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Full length article

Production of magnesium during carbothermal reduction of magnesium oxide by differential condensation of magnesium and alkali vapours[☆]

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Abstract

Most of researchers believed that the developments on the condensation of magnesium produced by carbothermic reduction just concentrated on two process routes: the “quench” route and the “solvent” route. But this paper will briefly analyzes the major challenges in magnesium vapor condensation during the vacuum carbothermic reduction of calcined dolomite, on equipment upgrade, heat transfers alter, to achieve condensation control and production collection. Solutions are then proposed using theoretical calculations and experiment results. Comparative analysis of the experiment results shows that the burning and even explosion of condensation products during the vacuum carbothermic reduction of calcined dolomite are mainly due to the burning of crystallized powder magnesium, which results from the self-ignition of alkali metals. Finally, this paper proposes a multistage condensation solution to improve traditional vacuum condensation equipment. And result show that the condensation equipment can effectively mitigate the burning and loss during condensation, also the morphology of the condensation products clearly improved, the grain size increased, and the oxidation rate decreased. The potassium/sodium vapor and the magnesium vapor were separately condensed.

Keywords: Carbothermic reduction; Calcined dolomite; Magnesium vapor; Condensation

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Carbothermic reduction is an alternative to both silicothermic and electrolytic processes for the production of magnesium. It has the advantages of low reductant cost, high equipment utilization rate, and environment-friendliness compared to the first two techniques. The current domination of world production by the Pidgeon process mainly reflects the economic conditions of China rather than any inherent advantages of the Pidgeon process over electrolytic routes. Under environment pressure, this process has always been favored by a great deal of researchers [1−3]. On condensation of magnesium vapor during Carbothermic reduction aspect, Hansgirg [4] tackled the problem of reversion and consolidation of fine magnesium using a wide variety of condensation techniques including quenching the magnesium vapor with water-cooled pipes and mechanically scraping the condensate from the tubes, shock cooling through

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injection of a cold gas, chilling with hydrocarbon oils and subsequently distilling magnesium from the slurry formed. All of these approaches failed to overcome the key problems of flammable powders and effective consolidation of fine powders, though his papers and numerous patents provide a thorough record of process development in this area and are essential reading for any researcher contemplating carbothermic production of magnesium. Cameron [5,6] and co-workers further investigated this route at the pilot plant level in the1980s, concentrating on the reduction stage of the process and providing useful thermodynamic analysis of impurity distributions between slag and vapor phases. However, the experiment was announced as a failure because the magnesium powders blocked the pipelines. Researchers of the CSIRO recently made a breakthrough in a semi-industrial experiment [7]. They used a Laval nozzle to create an environment where a supersonic gas is cooled rapidly at more than 10^6 °C S^{-1} [8–10], facilitating homogeneous nucleation and condensation of the magnesium vapour [11,12].

Meanwhile, our research group National Engineering Laboratory for Vacuum Metallurgy achieved significant progress in the carbothermic reduction of magnesia, which paved the way for the industrial applications of magnesium metal production using the vacuum carbothermic reduction method. Li [13] and Luo [14] conducted studies on the vacuum carbothermic reduction of magnesite and lateritic nickel ores, respectively. Li [15], Tian [16] and Yu [17] also made significant progress in reaction chamber spraying, catalytic mechanisms, and improvements in the reduction rate. At present, our experimental studies focus on the vacuum carbothermic reduction of calcined dolomite. However, the condensation of magnesium vapor fails to meet the collection requirements because of the small grain size of the obtained magnesium metal. In addition, the high oxidation rate easily leads to burning or even explosion, which significantly increases the damage rate due to burning of magnesium and reduces production safety. Therefore, the effective control of magnesium vapor condensation has become the key factor to the successful application of this method to industrial production. This paper is to probe into the reason of the problem, and try to resolve.

1. Theoretical analysis

1.1. Heat input to the condensing zone

Fig. 1 shows that the heat in the reaction zone (high-temperature A) is radiated into the condensing zone (low-temperature B) in three ways:

(1) The reaction zone radiates heat to the condensing zone through coupling sleeves (graphite condensing pipe C). Heat radiation is the major heat-transfer process at high temperatures. In addition, heat can be transferred through a vacuum without requiring any material media;

(2) Superheated magnesium vapor radiates heat when it enters the condensing zone and is condensed into solid magnesium; and

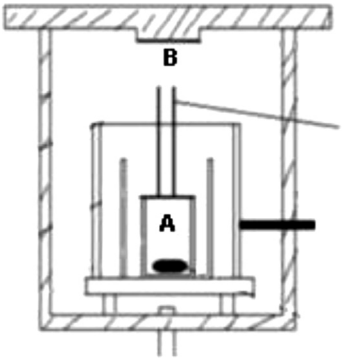

Fig. 1. Schematic model of a vacuum furnace.

(3) Heat is radiated to the condensing zone through the crucible surface in the reduction zone. Given that the crucible in the entire reduction zone is sealed and wrapped by an insulation quilt, only coupling sleeve C is connected to the condensing zone. Therefore, compared with the two previously described input processes, this process is not considered here.

Assume that high-temperature zone A is the surrounded object and condensing zone B is the peripheral object. Then [18],

$$Q_{1-2} = C_{1-2} A_1 \left[\left(\frac{T_1}{100} \right)^4 - \left(\frac{T_2}{100} \right)^4 \right].$$

Therefore, $C_{1-2} = \dfrac{C_0}{\left[\frac{1}{\varepsilon_1} + \frac{A_1}{A_2} \left(\frac{1}{\varepsilon_2} - 1 \right) \right]}$

where C_{1-2} is the emission coefficient, C_0 is the blackbody emission coefficient, ε_1 is the blackness of the surrounded object, ε_2 is the blackness of the peripheral object, A_1 is the radiation area of the surrounded object, A_2 is the radiation area of the peripheral object, T_1 is the temperature in the reaction zone, and T_2 is the temperature in the condensing zone. When the condenser size is specified and the temperature exceeds 1000 °C, then A_1, A_2, ε_1, and ε_2 can all be considered as fixed values [19], and T_1 is the only variable. By satisfying the technological conditions as well as the small variation range of the temperature, the reduction temperature T_1 contributes to the relative stability of the radiation heat in the condensing zone.

1.2. Heat released by the condensation of magnesium vapor into solid magnesium

The condensation of magnesium vapor into solid magnesium involves five steps [20]. The heat that enters the condensing zone is then calculated based on the five-step process. The five steps are as follows:

(1) The heat released by the cooling of the superheated vapor to the boiling temperature is denoted as Q^{I}:

$$Q^{\mathrm{I}} = C_{\text{vapor}}(t_s - t_b) \cdot m = 0.204(1350 - 1090) \cdot m = 53.04m$$