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KINETICS OF EXTRACTING MAGNESIUM FROM PREFABRICATED PELLETS BY SILICOTHERMIC PROCESS UNDER FLOWING ARGON ATMOSPHERE

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Abstract

The Pidgeon process is the main extraction method of magnesium, but its continuous production cannot be achieved due to the switch between vacuum and atmospheric pressure. Therefore, it is vital to realize continuous extraction of magnesium under atmospheric pressure. In this paper, the process of extracting magnesium from prefabricated pellets in flowing argon was proposed. The isothermal kinetic analysis of the reduction process was carried out. The results showed that the reduction process was controlled by diffusion process in 1 h, and the apparent activation energy of extracting magnesium from prefabricated pellets in flowing argon was 218.75 kJ/mol. Then the influence of experimental factors on the reduction rate was explored, including briquetting pressure, carrier gas flow rate, ferrosilicon content, reaction temperature and time. Through analysis and calculation, it was concluded that the main control step of diffusion process was silicon diffusion.

Keywords: *Isothermal kinetics; Flowing argon; Prefabricated pellet; Diffusion; Silicothermic process*

1. Introduction

Magnesium has a wide range of industrial applications, such as the preparation of titanium, zirconium, beryllium and other metal reducing agents [1], and hot metal desulfurizer [2]. Due to its high hydrogen storage capacity and electric energy density, it is also used as high-performance hydrogen storage materials [3, 4]. Magnesium alloy will become an ideal substitute for aluminum alloy due to its good specific strength, specific stiffness, and other properties, used widely in aviation, transportation, and 3C (computer, communications, and consumer electronics) industry [5-7]. The main extraction methods of magnesium include magnesium chloride smelting electrolysis and silicothermic process under high temperature and high vacuum.

Because of its low cost and simple technology, Pidgeon process has become the main method of magnesium smelting in China. However, with the progress of industrial technology, the Pidgeon process of extracting magnesium still remains challenging. In Pidgeon process, the raw material of extracting magnesium is MgO·CaO obtained by calcining dolomite. The calcined dolomite needs to be cooled at a certain temperature before mixing and briquetting, which wastes a lot of heat [8-9]. In addition, it is necessary to switch between vacuum and atmospheric pressure to ensure the loading of raw materials and the production of magnesium. Therefore, these processes result in high labor intensity, appalling working condition, and intermittent production [10-11]. Therefore, it is imperative to find an alternative extraction method of magnesium with low labor intensity, high efficiency, and continuous production.

Through the analysis, it can be seen that the continuous production of magnesium cannot be achieved due to vacuum smelting. Therefore, extracting magnesium under atmospheric pressure is the key to continuous smelting. Because the reaction can be continued by using flowing inert gas to carry magnesium vapor away from the pellet surface to reduce the partial pressure of magnesium, some researchers [12-17] had studied the extraction of magnesium by silicothermic reduction in flowing argon and it was proved that the reduction in argon flow was meaningful. Some researchers also put forward new technologies such as microwave smelting and thermal reduction of magnesium in seawater [18, 19], which have been in the exploration

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stage and did not solve the problem of continuous production. In this paper, the reduction process of prefabricated pellets [20] was proposed, which included first mixing dolomite, ferrosilicon, and fluorite to make pellets by pelletizing equipment, then reducing the pellets after calcination directly in flowing argon. Compared with traditional pellets, this process avoids the heat loss of high temperature MgO·CaO and connects the calcination and reduction processes. This paper is devoted to the isothermal kinetics of magnesium extraction under flowing argon, and to proving the type of elements diffusion, which provided a theoretical basis for the industrialization of continuous extraction of magnesium.

2. Experiments

2.1 Raw materials

Considering the high efficiency and comprehensive utilization of low-grade magnesite, magnesite and calcium carbonate were used instead of dolomite as raw materials for extraction of magnesium. Therefore, the experimental raw materials used in the research process were low-grade magnesite and ferrosilicon, as well as analytical-grade drugs, including calcium carbonate and calcium fluoride. Among them, ferrosilicon came from Henan, China, with 74.13% silicon content. Magnesite came from Liaoning, China. Its chemical composition is shown in Table 1.

Table 1. *Chemical composition of magnesite*

Ore	Ignition loss	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO
Magnesite/%	51.92	0.75	0.19	0.74	0.90	45.50

2.2 Apparatus

The experimental apparatus is shown in Fig. 1.

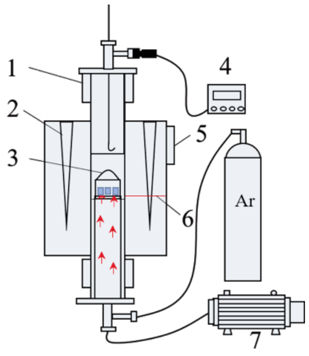

Figure 1. *Schematic diagram of the equipment; 1—Water jacket; 2—Silicon-Molybdenum bar; 3—Porous crucible; 4—Vacuum display instrument; 5—Control dial; 6—Thermocouple; 7—Vacuum pump*

The resistance furnace adopts the silicon molybdenum rod as the heating body, and the temperature can rise to 1600 °C. In addition, the furnace is equipped with a vacuum pump, which can remove the air in the furnace tube before putting the raw materials, and change in inert gas to reduce the experimental error.

2.3 Procedures

The magnesite and ferrosilicon were pulverized, respectively, and then mixed at a molar ratio of magnesite to calcium carbonate to ferrosilicon to be 2:2:1, and after that 3% of the total mass of calcium fluoride was added to the mix. The mixed powders were compacted into the cylindrical pellets with diameter of 15mm by the hydraulic pelletizing equipment with controllable pressure, and then the reduction of the pellets after calcination was carried out in the resistance furnace with flowing argon gas, and the reduced slag was weighed by electronic balance with a detection precision of 0.01 g. The content of magnesium in the slag was measured by inductive coupled plasma emission spectrometer (ICP-OES, Optima 8300DV, Perkin-Elmer, USA). The phase analysis and morphology observation were carried out by X-ray diffraction (XRD, Bruker D8, Germany) and scanning electron microscopy (SEM, su-8000, Japan), respectively. The reduction rate of magnesium and the utilization rate of silicon were calculated by equation (1) and (2), respectively.

$$\eta_{Mg} = \frac{M_1 \times \alpha - M_2 \times \beta}{M_1 \times \alpha} \tag{1}$$

$$\eta_{Si} = \frac{(M_1 \times \alpha - M_2 \times \beta) \times q_{Si}}{2q_{Mg} \times M_1 \times \theta} = \eta_{Mg} \cdot \frac{q_{Si} \cdot \alpha}{2q_{Mg} \cdot \theta} \tag{2}$$

where η_{Mg} and η_{Si} are the reduction rate of magnesium and the utilization rate of silicon, respectively, M_1 is the initial mass of prefabricated pellets, α is the magnesium contents of the prefabricated pellets, M_2 is the mass of magnesium slag, β is the magnesium content of magnesium slag, θ is the silicon content of prefabricated pellets, and q_{Mg} and q_{Si} are the atomic mass of Mg and Si, respectively.

2.4 Isothermal reduction method

In metallurgical process, isothermal operation is relatively difficult. Even though many researchers had made a lot of efforts, they were only close to isothermal operation and could not realize the real isothermal operation. Such as aluminothermic reduction of titanium oxide [21], the reacting substances were preliminarily placed in an alumina crucible and were preheated at a lower temperature. Before starting an experiment, the temperature was increased to the prescribed one as quickly as possible by applying full power of induction furnace. However, this method cannot control the temperature precisely through induction heating. Fu et al. [22] used two same furnaces to realize isothermal operation. The temperature of the first furnace was maintained at 1273K, and the temperature of the second one was set as the experimental temperature. The sample was put into the tube at room temperature and then the air in the tube was exhausted through argon. After the temperature of the tube reached 1273K, the tube was immediately changed to the second furnace to continue heating. The vacuum valve was opened when the temperature reached the prescribed one. The pressure in the tube was reduced to 4 Pa within 20 s by using the mechanical pump and the roots pump. After the experiment, the pressure in the tube was promptly recovered to 120 kPa by filling argon and the tube was taken out of the furnace to end the reduction. The method only realizes isothermal reduction in vacuum and the operation is complex.

In the present experiments, the whole experimental process was carried out under atmospheric pressure. Due to the difference between the temperature displayed in the furnace and the temperature at the pellet reaction position, in order to ensure the accuracy of the experimental data, the temperature of the pellet reaction position and the temperature of different positions in the furnace were remeasured by thermocouple. The temperature mentioned in this paper was the temperature remeasured by thermocouple. For the prefabricated pellets, they were first calcined at 1273K for 1h, and then the crucible containing the pellets was lifted 25cm away from the reaction position of the pellet (when the reaction position of the pellet was 1573K, the temperature at this position was 1173K). Then the resistance furnace continued to heat up. When the reaction temperature reached 1573K, the crucible was put back to its original position for reduction. Before the experiment, the temperature of different positions in the resistance furnace was measured when the temperature of reaction position was 1273K and 1573K respectively. It is assumed that the pellets are placed at point "0" (reaction position), so the relationship between the distance between point "0" and different positions in the resistance furnace and the temperature is shown in Fig. 2(a). In addition, XRD analysis was carried out on the pellets kept at 1273K for 2 h, as shown in Fig. 2 (b). It was found that there was no Ca_2SiO_4 phase in the product, so it was known that the reduction reaction would not take place at the temperature below 1273 K.

3. Results and discussion

3.1 Kinetic analysis

The pellets containing 15% of excess ferrosilicon were calcined at 1273 K for 1 h. The reduction experiments were carried out at 1473 K, 1523 K, 1573 K, and 1623 K for different times. Fig. 3(a) expresses the effects of reduction temperature and reduction

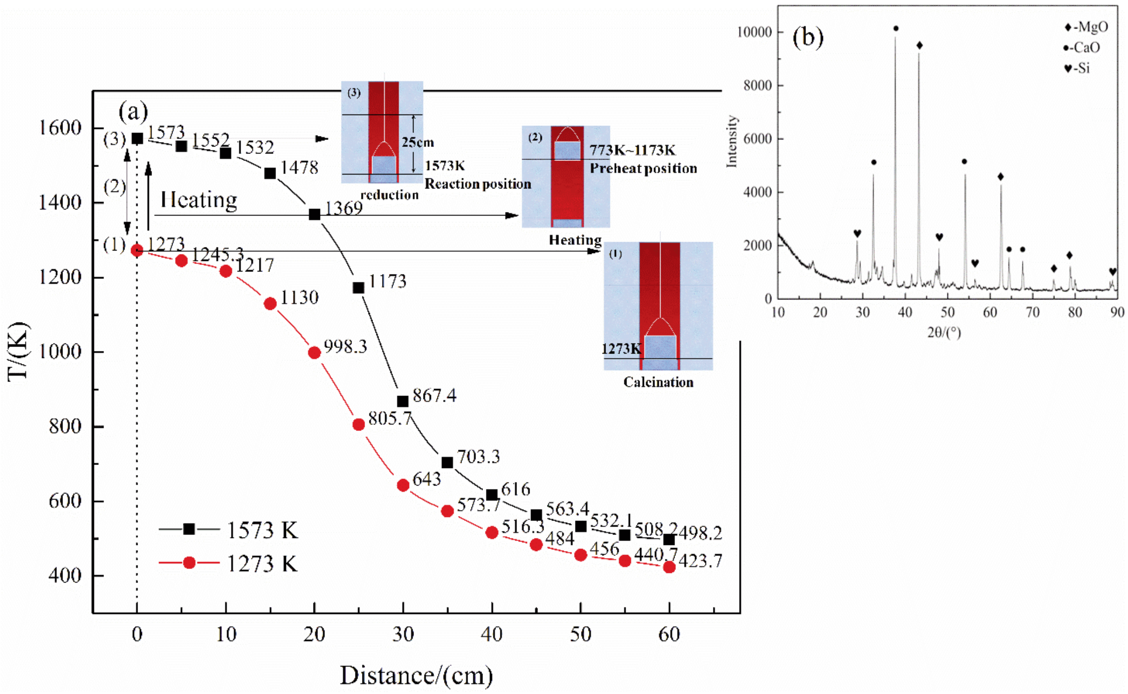

Figure 2. *(a) Relationship between temperature and distance between "0" point and different positions; (b) XRD analysis of pellets kept at 1273K for 2 h*

time on the reduction rate.

The kinetic formula is expressed as follows:

$$\frac{\mathrm{d}x}{\mathrm{d}t}=k(T)f(x) \tag{3}$$

where α is the reaction degree, which is defined by

$$x=\frac{\eta_t-\eta_0}{\eta_f-\eta_0} \tag{4}$$

where η_t is the reduction ratio at time t, η_f is the final reduction ratio at the end of the experiment, and η_0 is the initial reduction ratio. In the present case, $x=\eta_t$ can be obtained since $\eta_f=1$ and $\eta_0=0$. Integration of Eq. (5) gives the relationship between x and t. The functions F(x) are shown in Table 2.

$$F(x)=\int_0^x\frac{1}{f(x)}\mathrm{d}x=k(T)t \tag{5}$$

Table 2. *Different reaction models used to describe the reduction kinetics*

Number	Symbol	Kinetic characteristics of reaction process	Kinetic equation
1	R_1	Zero order reaction, Flat particles	x
2	R_2	Zero order reaction, Cylindrical particles	$1-(1-x)^{1/2}$
3	R_3	Zero order reaction, Spherical particles	$1-(1-x)^{1/3}$
4	F_1	First order reaction	$-\ln(1-x)$
5	D_1	One-dimensional diffusion	x^2
6	D_2	Two-dimensional diffusion	$(1-x)\ln(1-x)+x$
7	D_3	Three-dimensional diffusion (Jander)	$[1-(1-x)^{1/3}]^2$
8	D_4	Three-dimensional diffusion (G-B)	$1-2/3x-(1-x)^{2/3}$

The function F(x) in Table 2 is substituted into Eq. (5). The relationship between F(x) and t at different temperatures can get the fitting line by using the reduction rate in Fig.3 (a). According to the fitting lines, Adj. R-square values can be obtained. Fig.3 (b) shows the Adj. R-square values of the fitting lines. It can be found that the reduction stage can be well explained by the function D3. According to the kinetic models, the reduction stage is controlled by the diffusion process in 1 h.

According to the fitting results, the reduction rate of metal magnesium under different conditions was calculated, and the experimental value and calculated value were compared, as shown in Fig. 3 (a). Fig. 3 (a) shows that the calculated values of reduction rate are basically the same as the actual conversion rate of the reaction, which also shows that the selected kinetic model is accurate.

The activation energy of this reaction was calculated by plotting lnk with 1/T, based on the formula

$$k=A\exp\left(-\frac{E}{RT}\right) \tag{5a}$$

where A is the pre-exponential constant (s^{-1}), R is the gas constant (J/(mol·K)), and E is the apparent activation energy (J/mol). The intercept corresponds to ln$A$ and the slope corresponds to $-E/R$. The results are shown in Fig.3 (c). The apparent activation energy of extracting magnesium from prefabricated pellets in flowing argon atmosphere is 218.75 kJ/mol. The results found that the apparent activation energy of silicothermic reduction of the prefabricated pellets under argon atmosphere is lower than that of the traditional pellets, which is between 299 and 322 kJ/mol [13]. The result may be caused by the formation of Ca_2Si during the calcination of the prefabricated pellets. Above 1300 K, Ca_2Si is

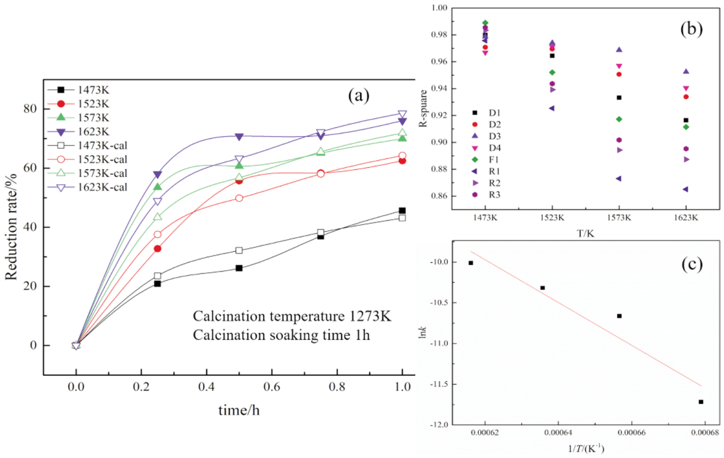

Figure 3. *(a) Reduction rate of magnesium in different times at different temperatures; (b) Adj. R-square values of the fitting lines; (c) Relationship between lnk and 1/T*

transformed into the liquid [23]. Therefore, the silicothermic process in the prefabricated pellets may be a solid-liquid reaction, which is conducive to the diffusion of Si and is beneficial to the reduction reaction.

Through kinetic analysis, it can be seen that the reduction reaction is controlled by diffusion, but it cannot determine which element diffusion controls the reaction process. Therefore, the effects of temperature, time, gas flow rate and ferrosilicon content on the reduction process of prefabricated pellets in flowing argon atmosphere were studied.

3.2 Effect of carrier gas flow rate on reduction rate

The gas flow rate directly affected the partial pressure of magnesium vapor at the reaction interface, so the effect of carrier gas flow rate on the reduction rate was explored. The experimental results are shown in Fig. 4. The results showed that when the argon flow rate increased from 0.1 m^3/h to 0.2 m^3/h, the reduction rate of magnesium increased by 5.23%. When the argon flow rate was 0.2 m^3/h, the reduction rate of magnesium was 70.37%, and the utilization rate of ferrosilicon was 70.12%. When the argon flow rate increased from 0.2 m^3/h to 0.5 m^3/h, the reduction rate of magnesium increased from 70.37% to 71.42%, with a small increase of 1.5%. In the reduction process, the magnesium vapor generated by the pellets diffused into the inert gas and was carried away from the reaction interface, resulting in the partial pressure of the magnesium vapor at the reaction interface being lower than the equilibrium partial pressure of the magnesium vapor, so the whole reduction reaction could occur. Due to the increase of carrier gas flow rate, the partial pressure of magnesium in the tube was decreased, and the escape of magnesium vapor from the reaction zone was not hindered, so the reduction rate of magnesium oxide was raised [24]. When the gas flow rate was higher than 0.2 m^3/h, the reduction rate of magnesium oxide would not continue to increase rapidly, which showed that the diffusion of magnesium was not the controlling step of the reaction.

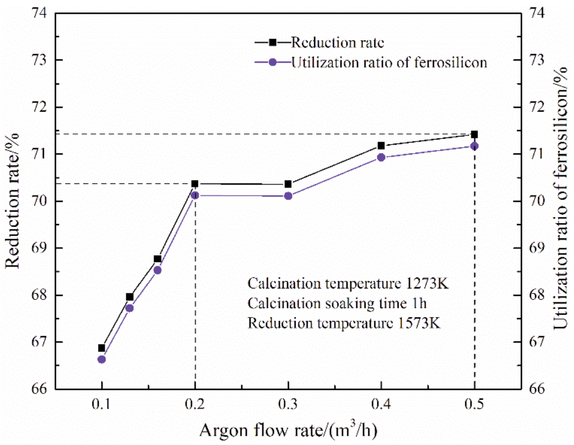

Figure 4. *Reduction rate of magnesium and utilization rate of silicon under different argon flow rates*

3.3 Effect of ferrosilicon content on reduction rate

The effect of the contents of ferrosilicon (assuming the molar ratio of Si to 2MgO is M, M = 1.00, 1.05, 1.10, 1.15, and 1.20) in the prefabricated pellets on the reduction process was investigated in the work. Fig. 5 shows the experimental results of calcination at 1273 K for 1h and reduction at 1573 K for 2h at argon flow rate of 0.2 m^3/h. The results showed that with the increase of ferrosilicon content, the reduction rate increased. When M was 1.15, the reduction rate was the highest, which was 72.28%. It could be inferred that this phenomenon was caused by

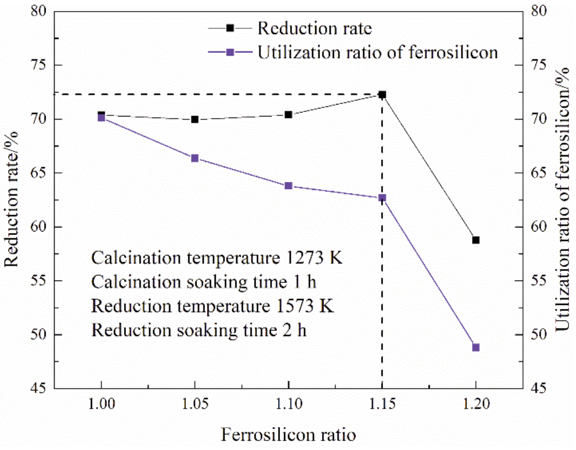

Figure 5. *Reduction rate of magnesium and utilization rate of silicon at different ferrosilicon content*

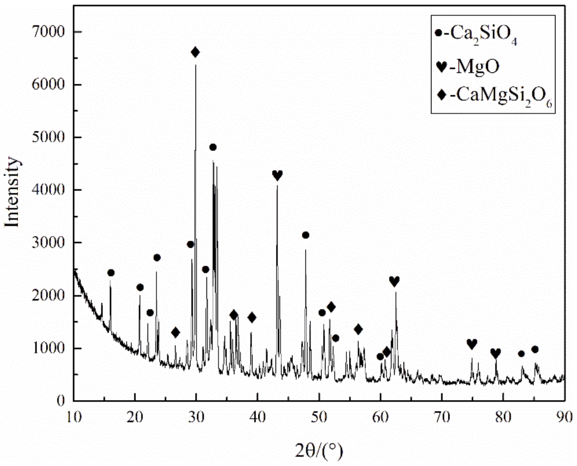

Figure 6. *Phase analysis of slag in excess of 20% ferrosilicon of the raw material*

Si diffusion. With the increase of ferrosilicon concentration, the number of Si atoms per unit volume in the pellets increased, the free path of atom migration decreased, and the rate of atom diffusion increased. Therefore, the reduction rate of magnesium increased in unit time. When the ferrosilicon content M was increased from 1.15 to 1.20, the reduction rate decreased to 58.75%. It could also be seen from the Fig. 5 that the utilization rate of silicon decreased with the increase of ferrosilicon content. The phase analysis of the reduction slag is shown in Fig. 6. The results showed that there was $CaMgSi_2O$ phase in the reduction slag. It could be inferred that when the amount of ferrosilicon was too large, MgO would participate in the slagging process, resulting that the reduction rate of magnesium greatly reduced.

3.4 Effect of temperature and time on reduction rate

The reduction rate of prefabricated pellets in the temperature range of 1473 K~1623 K for 0~4 h was investigated. Since the reduction reaction started when the furnace temperature rose to the set temperature, the reduction rate at the starting point of reduction reaction was 0. The experimental results are shown in Fig. 7. The results showed that the reduction rate changed rapidly in 1 h. At different temperatures, the reduction rates were 45.64%, 62.51%, 69.70%, and 76.18% in 1 h respectively. The reduction rates of 1473 K to 1523 K increased by 36.96%, and the reduction rates of 1523 K to 1573 K and 1573 K to 1623 K increased by 11.50% and 9.30%, respectively. The increasing degree of reduction rate decreased with the increase of reduction temperature. The reduction rate of 1623 K was about 1.7 times of 1473 K at 1 h, which indicated that the reduction rate increased with the increase of temperature. At different temperatures from 1473 K to 1623 K, the reduction rate increased by 33.64%, 21.15%, 9.66%, and 0.13%, respectively, from 1 h to 3 h. Because the reaction rate was faster in 1 hour and the content of reducing agent silicon in pellet was less at relatively high temperature, the reduction rate increased slowly with the increase of temperature after 1h.

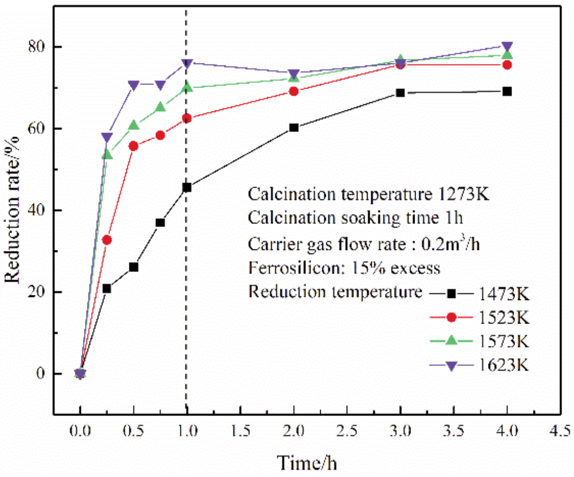

Figure 7. *Reduction rate of magnesium at different reduction temperatures and times*

It is assumed that the magnesium vapor diffused into the gas and mixed uniformly with the inert gas. The magnesium partial pressure in the inert gas is P_B, and the magnesium partial pressure on the pellet surface is P_S. The mass transfer process of magnesium vapor can be expressed as follows [14]:

$$j = \frac{M}{RT} K_c (P_B - P_S) \tag{6}$$

Equation (6) can be arranged into

$$\Delta P = \frac{RT}{Mk_c} \frac{N}{A} \tag{7}$$

where N is the generation rate of magnesium, g/s, A is the pellet surface area, m^2, k_c is the mass transfer coefficient, $\Delta P = P_B - P_S$.

Since the gas stream pass through an annulus formed by the cylindrical pellet and crucible, k_c is calculated by the following equation:

$$k_c = \frac{1.15D}{2r_s} (2.0 + 0.6 N_{Re}^{1/2} N_{Sc}^{1/3}) \tag{8}$$

The mass transfer process of magnesium vapor at 1473 K and 1573 K was calculated by equations (7) and (8), and the results are shown in Fig. 8. The results showed that when the reaction time was 0.25h at 1573 K, was 2.95 kPa, and when the reaction time was 4h, was 0.269 kPa. At the same reaction temperature, decreased with extension of time. The reason was that the concentration of reducing agent silicon in the pellet decreased and the reaction speed decreased with the reaction going on, which resulted

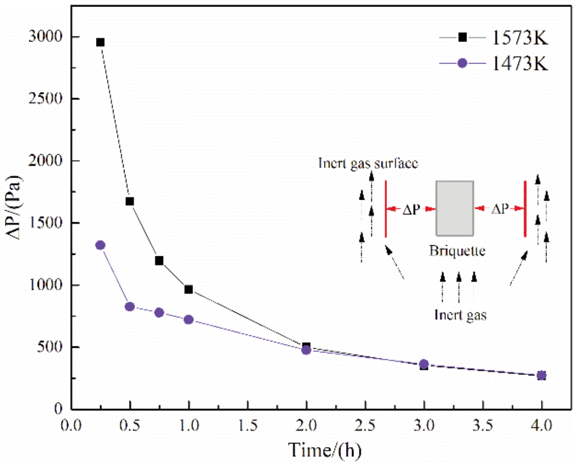

Figure 8. *Calculated differential pressure (ΔP) of magnesium vapour on the surface of briquette and bulk phase at 1473 K and 1573 K*

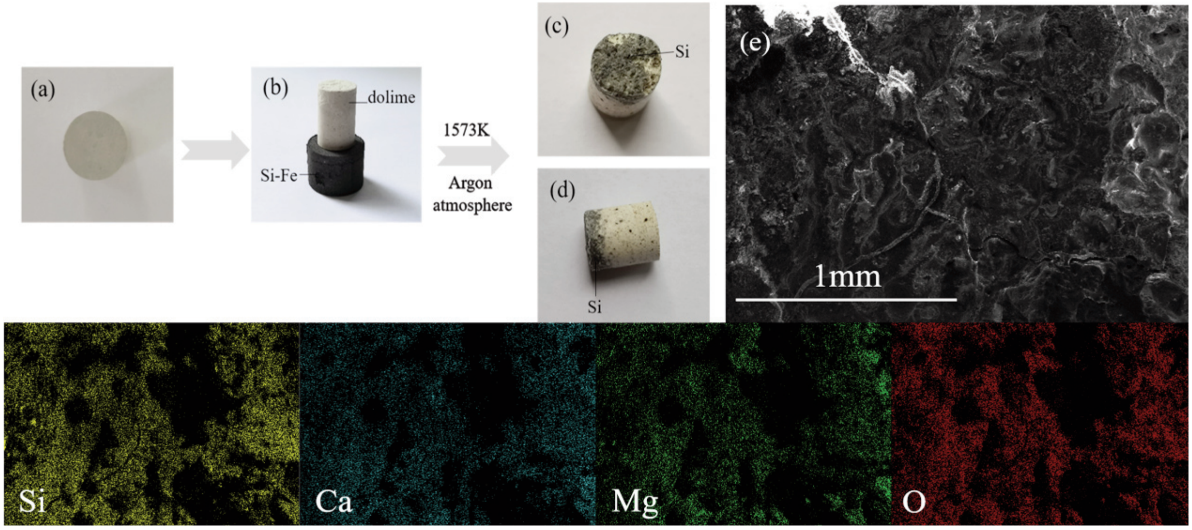

Figure 9. *Photos of dolime and Si during the experiment; (a) dolime briquette; (b) briquette stacking diagram; (c) and (d) dolime briquette after reaction; (e) EDS and SEM of the surface of the dolime briquette contacting with Si-Fe*

in the decrease of magnesium partial pressure on the surface of pellets. It could also be seen from Fig. 8 that at 1473 K was less than at 1573 K in the early stage of the reaction, because the diffusion of silicon atom accelerated with the increase of temperature and the reaction speed at 1573 K was faster, so the magnesium partial pressure on the pellet surface was higher. Through the above analysis, it was proved that the reaction control step was the diffusion of Si.

In order to further verify the diffusion of Si, dolime and Si-Fe were briquetted respectively, and the briquettes were then placed as shown in Fig. 9 (b). They were heated at 1573 K for 3 h with flowing argon. After the experiment, the dolime briquette was separated from Si-Fe briquette, the photos of dolime briquette are shown in Fig. 9 (c) and (d). It could be seen from the figure that the surface of the dolime briquette contacting with Si-Fe briquette changed from white to black and became rough, at the same time, black could be seen on the side of cylindrical dolime briquette. SEM and EDS analysis were carried out on the dolime surface which contacted with ferrosilicon after the reaction, as shown in Fig. 9 (e). The results showed that besides Ca, Mg, and O, there were a lot of Si elements on the surface of dolime pellets. Therefore, it could be inferred that the black on the surface of dolime was caused by silicon, so it was proved that Si diffused into dolime.

Conclusion

In order to solve the problem of continuous production of magnesium, the extraction of magnesium from prefabricated pellets under flowing argon gas was developed. In the work, the isothermal kinetic analysis of the reduction process was carried out. The experimental results are as follows:

(1) The process of extracting magnesium by prefabricated pellets in flowing argon gas can be well explained by the function D3, that is, the reduction stage was controlled by diffusion process, and the apparent activation energy value was 218.75 kJ/mol.

(2) The apparent activation energy of silicothermic reduction of the prefabricated pellets under argon atmosphere was lower than that of the traditional pellets, which was beneficial to the reduction reaction.

(3) The diffusion control step of reduction reaction was controlled by the diffusion of Si.

Acknowledgments

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KINETIKA EKSTRAKCIJE MAGNEZIJUMA IZ GOTOVIH PELETA SILIKOTERMIČKIM POSTUPKOM U PROTOČNOJ ATMOSFERI SA ARGONOM

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[b] Glavna laboratorija za ekološko iskorišćenje minerala koji se sastoje od više metala (Ministarstvo obrazovanja), Severoistočni univerzitet, Šenjang, Kina

Apstrakt

Pidgeon proces predstavlja glavnu metodu za ekstrakciju magnezijuma, ali njegova kontinuirana proizvodnja se ne može postići zbog prelaska iz vakuuma na atmosferski pritisak. Zbog toga je realizacija kontinuirane ekstrakcije magnezijuma od vitalnog značaja. U ovom radu je predložen postupak za ekstrakciju magnezijuma iz gotovih peleta u protočnoj atmosferi sa argonom. Izvršena je izotermalna kinetička analiza procesa redukcije gde su rezultati pokazali da je postupak redukcije kontrolisan postupkom difuzije u roku od jednog sata, a da je energija aktivacije ekstrakcije magnezijuma iz gotovih peleta u protočnoj atmosferi sa argonom iznosila 218,75 kJ/mol. Nakon toga je ispitan uticaj sledećih eksperimentalnih faktora na brzinu redukcije: pritisak prilikom briketiranja, protok gasa nosača, sadržaj ferosilicijuma, temperatura i vreme reakcije. Analiza i proračun su pokazali da glavni kontrolni korak prilikom procesa difuzije predstavlja difuzija silicijuma.

Ključne reči: *Izotermalna kinetika; Protočna atmosfera sa argonom; Gotov pelet; Difuzija; Silikotermički postupak*