissolution with copper(II) sulfate is similar (Eq 14) to that of Nd-Fe-B:



The recovery of  $\text{Sm}_2\text{O}_3$  from this solution requires precipitation of double salt  $\text{NaSm}(\text{SO}_4)_2$  (Eq 15), conversion to samarium(III) oxalate (Eq 16) and calcination at  $800^\circ\text{C}$  with  $\text{Sm}_2\text{O}_3$  as a final product (Eq 17):

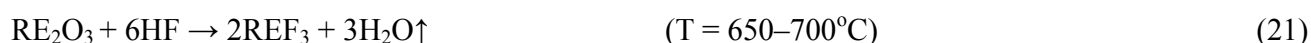


Cobalt, like the REEs, has been identified as a critical material. Successful application of the recycling process to Sm-Co magnets indicates the potential for waste Sm-Co alloys to be sources of significant amounts of cobalt for secondary supplies. The recovery of cobalt can be done in the form of  $\text{Co}_3(\text{PO}_4)_2$  (used as an inorganic pigment) (Eq 18) or cobalt(II, III) oxide (Eqs 19, 20; used for cathode material manufacturing):

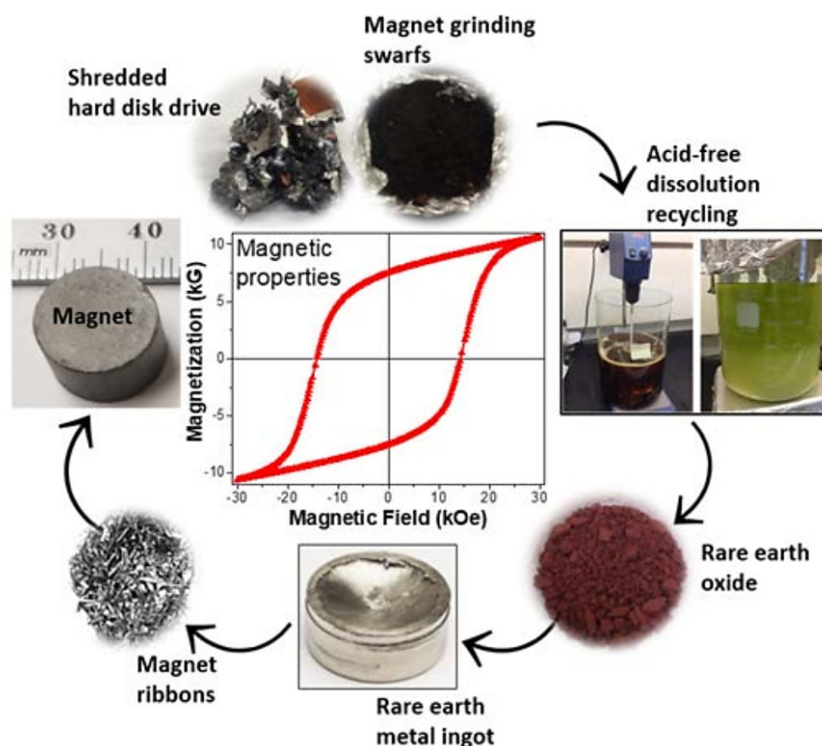
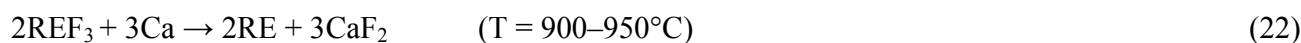


Another advantage here is reusable by-products ( $\text{Na}_2\text{SO}_4$  and copper oxides) which is important because it minimizes materials (processing) costs and being fully in line with some of the 12 principles of green chemistry (atom economy, use of renewable feedstock, and reduce derivatives) [52].

For making magnets using recovered REE oxide, the oxides were first reduced to RE metal ingot using the established Ames process [53] (Eqs 21, 22):



The oxides were converted to fluorides at  $700^\circ\text{C}$  under a flow of anhydrous HF and argon. The anhydrous fluorides were then reduced with high purity calcium in tantalum crucibles as described in (Eq 22):



**Figure 3.** The suitability for reinsertion of the recycled REEs into the magnet supply chain. Reprinted with permission from *ACS Sustainable Chem Eng* 2020, 8: 1455–1463 [38]. Copyright 2020 American Chemical Society.



Part of the obtained ingot was alloyed in an argon atmosphere with 99.9% pure Fe and Cu, and 99.5% pure boron via arc melting to produce a magnet feed-stock with composition  $(\text{Nd-Pr})_{2.3}\text{Fe}_{14}\text{B} + 0.5 \text{ wt.}\%$  Cu. The recycled REEs were used to make magnets as previously described in [38] (Figure 3) to exhibit the suitability for reinsertion into the magnet supply chain.

In conclusion, the development of efficient and safe recycling technology can help to minimize the consequences of supply disruptions. Recycling of REEs from magnet waste is one approach for addressing REEs materials criticality problems and outcomes in products suitable to be reinserted into the rare earth elements supply chain.

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## Conflict of interest

All authors declare no conflict of interest in this paper.

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