Recovering Magnesium from Ferronickel Slag by Vacuum Reduction: Thermodynamic Analysis and Experimental Verification

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ABSTRACT: The feasibility of recovering magnesium from ferronickel slag by vacuum reduction was evaluated. The thermodynamic calculations indicated that the magnesia in slag can be reduced to gaseous magnesium by Si, FeSi, Al, and C, with the minimum reduction temperatures of 2324, 2530, 1678, and 2580 K at 100 000 Pa, respectively. As the system pressure decreases, the minimum reduction temperatures decline significantly. Si maintains the minimum reduction temperature of 1585–1673 K at the atmospheric pressure of 10–100 Pa, acting as a suitable reducing agent for recovering magnesium. To verify the findings, preliminary vacuum reduction experiments, in which CaO was added to eliminate the adverse impact of SiO2 in slag, were carried out. By reducing slag with additions of 50 wt % Si and 30 wt % CaO at 1573 K for 3 h at 10 Pa, the recovery of magnesium reached 97.74%.

INTRODUCTION

With the continuously increasing demand of ferronickel alloy, smelting of nickel laterite resources for production of ferronickel has attracted considerable attention in the past decade.1 Owing to the advantages of good operational flexibility, high productivity, and high quality of the crude ferronickel product, the rotary kiln-electric furnace (RKEF) process has undergone rapid development around the world and become a major ferronickel smelting technology.1 However, the RKEF process also suffers from a few shortcomings, particularly high energy consumption and huge discharge of slag. It was reported that about 14 tons of slag are generated in the production of 1 ton of ferronickel. By now, slag has become the fourth largest industrial solid waste in China.2,3 Depending on the cooling methods, ferronickel slag can be classified into three types, namely, slowly-cooled slag, wind-cooled slag, and water-quenched slag.4 Among them, water-quenched slag is a major type in the industry. Many studies have been focused on the utilization of this type of ferronickel slag,5–14 including those on making construction and building materials,5,7 producing glass ceramics,9 and recovering the contained metals (e.g., nickel, cobalt).10,11 Because of the existence of magnesia in ferronickel slag, the applications in construction and building materials have potential swelling problems, and the addition of ferronickel slag should be limited in a small range. For the production of glass ceramics, the necessity of applying various additives increases the complexity of the process. For recovering metals from ferronickel slag, most relevant studies were focused on the extraction of iron, nickel, cobalt, and chromium.15–18 However, because of the high contents of magnesia (about 35 wt %) and silica (about 45 wt %) as well as low contents of the aforementioned metals (generally less than 10 wt %) in ferronickel slag,19,20 recovering metals from the slag in a cost-efficient way remains a challenge until the present moment.

In view of the high content of magnesia in ferronickel slag, it may be viable to extract magnesium from slag by using a proper method. Magnesium is the lightest metal among the commonly used structure metals and widely used in aircrafts, rockets, and automobile industries.21 It is produced by two principal routes: electrolysis of molten magnesium chloride and thermal reduction of magnesia.22 Because of the high cost of the electrolytic process, at present, the majority of magnesium is produced from dolomite by the so-called Pidgeon process based on thermal reduction at low pressure.23–25 In this process, calcined dolomite is mixed with the reducing agent (such as silicon, ferrosilicon, and aluminum) to form a mixture which is then charged into a batch vacuum reduction furnace to obtain magnesium vapor for condensation and collection.26–28 Regarding the similar content of magnesia in ferronickel slag to that of calcined dolomite, the necessity of applying various additives increases the complexity of the process. For recovering metals from ferronickel slag, the necessity of applying various additives increases the complexity of the process.
Dolomite, a new method for recovering magnesium from ferronickel slag by vacuum reduction was proposed in this study. Its feasibility was first examined by thermodynamic analysis of reduction of magnesia considering both effects of reducing agents and reaction pressure. Because silica is the main impurity component of ferronickel slag, which may play an important role in the reduction, its influence was also examined. The findings were then verified by preliminary experiments. It was found that by vacuum reduction of ferronickel slag with additions of Si and CaO, which controlled the adverse impact of silica, at 10 Pa, high recovery of magnesium could be obtained from the slag.

**EXPERIMENTAL SECTION**

Materials. The ferronickel slag used in this study was obtained from a ferronickel smelting plant in China, which produces Ni−Fe alloy by the RKEF process. This slag contained 48.29 wt % SiO₂, 30.95 wt % MgO, 2.11 wt % Cr₂O₃, 7.39 wt % FeO, and 4.04 wt % Al₂O₃. Its main mineral phase was olivine (Mg₁.₈Fe₀.₂SiO₄). Silicon and calcium oxide of the analytical grade were used as the reducing agent and additive, respectively.

Methods. Thermodynamic Calculation. The thermodynamic analysis of relevant reactions during reduction of ferronickel slag with the addition of various reducing agents was performed by establishing the corresponding Gibbs free energy changes of reactions versus temperature ($\Delta G_m$–$T$) graphs and phase diagrams using the software FactSage 7.2 (Thermfact/CRCT and GTT-Technologies, Montreal, Canada and Herzogenrath, Germany) based on its databases of FactPs, FToxid, and FTsalt.

Verification Tests. In order to verify the findings of thermodynamic analysis, the vacuum reduction experiments were carried out. Initially, the ferronickel slag sample with a particle size less than 0.074 mm was mixed homogeneously with predetermined amounts of the reducing agent and additives (100% particles passing 0.074 mm). Then, the mixture was used to produce briquettes of 15 mm in diameter and 15 mm in height by hydraulic pressing at 50 MPa. The briquettes were loaded in a graphite crucible and roasted in a vacuum furnace, which was maintained at a preset temperature (1573 K) and vacuum degree for a given period of time. After reduction, the briquettes were taken out for characterization and analysis. The magnesium contents in the briquettes before/after reduction were measured for calculating the recovery of magnesium using the following equation

$$\eta = \left( 1 - \frac{W_2M_2}{W_1M_1} \right) \times 100\%$$

where $\eta$ is the recovery of Mg; $W_1$ and $W_2$ are the Mg contents of the samples before reduction and after reduction, respectively; $M_1$ and $M_2$ are the masses of the samples before reduction and after reduction, respectively.

The chemical compositions of the samples were measured by using an X-ray fluorescence spectrometer (Axios Max, PANalytical, Netherlands). To confirm the phase transformation in ferronickel slag due to vacuum reduction, the phase compositions of the samples were determined by using the software FactSage 7.2.
an X-ray diffraction (XRD) spectrometer (D/Max 2550 PC, Rigaku Co., Ltd, Japan) with a Cu anode (wavelength 1.54056 Å, step scan mode, scan range from 10° to 80°, scan speed 5°/min, and step length 0.02°).

RESULTS AND DISCUSSION

Thermodynamic Analysis. Δ_rGm−T Graphs. In vacuum reduction, the reducing agent and reaction pressure are crucial for the process. In order to seek a suitable reducing agent for the recovery of magnesium from ferronickel slag, the reactions between ferronickel slag and different reducing agents, including Si, FeSi, Al, and C, were investigated. According to the chemical composition of ferronickel slag, the possible reactions between magnesia and reducing agents are as follows

\[
\begin{align*}
2\text{MgO}(s) + \text{Si}(s) &= 2\text{Mg}(g) + \text{SiO}_2(s) \\
2\text{MgO}(s) + \text{FeSi}(s) &= 2\text{Mg}(g) + \text{Fe}(s) + \text{SiO}_2(s) \\
3\text{MgO}(s) + 2\text{Al}(s) &= 3\text{Mg}(g) + \text{Al}_2\text{O}_3(s) \\
2\text{MgO}(s) + C(s) &= 2\text{Mg}(g) + \text{CO}(g)
\end{align*}
\]

The Δ_rGm−T graphs of the above reactions at different pressures are shown in Figure 1. As shown in Figure 1a, in the presence of Si, magnesia can be converted to gaseous magnesium at different temperatures, depending upon the system pressure. In specific, at the standard atmospheric pressure (100 000 Pa), the reduction of magnesia to gaseous magnesium demands a temperature higher than 2324 K (at which the Gibbs free energy change of reaction was 0). As the system pressure decreases, the minimum temperature required for the reduction declines significantly. For instance, when the system pressure decreases to 10 Pa, the minimum temperature drops to 1585 K. It will further decrease to 1417 K at 1 Pa.

As shown in Figure 1b, when FeSi is used as the reducing agent, the Δ_rGm value of the reaction between MgO and FeSi decreases evidently with increasing temperature and decreasing pressure. The minimum temperature required for reducing magnesia to gaseous magnesium at the standard atmospheric pressure was 2530 K. As the pressure decreases to as low as 10 and 1 Pa, the minimum temperatures required for the reduction decrease to 1598 and 1467 K, respectively.

By using Al as the reducing agent, magnesia will be reduced to gaseous magnesium above 1678 K at the standard atmospheric pressure, as shown in Figure 1c. As the system pressure decreases, the minimum temperature required for the reduction decreases significantly. When the system pressure decreases to 10 and 1 Pa, the temperatures decline to 977 and 909 K, respectively.

In the presence of C, magnesia can also be reduced to gaseous magnesium, in spite of stricter requirements for temperature and system pressure (Figure 1d). At the standard atmospheric pressure, the minimum temperature required for the reduction of magnesia to gaseous magnesium is 2580 K. As the system pressure declines to 10 and 1 Pa, the temperatures drop to 1622 and 1505 K, respectively.

Based on the above analysis, it is feasible to recover gaseous magnesium from the slag despite the high temperature requirement when the process is operated at the standard atmospheric pressure. By reducing system pressure, the minimum temperature required for reducing magnesia to gaseous magnesium decreases significantly. Among all the reducing agents, Al has the advantage of low reduction temperature. However, it is explosive and difficult to control during the reduction process. Conversely, by using C as the reducing agent, the minimum temperature required for the reduction is too high for practical operation, which limits its application. In addition, it causes emission of CO₂ which would enter into the condensing zone with magnesium vapor, reverting the reaction. By comparing Si and FeSi as the reducing agents, the former one requires lower temperatures for reducing magnesia to gaseous magnesium, in particular at the system pressure of 10–100 Pa. On the other hand, the latter one has a lower cost. However, some silicon in ferrosilicon may not participate in the reaction, leading to the slow reaction rate and low reduction percentage of magnesia. In this study, Si was chosen as the suitable reducing agent.

Phase Diagrams. In order to further reveal the phase transformation of ferronickel slag for recovery of magnesium in the presence of Si, relevant phase diagrams were calculated. The phase diagrams of the MgO–Si system at the atmospheric pressures of 100 000, 100, and 10 Pa are shown in Figures 2–4, respectively. As shown in Figure 2, MgO and Si remain...
unchanged until the temperature is higher than 1690 K at which a liquid phase appears. At 1690–1856 K, by keeping the value of MgO/(MgO + Si) above 0.02, Mg$_2$SiO$_4$ generates and remains stable. When the temperature is elevated to 2073 K, a gas phase emerges, indicating the possibility to obtain gaseous magnesium at the standard atmospheric pressure. As the system pressure decreases to 100 Pa, the formation temperature of the gas phase decreases by 728 K and the stability area of Mg$_2$SiO$_4$ changes significantly. As shown in Figure 3, at 1345–2237 K, when the value of MgO/(MgO + Si) exceeds 0.6, Mg$_2$SiO$_4$ remains stable. In other words, the value of MgO/(MgO + Si) should be lower than 0.6 to ensure the reduction of magnesia to gaseous magnesium. When the temperature is higher than 2400 K, only the gas phase exists. It is harmful for recovery of magnesium from the gas phase because Si also evaporates into the gas phase, resulting in the difficult separation of magnesium. As shown in Figure 4, when the system pressure decreases to 10 Pa, the phase diagram of the MgO–Si system changes slightly. The formation temperature of the gas phase decreases further to 1273 K and similarly only the gas phase exists at temperatures above 2223 K.

According to Figures 2–4, it is obvious that the minimum temperature required for reduction of magnesia to gaseous magnesium decreases significantly with decreasing atmospheric pressure. Meanwhile, the theoretical suitable areas (the green areas in Figures 2–4) for recovery of gaseous magnesium from the slag decrease. It shows stricter requirements for selecting addition of the reducing agent, indicated by the smaller value of MgO/(MgO + Si) and the lower temperatures for the process operated at lower pressures.

As aforementioned, SiO$_2$ is the main impurity component in ferronickel slag. It may significantly reduce the efficiency for recovery of magnesium due to its reaction with MgO which produces MgSiO$_3$ that is expected to suppress the reduction of magnesia. To avoid this negative impact, the activity of SiO$_2$ must be lowered and it can be realized by adding alkaline agents, such as low-cost CaO. Figure 5 shows the calculated phase diagram of the CaO–MgO–SiO$_2$ system. It reveals that the addition of CaO will induce its reaction with silica to produce Ca$_2$SiO$_4$, CaSiO$_3$, and Ca$_3$Si$_2$O$_7$. Meanwhile, MgO will remain stable when the mass fraction of CaO is less than 0.7 (the yellow area in Figure 5). From this perspective, with proper addition of CaO, it is feasible to eliminate the adverse impact of SiO$_2$ during recovery of magnesium from ferronickel slag by vacuum reduction.

**Verification Test of Recovery of Magnesium from Ferronickel Slag.** To verify the findings of thermodynamic analysis, the vacuum reduction experiments of slag were carried out by reducing the slag using Si as the reducing agent. The effect of Si addition on the recovery of magnesium from the slag was explored under the conditions of the addition of 30 wt % CaO, reduction temperature of 1573 K, reduction time of 3 h, and system pressure of 10 Pa. As shown in Figure 6, when the addition of Si increased from 20 to 50 wt %, the recovery of Mg increased from 13.30 to 97.74%. As the addition of Si was further increased, the recovery of Mg increased slightly. It was shown that recovering gaseous magnesium from the slag was feasible under vacuum conditions, agreeing with the thermodynamic analysis. The effect of the addition of CaO on the recovery of magnesium from the slag was examined by...
fixing the conditions of addition of 50 wt % Si, reduction temperature of 1573 K, reduction time of 3 h, and atmospheric pressure of 10 Pa. According to Figure 6, without the addition of CaO, the recovery of Mg was relatively low (35.73%). Under the same experimental conditions, the presence of 30 wt % CaO promoted the recovery of Mg considerably to 97.74%. When the addition of CaO increased further to 40 wt %, the recovery of Mg remained relatively stable. The effect of CaO was also verified by comparing the main chemical compositions of the ferronickel slag samples after vacuum reduction in the absence or presence of CaO, which are provided in Table 1. The contents of Mg in the samples after vacuum reduction with additions of 0 wt % CaO and 30 wt % were 12.55 and 0.30 wt %, respectively. The big difference between the two cases was believed to be associated with the phase transformation in slag during vacuum reduction. This speculation was confirmed by Figure 7 which shows the main phases of the slag after vacuum reduction. In the absence of CaO, the main phases of the slag after vacuum reduction were olivine and silicon, indicating incomplete conversion of magnesia in the slag even with the excessive addition of the reducing agent. On the contrary, in the presence of 30 wt % CaO, the main phases were changed to dicalcium silicate, silicon, and katite, partially confirming the desirable reduction of magnesia in the slag. The experimental results agreed well with the thermodynamic analysis, verifying the feasibility to recover magnesium from ferronickel slag by vacuum reduction. It should be pointed out that the process is not only economically viable but also environmentally friendly. After the reduction, the resulting slag has the potential to be used as a good construction material because it had a similar composition (mainly SiO2 and CaO) to a few types of metallurgical slag which have been widely applied in construction and relevant fields.30

![Figure 7. XRD patterns of ferronickel slag after vacuum reduction with additions of (a) 0 wt % CaO and (b) 30 wt % CaO.](image_url)

**Table 1. Main Chemical Compositions of Ferronickel Slag Samples before/after Reduction**

| CaO addition (wt %) | before reduction | after reduction |
|---------------------|-----------------|----------------|
| 0                   | 23.30 7.54 Si 58.04 Ca 1.75 Fe 0.61 Al 0.93 | 27.90 12.55 Si 38.80 Ca 5.35 Fe 0.17 Al 1.58 |
| 30                  | 27.20 5.89 Si 42.33 Ca 18.50 Fe 4.30 Al 0.71 | 29.80 0.30 Si 22.36 Ca 33.59 Fe 6.91 Al 1.05 |

**CONCLUSIONS**

The feasibility of recovering magnesium from ferronickel slag by vacuum reduction using different reducing agents (Si, FeSi, Al, and C) has been evaluated based on the thermodynamic analysis and experimental verification. The thermodynamic calculations indicated that magnesium can be reduced to gaseous magnesium by Si, FeSi, Al, and C, with the minimum reduction temperatures of 2324, 2530, 1678, and 2580 K at the standard atmospheric pressure, respectively. As the atmospheric pressure decreases, the minimum temperatures required for the reduction decline significantly. It was found that Si is suitable for recovery of magnesium from ferronickel slag with the minimum reduction temperature of 1585–1673 K at 10–100 Pa despite stricter requirements for the addition of the reducing agent and temperature with decreasing atmospheric pressure. For improving vacuum reduction, CaO has been found to be a good additive for eliminating the negative impact of SiO2 on the reduction. The experimental verification test showed that after vacuum reduction of ferronickel slag at 1573 K for 3 h in the presence of 50 wt % Si and 30 wt % CaO at 10 Pa, the recovery of Mg reached 97.74%, much higher than that without addition of CaO (35.73%). These results confirmed the good viability of recovering magnesium from ferronickel slag by vacuum reduction in the presence of CaO.

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**Notes**
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