



Iron extraction from lead slag by bath smelting

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Abstract: A new bath smelting process was proposed to recover iron for solid wastes reduction. 99.79% of iron metallization, 99.61% of iron recovery, pig iron with 93.58% Fe, 0.021% S, 0.11% P, 1.38% C, 0.22% Si, 0.01% Pb and 0.031% Zn were achieved after the wastes were smelted at 1575 °C for 20 min under C/Fe molar ratio of 1.6 and basicity of 1.2. The produced pig iron could be used in steel-making. This study provides a way for recycling iron from smelting slag and hydrometallurgical residue.

Key words: lead slag; iron extraction; bath smelting; pig iron

1 Introduction

Smelting process generates considerable amounts of solid wastes such as lead slag [1,2], copper slag and zinc residue [3,4], which contain hazardous compounds not to be discharged into the environment directly. Accumulation of these wastes not only occupies massive land but also aggravates a series of environmental problems [5–7]. Great research effort was taken to treat such solid wastes, including solidified and reduction processes [8–11]. It is a priority and real necessity to reduce solid waste rather than to solidify it in the consideration of environmental problem and land issue. Solid wastes reduction will be achieved by using them as ingredient of material or extracting valuable metals [12–16]. However, the consumption of solid wastes as ingredients of materials is limited. Moreover, leaching toxicity of the material would be over environmental restrict due to heavy metals [17,18]. On the other hand, the reduction of solid wastes is restricted due to most of work for extracting valuable metal from solid wastes only concerns extraction of lead, zinc and copper, etc, so that still left a lot of solid wastes. Extraction iron from solid wastes is an effective approach to reduce solid wastes because the average iron concentration in the

solid wastes is approximately 30%–50%.

Iron in solid wastes can be extracted as iron concentration by reduction roasting or direct reduction then magnetic separation [19,20]. However, the iron concentration needs to further smelt for obtaining metal iron. Furthermore, zinc and lead contained in solid wastes are harmful in iron smelting process [21]. Bath smelting process is recognized as one of the most efficient process to treat the waste and well suited for reducing zinc and lead oxides [21–24]. However, thermodynamic calculations of Gibbs free energy changes of reduction for zinc, lead and iron oxides indicate that zinc and lead oxides are easily reduction under low concentration of carbon monoxide, whereas the reduction of iron oxide needs high concentration of carbon monoxide. Hence, zinc and lead are reduced to metal, while iron is reduced to ferrous in bath smelting process. Abundant carbon needs to be added for reducing zinc, lead and iron oxides simultaneously, which makes carbon content in metal iron higher.

It is necessary to develop a more advanced method to solve above problems. The objective of this study is to recover iron to obtain low carbon pig iron for solid waste reduction in liquid iron bath smelting process. Iron oxide would be reduced to metal iron in one step smelting process by dissolved carbon other than solid carbon and

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carbon monoxide. Low carbon content of pig iron would achieve due to the fact that reactivity of dissolved carbon is significantly superior to solid carbon and carbon monoxide [25]. Furthermore, desulfurizing agent was added to obtain a high quality pig iron. The quality of pig iron depends on contents of phosphorus and sulfur except carbon content. To this aim, the present study investigates the reduction behavior of iron, as well as the reduction behavior of phosphorus and sulfur. The study intends to provide data about solid wastes reduction and improve the quality of pig iron produced from solid waste.

2 Experimental

2.1 Materials

The calcium oxide used in this study is analytical grade reagent (content of CaO $\geq 98.0\%$) and was supplied by Westlong Chemical Reagent Plant, China. The graphite used in this study is industrial pure.

The lead slag was obtained from a lead smelter in Hunan Province, China. The slag mainly contains Fe, Si and Ca, as shown in Table 1. The mineral phases identified by XRD analysis (Fig. 1) include fayalite $\text{Ca}(\text{Fe}_{0.69}\text{Mg}_{0.31})(\text{SiO}_4)$, iron and silicon oxide $\text{Fe}_{0.914}\text{Si}_{0.086}(\text{Fe}_{0.998}\text{Si}_{0.002})_2\text{O}_4$, and ferroferric oxide (Fe_3O_4). Ferrous oxide (FeO), calcium and zinc silicate ($\text{Ca}_2\text{ZnSi}_2\text{O}_7$), and calcium and iron silicate ($\text{CaFe}_2\text{SiO}_4$) are the minor phases present in the lead slag. SEM associated with energy-dispersive spectroscopy (EDS) was used for the morphology study of the slag. The results (Fig. 2) of the XRD and XRF analyses are consistent.

The chemical composition of the lead slag was analyzed by X-ray fluorescence diffraction (S4-pioneer;

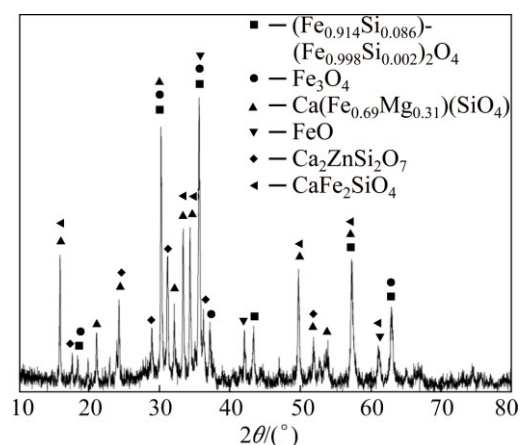


Fig. 1 XRD pattern of lead slag

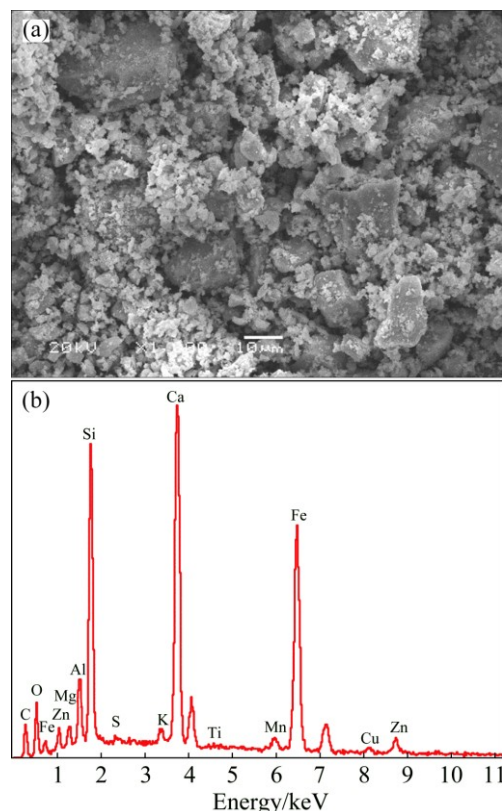


Fig. 2 SEM image (a) and EDS analysis (b) of lead slag

Table 1 Chemical composition of lead slag by XRF

Element	Mass fraction/%	Element	Mass fraction/%
O	31.9	Ca	14.59
Na	0.94	Ti	0.23
Mg	1.13	Rb	0.005
Al	3.29	Cr	0.12
Si	12.53	Mn	1.13
P	0.065	Fe	23.84
S	0.21	Cu	0.17
K	0.82	Zn	4.28
Co	0.017	Ge	0.004
Ga	0.006	Sr	0.12
Zr	0.012	Mo	0.013
Sn	0.062	Sb	0.072
Ba	0.13	Pb	0.076

Bruker, Germany). The crystalline phases were investigated by X-ray powder diffraction (TTR-III; Rigaku Corporation, the Woodlands, TX) on the 2θ scale using $\text{Cu K}\alpha$ radiation (40 kV and 250 mA) at a scanning rate of $10^\circ/\text{min}$ from 10° to 80° . The surface profile and semi-quantitative information were detected by SEM-EDS (JSM-6360 LV; JEOL, Ltd., Tokyo, Japan, and EDX-GENESIS 60S; EDAX, Ltd., Mahwah, NJ). The ferric chloride-sodium acetate volumetric method and potassium dichromate volumetric method were used to determine the contents of metallic iron and total iron. The content of phosphorus was analyzed by

spectrophotometry (721G). The content of sulfur was analyzed by sulfur determinator (KZDL-4).

2.2 Experimental set-up and procedure

A furnace of the thermal concept HTK40-18/S with maximal temperature of 1800 °C and ± 1 °C accuracy was used as the experimental apparatus. All samples were dried, ground and sieved to approximately 90% particles below 74 μm before smelting. 100 g of lead slag was blended with graphite powder and calcium oxide in a mortar and pestle for 20 min and was placed in a magnesium oxide crucible and heated in the melting furnace for a desired time and temperature under an argon atmosphere. Zinc and lead oxides were reduced and volatilized to obtain the smoke of zinc oxide. Iron oxide was reduced to liquid iron to obtain pig iron. Then, the samples were cooled to room temperature and removed to separate the pig iron and slag. The pig iron was weighed, and the quantities of total iron, phosphorus and sulfur were determined. The slag was weighed, and the quantities of total iron and metal iron were determined.

The iron yield (η) and iron reduction rate (ε) were calculated according to following equations:

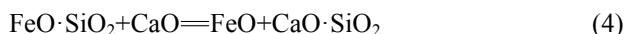
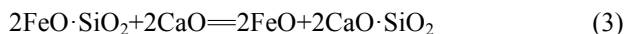
$$\eta = \frac{m_i w_i}{m_0} \times 100\% \quad (1)$$

$$\varepsilon = \frac{m_i w_i + m_s w_s}{m_0} \times 100\% \quad (2)$$

where m_i is the mass of the pig iron (g), w_i is the iron content of the pig iron (%), m_0 is the iron mass of the lead slag (g), m_s is the mass of the slag (g), and w_s is the metal iron content of slag (%).

2.3 Experimental mechanisms

The iron present in lead slag is mainly $\text{CaFe}_{0.69}\text{Mg}_{0.31}\text{SiO}_4(\text{CaO} \cdot \text{Fe}_{0.69}\text{O}_{0.69} \cdot \text{Mg}_{0.31}\text{O}_{0.31} \cdot \text{SiO}_2)$ and Fe_3O_4 according to Figs. 1 and 2. The $\text{FeO} \cdot \text{SiO}_2$ is hard to be reduced without adding calcium oxide due to low ferrous activity. The reactions occurring in the process are as follows:

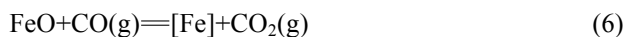


The theoretically calculated results indicate that the ferrous activity increases as the basicity increases from 0.8 to 1.2. The ferrous activity is at the maximum at a basicity of 1.2, and above that value, the activity remains constant [25].

Another iron phase present in lead slag is Fe_3O_4 , which is unstable due to existence of liquid iron [24]. The reaction is as follows:



Hence, the main iron reaction is the reduction of FeO in this process. Abundant dissolved carbon ($[\text{C}]$) is produced due to the existence of liquid iron [25]. Thus, FeO is reduced not only by CO(g) and solid carbon (C(s)) but also by dissolved carbon ($[\text{C}]$). The reactions are as follows:



The reduction rate of the dissolved carbon (Eq. (8)) is the fastest and is 10–1000 times that of Eqs. (6) and (7) [25]. Moreover, the separation of slag and iron is more complete in the molten state. Hence, a better iron reduction and quality of molten iron are obtained in the liquid iron medium.

The vapor pressure of lead is 1×10^5 kPa at 1500 °C and increases with temperature [26]. Zinc and lead oxides in the lead slag are reduced by liquid iron and dissolved carbon ($[\text{C}]$) in the following equations, except solid carbon (C(s)) and CO(g) [27].



Moreover, the volatilization rate of zinc increases with the increase of ferrous activity [28]. Therefore, the reduction rate and vapor pressure of zinc and lead are improved to promote volatilization.

The reduction of iron, zinc and lead and the volatilization of zinc and lead are more efficient in a liquid iron medium.

3 Results and discussion

3.1 Effect of smelting temperature

The effects of the smelting temperature on the iron content, iron reduction and iron yield are shown in Fig. 3. The iron content, iron reduction and iron yield increased as the smelting temperature increased from 1500 to 1575 °C. The iron content, iron yield and iron reduction reached maxima of 92.56%, 97.00% and 97.10%, respectively, at a smelting temperature of 1575 °C. Above this temperature, the iron reduction and iron yield remained approximately constant, whereas the iron content dropped slightly. The iron reduction demonstrated better separation of iron and slag in the way of experimental mechanisms. The iron reduction and iron yield increased with the increase of smelting

temperature below 1575 °C due to decreased molten slag viscosity, which improved the conditions of iron reduction and iron-slag separation [25].

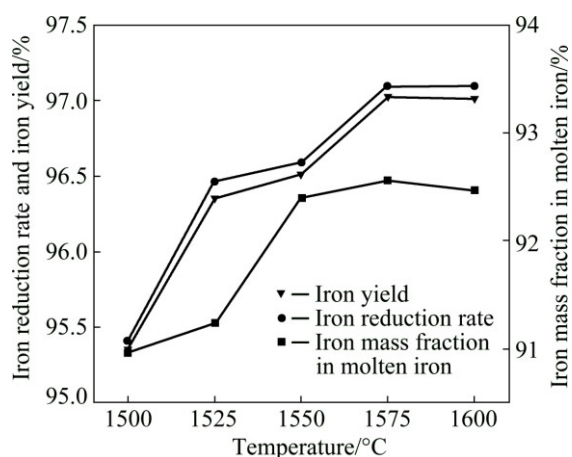


Fig. 3 Effect of temperature on iron content, reduction and yield

The effects of the smelting temperature on sulfur and phosphorus removal from molten iron are shown in Fig. 4. The sulfur content of molten iron decreased to the lowest level of 0.15% as the smelting temperature increased from 1500 to 1600 °C. This phenomenon was a result of the endothermic reaction of desulfurization, in which increasing temperature accelerated desulfurization. The phosphorus content of molten iron increased with the increase of the temperature due to the exothermic reaction of dephosphorization, in which the increasing temperature is unfavorable for dephosphorization [29]. The smelting temperature should be 1575 °C to obtain the best iron reduction and the lowest sulfur content.

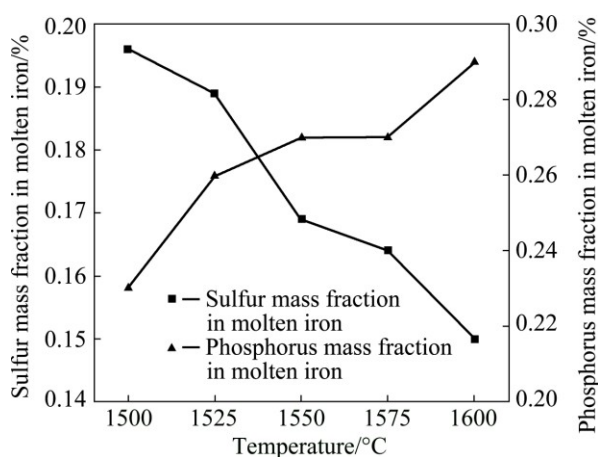


Fig. 4 Effect of temperature on sulfur and phosphorus contents in molten iron

3.2 Effect of holding time

Figure 5 shows the effects of the holding time on the iron content, iron reduction and iron yield. The iron yield and iron reduction increased from 96.66% and

98.46% to 98.74% and 98.78%, respectively, as the holding time extended from 10 to 30 min. After 30 min, the iron reduction and iron yield were constant. The iron yield and iron reduction over 96% within 10 min indicated a rapid reduction of iron and separation of iron and slag. The iron content increased from 92.79% to 93.64% as the holding time increased from 10 to 40 min and then remained approximately constant.

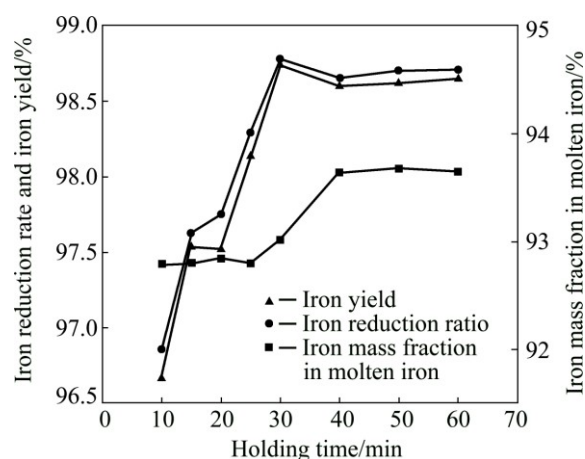


Fig. 5 Effect of holding time on iron content, reduction and yield

The effects of holding time on sulfur and phosphorus removal from molten iron are shown in Fig. 6. The sulfur and phosphorus content in molten iron increased and decreased in the holding time period from 10 to 40 min, and dephosphorization and rephosphorization, as well as desulfurization and resulfurization occurred every 5 min. After 40 min, the sulfur and phosphorus contents of the molten iron reached a low level.

The iron reduction and iron yield were greater than 97.5% at 20 min (Fig. 5), with a low concentration of sulfur (0.074%) and phosphorus (0.22%). Therefore, 20 min was set for the holding time of smelting.

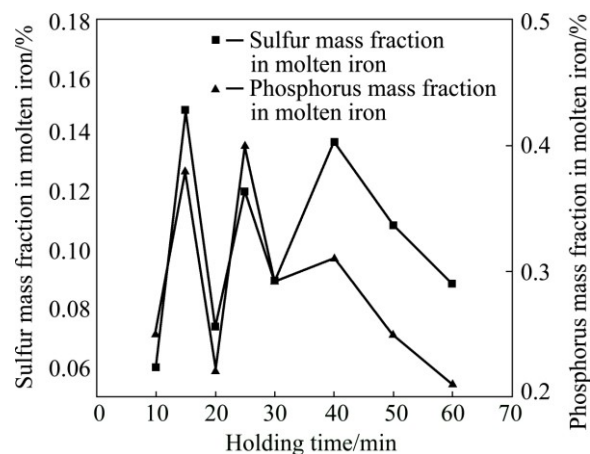


Fig. 6 Effect of holding time on sulfur and phosphorus contents in molten iron

3.3 Effect of C/Fe mole ratio

The effects of the C/Fe mole ratio on the iron content, iron reduction and iron yield are shown in Fig. 7. The iron content, iron reduction and iron yield increased sharply to 93.94%, 97.93% and 97.81%, respectively, at a C/Fe mole ratio of 1.6. Further increase of the C/Fe mole ratio led to a decrease of the iron content, iron reduction and iron yield.

Figure 8 indicates that the main component of the molten slag was silica (Fig. 8(a)), whereas the main

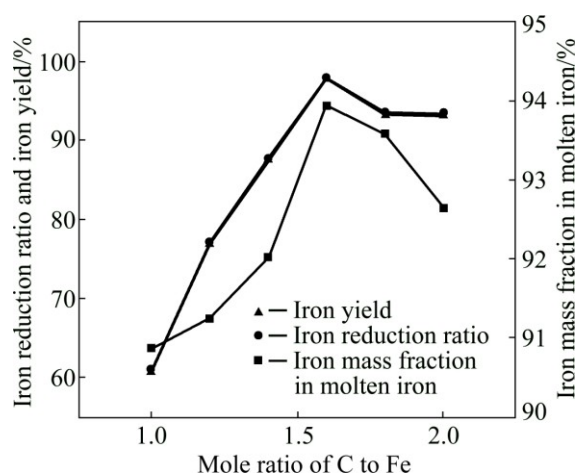


Fig. 7 Effect of C/Fe mole ratio on iron content, reduction and yield

content of the iron–slag interface was silicon (Fig. 8(b)). This result indicated that some silica was reduced to silicon at the iron–slag interface. Hence, the decrease of the iron content, iron reduction and iron yield was due to the reduction of silica, which thickened the metal–slag interface layer and affected the reduction and migration of iron.

Iron reduction had a positive effect on the increase of phosphorus in molten iron due to decrease of ferrous, the reaction is as



The sulfur in molten iron decreased with the increase of the C/Fe mole ratio, which benefited desulfurization [30]. The results of Figs. 7 and 9 indicate that increase of the C/Fe mole ratio was helpful to iron reduction and desulfurization but unfavorable to dephosphorization. Therefore, the C/Fe mole ratio was set at 1.6 in consideration of iron reduction and desulfurization.

3.4 Effect of basicity

Figure 10 shows the effects of the basicity on the iron content, iron reduction and iron yield. The best iron reduction of 99.79% and iron yield of 99.61% were obtained at a basicity of 1.2, which was the same as the theoretically calculated result [25]. The results of Fig. 10

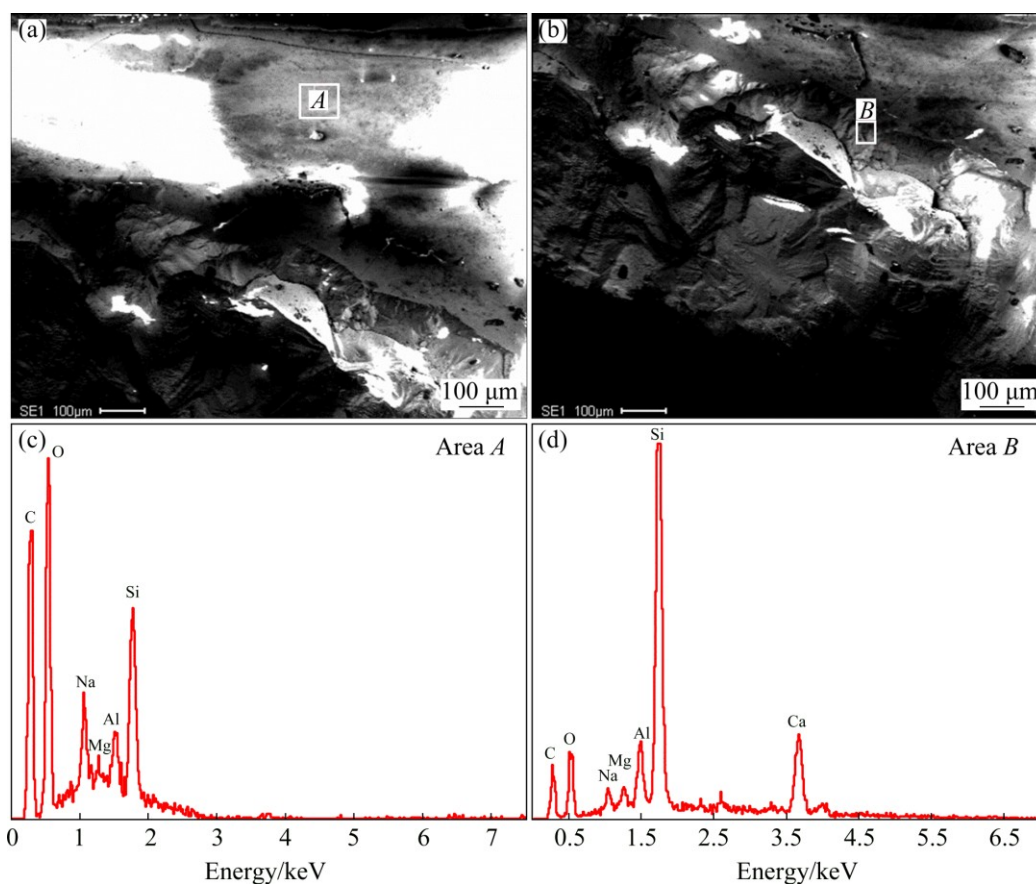


Fig. 8 SEM images (a, b) and EDS analysis (c, d) of slag–iron interface

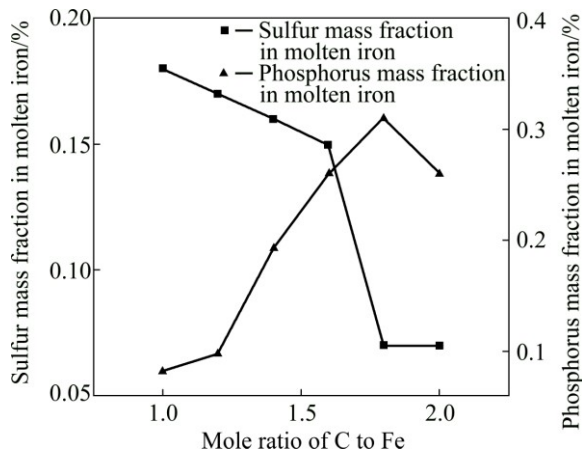


Fig. 9 Effect of C/Fe mole ratio on sulfur and phosphorus contents in molten iron

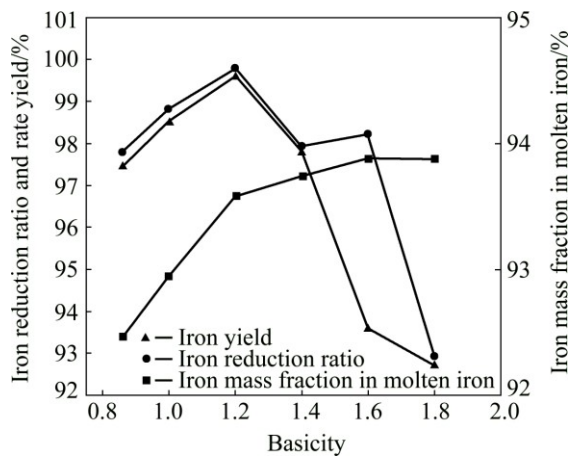


Fig. 10 Effect of basicity on iron content, reduction and yield

and the theoretically calculated results illustrated that increased basicity enhanced the ferrous activity to promote iron reduction. Moreover, the molten slag viscosity decreased to improve iron reduction at high basicity. Further increase of the basicity resulted in an increase of the molten slag viscosity, diffusion resistance of C and migration resistance of metal iron to decrease iron reduction and iron yield [31].

The effects of the basicity on the sulfur and phosphorus removal (Fig. 11) indicated that the phosphorus content of molten iron increased as the basicity increased from 1.0 to 1.2 due to the increased iron reduction, which decreased the ferrous concentration and resulted in rephosphorization (Eq. (15)). The phosphorus content of molten iron decreased as the basicity increased from 1.2 to 1.6 due to the increase of basicity increasing the concentration of O and promoting dephosphorization [32], the reactions are as follows:



The phosphorus content of molten iron increased with further increasing basicity over 1.6 due to increased viscosity, which hindered dephosphorization [32]. The sulfur content of molten iron decreased as the basicity increased from 1.0 to 1.4 due to increased basicity, which promoted desulfurization. Above 1.4, the sulfur content of molten iron increased as the basicity increased due to the increase of viscosity, which hindered desulfurization [32]. Increased basicity contributed to desulfurization and dephosphorization, but higher basicity was disadvantageous. Thus, 1.2 was the most favorable basicity for iron reduction and desulfurization.

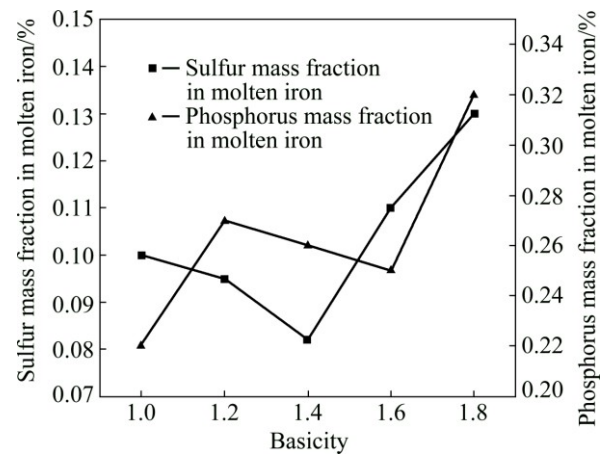


Fig. 11 Effect of basicity on sulfur and phosphorus contents in molten iron

3.5 Quality of pig iron

The pig iron obtained under the optimal conditions (Table 2) was composed of 93.74% Fe, 1.38% C, 0.021% S, 0.22% Si, 0.11% P, 0.01% Pb and 0.031% Zn. The contents of S and P accord with the first class of the Pig Iron Standard of Steel Making of China. The content of C is lower than the standard. The content of Si conforms to the standard of L04. Above all, the pig iron produced meets the requirements of steel-making. These results illustrate that a better iron quality was obtained, which is consistent with the experimental mechanism.

Table 2 Element content of produced pig iron (mass fraction)

Element	Content/%	Stand content/%	Standard grade
Fe	93.74	—	—
P	0.11	0.10–0.15	The first class standard
S	0.021	0.02–0.03	The first class standard
Si	0.22	≤0.45	L04
C	1.38	≥3.50	Standard

*Stand content: Pig iron standard for steel making in China

4 Conclusions

1) Under smelting temperature of 1575 °C, holding time of 20 min, C/Fe mole ratio of 1.6 and basicity of 1.2, 93.58% of iron content, 99.79% of iron reduction and 99.61% of iron yield are obtained.

2) Pig iron with 1.38% C, 0.021% S, 0.22% Si, 0.11% P, 0.01% Pb and 0.031% Zn is obtained. The produced pig iron meets the requirements of steel-making.

3) Reduction of iron, separation of iron and slag, and volatilization of zinc and lead are improved by liquid iron and dissolved carbon.

4) This study provides a method for recycling iron from smelting slag and hydrometallurgical residue.

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铅渣熔池熔炼提取铁

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摘 要: 为实现废渣减量化, 提出了一种熔池熔炼处理废渣回收铁的新工艺。在熔炼温度 1575 °C、保温时间 20 min、C/Fe 摩尔比 1.6、碱度 1.2 的优化条件下, 铁金属化率和回收率分别为 99.79%和 99.61%。获得含 93.58% Fe、0.021% S、0.11% P、1.38% C、0.22% Si、0.01% Pb 和 0.031% Zn 的生铁, 达到国家炼钢生铁标准, 可用于炼钢。

关键词: 铅渣; 铁提取; 熔池熔炼; 生铁

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