The Feasibility Analysis of Decrease the Reverse Reaction Rate in the Carbothermic Reduction of Magnesia in a Vacuum

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***Abstract*—In the present work, the behavior of un-coagulable CO was experimentally investigated during the condensation process of carbothermic reduction of magnesia at condensing zone temperatures ranging from 923 K to 1223 K. Magnesium powders and magnesium lump condensates were produced under different conditions and characterized by scanning electron microscopy and energy-dispersive X-ray spectroscopy. The reverse reaction products C and MgO were formed following the process of magnesium vapor condensation and we just found the nearer the temperature of the condensation zone approached the liquid transition temperature, the lower the rate of the reverse reaction between CO and magnesium vapor. So, decreases in the rate of the reverse reaction of magnesium were possible by controlling the condensation temperature in experiments.**

Keywords-Magnesium Vapor, Vacuum, Reverse reaction, Carbothermic reduction.

I. INTRODUCTION

At present, metal magnesium smelting methods mainly include electrolysis and thermal reduction methods [1]. Based on the findings of various studies, such as those by Zhong [2], Li [3], Tian [4], Yu [5], we found that condensation temperature and temperature gradients have a significant impact on the condensation rate of magnesium vapor. The condensation temperature and pressure in a vacuum system are difficult to control effectively because of experimental equipment restrictions. In order to avoid the reversion reaction, Hori points that thermal control of the product gases is important throughout the process from the reaction chamber to the product collection point via the nozzle [6-7]. Now Tassios's invention involves the manner of heated nozzle. The invention heat is supplied to the nozzle over-and-above any heat that is supplied to the nozzle by gas flow [8]. In this work, the feasibility of decrease the rate of reverse reaction by control the phase transition and formation of magnesium crystals in a vacuum system were discussed.

II. THEORETICAL ANALYSIS

Under a vacuum pressure of 60 Pa, a number of possible reversion reactions can take place during cooling of an Mg/CO/inert gas mixture:

Gas-phase reversion reaction:

$$Mg_{(g)}+CO_{(g)}=MgO_{(s)}+C_{(s)} \quad (1)$$

Liquid-phase reversion reaction:

This work was financially supported by Science Research Fundation of Education Department of Yunnan Province, China (No. 2010C257).

$$Mg_{(l)}+CO_{(g)}=MgO_{(s)}+C_{(s)} \quad (2)$$

Solid-phase reversion reaction:

$$Mg_{(s)}+CO_{(g)}=MgO_{(s)}+C_{(s)} \quad (3)$$

The Gibbs free energies of reactions (1), (2), and (3) at different temperatures and pressures were evaluated, and the results are reported in Figure 1. Thermodynamically, these reversion reactions were exothermic reaction, the gas-phase reversion will commence as soon as a saturated gas mixture was cooled. When magnesium steam enters the condensation area, so long as the condensation temperature at the temperature of magnesium gas-liquid transition is controlled, the rate of the reverse reaction should decrease because the reaction between CO and liquid-magnesium has a larger free energy barrier that must be overcome.

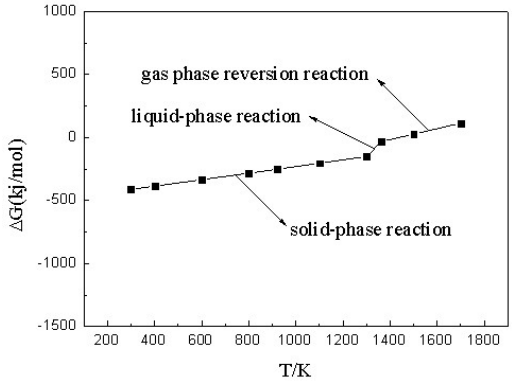


Fig. 1 Free energy changes related to the temperature of reactions (1), (2), and (3) at 60 Pa

III. Experimental

A. Raw material.

Carbothermic reduction process: analytical grade of magnesia, carbon were used as the raw materials in experiments.Vacuum condensation process: binary vapors CO and magnesium vapor cooling at the multistage condenser.

B. Condensation equipment.

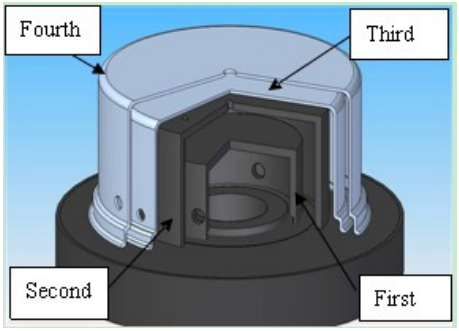


Fig.2 The longitudinal section of the multistage condenser

We have materials vacuum distilling by using internally heated vacuum furnace at 60 Pa, the longitudinal section of the multistage condenser shown in figure 2.

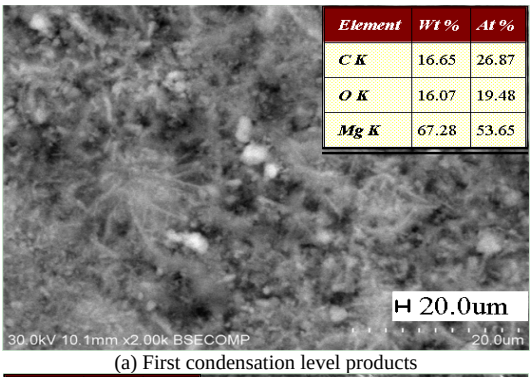
The temperature gradient between evaporation area and the first condensation area of the multi-level condensing collection devices is ΔT_1 =7 K/mm, temperature gradient between the first level and the second is ΔT_2 =6 K/mm, temperature gradient between the second level and the third is ΔT_3 =0.5 K/mm, and temperature gradient between the third level and the last is ΔT_4 =0.3 K/mm Where T_{min} is the minimum of the condensation temperature.

IV. Analysis Methods

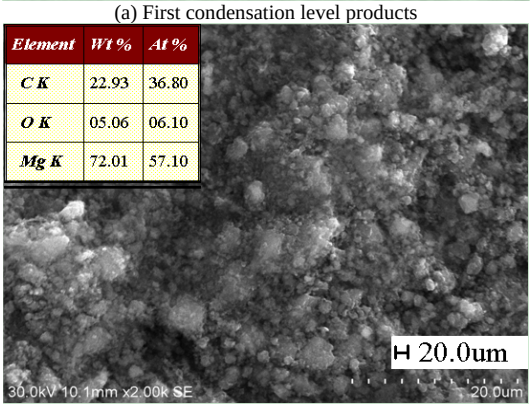
Surface morphology of the condensing product was characterized by scanning electron microscopy (XL30ESEM-TMP, Phillips, Holland).

V. Results and Discussion

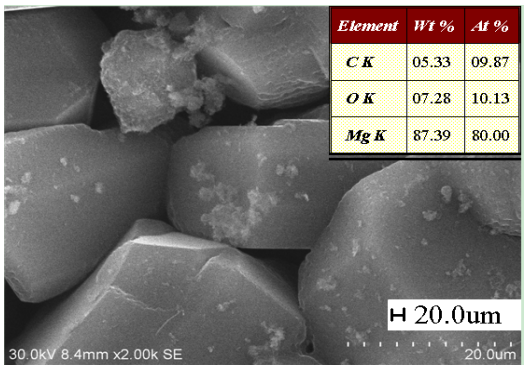
As shown in Figure 3, the crystal morphologies and impurity contents of the magnesium crystals varied with decreasing condensation temperature and gradient. In the first layer, where a large amount of heat exchange and heat loss occurs because of the large gradient, the magnesium vapor crossed the liquid phase and directly condensed into solid powder. Thus, we obtained flocking floe magnesium, which has a higher degree of oxidation. Bulk magnesium began to appear in the second condensation layer but some flocking floe products remained. We obtained the best condensation products from the third layer, which the phenomenon of super-saturation steam through liquid phase uniform growth is even more apparent after the gas-liquid phase transformation happened, as well as clear crystal boundary, compact structure without obvious oxidation. Bulk magnesium was still obtained but it was oxidized by CO in the last layer.



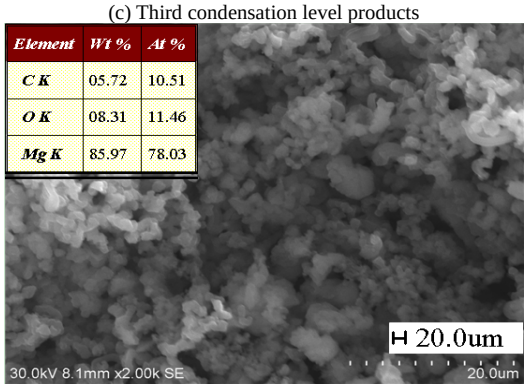
(a) First condensation level products



(b) Second condensation level products



(c) Third condensation level products



(d) Fourth condensation level products

Fig. 3 SEM image of condensed products when T_{min} = 923 K from the first to the fourth (a to d) condensation level

The difference of reverse reaction rate was showed from energy-dispersive X-ray spectroscopy in figure 3, that is, the levels of magnesium decreased in the first layer of the condenser, which had the largest temperature gradient, but increased in the third layer of the condenser, where the condensation temperature approached the liquid transition temperature.

Thus, control of the condensation temperature and the gradient in the condensation zone tend to change the rate of the reverse reaction of magnesium.

VI. Conclusion

Controlling the temperature of condensation zone approach to the temperature of liquid transition and a smaller temperature gradient not only decrease heat loss but also increase the liquid nucleation rate and improve the magnesium steam concentration. Consequently, crystal quality is also improved. Setting a mild temperature gradient helps avoid rapid solidification while extending the fusion time of the magnesium clusters. Under our experimental conditions, when the pressure of system at 60 pa, the third condensation level's condensation temperature at about 890 K, the condensed products was obtained at last.

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