THERMODYNAMIC SIMULATION AND VALIDATION EXPERIMENT OF NEODYMIUM OXIDE REDUCTION INTO METALLIC NEODYMIUM BY METALLOTHERMIC PROCESS

SIMULASI TERMODINAMIKA DAN EKSPERIMEN VALIDASI REDUKSI NEODIMIUM OKSIDA MENJADI LOGAM NEODIMIUM MENGGUNAKAN METODE METALOTERMIK

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ABSTRACT

Neodymium (Nd) is one of the rare-earth elements (REEs) found in significant amount within monazite and bastnasite minerals. Nd is a ferromagnetic metal that is often used as a material to produce magnet, commonly known as a permanent magnet. Neodymium is alloyed with other metals such as iron and boron to form one of the strongest types of permanent magnet. This research aim is to study the reduction process of Nd-oxide into Nd-metal through the metallothermic process. The Nd metal product is expected to fulfill the material specification for a permanent magnet. Thermodynamics simulation of Nd reduction into its metal was conducted using ITB"s licensed Factsage software. A validation experiment was conducted only to the Nd metal resulting simulation. The simulations involved some parameters, i.e. temperatures (600, 700, 800, 900, 1000, 1100 and 1200 °C), types of fluxes (CaCl₂ and Ca(OH)₂), composition of the reducing agent (1x, 2x dan 3x of the stoichiometric calculations), types of the reducing agents (Ca and Mg), and types of feeds used (Nd-oxide and Ndchloride). The thermodynamic simulation shows that Nd metal was produced in a condition where the temperature should be1100-1200 °C using Ca as the reducing agent and CaCl₂ as the flux, while the amount of reducing agent has no effect on the resulted product. Validation result of the simulations shows that the Nd metal is formed up to 49% metal in a non-oxidative condition.

Keywords: neodymium, rare earth oxides, metallothermic, reduction, thermodynamic simulation

ABSTRAK

Neodimium (Nd) adalah salah satu unsur logam tanah jarang yang keberadaannya sangat signifikan didalam mineral monasit dan basnasit. Logam Nd memiliki sifat feromagnetik sebagai bahan magnet, yang lebih dikenal dengan magnet permanen. Nd jika dipadukan dengan logam lain seperti besi dan boron dapat membentuk magnet permanen yang memiliki sifat magnet sangat kuat. Penelitian ini difokuskan untuk mempelajari proses reduksi Nd-oksida menjadi logam Nd dengan metode metalotermik. Logam Nd yang dihasilkan diharapkan dapat memenuhi spesifikasi untuk bahan baku magnet permanen. Simulasi termodinamika proses reduksi untuk memprediksi proses reduksi dari oksida logam tanah jarang menjadi logamnya menggunakan FACTSAGE Software *lisensi Institut Teknologi Bandung*

(ITB). Validasi simulasi dilakukan melalui percobaan terhadap proses simulasi yang menghasilkan logam Nd. Parameter yang dilakukan pada proses simulasi adalah; suhu reduksi (600, 700, 800, 900, 1000, 1100 dan 1200 °C), jenis fluks (CaCl² dan Ca(OH)2), komposisi reduktor (1x, 2x dan 3x stoikiometri), jenis reduktor (Ca dan Mg) dan jenis umpan (Nd-oksida dan Nd-klorida). Hasil simulasi termodinamika menunjukkan bahwa logam Nd dapat terbentuk pada kondisi proses: suhu 1100-1200 °C dengan reduktor Ca dan penambahan fluks CaCl2, jumlah reduktor tidak mempengaruhi produk yang dihasilkan. Hasil validasi dari simulasi menunjukkan bahwa logam Nd terbentuk dengan kadar 49% dalam suasana non-oksidasi.

Kata kunci: neodimium, oksida logam tanah jarang, metalotermik, reduksi, simulasi termodinamika

INTRODUCTION

Rare earth elements (REEs) are a group of 17 chemical elements on the periodic table that consists of lanthanides group (from lanthanum to lutetium), yttrium, and scandium. Neodymium is one of the REEs symbolized with Nd and an atomic [number](https://en.wikipedia.org/wiki/Atomic_number) of 60. It is present in a significant quantities in [monazite](https://en.wikipedia.org/wiki/Monazite) and [bastnäsitem](https://en.wikipedia.org/wiki/Bastn%C3%A4site)inerals (Gupta and Krishnamurthy, 2005). The monazite deposits in Indonesia can be found in Bangka Belitung, Karimata/Ketapang, Rirang-Tanah Merah (Atmawinata, 2011).

REEs share some common properties; such as softness, ductility, malleability, reactivity and magnetic properties. Those properties, especially the latter, and the fact that rare earth magnets are the strongest type of magnets, have made REE useful for many applications (Manaf, 2007). Nd is considered a critical technology metal mostly used for permanent magnets, electric vehicles, harddisc drives, mobile phones and more (Riaño and Binnemans, 2015).

There are two important process steps of REEs extraction, one of them is the rareearth oxide separation in large scale, and the other is the production of rare-earth metal or rare-earth magnetic alloy from the oxide. This article is mainly devoted to the latter: obtaining the Nd metal from its oxide.

Rare earth oxides (RE-oxides) can be reduced into its metal through metallothermic or electrolysis processes. In electrolysis, the process can be carried out continuously (Claux, Serp and Fouletier, 2011). However, it requires relatively expensive and quite consumptive electrodes operating under high temperature (> 1000°C), with a relatively low recovery of rare earth metal (REM) of <40% (Zhu, 2014). In the metallothermic process, a nonoxidative condition is required and high energy is consumed while producing > 90% recovery of REM (Weber and Reisman, 2012).

Standard free energy data (ΔG^0_{1000}) shows that Ca, Y, and heavy rare earth elements (Tb, Dy, Ho, Er, Tm, Yb, and Lu) can reduce $Nd₂O₃$ (Gupta and Krishnamurthy, 2005). At the beginning of 2013, there was no commercial producer of the reduction of REoxide into its rare-earth metal in the United States, while in Europe the situation is different; the production of rare-earth metal using molten salts announced in mid-2013 (Koltun and Tharumarajah, 2014). Brazil was a producer of misch metal in the early 1990s at a company named CIF. However, since 1996. CIF has been focusing on tantalum production using fluoride molten salts (De Campos, Rodrigues and De Castro, 2014) and no longer produce the rare-earth metal. Present day technology usually uses fluoride salts for the reduction of Nd oxide.

Reduction of RE-oxide for the preparation of high purity rare earth metal (REM) is a descendant of the Ames Process for the preparation of uranium for the Manhattan Project - the metallothermic reduction of an RE-fluoride using calcium as the reducing agent. RE-fluoride is prepared from high purity RE-oxide reacted with a fluorine bearing compound (Ames Laboratory, 2017).

Research on the reduction of Nd-oxide has been done by researchers worldwide. Lee *et al.* (2016), studied the electrochemical reduction of $Nd₂O₃$ through co-reduction with NiO to increase the reduction degree of rare earth oxides in molten LiCl containing 1 %wt of $Li₂O$. Nd₂O₃ was successfully converted into its metal in the presence of NiO through the reduction of NiO to Ni, followed by that of

 $Nd₂O₃$ on the produced Ni to form an intermetallic $NdNi₅$, continued with the reduction of NdNi5. Biswas *et al.* (1998), Nd-aluminide aluminothermic reduction of neodymium oxide, even though the standard free energy change associated with the reduction of $Nd₂O₃$ by AI is positive (unspontaneous).

In Indonesia, the magnet products, particularly the permanent magnets (NdFeB) available on the market, are still entirely dependent on the imported product. The needs of permanent magnets in Indonesia are very high (Benecki, 2008). Therefore, mastering manufacture technology of permanent magnets from national resources, in this case, the rare earth metal as raw materials, is the utmost importance. Sharma (1986) in US Patent No. 4.578.242 carried out a reduction process of Nd-oxide using Na metal as reducing agent and $CaCl₂$ as flux and added by Zn metal which forms a low eutectic melting temperature with Nd (around 630°C) and resulted in alloy metal of Nd-Zn. Refining was done by evaporating Zn at its boiling point $(907^{\circ}C)$ and left Nd in a form of its metal. This was due to the high boiling point of Nd (3150°C). In this research, some innovations were done, i.e. reducing Nd-oxide without going through the formation of Nd-Zn alloy (direct reduction) and replace reducing agent with other metals that are less explosive than Na metal. Thus, it was expected that the Nd metal produced yields high purity (higher than 95%) in a shorter processing time.

Similar research was done by Saleh (2015) with the purpose of reducing cerium oxide $(CeO₂)$ into its cerium (Ce) metal. The reduction was carried out through the metallothermic process using magnesium powder (Mg) as the reducing agent and calcium chloride $(CaCl₂)$ as flux. From the research, 50% of Ce metal was recovered with the purity of 91% at the temperature of 1200 °C for 3 hours using 1% of flux and1:1 composition of $CeO₂$ and Mg.

Prior to the experimental research of Ndoxide reduction into its metal, the thermodynamic simulation was done using Factsage 6.4 (license owned by Bandung Institute of Technology). Factsage was used to simulate/predict the reduction and refining process of the rare earth without doing any experimental research. Nevertheless, experimental research is important and needs to be done in order to validate the calculation/simulation result from Factsage. Thus, Factsageacts as a "preliminary study" to obtain the illustration/prediction of the reduction process and its result.

The purpose of this research was to study the reduction process of Nd-oxide into its metal through thermodynamics simulation using Factsage software (ITB's licensed) to predict the result of the reduction and the refining processes without doing any experimental research. It was expected that by doing the thermodynamic simulation, the preparation of Nd-metal from its oxide through experimental research can be more efficient, time-effective with greater probability to be succeeded.

METHODOLOGY

Nd-oxide used as raw material in this study was obtained from commercial Nd-oxides. Reduction of Nd-oxide (Nd_2O_3) was conducted using metal powder of Ca (calcium), Al (aluminium) and Mg (magnesium) as the reducing agents with the addition of $Ca(OH)_2$ and $CaCl_2$ as the fluxes. Flow diagram of Nd-oxide reduction can be seen in Figure 1. RE-oxides can be reduced by a metallothermic method using alkali metals as reducing agents such as Li, Na, K, Mg, Ca and Al because they are more stable than RE-oxides (Krishnamurthy and Gupta, 2015).

Equipment used for the reduction of Ndoxide were as follows: press machine, tube furnace, combustion boat, crucible, muffle furnace and resistance furnace.

The parameters studied for simulation process were the types and amount of the reducing agents of Ca and Mg (1x, 2x and 3x of stoichiometric calculation), the types of feed $(Nd₂O₃$ and $NdCl₃$), and the reduction of temperature (600, 700, 800, 900, 1000, 1100, 1200°C).

Figure1. Flowchart of the reduction of Nd-oxide into Nd metal based on metallothermic process.

The reduction process for validation was carried out in a non-oxidative condition using inert gases (nitrogen and argon) to dissipate oxygen gas. After the reduction process, the metal phase and the slag produced were then separated using hand magnet (slag is non-magnetic material while metal is magnetic material) and analyzed using XRD and SEM analysis to further clarify the composition of the elements contained in the resulted product.

RESULTS AND DISCUSSION

This research was based on thermodynamics simulation to predict the condition of the reduction process using Factsage software. Factsage has been widely used in the calculation of thermodynamic equilibrium in combustion, slagging, corrosion, and reduction process taking place in a metallurgical plant. The calculation principle was based on the minimum total free energy in a system. When the total free energy in a system is minimum, the system reaches equilibrium, where all occurring reactions reaches the equilibrium. Thus, the thermodynamics calculation can be used to predict chemical and physical process rapidly.

The variable parameters applied in this simulation to determine the most suitable process were types of reducing agent and types of feed.

1. Types of reducing agent

This simulation was conducted to determine the best reducing agent to produce the Nd-metal. The reducing agents used in this simulation were Ca and Mg. The result of simulation using Ca are shown in Figure 2 while that of Mg are in Figure 3.

Figure 2 shows that Ca metals started to reduce the Nd-oxide (Nd_2O_3) at the minimum temperature of 1100°C. At the temperature of 600 – 1000°C, there were no Nd metals formed. Reaction during the process is as follows:

$$
Nd_2O_{3(s)} + 3Ca_{(s)} \rightarrow 2Nd_{(s)} + 3CaO_{(s)}
$$

Figure 3 shows that the reduction of Ndoxide using Mg did not produce its metal, even if the temperature was raised up to 1200°C.

Thus, it can be stated that the suitable reducing agent to reduce Nd-oxide into its metal is Ca at the minimum temperature of 1000°C.

Figure 2. The relation between temperature and neodymium recovery using calcium as reducing agent

Figure 3. The relation between temperature and neodymium recovery using magnesium as reducing agent

The selection of the suitable reducing agent can be predicted from the standard free energy formation (ΔG^0) .). The reduction process occurs if the difference of ΔG^0 of Nd₂O₃ with ΔG^0 of CaO is negative. The Ellingham Diagram given in Figure 4 shows the relation of free energy and the temperature of the reaction.

Figure 4 shows that the elements that were able to reduce rare earth-oxides were very limited. In the standard condition, the order of the oxide stability is $CaO > RE₂O₃ > MgO > Al₂O₃ > SiO₂.$ However, there are more elements that able to reduce the rare earth-chlorides, i.e.KCl>NaCl≈LiCl≈CaCl₂>RECl₃>MgCl₂>

>AlCl3. Calcium is the only metal element able to reduce both rare earth-oxides and chlorides into their metals.The statement is true based on both simulation results and the Ellingham Diagram.

2. Thermodynamic simulation using various types of feed

This simulation was done to determine which types of feed (types of Ndcompound) able to produce highest recovery of Nd through metallothermic reduction. Based on Ames Process, it was suggested to use RE-chlorides as the feed instead of RE-oxides in order to obtain higher recovery (Riedemann, 2011). Figure 5 shows the simulation results using Nd-chloride as the feed and Ca as the reducing agent, while Figure 6 shows simulation results using Ndchloride with Mg as the reducing agent.

Figures 5 and 6 show that Ca and Mg powder, acts as the reducing agent, was able to reduce Nd_2Cl_3 at a minimum temperature of 1100 °C.

Figure 4. (a) and (b) Standard free energy formation of various rare earth elements and metal chlorides as the function of temperature, (c) and (d) Standard free energy formation of the various rare earth elements and metal oxides as the function of temperature (Ellingham, 1944; Richardson and Jeffes, 1948)

Figure 5. Recovery of metallic-Ndvs temperatures using NdCl₃ as the feed and Ca as the reducing agent.

Figure 6. Recovery of metallic-Ndvs temperature using $NdCl₃$ as the feed and Mg as the reducing agent.

Based on Figures 5 and 6, it can be stated that by combining $Nd₂O₃$ as the feed and Mg as the reducing agent did not produce Nd metal. However, by converting the Nd_2O_3 into its chloride salt first $(NdCl₃)$, at the temperature of 1100 and 1200 °C, the Nd metal was started to be formed.

After completing the thermodynamics simulation, the next step was validation experiment. The validation experiment was done only to the Nd metal resulting simulations, i.e. using both types of feed $(Nd₂O₃$ and $Nd₂Cl₃)$ with Ca as the reducing agent at the temperature of 1100-1200 °C. The reduction of Nd-oxide into its metal was conducted using commercial Nd-oxide. The ICP analysis to determine the chemical composition of the commercial Nd-oxide can be seen in Table 1.

The experiments were carried out using various compositions of fluxes and a reducing agent at the temperature of 1200 °C for 4 hours using Ca as the reducing agent with the addition of $CaCl₂$ and NaCl as the fluxes. Prior to the experiments, the parameters were varied (Table 2) and simulated (Figure 7) to predict the amount of Nd metal formed.

Figure 7. Recovery of Nd metal vs variation of the feeds at the temperature of 1200 °C.

Table2. Variation of the experiment's parameters

Sample 12 NdCl₃ as the feed with 10% excess of Ca;flux consisted of 70% of CaCl₂ 70% and 30% of NaCl

Figure 7 shows that Nd metal was formed in samples no. 2, 3, and 6. The formation of liquid Nd from Factsage simulation indicated the formation of Nd metal in a form of liquid. The highest Nd metal recovery was 20 moles (sample no 3) achieved using $Nd₂O₃$ as the feed with 10% excess of Ca; 70% excess of $CaCl₂$; and 30% excess of NaCl, reduced at the temperature of 1200 °C for 4 hours in a non-oxidative condition. Ndchloride $(NdCl₃)$, an intermediate product, was formed using samples no 5, 6, and 7, while the products of samples 3 and 4 were still in a form of Nd-oxide.

Figure 8 to Figure 14 show the SEM-EDS result analysis for the validation experiment for samples no. 2, 3, 4, 7, 8, 9, and 10 respectively (produced the intermediate product Nd-chloride or Nd metal).

Figure 8. SEM-photomicrograph (left figure) and SEM-EDS (right figure) analysis results on sample 2.

Figure 9. SEM-photomicrograph (left figure) and SEM-EDS (right figure) analysis results on sample 3.

Figure 10. SEM-photomicrograph (left figure) and SEM-EDS (right figure) analysis results on sample 4.

Figure 11. SEM-photomicrograph (left figure) and SEM-EDS (right figure) analysis results on sample 7.

Figure 12. SEM-photomicrograph (left figure) and SEM-EDS (right figure) analysis results on sample 8.

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Figure 13. SEM-photomicrograph (left figure) and SEM-EDS (right figure) analysis results on sample 9.

Figure 14. SEM-photomicrograph (left figure) and SEM-EDS (right figure) analysis results on sample 10.

Based on the SEM-photomicrograph analysis results on Figures 8 to 14, the Nd elements were shown with the brighter colours on the left side of the figures. While based on the SEM-EDS mapping on the right side of the figures, it can be concluded that the Nd element was well-scattered, shown by the red colour on each of the element"s mapping results.

From the validation experiment, only samples 2 and 3 produced Nd metal. However, sample 6 with the same composition/parameter as sample 2 did not produce Nd metal. This might be caused by different treatment applied to the sample. Sample 2 was pelletized prior to the reduction process, while sample 6 was used directly in its existing form (powder).

Table 3 shows the result of the elemental composition analysis on sample 3 using $Nd₂O₃$ as the feed with the amount of Ca excessed twice of the stoichiometric calculation.

Based on Table 3, it can be concluded that Nd metal was formed through the reduction process with a grade of 49.25%. However, Na, Ca, and Cl were still contained in the product (as the impurities of the Nd metal). Nd metal produced from the validation

experiment for sample no. 3 shown in Figure 15.

Table 3. Result of the elemental composition analysis on sample 3 using Nd_2O_3 as the feed with the amount of C at twice excessed of the stoichiometric calculation

Experimental results carried out to validate Factsage"s thermodynamics simulations generally shows similar results. Factsage result showed that both samples no. 2 and 3 produced Nd metal. However, from the validation experiment, only sample no. 3 produced Nd metal. The difference in the results can be caused by the non-ideal condition in the validation experiments, on the contrary to the conditions in the Factsage"s thermodynamics simulation. For example, the presence of oxygen in the reduction process; it was an 'easy' task to eliminate oxygen in the simulation but not in the real experiment. Thus, this may lead to the result"s difference, including the formation of the metal.

Figure 15. Nd metal produced using 1 Gram of Nd_2O_3 .

CONCLUSION

The validation experiment shows a similar result with the preceding thermodynamic simulation using Factsage software. However, some difference in the product resulted might be due to the different condition between the real experiment (nonideal) and thermodynamic simulation (ideal).

Reduction of rare earth metal oxides into their metals, particularly for Nd, can be carried out through metallothermic methods and generate a product with more than 49% of metal.

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