Journal of Alloys and Compounds 613 (2014) 102–106

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jalcom

Preparation of reduced iron powder using combined distribution of wood-charcoal by microwave heating

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ARTICLE INFO

Article history:
Received 28 April 2014
Received in revised form 26 May 2014
Accepted 4 June 2014
Available online 12 June 2014

Keywords:
Microwave heating
Combined distribution of wood-charcoal
Reduced iron powder
Wood-charcoal

ABSTRACT

In this paper, the influences of microwave heating with wood-charcoal as the reducing agent, on the reducing characterization of mill-scale were systematically investigated. The microstructures of the samples were characterized before and after microwave heating using SEM. The SEM analysis results indicated that the high-grade reduced iron powders were prepared using microwave heating. It was concluded that microwave heating technologies can be applied effectively and efficiently to the reduction process processes of iron ore concentrate.

1. Introduction

Iron powder with high quality is an indispensable raw material for many fields, such as powder metallurgy [1–3], magnetic materials [4], metal welding cutting, coating [5,6], wastewater treatment [7,8], medicine and food [9–11]. Its demands have been increasing stably with the booming development of auto industry.

Reduced iron powder [12,13] and atomized iron powder [14,15] are the two largest outputs at present. The processes of Sweden Höganäs Co. (the major reduced iron powder manufacturer) include two steps [16]: first, to reduce the high-purity iron ore concentrate (total iron $\geqslant$71.5%); then, the second reduction in the hydrogen gas which is processed in the belt furnace for the previous products after crush, magnetic separation and drying. During the processes, the first reduction is the key step to producing high quality iron powder. The circular crucible ring-like charging method is used – the inner and outer layers are the reduction coal, the middle layer is the iron ore concentrate, which we called the outer distribution coal. Tunnel kiln is used and heated by heavy oil, the reduction period is 53–90 h, and the total iron and metallization ratio are about 97% and 95.9%, respectively. The largest advantage of this method is that the iron ore concentrate and coal are contacted without mixing. Thus the reducing agent will not pollute the products and then the high total iron and low impurity content of reduced iron powder are assured. However, the advantage means the largest disadvantage at the same time for this method in that the non-mixing of the iron ore concentrate and coal leads to the complex reduction process, long period, energy consumption and hard-to-grind products. The reduction reaction of iron oxide of this outer distribution coal is largely influenced by the condition of mass transfer. And the conventional heating method cannot meet the energy requirement of reduction and Boudouard reaction. In addition, it leads to the products which tend to be densifying.

The characteristic of direct reduced iron (DRI) [17,18] is that the iron oxide and reducing agent are mixed completely, namely the inner distribution coal. The advantages of this method are the short production period and high metallization due to the fast speed of reduction reaction. While it also leads to both high impurity of the content of the products and low rate of total iron.

To solve the problem of long reaction period for outer distribution coal under conventional heating and the high impurity content of products for inner distribution coal, a combination of the outer and inner distribution of wood-charcoal is used in this paper

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http://dx.doi.org/10.1016/j.jallcom.2014.06.016
0925-8388/

to prepare high quality reduced iron powder in the microwave field [19–22] considering the fact that mill-scale, coal and wood-charcoal all have a good property of microwave absorbing.

2. Experimental

2.1. Materials

The compositions of the mill-scale and wood-charcoal are shown in the following Tables 1 and 2, respectively. The particle size distribution of the material and wood-charcoal used is represented in Fig. 1. All these materials are milled by the ball grinding mill. The percentages shown in full paper are weight percentages.

2.2. Methods

A self-made microwave tube furnace, which utilizes a single-mode continuous controllable power is utilized for all experiments and shown in Fig. 2. The microwave frequency is 2.45 GHz, while the output power is controlled within the maximum of 1500 W. The activation temperature is controlled by varying the input microwave power. The activation temperature is measured by nickel chrome–nickel silicon armor type thermocouple which is in contact with the material. The thermocouple has dimension of length of 1000 mm, 3 mm diameter, with the temperature range of 0–1250 °C, and a measurement precision of ±0.5 °C.

Distribution: the materials are divided into three layers. At the bottom layer is the mixture of wood-charcoal and calcium carbonate, and at the mid-layer is the mixture of wood-charcoal and mill-scale while at the upper layer is wood-charcoal.

Note: a, b, c and d denote the mass of the corresponding materials in Fig. 3. Wood-charcoal at the mid layer is called inner distribution wood-charcoal and can be defined as:

$$\text{inner distribution wood} - \text{charcoal} = \frac{b}{a+b} \times 100\% \quad (1)$$

Wood-charcoal at the bottom is named as outer distribution wood-charcoal and can be defined as:

$$\text{outer distribution wood} - \text{charcoal} = \frac{b+c}{a+b+c} \times 100\% - \frac{b}{a+b} * 100\% \quad (2)$$

Combined distribution of wood-charcoal means the combination of the inner distribution wood-charcoal and outer distribution wood-charcoal. If $c = 0$ g, $d = 0$ g and the reductant is carbon, the stoichiometric content of reductant required for reduced completely of mill-scale is 15.78% [20].

2.3. Design of experiments

Experiment Process: Charge as Fig. 2. Subsequently, pass N_2 for 30 min, start the microwave, stop passing N_2. Then start the experiment according to the process.

Experiment 1: $a = 27$ g, $b = 3$ g, $c = 3.75$ g, $d = 2.5$ g. Namely, both the percentages of the inner distribution wood-charcoal and the outer distribution wood-charcoal are 10%. The gas released at 550 °C, 850 °C, 950 °C, and 1150 °C was collected by displacement of water and the gas composition was analyzed by gas chromatography. The reduction temperature was set at 1150 °C and the holding time were 5 min and 50 min, respectively. Then the products were examined by SEM, and the total iron and metallization ratio of the sample reduced by 50 min were measured.

Experiment 2: $a = 0$ g, $b = 30$ g, $c = 0$ g, $d = 0$ g. This means that the material used in this experiment is only the reduced agent, wood-charcoal. The components of the gas released by wood-charcoal during the heating process were collected and measured.

$$\text{total iron} = \frac{M_1}{M_0} \times 100\% \quad (3)$$

$$\text{metallization ratio} = \frac{M_2}{M_1} \times 100\% \quad (4)$$

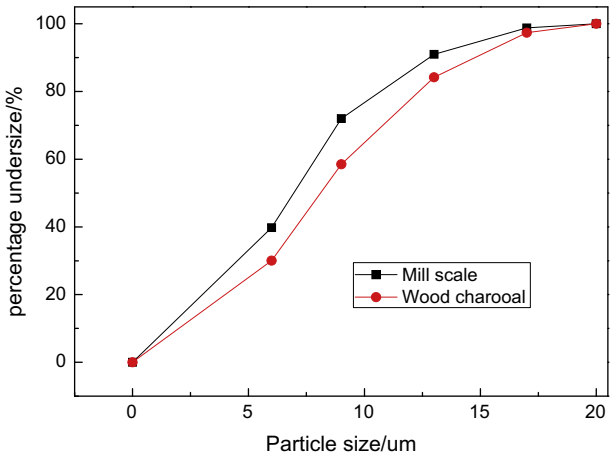

Fig. 1. Particle size distributions of mill-scale and wood-charcoal.

M_0 is the mass of a sample, M_1 is the mass of Fe, M_2 is the mass of zero valent iron. Determination of total iron and metallization ratio is measured using potassium dichromate: Redox indicators.

3. Results and discussion

The total iron of the reduced iron powder obtained (with a holding time 50 min at 1150 °C) in experiment 1 was 98.56%, and the metallization ratio was 99.25%. Based on the analysis of XRF (Shimadzu, XRF-1800 Sequential WDXRF), the detailed composition of iron powder is listed in Table 3. From the table, it can be seen that the chemical composition of the reduced iron powder meets the specification of the HY100.23 first-class iron powder standard.

Fig. 4 shows the gas composition produced by wood-charcoal at different temperature under microwave heating in experiment 2. Fig. 5 shows the off-gas composition produced at different temperature in the microwave field under the condition that the ratio of both the inner and outer distribution wood-charcoal is 10% in experiment 1.

Fig. 6 is the SEM of the mixed raw material with 10% of wood-charcoal. Wood-charcoal corresponds to the darker color, with smooth surface and clear profile, while the rest of the portion corresponds to the mill-scale.

Figs. 7 and 8 are the SEM of the reduced iron powder with an inner distribution wood-charcoal of 10%, outer distribution wood-charcoal of 10%, and reduction temperature of 1150 °C. But the holding time is 5 min and 50 min, respectively.

The reduced iron powder was prepared in microwave field by combined distribution of wood-charcoal with the holding time 50 min (using Höganäs process-ring-like charging method, heavy oil was used as fuel and the reduction period was 172 h). The chemical composition of the products meets the HY100.23 first-class iron powder standard. There are four reasons accounting for this.

Table 1
Compositions of mill-scale (total iron 74.25%) (%).

FeO	Fe_2O_3	Fe_3O_4	SiO_2	MnO	P	S	CuO	SnO_2	CaO	Cr_2O_3
61.31	36.36	1.61	0.096	0.30	0.014	0.017	0.12	0.09	0.032	0.029

Table 2
Compositions of wood-charcoal (%).

Fixed carbon	Volatile organic matter	H_2O	CaO	FeO	MgO	S	K_2O	Na_2O
72.58	22.26	4.67	0.40	0.01	0.014	0.0028	0.069	0.0005