Thermodynamic analysis and experimental studies of magnesium extraction from szaibelyite—suanite ore by aluminium

by J.P. Peng*†, X.L. Wu*‡, Y.Z. Di*†, N.X. Feng*†, and S.G. Zhou*

Synopsis

Szaibelyite–suanite type ore is rich in magnesium oxide and boron oxide. We propose a new method to extract both metallic magnesium and valuable residues rich in boron by reduction using metallic aluminium.

Thermodynamic analysis of the reactions between aluminum and MgO, $Mg_2B_2O_5$, and $Mg_3B_2O_6$ was carried out. The effects of CaF_2 addition, temperature, mass of Al, and pellet formation pressure on the extraction of magnesium were also investigated experimentally.

The results indicate that the magnesium oxide phase can be displaced from $2MgO\cdot B_2O_3$ and $3MgO\cdot B_2O_3$ by CaO in the reduction system. The reduction ratio of magnesium oxide was 38% without calcium fluoride addition, increasing to 94% with 5.1% calcium fluoride. The reduction ratio increased with increasing temperature and mass of Al. To obtain a higher reduction ratio, the reduction pellet should be formed under a reasonable pressure.

Keywords
szaibelyite–suanite, magnesium, aluminum, thermodynamic analysis.

Introduction

Boron and its compounds are widely used in textiles, borosilicate glass, metallurgy, fertilizers, and nuclear shielding. Boron resources are mainly found in Turkey, the USA, Russia, and China. In Liaoning, China, there are abundant szaibelyite–suanite type ores that are rich in magnesium oxide and boron oxide. Szaibelyite and suanite ores are used to produce borax by a carbon dioxide–soda process. Annually, 2 Mt of waste materials containing 3–20% B_2O_3 and 2–25% MgO are produced during the process. It is necessary to make use of these waste materials containing high concentrations of boron oxide and magnesium oxide to avoid environmental problems as well as for economic reasons (Özdemir and Kıpçak, 2007).

Many studies have been performed on boron recovery from boron-containing waste materials (Özdemir and Kıpçak, 2007; Wang 2012; Özdemir and Kıpçak, 2003). Some studies have focused on the preparation of magnesium sulphate, refractory materials, and fire retardants. Metallic magnesium can be extracted from borax sludge by vacuum thermal reduction (Wu et al., 2009). Boron oxide remains in the reduction residue, and thus the residue can be used to produce non-alkali glass fibre.

Szaibelyite–suanite type ores can be used to produce metallic magnesium using calcium carbide (Peng et al., 2011), metallic silicon (Wu et al., 2011), and metallic aluminum as reducing agents. Furthermore, the reduction residue also contains the main components of non-alkali glass fibre.

In this study, thermodynamic and experimental investigations of magnesium extraction by metallic aluminum were carried out.

Raw materials

The szaibelyite–suanite ore was supplied by the Kuandian boron–magnesium plant in Liaoning Province, China. Table I lists the chemical composition of the ore. Figure 1 shows a comparison of the X-ray diffraction (XRD) patterns of the ore before and after calcination. The XRD patterns show that the raw ore contains mainly $MgCO_3$, $Mg_2B_2O_5$, and $MgBO_2(OH)$. After calcination, the product consists of MgO, $2MgO^aB_2O_3$, and $3MgO\cdot B_2O_3$.

Thermodynamic considerations

From a comparison of the XRD patterns (Figure 1), it is clear that MgO and $2MgO\cdot B_2O_3$ were generated through several important decomposition reactions during the calcination process:

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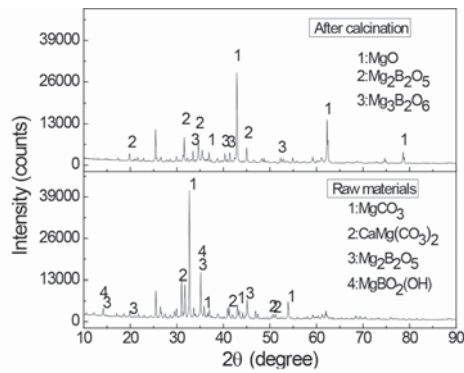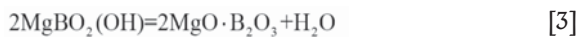# Thermodynamic analysis and experimental studies of magnesium extraction
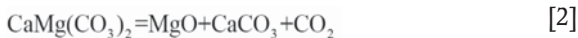

Figure 1—XRD patterns of the szaibelyite-suanite ore before and after calcination

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Table I

Chemical composition of the szaibelyite-suanite ore used in this study (wt%)

MgO	B_2O_3	SiO_2	CaO	Al_2O_3
44.67	26.08	6.18	3.19	1.43

$$MgCO_3 = MgO + CO_2 \quad [1]$$

$$CaMg(CO_3)_2 = MgO + CaCO_3 + CO_2 \quad [2]$$

$$2MgBO_2(OH) = 2MgO \cdot B_2O_3 + H_2O \quad [3]$$

The $CaCO_3$ generated from Equation [2] decomposes at high temperatures:

$$CaCO_3 = CaO + CO_2 \quad [4]$$

The existence of $3MgO \cdot B_2O_3$ proves that a reaction between MgO and $2MgO \cdot B_2O_3$ occurred:
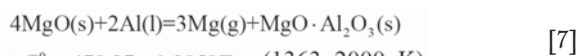
$$2MgO \cdot B_2O_3 + MgO = 3MgO \cdot B_2O_3 \quad [5]$$

According to Hauck and Muller (1979), the formation of $3MgO \cdot B_2O_3$ occurs easily at high temperatures, and the standard Gibbs free energy ΔG^0 (in kJ mol^{-1}) for Equation [5] can be calculated by

$$\Delta G_5^0 = -7.4 - 33.27 \times 10^{-3} T \quad [5']$$
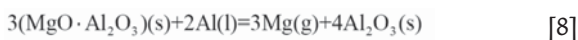
Based on thermochemical data (Liang and Che, 1993), thermodynamic analysis of the reduction of magnesium oxide by aluminium proceeds as follows:
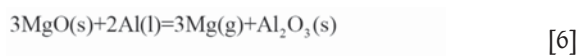
$$3MgO(s) + 2Al(l) = 3Mg(g) + Al_2O_3(s)$$
$$\Delta G^0 = 509.08 - 0.2911T \quad (1363-2000 \text{ K}) \quad [6]$$

$$4MgO(s) + 2Al(l) = 3Mg(g) + MgO \cdot Al_2O_3(s)$$
$$\Delta G^0 = 478.27 - 0.2958T \quad (1363-2000 \text{ K}) \quad [7]$$

$$3(MgO \cdot Al_2O_3)(s) + 2Al(l) = 3Mg(g) + 4Al_2O_3(s)$$
$$\Delta G^0 = 601.5 - 0.277T \quad (1363-2000 \text{ K}) \quad [8]$$

For $2MgO \cdot B_2O_3$ and $3MgO \cdot B_2O_3$, we first consider the following possible reactions:
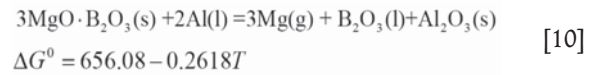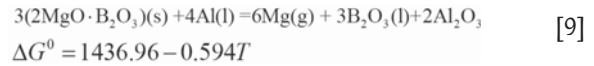
$$3(2MgO \cdot B_2O_3)(s) + 4Al(l) = 6Mg(g) + 3B_2O_3(l) + 2Al_2O_3$$
$$\Delta G^0 = 1436.96 - 0.594T \quad [9]$$

$$3MgO \cdot B_2O_3(s) + 2Al(l) = 3Mg(g) + B_2O_3(l) + Al_2O_3(s)$$
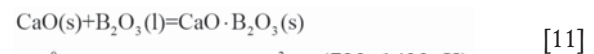$$\Delta G^0 = 656.08 - 0.2618T \quad [10]$$

The relationships between equilibrium magnesium partial pressure and temperature calculated from Equations [6], [7], [8], [9], and [10] are plotted in Figure 2. From Figure 2, it is evident that reaction [7] easily takes place because its critical reaction temperature is lower than that of reaction [6] under the same magnesium partial pressure. At temperatures below 2000 K, the Gibbs free energy change of reaction [8] is positive.
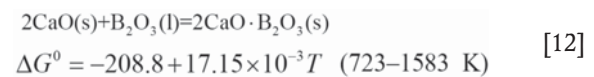
The boiling point of magnesium (1363 K) is lower than that of aluminium (2723 K). When the magnesium produced is removed as vapour, reactions [6] and [7] can be accelerated. In addition, reaction [8] can also occur when the equilibrium magnesium partial pressure is decreased, as shown in Figure 2. Hong *et al.* (1999) and Yang *et al.* (2006) proposed that the reduction of magnesium oxide by aluminium should take place in two stages: reaction [7] to produce magnesium vapour and $MgO \cdot Al_2O_3$ spinel and reduction (reaction [8]) of the spinel by aluminium.

However, Figure 2 indicates that the equilibrium partial pressures of magnesium for reactions [9] and [10] are less than 10^{-3} atm. below 1500 K. Thus, it is difficult to directly extract magnesium from $2MgO \cdot B_2O_3$ and $3MgO \cdot B_2O_3$.
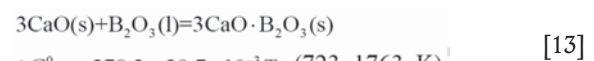
We propose to add $CaCO_3$ into the reduction system, which is converted to CaO at high temperatures, because some reactions between CaO and B_2O_3 can easily take place (Turkdogan, 1980):

$$CaO(s) + B_2O_3(l) = CaO \cdot B_2O_3(s)$$
$$\Delta G^0 = -149.4 + 30.96 \times 10^{-3} T \quad (723-1433 \text{ K}) \quad [11]$$

$$2CaO(s) + B_2O_3(l) = 2CaO \cdot B_2O_3(s)$$
$$\Delta G^0 = -208.8 + 17.15 \times 10^{-3} T \quad (723-1583 \text{ K}) \quad [12]$$

$$3CaO(s) + B_2O_3(l) = 3CaO \cdot B_2O_3(s)$$
$$\Delta G^0 = -278.2 + 29.7 \times 10^{-3} T \quad (723-1763 \text{ K}) \quad [13]$$

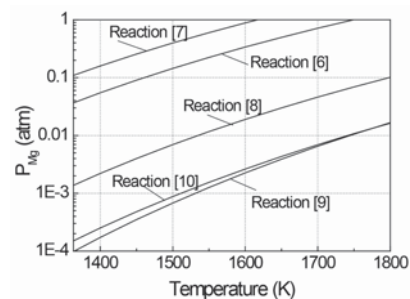Therefore, in the reduction system with CaO, we consider the following reactions:

Figure 2—Effect of temperature on the equilibrium partial pressure of magnesium

$$3(2MgO \cdot B_2O_3)(s) + 3CaO + 4Al(l) = 6Mg(g) +$$
$$3(CaO \cdot B_2O_3)(s) + 2Al_2O_3(s) \quad \Delta G^0 = 988.8 - 0.5011T \qquad [14]$$

$$3(2MgO \cdot B_2O_3)(s) + 6CaO + 4Al(l) = 6Mg(g) + \qquad [15]$$
$$3(2CaO \cdot B_2O_3)(s) + 2Al_2O_3(s) \quad \Delta G^0 = 810.6 - 0.5426T$$

$$3(2MgO \cdot B_2O_3)(s) + 9CaO + 4Al(l) = 6Mg(g) + \qquad [16]$$
$$3(3CaO \cdot B_2O_3)(s) + 2Al_2O_3(s) \quad \Delta G^0 = 602.4 - 0.5049T$$

$$2(3MgO \cdot B_2O_3)(s) + 2CaO + 4Al(l) = 6Mg(g) + \qquad [17]$$
$$2(CaO \cdot B_2O_3)(s) + 2Al_2O_3(s) \quad \Delta G^0 = 1013.36 - 0.4617T$$

$$2(3MgO \cdot B_2O_3)(s) + 4CaO + 4Al(l) = 6Mg(g) + \qquad [18]$$
$$2(2CaO \cdot B_2O_3)(s) + 2Al_2O_3(s) \quad \Delta G^0 = 894.56 - 0.4893T$$

$$2(3MgO \cdot B_2O_3)(s) + 6CaO + 4Al(l) = 6Mg(g) + \qquad [19]$$
$$2(3CaO \cdot B_2O_3)(s) + 2Al_2O_3(s) \quad \Delta G^0 = 755.76 - 0.4642T$$
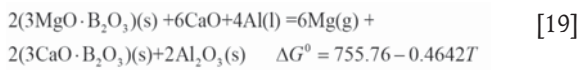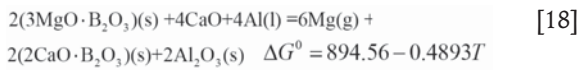
Figure 3 shows the relationships between equilibrium magnesium partial pressure and temperature calculated from Equations [14]–[19].

It is clear from Figure 3 that reaction [16] is most likely to occur because it has the highest equilibrium partial pressure of magnesium vapour. In addition, the figure shows that the equilibrium partial pressures of magnesium for reactions [14]–[19] are higher than 0.01 atm. at 1500 K; *i.e.*, these reduction reactions with CaO could easily occur under vacuum conditions.

Experimental procedure and apparatus

Figure 4 shows a flow diagram of the experimental procedure. First, szaibelyite–suanite powder of less than 125 μm in size and $CaCO_3$ were mixed prior to compaction into briquettes. The briquettes were calcined at 1273 K for 30 minutes in a resistance furnace to eliminate H_2O and CO_2.

The calcined products were then ground into powder of particle size less than 120 μm. A homogeneous mixture of the calcined product powder and aluminum powder was compacted into pellets 27 mm in diameter and about 10 mm in thickness. Pellet samples were then fed into a retort fitted with a condenser to collect metallic magnesium. The retort was heated to a given experimental temperature and held at this temperature for a given period. The pressure in the retort was controlled by a vacuum system. Figure 5 shows a photograph of the apparatus and a schematic diagram of the retort.

In the present study, experiments were performed at various operation conditions to study the effects of CaF_2 addition, temperature, and pellet formation pressure on the reduction ratio of magnesium oxide. In general, the pellet mass was 130–150 g.

Results and discussion

Table II lists the experimental conditions and results of the reduction experiments.

In Table II, the reduction ratio of magnesium oxide is used to estimate the level of the reduction process. It is defined as the ratio between the mass of magnesium extracted and the initial magnesium mass in the pellet used for reduction:

$$S = (m_0 - m_1)/m^* \times 100\% \qquad [20]$$

where m_0 is the pellet mass before reduction, m_1 is the mass of the residue after reduction, and m^* is the mass of magnesium contained in the pellet.

If only magnesium vapour is released from the pellet, the change in the pellet mass is taken as the mass of magnesium extracted. In fact, the magnesium products contained a low level of calcium. At high temperatures, calcium oxide in the pellet would also react with aluminum and form metallic calcium. Nevertheless, most of the metallic calcium would rapidly displace magnesium and form calcium oxide (Liang and Che, 1993):

Figure 4—Flow diagram of the experiments

Figure 5—Apparatus for the vacuum thermal reduction experiments. (A) Thermocouple, (B) heating elements, (C) pellets, (D) condenser, (E) water outlet, (F) water inlet, (G) to vacuum

Figure 3—Effect of temperature on the equilibrium partial pressure of magnesium in reaction systems with CaO
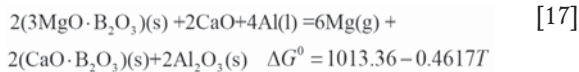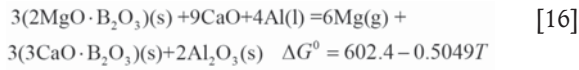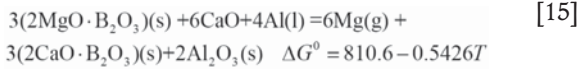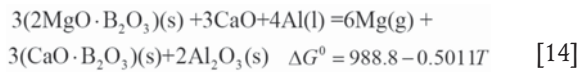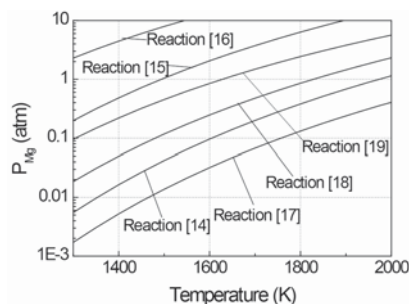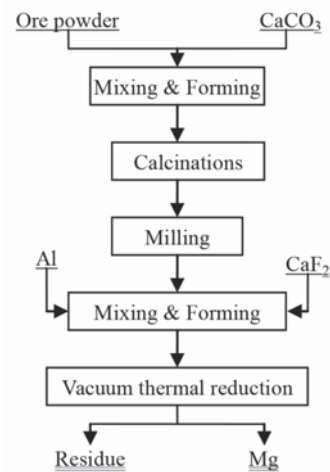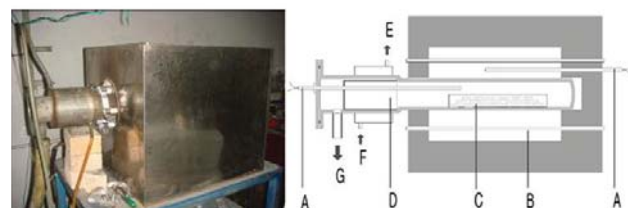
Thermodynamic analysis and experimental studies of magnesium extraction

Reduction ratios for experiments at various conditions

	Ore (g)	CaCO$_3$ (g)	Al (g)	CaF$_2$ (g)	Pelletizing pressure (MPa)	Temperature (K)	Holding time* (min)	Reduction ratio (%)
M-1	80	110	35	0	90	1473	0	16
M-2	80	110	35	0	90	1473	30	39
M-3	80	110	35	0	90	1473	60	50
M-4	80	110	35	0	90	1473	90	78
M-5	80	110	35	0	90	1473	120	94
C-1	80	110	18.3	0	90	1473	120	38
C-2	80	110	18.3	3.5	90	1473	120	58
C-3	80	110	18.3	4.1	90	1473	120	74
C-4	80	110	18.3	5.1	90	1473	120	94
C-5	80	110	18.3	5.3	90	1473	120	92
T-1	80	151	18.3	5.1	90	1273	120	3
T-2	80	151	18.3	5.1	90	1323	120	15
T-3	80	151	18.3	5.1	90	1373	120	30
T-4	80	151	18.3	5.1	90	1423	120	55
T-5	80	151	18.3	5.1	90	1473	120	95
A-1	80	110	16.6	5.1	90	1473	120	85
A-2	80	110	17.0	5.1	90	1473	120	88
A-3	80	110	17.5	5.1	90	1473	120	89
A-4	80	110	17.9	5.1	90	1473	120	92
A-5	80	110	18.3	5.1	90	1473	120	94
F-1	80	110	18.3	5.1	0	1473	120	83
F-2	80	110	18.3	5.1	45	1473	120	89
F-3	80	110	18.3	5.1	90	1473	120	94
F-4	80	110	18.3	5.1	135	1473	120	91
F-5	80	110	18.3	5.1	180	1473	120	88

*The holding time was recorded from when the temperature reached the required experimental temperature.

$$Ca(l) + MgO(s) = CaO(s) + Mg(g)$$
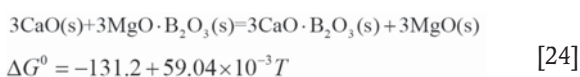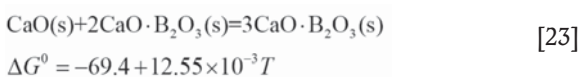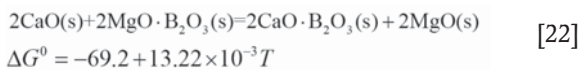$$\Delta G^0 = 92.55 - 97.42 \times 10^{-3} T \qquad [21]$$
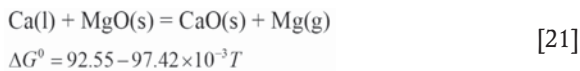
Hence, a very small amount of calcium is released from the pellet. The change in the pellet mass is taken as the mass of magnesium vapour because the mass of calcium is ignored. The error is likely to be less than 2%.

Role of CaCO$_3$ addition

An additional experiment was performed to study the behaviour of CaO during the calcination process. The material mixture consisted of 80.00 g ore powder and 151.10 g lime. The briquettes were formed under a pressure of 135 MPa for 2 minutes and were then calcined at 1273 K for 30 minutes.

Figure 6 shows XRD patterns of the mixed material after calcination. The products contained CaO, MgO, and Ca$_3$B$_2$O$_6$. Comparison with the components of the raw ore after calcination (see Figure 1) indicates that the following reactions occurred during the calcination process:

$$2CaO(s) + 2MgO \cdot B_2O_3(s) = 2CaO \cdot B_2O_3(s) + 2MgO(s)$$
$$\Delta G^0 = -69.2 + 13.22 \times 10^{-3} T \qquad [22]$$

$$CaO(s) + 2CaO \cdot B_2O_3(s) = 3CaO \cdot B_2O_3(s)$$
$$\Delta G^0 = -69.4 + 12.55 \times 10^{-3} T \qquad [23]$$

$$3CaO(s) + 3MgO \cdot B_2O_3(s) = 3CaO \cdot B_2O_3(s) + 3MgO(s)$$
$$\Delta G^0 = -131.2 + 59.04 \times 10^{-3} T \qquad [24]$$

Thus, the magnesium oxide phase is displaced from 2MgO·B$_2$O$_3$ and 3MgO·B$_2$O$_3$ by CaO to form 2CaO·B$_2$O$_3$ and 3CaO·B$_2$O$_3$. Therefore, magnesium can be extracted from szaibelyite–suanite ore by the addition of CaCO$_3$.

Effect of CaF$_2$ addition on the reaction

The Pidgeon process is the main method of magnesium metal production. In the process, calcium fluoride is added to the mixture of calcined dolomite and FeSi to improve the reduction ratio of magnesium oxide. In this study, a small amount of CaF$_2$ was also added into the mixture (see Figure 4). The effect of CaF$_2$ addition on the reduction ratio is shown in Table II.

For experiment C-1 without CaF$_2$, the reduction ratio of magnesium oxide is lower than for the experiments with CaF$_2$ under the same conditions. Figure 7 shows XRD patterns of the reduced residue from C-1. The chemical compositions of the residues from experiments C-1 and C-4 are listed in Table III. The residues contain mainly 3CaO·B$_2$O$_3$, 12CaO·7Al$_2$O$_3$, spinel (MgO·Al$_2$O$_3$), MgO, and Al. The existence of spinel proves that reaction [7] occurred. Calcium aluminate (12CaO·7Al$_2$O$_3$) forms by the reaction between calcium oxide and alumina:
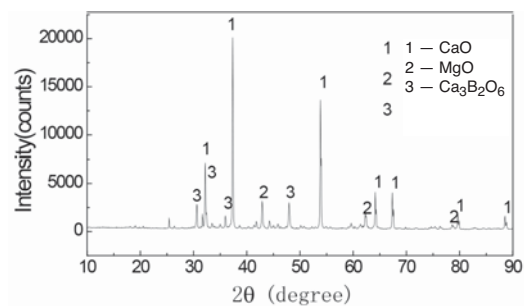

Figure 6—XRD patterns of the mixed material after calcination at 1273 K for 30 minutes

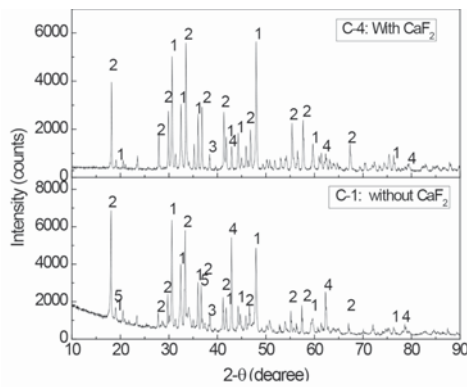# Thermodynamic analysis and experimental studies of magnesium extraction

Figure 7—XRD patterns of the reduction residues from experiments C-1 and C-4. (1) $Ca_3B_2O_6$, (2) $Ca_{12}Al_{14}O_{33}$, (3) Al, (4) MgO, and (5) $MgAl_2O_4$

Table III

Chemical compositions of the reduction residues (wt%)

	MgO	B_2O_3	SiO_2	CaO	$Al+Al_2O_3$	Others
C-1	16.72	14.52	3.2	52.04	13.07	0.45
C-4	3.49	14.7	3	56.42	21.89	0.5

$$12CaO+7Al_2O_3=12CaO\cdot 7Al_2O_3$$
$$\Delta G^\theta = -6.56-0.222T \qquad [25]$$
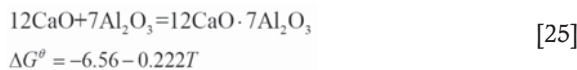
The formation of $12CaO\cdot 7Al_2O_3$ favours the reduction of MgO by aluminum, and the following reaction takes place:

$$12CaO(s) + 21MgO(s) +14Al(l) = 21Mg(g) +$$
$$12CaO\cdot 7Al_2O_3(s) \qquad \Delta G^\theta = 3557-2.26T \qquad [26]$$
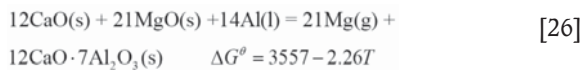
However, for experiment C-1, the high content of MgO in the residue verifies that the reduction ratio of magnesium oxide is low. The experimental results in Table II show that the reduction ratio of magnesium oxide increases markedly with increasing amounts of CaF_2, reaching 94% in experiment C-4.

Figure 7 also shows XRD patterns of the reduction residue from experiment C-4. Compared with the patterns of the residue obtained from experiment C-1, the XRD peaks of $MgAl_2O_4$, MgO, and Al for experiment C-4 are less intense. Thus, reactions [6] and [8] occurred.

Effect of temperature on the reaction

Table II also lists the results for experiments performed at various temperatures. The experimental temperatures were set to 1273–1473 K, which are higher than the melting point of pure aluminum in ambient atmosphere (933 K). Molten aluminum in the briquettes easily penetrates into the magnesium oxide phase. Furthermore, molten aluminum favours mass and heat transfer. Thus, the reaction between aluminum and magnesium oxide easily occurs.

The results show a remarkable improvement in the reduction ratio with increasing temperature. For experiment T-1 carried out at 1273 K, the reduction ratio is very poor. At a temperature of 1373 K (experiment T-3), the reduction ratio of magnesium oxide increases to about 30%. With increasing temperature, both the penetration rate of molten aluminum and the rate of release of magnesium vapour increase, so the reduction of MgO by Al is faster. At a temperature of 1473 K (experiment T-5), the reduction ratio is 95%, which is the highest reduction ratio achieved in the present study. Nevertheless, it must be pointed out that 151 g of calcium carbonate was used in experiment T-5. Under the same operation conditions, experiment C-4 achieved a 94% reduction ratio using only 110 g calcium carbonate (see Table II). Thus, experiment C-4 is considered more economical than experiment T-5.

Effect of the reactant stoichiometry on the reaction

According to Equation [7], the maximum reduction ratio of MgO is 75% because of the formation of $MgO\cdot Al_2O_3$. If all of the $MgO\cdot Al_2O_3$ reacts with Al to form Mg and Al_2O_3 (Equation [8]), the maximum reduction ratio of MgO is 100%. The molar ratio of Al: MgO must be higher than 2:3.

According to the chemical composition of the szaibelyite–suanite ore (Table I), 80 g of the ore contains 35.74 g of MgO, therefore the stoichiometric requirement of Al is 16.08 g. Figure 8 shows the results of experiments with various amounts of aluminum. With increasing amounts of aluminum, the contact area of MgO with aluminum particles increases and thus the reduction ratio of magnesium oxide improves.

Effect of pelletizing pressure on the reaction

The effect of the pelletizing pressure on the reaction is shown in Figure 9. For experiment F-1, the uncompacted material was fed directly into the retort for thermal reduction. The reduction ratio for experiment F-1 is 80%. By increasing the pelletizing pressure to 90 MPa, the contact area between aluminum and MgO increases, and thus the reduction ratio increases.

However, with increasing pelletizing pressure, the porosity of the pellet decreases. Lower porosity hinders the release of magnesium vapour and penetration by unreacted aluminum into the magnesium oxide phase. For this reason, the reaction rate slows and the reduction ratio decreases. Figure 9 shows that a reasonable pelletizing pressure is about 90 MPa.
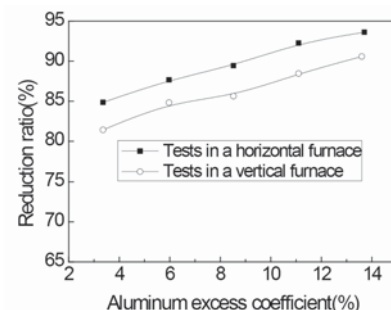

Figure 8—Effect of excess Al on the reduction ratio

Thermodynamic analysis and experimental studies of magnesium extraction
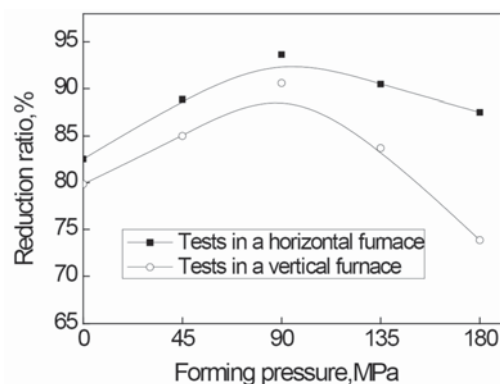

Figure 9—Effect of pelletizing pressure on the reduction ratio

Conclusions

Thermodynamic analysis of the reduction reactions of magnesium in MgO, $Mg_2B_2O_5$, and $Mg_3B_2O_6$ by aluminum was carried out. The effects of CaF_2, temperature, mass of Al, and pelletizing pressure on the reduction ratio were also investigated experimentally. The following conclusions can be drawn.

(1) It is difficult to directly extract magnesium from $2MgO \cdot B_2O_3$ and $3MgO \cdot B_2O_3$. With CaO in the system, the magnesium oxide phase can be displaced from $2MgO \cdot B_2O_3$ and $3MgO \cdot B_2O_3$ due to the formation of $2CaO \cdot B_2O_3$ and $3CaO \cdot B_2O_3$. Thus, most of the magnesium in szaibelyite–suanite ores can be extracted under reasonable conditions

(2) The reduction of magnesium oxide by aluminum occurs in two stages: the reaction to produce magnesium vapour and $MgO \cdot Al_2O_3$ and the reduction reaction between $MgO \cdot Al_2O_3$ and aluminum under vacuum conditions. The formation of $12CaO \cdot 7Al_2O_3$ favours the reduction of MgO by aluminum

(3) Calcium fluoride addition markedly improves the reduction ratio of magnesium oxide. The reduction ratio increases with increasing experimental temperature: from 3% at 1273 K to 95% at 1473 K

(4) Increasing the pelletizing pressure up to 90 MPa improves the reduction ratio from 83% (zero pressure) to 94%. Further increases in pelletizing pressure decrease the porosity of the pellets, hindering the release of Mg vapour and thus slowing the rate and extent of the reaction.

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