

Full length article

Magnesium production by carbothermic reduction in vacuum[☆]

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Abstract

In present work, we investigate production of magnesium by carbothermic reduction under vacuum conditions. The process was divided into two parts, one is reduction process, and the other one is condensation process. The experimental results revealed that during reduction process, the gas–solid reaction between MgO and CO was not occurred at a temperature and pressure of 1723 K and 30–100 Pa respectively. So the main reduction reaction was $\text{MgO}_{(s)} + \text{C}_{(s)} = \text{Mg}_{(g)} + \text{CO}_{(g)}$ (under vacuum) and reaction type belonged to solid–solid reaction. In Condensation Process, according to a contrast and analysis, the condensation quality of magnesium is associated with CO concentration. The resultant product C was formed and it followed magnesium vapor condensation which prevents mutual combination of two metal droplets to forms the compact condensation produces. Therefore, in order to compact morphological forms of magnesium crystal whiskers, we must control the technical conditions and find the method to separate the magnesium vapor and carbon monoxide. That's the key factor to get better crystalline structure. Copyright 2015, National Engineering Research Center for Magnesium Alloys of China, Chongqing University. Production and hosting by Elsevier B.V. All rights reserved.

Keywords: Metal magnesium; Carbothermic reduction; Condensation; Vacuum

1. Introduction

Generally, magnesium is produced by two main processes: electrolysis of molten magnesium chloride and thermal reduction of magnesia [1]. Electrolysis is the pre-dominant

route in western countries, accounting for about 77 pct of western countries total production, however, this process is characterized by high-energy requirements [2]. In recent years, production of magnesium in China by the Pidgeon process has dominated world production. In this process, ferrosilicon reduces magnesia from calcined dolomite under vacuum. The process suffers from high energy usage and low productivity [3]. Our research group is exploring the carbothermic reduction of magnesia area of study for many years, and the process of carbothermic reduction and magnesium vapor condensation were the important link to solve the problem of industrialization. The reaction between MgO and C may provide a new route toward the production of magnesium. But for the carbothermic reduction reaction type, one point of view was that the main reaction is solid–solid

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reaction, the other was that the main reaction is gas–solid reaction [4–6].

Qu analyzed the uniform nucleation of water from nucleation rate and randomness, while investigating magnesium vapor condensation process. Zhang theoretically studied the gas bubble nucleation of molten metal. Zeng evaluated the properties of metastable equilibrium, and the mechanism of gas bubble nucleation through thermodynamics and kinetic theory. Izmailov studied the statistical mechanics of nucleation. Ring researched the reason of nano-clusters nucleation. Leubner established nucleation and growth model. In Lüm-men's paper, the uniform nucleation of supersaturation iron vapors has been investigated by molecular dynamics simulation method [7–15]. Therefore, analysis of the magnesium vapor nucleation in theory is helpful to find the key factors which affect the way of magnesium vapor condensation. In addition the reactions of carbothermic reduction process were discussed. In order to investigate the mechanism of the carbothermic reduction process in vacuum, XRD analysis was also proposed.

2. Reduction process

2.1. Raw material

Analytical-grade magnesia and carbon were used as the raw materials in our experiments.

2.2. Schematic diagram of vacuum furnace

The experiments were carried out in a self-made vacuum furnace shown in Fig. 1.

2.3. Experimental procedure

The raw materials, magnesia (20 g) and carbon (12 g) grounded to –140 mesh after drying were mixed in a molar ratio ($MgO/C = 1/2$). The mixed materials were compacted into pieces of $\Phi 30\text{ mm} \times 10\text{ mm}$ dimensions under 6 MPa. The compacts were placed in a graphite crucible and heated at the heating rate of $15\text{ K} \times \text{min}^{-1}$. The system temperature needed to be kept at 1723 K for 1–2 h under vacuum. At high

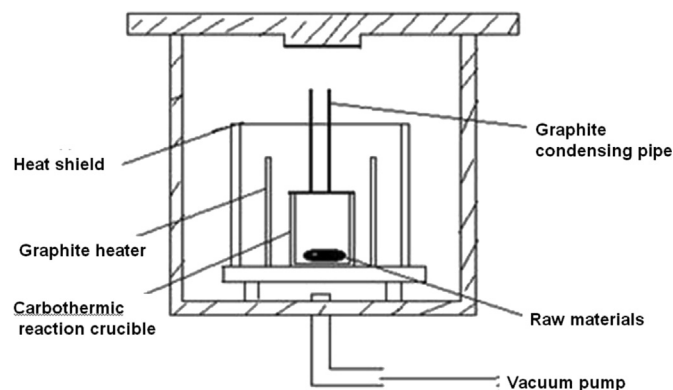


Fig. 1. Schematic diagram of vacuum furnace.

reduction temperature, Mg vapors and CO were produced and moved into the upper condensing towers. The temperature of the condensing surfaces was kept between 773 and 973 K. The reversion reaction occurred during the condensing process.

2.4. Analysis method

The crystalline phase of the resulted products was identified by X-ray diffraction (XRD) instrument (D/max-3B) using $\text{Cu K}\alpha$ radiation with a scanning rate of $2\text{ (}^\circ\text{)}/\text{min}$ which was made by Rigaku Corporation of Japan.

The mass loss ratio (α) is defined as

$$\alpha = \frac{M_0 - M_1}{M_0} \times 100\% \quad (1)$$

where M_0 is the mass at the start of the reduction reaction and M_1 is the reaction equilibrium mass at the end of reduction. The term $M_0 - M_1/M_1$, represents the reduction degree of magnesia available for reaction.

2.5. Analysis of experimental results

Carbothermic reduction was carried out under the following conditions: $MgO:C = 1:2$ (molar ratio), temperature 1073–1773 K, hold temperature time 1–2 h, system pressure 30–100 Pa, compacting pressures 6 MPa of the blend MgO . During the thermo gravimetric reduction process, the mass loss of the sample was monitored as a function of time. It was calculated by formula (1). The photographs of materials ($MgO + C$) before and after reaction at 1723 K are shown in Fig. 2. A typical mass-loss percentage vs. temperature curve of reduction of magnesia by carbon is shown in Fig. 3.

Fig. 2 shows that the structure of briquetted materials was changed into porous and loose after reduction reaction, which revealed that the solid–solid reaction occurred between MgO and C. It can be noticed that there was no sharply mass loss detected when the temperature was below 1553 K as shown in Fig. 3. The values below 15% indicate that there was not any reduction reaction. When the temperature was higher than 1553 K, the sample weight changes sharply with increase of temperature, and there was an obvious inflection point of mass loss at 1553 K. Therefore, this temperature was considered as initial reaction temperature. It accorded with the theoretical calculation. It can also be seen that the maximum of mass loss reached 75% in the experimental temperature range, the solid–solid reduction reaction was completely finished under the experimental condition.

The reaction between MgO and CO may provide a new method toward investigation of gas–solid reaction. A stomatal dish was added in the carbothermic reaction crucible to divide reduction zone into two parts. The pieces of MgO were placed on the Al_2O_3 flats (above the stomatal dish) in order to avoid contacting the graphite, so that the reaction between MgO and crucible(C) was not occurred. Under the stomatal dish, magnesia and carbon as the starting materials were mixed in a molar ratio ($MgO/C = 1/2$) with addition of 5% CaF_2 and

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