

EXTRACTING OF RARE EARTH METALS FROM SECONDARY RAW MATERIALS

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A review of new publications on rare earth extraction technologies from various types of secondary raw materials: permanent magnet scrap made of $Nd_2Fe_{14}B$ alloy and nickel-metal hydride batteries, spent catalysts, electronics scrap, waste luminophores and scrap fluorescent lamps.

INTRODUCTION

In the group of rare metals, a subgroup of rare earth metals (REM) consists of 17 elements of the III group of the periodic system: scandium, yttrium, lanthanum and a family of 14 lanthanides from cerium (serial number 58) to lutetium (71).

The last decades have shown a significant increase in the production and consumption of rare earth products. Today more than 80% of the total volume of REM produced comes in three areas of use: permanent magnets based on *Sm-Co* and *Nd-Fe-B* systems, nickel-metal hydride (*NiMH* type) batteries (where *M* - *La*, *Nd* and other REM) and phosphorus fluorescent lamps [1].

The structure of consumption of REM is generally fairly stable, however, in recent years, the use of rare earths in the production of magnets has increased - up to 23%. As for individual REMs, so according to the materials of the academic paper [2], cerium (about 45 thousand tons), lanthanum (about 30 thousand tons), neodymium (about 20 thousand tons) and yttrium (12 thousand tons) are most widely used. The share of these metals accounts for 87...88% of the world's consumption of REM. Consideration of demand for REM in the coordinate system of the global economy makes it possible to distinguish three main areas of their use, which in the coming years will be characteristic development trends. These are «clean» technologies (so-called «green energy»), commodities of everyday use ("goods for life") and military-industrial complex (weapons and defense means). It is believed that the demand for REM in the short and long term will be supported by the development of markets based on clean technologies such as the production of electric vehicles and wind turbines. According to the forecast, in 2020 the world consumption of REM will increase to 180 thousand tons at an annual growth rate of $\approx 5.7\%$. At the same time, the volume of consumption of REM by China by 2020 will be about 120 thousand tons, the production of REM outside China will be no more than 65 thousand tons [2].

SECONDARY RAW REM

Due to the high demand for REM and their high cost, lately, increased attention has been paid to the use of secondary raw materials to produce these metals. First of all, these are REM-containing waste products

and materials, such as permanent magnets, *NiMH* accumulator batteries, phosphors, catalysts. The problem of extracting REM from various types of secondary raw materials by the authors of this paper was devoted to several papers [3–8]. This report reviews recent (2016–2017 years) works on this issue, which are not discussed in previously published articles.

The production cycles of the REM industry include numerous phases, each of which is characterized by its complex market dynamics: ore mining, retrieval of REM oxides, their purification, oxide reduction to metals, the inclusion of metals and alloys into components, and the production of final products. Thus, the development of the REM industry involves a wide range of issues from extraction of ores and the use of secondary raw materials to obtain clean and especially clean individual rare earth elements and their applications in the defense and civilian sectors.

Only 1% of the used rare earth metals are returned to the category of secondary raw materials of REM due to their strong scattering and difficulty in collecting waste. In this regard, in [9], the need to increase the efficiency of the technologies of extraction of REM from secondary raw materials is emphasized.

In the analysis of reverse logistics and resource allocation in the REM industry, [10] introduced a reverse logistics scheme in accordance with the REM recycling procedure. The key point in this network was the choice of equipment and the design of a reversible logistic level to reduce pollution and damage the natural environment. The optimal use of rare earth metals should increase the development of reverse logistics in the REM industry and support the benefits of recycling rare earth metals.

WASTE MAGNETIC ALLOYS

Creating a new class of permanent magnets from alloys of lanthanides with cobalt, iron and boron was an outstanding event of the late 60's (1966) and 80's (1984) of the twentieth century. Samarium-cobalt magnets (*SmCo₅* alloy) surpass other common magnetic materials by 2–4 times the magnitude of the magnetic energy per unit volume and 5–10 times by the magnitude of the coercive force. An even greater magnetic field of energy Alloys are produced on the basis of the neodymium-iron-boron (*Nd₂Fe₁₄B* alloy).

Questions for improving the technology of *Nd-Fe-B* magnets production and recommendations for its optimization are considered in [11]. The issues of recycling of rare earth metals in the processing of production and consumption waste of permanent magnets and reduction of its value are also considered there. It is recommended in the processing of waste to separate the collection of light REM (neodymium, praseodymium) and heavy (dysprosium, terbium).

The technology of the smelting of alloys of the *REM-Fe-Co-B* system for thermally stable magnets, which ensures the stability of the chemical composition (deviations in the content of REM ± 1.0 , cobalt and boron by $\pm 0.5\%$ by weight of the calculated and purity by impurities: aluminum and nickel < 0.2 wt% of each, oxygen < 0.1 wt%), developed in [12]. This technology allows both *Pr-Dy-Fe-Co-B* systems to be obtained as well as more complex compositions including additional REMs, for example gadolinium.

In the first stages of the production of permanent magnets based on the *Nd-Fe-B* alloy, a portion of the powder material falls into the waste. In the course of studies [13], it was found that magnetic separation could be used to clean powdered waste from fractions contaminated with neodymium oxide above a permissible norm. Samples of permanent magnets, which showed higher magnetic parameters (residual induction, coercive force), were made from the powders purified by this method, than those obtained by other methods of purification.

An article by Japanese scientists [14] devoted to the processing of permanent magnets of the composition *Nd₂Fe₁₄B* containing additives *Pr* and *Dy*, presents the latest advances in the technologies of extracting REM and their rational use. Japan exports all rare earth metals from China, so recycling from waste is a topical task for it. Recycling of REM is carried out by *Hitachi*, *Mitsubishi Materials* and *Showa Denko KK*.

Thermodynamic simulation and experimental study of the *B₂O₃-FeO-Fe₂O₃-Nd₂O₃* system have been carried out in [15]. The thermodynamic calculations of this system have made it possible to optimize the selective process of oxidation of neodymium in the processing of magnesium waste and thereby increase its extraction.

The main technology for the processing of permanent magnets scrap on the basis of the *Nd-Fe-B* system is hydrometallurgy, which involves the leaching of waste. Thus, in [16], it was shown that under optimal leaching conditions of the scrap, iron, boron, neodymium and 85.87% cobalt are completely removed. Upon subsequent electrolysis of the solution, high-purity metals, including neodymium, were obtained.

In [17], the scrap of permanent magnets, containing, %: 64.37 *Fe*; 30.57 *Nd*; 0.42 *Pr* and 0.71 *B* were leached with a solution of hydrochloric acid. The optimum mode for maximum extraction of neodymium (99.9%) and iron (61.36%) was: 0.2 M *HCl*, 90 °C, duration 4 hours, material thickness 76-105 micron, agitation 800 rpm, pulp density 1%. From the solution, iron was precipitated by adding *Ca(OH)₂* to pH=4.0. After removal of the iron, 0.8 M *NaD₂EDPA* was extracted with liquid extraction followed by re-extraction to obtain a solution containing 72 g/l of

neodymium and 1.088 g/l of praseodymium. As a result of crystallization, the salts of *Nd₂(SO₄)₃* and *Pr₂(SO₄)₃* were precipitated.

In the processing of waste from the production of permanent magnets for extraction from solutions after leaching of dysprosium and neodymium, two extractants, D2ЭГФК and PC88A, were used in [18]. The paper proposes optimal modes of extraction and re-extraction of dysprosium and neodymium for their industrial regeneration.

As shown in [19], the extraction of neodymium and praseodymium from chloride solutions after the leaching of *Nd-Fe-B* magnets in bifunctional ionic fluids *R₄NCy* and *R₄ND* is more efficient than in conventional extragens. At optimum regimes, extraction of neodymium was 98.79% and praseodymium was 99.02%. The re-extraction was conducted with sulfuric acid.

Often, before leaching the scrap of magnets, oxidizing firing is carried out. Thus, in [20] the method of 95...100% extraction of *Nd*, *Pr*, *Dy* and *Gd* with the transfer of iron to hematite and goethite is developed. Initially, the waste is subjected to low temperature (200 °C) firing, and then water leaching and cleaning of the solution from impurities. In [21], oxidative calcination was carried out before hydrochloric acid leaching of scrap. At burning, the degree of iron oxidation to *Fe³⁺* reaches a maximum at 700 °C for 1.5 hours. The degree of leaching of neodymium was 99.33% at pH=3.5 for 2 hours, the residual iron content in the solution was 0.00014 g/l.

Fluorination and electrolysis are the basis of the technology for the processing of *Nd-Fe-B* magnets waste, presented in [22]. During fluorination of the scrap using the fluxes of *AlF₃*, *ZnF₂*, *FeF₃* and *Na₃AlF₆*, neodymium and dysprosium are converted to *LiF*-based melt in the form of fluorides, while iron and boron remain in elemental state. When electrolysis of REM is dissolved in the anode and deposited on the cathode.

A method for processing sanding waste produced during the production process REM-containing magnets are proposed in the patent [23]. The shaving waste is mixed with concentrated (not less than 92%) sulfuric acid in the amount needed to obtain a solid agglomerated product. Subsequently, the solid-phase sulphation process is carried out at a temperature of 200...250 °C, followed by firing at a temperature of 650...850 °C. Further, leaching in percolation columns of sulphates of REM and other metals with acidified water and the release of REM from productive solutions by precipitation. The solid leach residue (cup) is a practically pure trivalent iron oxide. At the same time it is achieved 94...98% recovery of the amount of REM in the solution with a minimum transition of iron and other impurities into the solution in the form of sulfates.

OTHER WASTE WAYS

Nickel-metal hydride, along with lithium ions, relate to a new class of rechargeable batteries. Used *NiMH* rechargeable batteries, except nickel and cobalt, contain *La*, *Sm*, *Nd*, *Pr*, *Ce*, so the scrap of such batteries is considered as a secondary raw material of REM.

The optimal leaching mode of *Ni-MH* scrap batteries is given in [24]: 2 M H_2SO_4 , 348 K, 120 min, a pulp density of 100 g/l. Extract of REM to the solution was: 98.1 *Nd*, 98.4 *Sm*, 95.5 *Pr*, 89.4 *Ce*. At the same time, heavy metals (*Ni*, *Co*, *Mn*, *Zn*) dissolved more than 90%.

Electrode materials of the AB_5 -type *Ni-MH* batteries can be refined to produce nickel-cobalt alloy and slag enriched with REM-oxides. In [25], the melt was conducted at a temperature of 800...900 °C with the addition of H_2/H_2O . At the same time, REM, aluminum and manganese were oxidized, and more inert nickel and cobalt passed into elemental state. The second remelting of electrode materials was carried out at 1550 °C with the addition of SiO_2 and Al_2O_3 fluxes and *Ni-Co* alloy and slag of $REM-SiO_2-Al_2O_3-MnO$ were obtained. Nickel-cobalt alloy can be used to produce an AB_5 -alloy for storage of hydrogen, and individual rare earth metals are extracted from slag containing up to 48.5% REM-oxides.

One of the types of secondary raw materials of REM are *spent catalysts*. The multi-stage technological scheme for the extraction of cerium from spent iron-potassium catalysts of dehydrogenation of olefin hydrocarbons proposed in the patent [26] is completed by crystallization of cerium dioxide at an extract of the solution for 3...12 hours at temperatures up to minus 20 °C. The separated precipitate is separated from the mother liquor by decantation and filtration, washed on a filter from iron compounds and dried to a constant mass.

Bio-leaching can be used to extract REM from waste oil catalysts. In [27] *Gluconobacter oxydans* strain was used for this purpose, while the recovery of REM was only 49%, but it was shown that it increases with leaching with the addition of gluconic acid in a quantity up to 90 mmol/l.

Especially increased demand for REM due to the rapid development of electronics. In electronic devices of rare earth metals lanthanum is most often used. In [28], for the recovery of lanthanum from *electronics scrap*, he was dissolved in acid and sorption of lanthanum was carried out with ordinary clay or organochlorine (polyamine clay). It has been shown that clays have a cation-exchange property, sorbing metals (in this case, lanthanum and copper).

The most widely used luminophores are used in fluorescent lamps – the most economical sources of light, in color and black and white television, as well as for the production of glowing paints (luminescent compositions based on yttrium, europium, terbium and other lanthanides). In particular, chlorination technology is used for the treatment of *luminophores waste*. Patent [29] proposes a method of producing yttrium and europium enriched concentrate in the processing of zinc sulfide phosphorous waste. The method involves drying the raw material at a temperature of 150...320 °C for 2...3 hours and subsequent chlorination at a temperature of 800...900 °C in a melt of chloride salts of yttrium and europium at a mass ratio of yttrium chloride to chloride europium 1:(0.1...0.3).

The production of individual REM oxides (yttrium and europium) from luminophores waste by methods of chlorination of the raw material at the stage of its

opening and extraction with oleic acid at the stage of separation of yttrium and europium was considered in [30]. Investigations of the extraction process performed on model solutions are confirmed by tests on technological solutions containing 125 g/l of the sum of lanthanides (96% yttrium and 4% europium) and up to 10 g/l zinc. The purity of the oxides obtained was 99.91% europium and 99.90% yttrium. Experimentally it has been shown that oleic acid has several advantages over other extractives. In addition to high values of the coefficients of separation of the pair of europium/yttrium (not lower than 4.0), it is characterized by a significant capacity (from 12 to 26 g/l, the sum of yttrium and europium in terms of oxides – depending on the concentration of the extractant in the diluent) and low cost.

Pre-treatment of waste fluorescence powder can affect the degree of recovery of them by REM with subsequent leaching. Thus, it was shown in [31] that the powder exposure for 2 hours at a temperature of 1050 °C in a melt of *NaOH* at ratio a *NaOH*: a powder of 2.5:1 allows an increase in the extraction of REM to more than 98%.

The *fluorescent lamp* scrap can serve as a secondary raw material for the extraction of *Eu*, *Y*, *Ce*, *Tb*, *Gd*, *La*. Their average content in scrap is estimated at 8,4 g/kg, and the mercury content is (0.7±0.1) g/kg. At the processing of scrap fluorescent lamps on an industrial scale, the technology of extraction is used. Reagent *Cyanex 572* dissolved in kerosene was used in [32] for liquid extraction of yttrium and europium to produce oxides of purity of 99.82% Y_2O_3 and 91,6% Eu_2O_3 . An optimum regimen for separating REM was set to pH=0 for the separation of yttrium, followed by the release of europium at pH=1. Increasing the temperature from 25 to 65 °C resulted in a deterioration of the separation of metals. Re-extraction was carried out with hydrochloric acid.

Extracting REM from *phosphorus lamp* waste through reductive alkaline smelting followed by acid leaching was considered in [33]. An iron powder was used as a reducing agent, the ratio of alkali to phosphorous mass was 2:1, temperature 700 °C, duration 3 hours. The leaching was carried out by hydrochloric acid at a concentration of 3 mol/l at of L:S=7.5:1 at a temperature of 70 °C during 1 h. The total extraction of REM reached 99.35%, and for cerium, europium, terbium and yttrium, respectively, 98.12; 99.93; 99.97, and 99.60%.

In conclusion, we note that the proposed technologies have not yet received due economic justification. Most of them are inherent factors constraining their implementation, including not only economic, but also environmental.

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ИЗВЛЕЧЕНИЕ РЕДКОЗЕМЕЛЬНЫХ МЕТАЛЛОВ ИЗ ВТОРИЧНОГО СЫРЬЯ

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Дан обзор новых публикаций, посвященных технологиям извлечения редкоземельных металлов (РЗМ) из различных видов вторичного сырья: скрапа постоянных магнитов, изготовленных из сплава $Nd_2Fe_{14}B$, и никель-металлгидридных аккумуляторных батарей; отработанных катализаторов; скрапа электроники; отходов люминофоров и скрапа флуоресцентных ламп.

ВИЛУЧЕННЯ РІДКОЗЕМЕЛЬНИХ МЕТАЛІВ ІЗ ВТОРИННОЇ СИРОВИНИ

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Дано огляд нових публікацій, що присвячені технологіям вилучення рідкоземельних металів (РЗМ) із різноманітних видів вторинної сировини: скрапу постійних магнітів, виготовлених із сплаву $Nd_2Fe_{14}B$, і нікель-металгідридних акумуляторних батарей; відпрацьованих катализаторів; скрапу електроніки; відходів люмінофорів і скрапу флуоресцентних ламп.