Influence of Dysprosium Compounds on the Extraction Behavior of Dy from Nd-Dy-Fe-B Magnet Using Liquid Magnesium

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Abstract: During the liquid metal extraction reaction between a Nd-Dy-Fe-B magnet and liquid Mg, Nd rapidly diffuses out of the magnet, whereas Dy is not extracted due to the reaction with the matrix and the formation of Dy2Fe17 phase. In addition, the Dy2O3 phase exists at the grain boundaries. Until now, only the effect of the Dy2O3 phase on the extraction of Dy has been reported. In this study, the effect of the Dy2Fe17 phase on the extraction of Dy from the Nd-Dy-Fe-B magnet was investigated in liquid Mg. The formation of the Dy2Fe17 phase during the reaction between Mg and matrix (RE2Fe14B) was first examined using a thermodynamical approach and confirmed by microstructural analysis. It was observed that Dy extraction was dominated by Dy2Fe17 phase decomposition from 3 h to 24 h, followed by Dy2O3 phase dominant reaction with Mg. Comparing the activities of the Dy2Fe17 phase and the Dy2O3 phase, the reaction of Dy2Fe17 is dominant, as compared to the Dy2O3 phase. Finally, at 48 h, the high Dy extraction percentage of 93% was achieved. As a result, it was concluded that the Dy2Fe17 phase acts as an obstacle in the extraction of Dy. In the future, if research to control the Dy2Fe17 phase proceeds, it will be of great importance to advance the recycling of Dy.

Keywords: recycling; pyrometallurgy; dysprosium; liquid metal extraction; phase transformation

1. Introduction

Rare earth (RE) permanent magnets have recently been employed in high-tech industrial applications, such as electric vehicles, renewable energy, robotics, and their utilization is increasing as the world shifts towards a green economy [1,2]. Even though there are various types of magnets, such as ferrite, AlNiCo, etc., the reason for the domination of the permanent market by the RE magnets is due to their superior magnetic properties, such as high remanent magnetization and coercivity [3]. The rare-earth sublattice (4f electrons) comprises essential components to stabilize the magnetization direction for the crystal axes, i.e., high magnetic anisotropy.

Recently, the focus on carbon neutrality has led to a sharp increase in the consumption of permanent magnets to achieve net-zero carbon dioxide emission. Among the RE permanent magnets, the Nd-Dy-Fe-B magnets are widely used because of their relatively low cost and high productivity, compared to the SmCo magnets. The Nd-Dy-Fe-B magnets are generally known to be composed of about 30 wt.% REEs contents. Moreover, Dy is integrated into the magnets to enhance their thermal stability and corrosion resistance [4]. Thus, the demand for Dy has been also gradually increasing with the development of green technology. However, the production of heavy REEs (HREEs) has undergone serious balance problems related to political, geological, and technical issues as the HREEs like...
Dy are only produced from the ion-adsorbed ores in southern China [5]. In the case of ion-adsorbed ores, the REEs are absorbed on the clay, which is formed with the hydrated cations. As opposed to other rare earth ore, such as the bastnaesite, monazite, xenotime, and so on, a large number of HREEs can be effectively obtained without the emission of radioactive substances by using the chemicals. However, the usage of acids and base solutions is a cause of environmental concerns.

Therefore, diversifying the supply of HREEs is significant to improve the supply chain for sustainability. For recycling, the ways to recover end of life (EOL) magnets or magnet scraps are suggested. During the magnet fabrication process, the amount of magnet scrap can be generated to about 20% to 30% [6]. Besides, the EOL magnets can be accumulated, depending on the applications, such as small electronics, electric vehicles, and wind turbines with various shapes and sizes.

One of the recycling processes for recovering HREEs from EOL magnets or scraps, the pyrometallurgy method, is a potential alternative to collect REEs from Nd-Dy-Fe-B magnet [7–9]. Among these processes, liquid metal extraction (LME) is based on a selective reaction with target metals by using solvent metals, such as Mg, Ag, Bi, and Cu [10–13]. It has the advantage of being an environmentally friendly chemical-free process without the emission of wastes and no requirement of additional reduction processes due to the direct recovery of REEs in metal form.

The Mg is a strong candidate for being an extraction agent in the LME process. It can be selectively reacted with REEs (Nd, Dy) without Fe and B intermetallic compounds due to higher chemical affinity with REEs (Nd, Dy) compared to with Fe [13]. Previous studies have shown that Nd is easily extracted from the Nd-Dy-Fe-B magnet and the reaction mechanism is successfully demonstrated [10,14–16]. On the other hand, the low extraction efficiency of Dy was reported because of the small amount of Dy present in the Nd-Dy-Fe-B magnet and quite a different reaction behavior with Mg. Akahori et al. thermodynamically demonstrated that the oxidation of Dy can be affected by the decreasing extraction by forming Dy2O3 and not DyNdO3. It is shown that preventing the oxidized phases is decisive in improving extraction efficiency [16]. To understand the Dy extraction mechanism, Kim et al. investigated the development of (Nd and Dy)-oxide phases in the microstructures as a result of formation during the process. They offer experimental evidence that the limited extraction of Dy is caused by the formation of Dy oxide [17]. Park et al. conducted a comparison of the extraction efficiency with increasing oxide composition. It is shown that Dy was not easily extracted in the form of Dy-oxide and Dy2Fe17, while Nd was completely extracted [18]. Even though the scrap was heavily oxidized due to the small scrap size, the Dy2Fe17 phase remained as the result of the decomposition of REFeB grain. It is supported by the experimental results of Nam et al., that the phase transformation of direct reaction between DyFeB and Mg phenomenologically shows that Dy2Fe17 is first formed in the pure DyFeB phase as a byproduct, while liquid Mg is infiltrated into the grain [19]. However, the reasoning behind the influence of the Dy2Fe17 phase in the extraction process is still unclear.

In this work, the entire extraction behavior of Dy is systematically investigated, depending on the time, activity with Mg, intermediate phase, and oxides, infiltrating Mg into the Nd-Dy-Fe-B specimen. The generation and decomposition of intermediate phases are observed in detail and the Dy2Fe17 phase is clearly identified. This demonstrates that the interplay between Dy2Fe17 and oxides affects the extraction behavior of Dy.

2. Experimental

The permanent magnets for the liquid metal extraction (LME) process were supplied by Jahwa Electronics Co. Ltd., Cheongju, Korea. Pure Mg was purchased from JC Magnesium Co., Burnaby, BC, Canada. The chemical composition of the magnet is shown in Table 1 and were determined by X-ray fluorescence (XRF; Thermo Fisher Science ARL PERFORM’X, Middlesex County, MA, USA). The preparation size of the magnet sample and pure Mg is 10 × 10 × 3 mm and 10 × 10 × 10 mm, respectively. To confirm the reaction
behavior, the magnets were placed at the bottom of a mild steel crucible with Mg on top. The crucible was then placed inside a high-frequency induction furnace and heated in an atmosphere-controlled chamber for a reaction time between 30 min and 48 h. The Mg to magnet mass ratio was 15 to 1 and LME reactions were observed at 900 °C. This has been known, as an ideal condition, to maximize the extraction ratio [20]. For analyzing the characteristics of the magnet obtained by furnace cooling following the change in periods at 900 °C, the reaction sample was cut into properly sized samples using a diamond wheel cutter with a thickness of 0.3 mm. The specimens were ground using abrasive papers, which were scaled from 200 to 4000 grit SiC. Subsequently, the samples were polished using a 0.1 µm Al₂O₃ suspension. After polishing, the specimens were cleaned in an ultrasonic cleaner for 5 min by steeping them in ethanol. Then, the specimens were dried using a high-pressure air spray gun. The microstructure of the samples was characterized using an FE-SEM (JSM-5310, JEOL, Tokyo, Japan) and a transmission electron microscope (TEM; JEM-F200, JEOL, Tokyo, Japan). The thickness of the diffusion layer in the magnet was measured using the BSE mode in FE-SEM and the concentrations of the REE (Nd, Dy) and Mg were investigated using XRF and EDS analyses in both the diffusion layer and Mg-zone.

Table 1. Chemical composition of the magnet by XRF.

| Elements | Fe (wt.%) | Nd (wt.%) | Dy (wt.%) | Minor (wt.%) |
|----------|-----------|-----------|-----------|--------------|
| Magnet   | 69.0      | 25.8      | 3.50      | 1.72         |

3. Results and Discussion

Table 1 shows the chemical composition of the magnet which is the well-controlled type of oxygen content at 800 ppm. The minor components are Cu, Al, Co, Nb, etc.

Considering the magnet composition, a 100% extracted ratio of REE into Mg can be calculated to be 1.730% in Nd and 0.233% in Dy, respectively. Table 2 shows the characterization of the extracted concentration of Nd and Dy with increasing reaction time by XRF measurement. Figure 1 reveals converted values with the extraction efficiency by using Equations (1) and (2). While all of Nd is completely reacted at 6 h, the extraction of Dy is 72% at the same reaction time.

\[
\text{Percentage of extraction Nd (\%)} = \left( \frac{C_{\text{Nd in Mg}}}{1.730} \times 100 \right) \quad (1)
\]

\[
\text{Percentage of extraction of Dy (\%)} = \left( \frac{C_{\text{Dy in Mg}}}{0.233} \times 100 \right) \quad (2)
\]

Table 2. The concentration of elements in the Mg region by XRF.

| Time (Hours) | Concentration (wt.%) | C_{\text{Nd}} | C_{\text{Dy}} |
|--------------|---------------------|---------------|---------------|
| 0.5          | 0.902               | 0.041         |
| 1            | 1.08                | 0.063         |
| 3            | 1.48                | 0.117         |
| 6            | 1.73                | 0.155         |
| 12           | 1.73                | 0.158         |
| 24           | 1.73                | 0.193         |
| 48           | 1.73                | 0.219         |
Figure 1. Variation of Nd and Dy extraction efficiency with increasing time.

To understand the extraction behavior of Dy in detail, the microstructures are observed with increasing reaction time on the magnet side. Figure 2 shows that liquid Mg diffused inside the magnet along the grain boundaries and formed ligaments (dark regions) around the magnet (grey region), forming a reaction zone inside the magnet. In morphology, the pattern of Mg infiltration is developed as the changing size and the number of Mg zone with increasing reaction time. Because the Mg can be infiltrated in the entire magnet area in just 30 min, the REEs in the magnet can be totally extracted by the strengthened Mg ligament networking. The liquid Mg diffuses into a magnet to form a diffusion layer on the surface which, in turn, alters the chemical composition of the surface layer. This change in the composition of the surface layer decreases the surface energy and melting point of the solid, allowing it to transform into a liquid state by melting.

The liquid Mg first reacts with RE-rich and RE-oxide phases through the grain boundary in magnets as the diffusion path. Due to the low melting point in the RE-rich phase, which is only 600 °C, the initial extraction curves of Nd and Dy are rapidly increased in Figure 1 [21]. The high reaction temperature of 900 °C promotes the liquid-liquid reaction between RE-rich and Mg. On the other hand, the liquid–solid reaction arises from the high melting points, which are 2230 °C in Nd$_2$O$_3$ and 2408 °C in Dy$_2$O$_3$. Thus, the oxides remained with small particles in the microstructures due to the relatively slow reactivity. Approximately 1 µm-sized white particles, which are identified as the RE oxide phase, were observed, as shown in Figure 2a–g.

With increasing reaction time, it is observed that the Mg infiltration is gradually increased and the distribution of oxides is decreased. This means that the oxides can be decomposed despite the slow reactivity with Mg. Considering the total extraction time of Nd at 6 h, the remained oxides indicate Dy$_2$O$_3$ after 6 h. It was thermodynamically demonstrated that the Nd$_2$O$_3$ reacts faster with Mg when comparing Gibbs free energy of Nd$_2$O$_3$ and Dy$_2$O$_3$ [16].

Figure 3 shows the change in the matrix (RE$_2$Fe$_{14}$B) with increasing reaction time. At 30 min, the co-existence between needle-shaped particles and the oxide phase is observed in the matrix. The reaction with the particles in the matrix complies with the tendency of oxides in the grain boundary. To identify the phase of needle-shaped particles and the oxide phase is observed in the matrix. The reaction with the particles in the matrix complies with the tendency of oxides in the grain boundary. To identify the phase of needle-shaped particles, the XRD and the STEM-EDS experiments are conducted. The XRD analysis is conducted on the reacted specimen for 3 h. The phases are defined with Fe, Fe$_2$B, Mg, RE$_2$O$_3$, Mg$_{12}$RE, and RE$_2$Fe$_{17}$ in Figure 4. Because of the gathering of all of the phase information about both grain boundary and the matrix, the Dy oxide phases and RE$_2$O$_3$ are discovered in the grain boundary, while the remaining phases in the matrix are defined to be Fe, Fe$_2$B, Mg, and Mg$_{12}$RE. Interestingly, the needle-shaped particles are characterized with the
RE-Fe intermetallic phases using phase elimination. Because of the chemical similarity, the RE$_2$Fe$_{17}$ phase cannot be clearly determined to be Nd$_2$Fe$_{17}$ or Dy$_2$Fe$_{17}$, even though the Nd is known to generate only the Nd-Mg-based intermetallic phase, and the formation of Nd$_2$Fe$_{17}$ is not reported [14].

**Figure 2.** The microstructure of the reaction region in the magnet with Mg for different reaction times: (a) 30 min, (b) 1 h, (c) 3 h, (d) 6 h, (e) 12 h, (f) 24 h, and (g) 48 h.
Figure 3. Microstructure of the matrix in reaction region with respect to the reaction Table (a) 30 min, (b) 1 h, (c) 3 h, (d) 6 h, (e) 12 h, (f) 24 h, and (g) 48 h.

Figure 4. XRD pattern of the matrix at a reaction time at 3 h.
To precisely define the intermetallic phase, Figure 5 shows the results of characterized phases in the matrix by STEM-EDS. To observe the morphologies, the areas in the reacted specimen at 3 h are divided into three zones, marked as 1, 2, and 3. The chemical compositions for each zone are collected in Table 3 by EDS in STEM. While there is little Nd and Dy inside zone 3, as most of the REEs were swept to the Mg side, it is observed that REE-Fe intermetallic compounds in zones 1 and 2 are indeed Dy$_{2}$Fe$_{17}$. These results match well with the XRD results.

![Figure 5. TEM image of the matrix at a reaction time at 3 h.](image)

**Table 3.** TEM-EDS results of reaction zone after 3 h.

| No. | Fe (wt.%) | Nd (wt.%) | Dy (wt.%) |
|-----|-----------|-----------|-----------|
| 1   | 90.2      | 0.660     | 9.00      |
| 2   | 91.1      | 0.550     | 8.16      |
| 3   | 99.2      | 0.160     | 0.660     |

The formation of Dy$_{2}$Fe$_{17}$ can be inferred by the heat of the mixing values between Dy-Mg and Nd-Mg. Even though the values of heat of the mixing between Mg and REEs (Nd and Dy) are the same with $-6$ cal/mol during the reaction, the values between Dy-Fe and Nd-Fe is different to be $\Delta H_{\text{mix}}^{\text{DyFe}} = -3 \text{ cal/mol}$, and $\Delta H_{\text{mix}}^{\text{NdFe}} = +1 \text{ cal/mol}$, respectively [22]. Thus, the diffusion of Dy to Mg can be interfered with due to its reaction with Fe as Fe exhibits a higher affinity towards Dy as compared to Nd. Similar to the results of the analysis, the Dy-Fe intermetallic compounds, Dy$_{2}$Fe$_{17}$, are observed in the results of the phase analysis on the matrix.

To investigate the reactivity between Mg and Dy$_{2}$Fe$_{17}$, the thermodynamic calculations were carried out using Equation (3):

$$2\text{Mg}(l) + \text{Dy}_{2}\text{Fe}_{17}(s) \rightarrow 2\text{MgDy}(s) + 17\text{Fe}(s) \quad (3)$$

The standard Gibbs free energy change, $\Delta G^\circ$, of the reaction between Mg and Dy$_{2}$Fe$_{17}$, shown in (3), is 85,778.2 J/mol (at 900 °C) [23], and the Gibbs free energy change for reaction (3), $\Delta G$, is expressed as follows:
\[ \Delta G = \Delta G^\circ + RT \ln K \left( \frac{\alpha_{\text{Fe}}^{17} \alpha_{\text{Mg}}^2}{\alpha_{\text{Mg}} \alpha_{\text{Dy2Fe17}}} \right) \] (4)

The activity of the chemical species \( i \) at temperature \( T/K \) is \( \alpha_i \) and the condition for reaction (3) and (4) to proceed are:

\[ \Delta G = 85,778.2 + 2RT \ln \alpha_{\text{MgDy}} < 0 \] (5)

The activity of \( \alpha_{\text{Mg}}, \alpha_{\text{Dy2Fe17}}, \alpha_{\text{Dy2O3}}, \) and \( \alpha_{\text{Fe}} \) are defined as 1. The condition for reaction (5) to progress is as follows at 900 °C:

\[ \alpha_{\text{MgDy}} < 0.01218 \]

It is indicated that the reaction priority can be derived, considering the grain boundary as well as the matrix, during Mg infiltration. According to the Ellingham diagram, the reaction of REEs with Mg is inevitably hindered because of the high-affinity properties with oxygen in REEs give rise to RE\(_2\)O\(_3\) formation around the grain boundary in advance. Despite the same group, there is a large difference in reactivity of Nd\(_2\)O\(_3\) and Dy\(_2\)O\(_3\) with Mg which is \( \alpha < 0.0433 \) and \( \alpha < 0.00535 \), respectively [16]. Moreover, the Dy-Fe reactivity is considered with \( \alpha < 0.01218 \) in the matrix, which is five times higher than Dy\(_2\)O\(_3\) reactivity. It is suggested that the decomposition of Dy\(_2\)Fe\(_{17}\) in the matrix leads to the Dy extraction process after Dy in the RE-rich phase on the grain boundary can be initially swept to Mg.

To check the tendency of reaction priority, based on the thermodynamics, the reaction time dependence of volume fractions in Dy\(_2\)O\(_3\) and Dy\(_2\)Fe\(_{17}\) is experimentally revealed in Figure 6a,b, respectively. The phase fractions are estimated by the SEM image analysis. It is observed that the decomposition rate of Dy\(_2\)Fe\(_{17}\) is gradually decreasing with increasing reaction time while Dy\(_2\)O\(_3\) is relatively stable except for 48 h. Considering the curves of extraction efficiency in Figure 1, the slope of extraction efficiency is drastically increased before 3 h because the RE-rich reaction with initial infiltrated Mg into the grain boundary is almost complete. Steadily expanding Mg reaction zone into the matrix, while the Nd and Nd\(_2\)O\(_3\) are totally reacted, the slope of Dy extraction is simultaneously found to be gradual. The decomposition of Dy\(_2\)Fe\(_{17}\) is mainly contributed to improving Dy extraction because the volume of Dy\(_2\)Fe\(_{17}\) is started to be dramatically decreased from 3 h to 6 h in Figure 6b. Nevertheless, the reason for the gradual slope is that the relatively small amount of Dy compared to in the RE-rich phase. Finally, Dy\(_2\)O\(_3\) phases, which are the most stable phase, started to decompose with increasing time once the decomposition of Dy\(_2\)Fe\(_{17}\) was almost complete. Infiltrating Mg into the magnet with increasing time, the oxide and intermetallic phases are generated depending on the reaction zones, which are the grain boundary and the matrix. Before decomposing Dy\(_2\)O\(_3\), the Dy\(_2\)Fe\(_{17}\) phases are induced by a reaction between magnets and liquid Mg in the matrix via RE-rich reaction. It is suggested that the Dy\(_2\)Fe\(_{17}\) phases are attributed to the first main hurdles, considering thermodynamic activity and the analysis of extraction behavior, prior to the decomposing Dy\(_2\)O\(_3\) phases.
4. Conclusions

The phase transformation in the microstructures of magnets during the LME process is systematically investigated with increasing reaction time. The liquid Mg is diffused into the magnet area and it gradually expanded its reaction zones inside the grain boundary and matrix. The REEs in the RE-rich phases rapidly swept to the Mg side. Since then, in spite of the magnet with well-controlled oxygen contents, the RE-oxide phases with a foam of RE$_2$O$_3$ are distributed in the grain boundary and the RE-Fe intermetallic compounds are simultaneously discovered with needle-shaped particles in the matrix. The RE-Fe phases are precisely defined with Dy$_2$Fe$_{17}$ by quantitative analysis and comparing thermodynamic reactivity. While the extraction of Nd is complete in the RE-rich reaction without the formation of intermetallic compounds, the extraction curves of Dy behave quite differently due to Mg reactivity. In terms of thermodynamics, the formation of Dy-Fe intermetallic compounds is inferred, and their reactivity is compared with oxides. The extraction behavior of Dy shows that the de-oxidized Dy in the RE-rich phase of the grain boundary is quickly reacted with Mg in 3 h. Even though there is a Dy$_2$O$_3$ phase in the same area, the Mg reaction preferentially arises with Dy$_2$Fe$_{17}$ in the matrix due to differences in stability and reactivity. Afterwards, the Dy extraction is finalized to the reaction of Dy$_2$O$_3$ with Mg. It is suggested that the appearance of Dy$_2$Fe$_{17}$ phases is intrinsic in the decomposing behavior of magnet, unlike Nd, and is attributed to the first hurdles prior to the decomposing Dy$_2$O$_3$ phases.

Author Contributions: Conceptualization, T.-S.K. and S.-W.N.; methodology, S.-W.N., S.-M.P. and M.Z.R.; validation, T.-S.K. and S.-W.N.; analysis, S.-W.N. and S.-M.P.; writing—original draft preparation, S.-W.N.; writing—review and editing T.-S.K. and M.-S.S. supervision T.-S.K. and D.-H.K.; project administration, T.-S.K.; All authors have read and agreed to the published version of the manuscript.

Funding: This research was supported by a grant from project of development of environment friendly pyrometallurgy process for high purity HREE and materialization (Project number: 20000970) by Korea evaluation Institute of Industrial Technology (KEIT) in Republic of Korea.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no confliction of interest.

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