

Microstructure Characteristic and Phase Evolution of Refractory Siderite Ore during Sodium-carbonate-added Catalyzing Carbothermic Reduction

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Abstract: Thermodynamic analysis of refractory siderite ore during carbothermic reduction was conducted. Microstructure characteristics and phase transformation of siderite ore during sodium-carbonate-added catalyzing carbothermic reduction were investigated. X-ray diffraction (XRD), scanning electron microscopy and energy-dispersive analysis of X-rays were used to characterize the reduced samples. Results indicate that the solid reaction between FeO and SiO₂ is inevitable during carbothermic reduction and the formation of fayalite is the main hindrance to the rapid reduction of siderite. The phase transformation of present siderite ore can be described as: siderite-magnetite-metallic iron, complying with the formation of abundant fayalite. Improving the reduction temperature (≤ 1050 °C) and duration is helpful for the formation and aggregation of metallic iron. The iron particle size in the reduced ore was below 20 μm , and fayalite was abundant in the absence of sodium carbonate. With 5% Na₂CO₃ addition, the iron particle size in the reduced ore was generally above 50 μm , and the diffraction intensity associated with metallic iron in the XRD pattern increased. The Na₂O formed from the dissociation of Na₂CO₃ can catalyze the carbothermic reduction of the siderite. This catalytic activity may be mainly caused by an increase in the reducing reaction activity of FeO.

Key words: siderite ore; microstructure characteristic; phase evolution; catalyzing; carbothermic reduction

With the continuous and rapid development of iron and steel industries around the world, many countries are facing the risk of a shortage in raw materials for the industry. Especially in China, the production of iron ore is seriously deficient, with a self-sufficiency rate less than 50%, and the annual imported iron ore reaches 619 Mt^[1,2]. Thus, to mitigate the risk of iron ore shortages, efficient exploitation of domestic iron ore becomes inevitable. The resource reserve of iron ore in China is abundant; however, most of them are either containing high content of harmful impurities (sulphur and phosphorus) or of low quality grade^[3,4]. This makes it very difficult to obtain high grade iron concentrates with a qualified level of harmful elements using traditional beneficiation methods, e. g. , magnetic separation, floatation and gravity concentration. Hence,

the cheap and abundant iron reserves have remained largely unexploited.

Nonetheless, siderite ore (FeCO₃) is a valuable raw material used in the production of high-quality pig iron and steel. The ore has a stable chemical composition, which is easily reduced to strongly magnetic mineral, and has a high tensile strength in the roasted state^[5,6]. Metallurgical approach is usually employed to process siderite ore. During this process, the siderite mineral phase can be transformed to strongly magnetic mineral phase, and then the strongly magnetic mineral grows and reconstructs, while the magnetism of gangue minerals changes little. This promotes magnetic separation of the iron and slag effectively. However, when the raw ore contains large amounts of acidity gangue minerals (e. g. , SiO₂, Al₂O₃), the conversion and reconstruction of

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magnetic minerals becomes very difficult^[7-9].

Up to now, many studies can be found in literatures. The studies are related to reduction in composite of iron oxides and carbon, and mainly focus on the effects of alkali metal oxides. It was established that the reduction sequence can be described as: $\text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4 \rightarrow \text{FeO} \rightarrow \text{Fe}$, and the reduction of wustite to metallic iron was verified to be the slowest step during the reduction process. Geva et al.^[10] investigated the critical influence of potassium on the reduction of wustite. It was demonstrated the potassium markedly increased the rate of reduction of wustite and changed the iron properties in subsequent reduction. Huang et al.^[11] also studied the reduction of iron oxides of red mud reinforced by Na_2CO_3 and CaF_2 . The results showed that the carbothermic reduction of hematite can be reinforced by adding Na_2O , and the mass and heat transfer during the reducing reaction can be optimized by adding CO_2 . Additional, similar conclusions have been drawn by Bahgat et al.^[12], Li et al.^[13] and Guo et al.^[14]. The results indicated that the alkali metal oxides played an important role in the reduction of iron oxides. However, little work has been performed related to the influence of alkali metal oxides on the reduction of siderite ore. The main reasons for this lie in the low grade of the raw ore and the complicated phase change associated with siderite^[15-17]. Consequently, investigating the reduction of siderite is critical for the utilization of refractory siderite ore.

The objective of the present study was to conduct the thermodynamic analysis of refractory siderite ore during sodium-carbonate-added carbothermic reduction and to investigate the main operating parameters affecting the microstructure characteristics and phase evolution of siderite during catalyzing carbothermic reduction (CCR). The reduction of this ore containing pulverized coal and sodium carbonate was studied. More attention was paid to the effect of sodium carbonate and its dosage on the reduction of siderite, and the mechanism of CCR of siderite ore was also investigated using scanning electron microscopy (SEM), X-ray diffraction (XRD) analysis and energy dispersive analysis of X-rays (EDS).

1 Experimental

1.1 Raw materials

The chemical analysis of raw ore is shown in Table 1. The mineralogical analysis result of the sample indicates that the iron ore predominantly consists of siderite and a small quantity of iron min-

erals in the form of magnetite and daphnite. The gangue mineral is mainly quartz. The results of industrial and fusibility analyses and the reactivity of pulverized coal were listed in previous study^[9]. The solid Na_2CO_3 used as the alkaline additive in the experiments was of reagent grade (Tianjin Chemical Plant).

Table 1 Chemical analysis of raw ore mass%

TFe	P	S	SiO ₂	Al ₂ O ₃	CaO	MgO	Na ₂ O
31.64	0.50	0.36	27.62	3.68	2.63	1.88	0.18

1.2 Experimental procedure

Reduction experiments were performed in a furnace with temperature controlled within ± 5 °C. Raw ore (50.0 g) was mixed with an appropriate amount of pulverized coal and sodium carbonate for every experiment. The mixture was placed in a graphite crucible ($\phi 60$ mm \times 80 mm). The sample powders were loosely filled in the crucible and the depth of mixture before reaction accounts for about 2/3 of the graphite crucible's depth. Upon the completion of experiments, the samples were taken out from the furnace and cooled down to ambient temperature with N_2 protective atmosphere (1 L/min).

1.3 Analysis and characterization

The reduced ores were divided into two portions. One portion was crushed and milled below 75 μm for XRD analysis, and the other portion was for microtopographic analysis. The reduced samples used for SEM-EDS were fracture surfaces and the evaluated region was the most dense metal region. The microtopographic analysis were conducted by the Kunming Institute of Precious Metals. The XRD patterns of the raw material and the reduced samples were acquired with a Rigaku diffractometer using $\text{CuK}\alpha$ radiation, and the scanning angle varied from 10° to 90° (2θ) at a speed of 1.2 (°)/min. The microstructure of the reduction product was observed with a scanning electron microscope (FEI Holland, Philip).

2 Results and Discussion

2.1 Thermodynamic analysis of refractory siderite ore during carbothermic reduction

The thermodynamic analysis of the main chemical reactions occurring during carbothermic reduction is shown in Fig. 1. It can be concluded that the solid reaction between FeO and SiO_2 is inevitable during

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