



Phase evolution of tin, iron and calcium oxides roasted in a simulative sintering atmosphere



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ABSTRACT

An investigation on the behavior of SnO₂ in the tin-bearing iron concentrates during the reduction sintering process (RSP) has been conducted by the authors' group. It was found that only about 30 wt.% of tin was removed under the optimal conditions. However, the underlying reason why it was so difficult to remove tin by the RSP was not understood. In this study, the phase evolution of tin oxide, iron oxide and calcium oxide under simulative sintering conditions was investigated by varying the gas compositions, sintering temperature and basicity (CaO/SiO₂). The results indicated that the Ca-Sn oxides were prior formed at 900 °C–1000 °C in the reduction atmosphere with CO / (CO + CO₂) = 10–40 vol.%, while the phases of Fe-Sn spinel (Fe_{3-x}Sn_xO₄) and Ca-Fe-Sn oxides were easily generated as the temperature increased to 1100 °C–1200 °C. The phases of Fe_{2-4x/3}Sn_xO₃ and Ca-Fe-Sn-Si oxides began to generate at 1100 °C in an oxidative atmosphere with O₂/(O₂ + CO₂) = 25–100 vol.%. In addition, increasing alkalinity accelerated the formation of Ca-Sn oxides under reduction atmosphere, which brought about an adverse effect on the tin removal in the sintering process.

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1. Introduction

Tin-bearing iron ore, with the reserve more than 0.5 billion tons, is one of typically complex iron ore resources in China, [1]. Because cassiterite is closely embedded and fine-grained dissemination in the iron ores, it is difficult to separate tin from them via physical mineral processing methods [2–4]. After multistage beneficiation process, the tin content in the final iron concentrates is still in excess of 0.1 wt.%. Steel production practice has proved that it is impossible to remove tin from the raw materials in the subsequent ironmaking and steelmaking processes, which causes the mechanical performance deterioration of the products. Therefore, these tin-bearing iron concentrates can't be used as ironmaking burdens directly [5–7]. Great efforts have been made to utilize the tin-bearing iron concentrates. For instance, selective sulfurization and chlorination roasting processes are effective to separate tin from the concentrates, but the environmental pollution and equipment corrosion are unavoidable [2]. Another selective reduction volatilization process of tin recovery and pellet preparation for blast furnace iron-making from the tin-bearing iron concentrates, namely a coal-based rotary kiln direct reduction (DR) process, has been developed and successfully performed in the pilot-scale and semi-industrialization tests by the authors' group [4–8]. However, the coal-

based rotary kiln DR process is not productive enough to utilize the huge reserve of the tin-bearing iron ores.

It is known that iron ore sintering and pelletizing are the predominant processes in the iron-making industry [9–13]. It was also reported that reduction sintering process (RSP) was effective to remove nonferrous metals, such as Pb and Zn, from iron-bearing dusts collected by iron and steel works [12]. The use of pre-reduced agglomerates as the BF burdens can reduce CO₂ emission [13]. In addition, previous investigation suggested that part of tin could be removed from the tin-bearing iron concentrates by the RSP [2]. It seems that the RSP is a promising way to utilize the tin-bearing iron concentrates. Nevertheless, the RSP has still not been put into practice. Especially, there is no knowledge on the removal behavior of tin oxide from the tin-bearing iron concentrate by the RSP.

An investigation on the removal behavior of tin in the RSP of the tin-bearing iron concentrates has been performed by the authors' group. Tin-bearing iron concentrates with iron grade of 64.48 wt.% and tin content of 0.23 wt.% were used as raw material. In addition, the main impurities compositions included 4.40 wt.% SiO₂, 2.49 wt.% CaO, 0.87 wt.% Al₂O₃ and 0.69 wt.% MgO [14]. The results indicated that only about 30 wt.% tin was removed from the tin-bearing concentrates under the optimal sintering conditions for the RSP. The tin-bearing phases which remained in the finished sinters exist in the form of stannic (Sn⁴⁺), and stannous (Sn²⁺) compounds and metallic Sn (Sn⁰). However, it is difficult to determine the detailed phase compositions of tin in the sinters because of the trace amount of Sn in the raw materials.

Previous researchers have reported the main phase compositions in the slag by reduction smelting processes [15–19]. However, little

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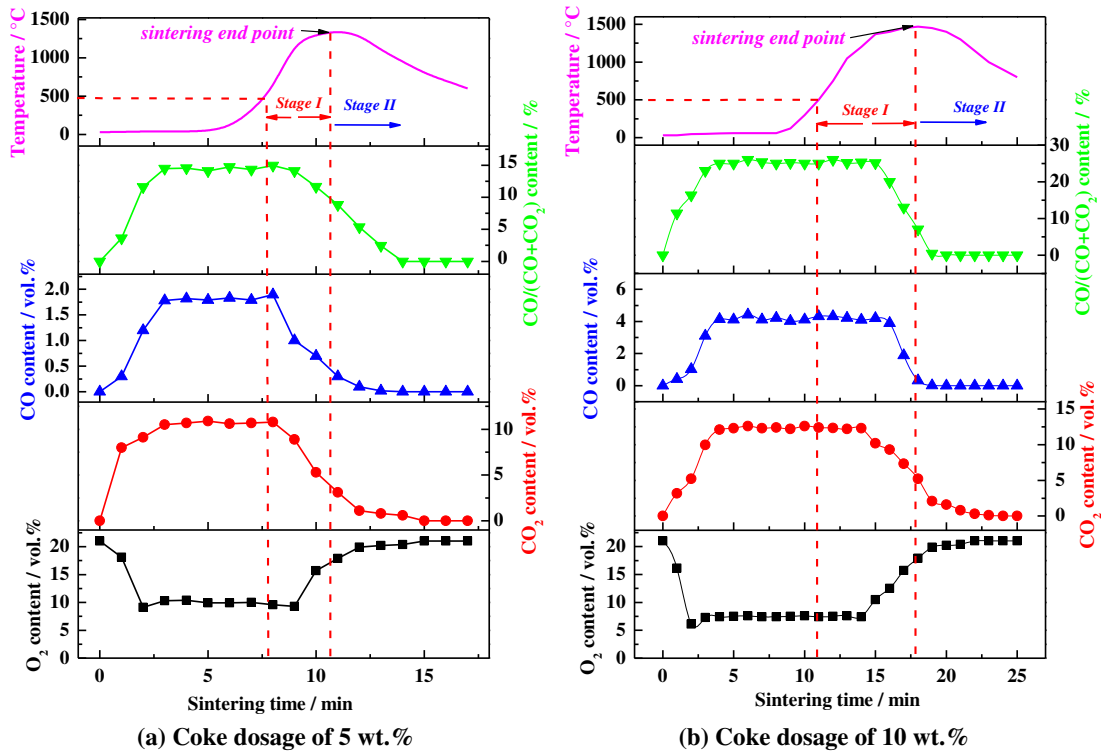


Fig. 1. Changing rules of O₂, CO, CO₂, CO/(CO + CO₂) content and the temperature in the sinter layer.

information related to solid state reactions among the tin oxide, iron oxide and calcium oxide was reported. In order to determine the phase evolution of cassiterite in the tin-bearing iron concentrates during the RSP, main constituents of CaCO₃, SiO₂ and Fe₃O₄ were used as raw materials in this study. The effects of sintering temperature, atmosphere and basicity ($R = \text{CaO}/\text{SiO}_2$) on the phase transformation of SnO₂ were investigated in a simulative sintering atmosphere.

2. Materials and method

2.1. Materials

The testing samples were analytical reagents including Fe₃O₄, SiO₂, CaCO₃ and SnO₂ (Aladdin, Shanghai), the purities of which were beyond 99.9 wt.%.

All of the reagents were pre-ground until the particle size passed through a 0.074 mm sieve. It was known from the previous sinter pot experiments that the volatilization ratio of Sn was affected significantly by the basicity (CaO/SiO_2) [14]. Therefore, the variation in basicity of the raw materials was taken into consideration. The powder samples consist of Fe₃O₄, SnO₂, CaCO₃, and SiO₂ and the mole ratio of Fe₃O₄:SnO₂:CaCO₃:SiO₂ for each test with the adjusted basicity ($R = \text{CaO}/\text{SiO}_2$) was 2:1:1:1 ($R = 1$), 2:1:2:1 ($R = 2$) and 2:1:3:1 ($R = 3$), respectively.

The purity of gases (CO, CO₂, O₂ and N₂) was higher than 99.99 vol.%.

2.2. Methods

2.2.1. The determination of sintering atmosphere and temperature

The roasting parameters were determined based upon the results of previous sintering pot tests. In the sintering process, the flue gas components in the sinter layers with different coke dosages (5 wt.% to 15 wt.%) were analyzed with a flue gas analyzer (MRU-VARIO PLUS, Germany).

Changing curves of the flue gas and temperature in the sinter layer with 5.0 wt.% and 10.0 wt.% coke are shown in Fig. 1. As presented in Fig. 1-(a), the O₂ content of the flue gas decreased obviously from 21.0 vol.% to 9.0 vol.% at the ignition stage then gradually increased and

maintained around 10.0 vol.%. After the ignition stage, the CO, CO₂ and CO/(CO + CO₂) content of the outlet flue gas was stable at 1.5–2.0 vol.%, 10.0–11.0 vol.% and 14.5–15.0 vol.%, respectively. The sinter bed temperature increased sharply to 1350 °C approaching the sintering end-point, and then CO, CO₂ and CO/(CO + CO₂) content decreased to nearly zero while the O₂ content increased rapidly to 21.0 vol.%.

The sintering time increased obviously when the coke dosage increased from 5.0 wt.% to 10.0 wt.% as presented in Fig. 1. The changing of the flue gas presented in Fig. 1-(b) was very similar to that shown in Fig. 1-(a). In addition, the content of O₂, CO, CO₂ and CO/(CO + CO₂) in RSP (coke dosage of 10.0 wt.%) was 7.0–8.0 vol.%, 4.0–4.5 vol.%, 12.0–12.5 vol.% and 24.5–25.5 vol.%, respectively.

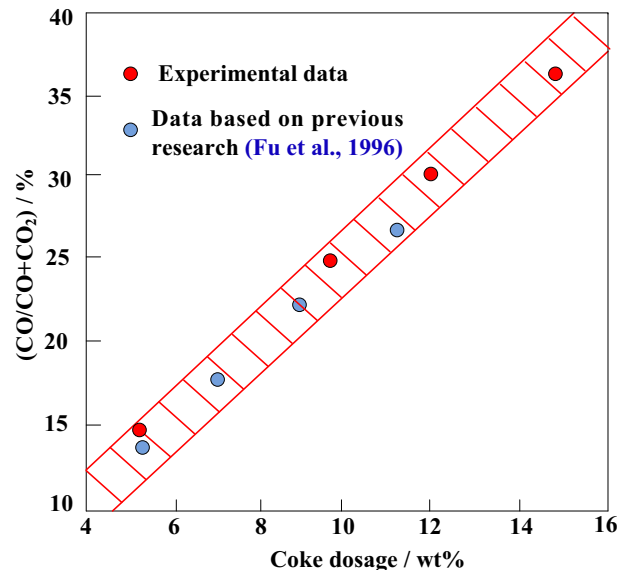


Fig. 2. Effect of coke dosage on the CO/(CO + CO₂) content of the sintering flue gas. [19]

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