Iron ore pellet disintegration mechanism in simulated shaft furnace conditions

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Abstract: The disintegration behavior and mechanism of iron ore pellet in simulated shaft furnace conditions were investigated in details. Effects of temperature, reducing gas and gangue compositions were emphatically discussed. It was found that pellet disintegrated acutely as reduced by $H_2/CO=0.8$ at 550 °C. Gangue minerals in pellet were proved to ease the disintegration in various degrees. SiO_2 or CaO could remarkably lower reduction disintegration index (RDI). But the effect of MgO or Al_2O_3 was much weaker. Furthermore, intrinsic relation between disintegration and reduction was revealed from microstructure aspect. Results demonstrated that lattice transition stress and crack generation brought about by the low temperature reduction led to pellet structure failure and therefore disintegration. The reduction degree showed positive correlation with disintegration level of pellet. Low melting point phase formed by SiO_2 or CaO lessened crack formation in pellet. However, MgO or Al_2O_3 could not play a similar role.

Keywords: Iron ore pellet, Gas-based reduction, Disintegration behavior, Shaft furnace, Hydrogen, Carbon monoxide

1 Introduction

Approximately 10% of anthropogenic CO₂ emissions in the world derive from the iron and steel industry [1, 2]. However, the fossil fuel energy (especially coke), performs as heat source, reducing agent and skeleton in blast furnace (BF), is essential to the conventional ironmaking process. Take H₂ as the substitution for coal in BFs may be a potential solution to overcome this problem. For the partial use of H₂ (rest is CO), shaft furnace direct reduction process (such as Midrex and HYL-III) has become the focus in ironmaking research field [3-6]. But the reduction disintegration of furnace burden at low temperature, exists both in BF and shaft furnace processes, strongly affects the permeability and thus the stable operation [2, 7-9]. Therefore, a better understanding of iron ore pellet disintegration behavior in shaft furnace is urgently required.

It is well known that disintegration of burdens always occurs around 500–600 °C in the upper zone of blast furnace. The main cause for disintegration is lattice expansion of iron oxide phase, due to its reduction from hematite to magnetite [9-14]. But hematite with different morphology effects variously. Large cracks usually form at reduced skeletal hematite along with net-like fine cracks near the reduced hematite. Therefore, approximately 30% of reduction disintegration index (RDI) for skeletal hematite was reported [2, 7, 15-17]. An appropriate increase in basicity (CaO/SiO₂) or MgO addition improves resistance to reduction disintegration for their strength rise effect on burdens [18, 19]. In some cases, increasing the FeO content of sinter also improves reduction disintegration by lessening the hematite reduction to magnetite

[20]. But limestone addition or TiO_2 -rich materials exhibits poor reduction disintegration properties [8, 21]. Generally, the RDI for sinter reduced by CO is always higher than that of the sinter processed by H_2 at the same reduction degree. Reduction by CO gas leads to the formation of magnetite phases with not only long-thick but also fine cracks near the surface, opposite to that of H_2 reduction (small amounts of fine cracks) [2, 7, 15, 22]. And therefore lower burden strength in CO reduction.

An X-ray imaging technology was applied to the real-time observation of iron ore disintegration behavior [23]. It is found that cracks always initiate from pores and propagate through neighboring pores. And the crack initiation temperature is as low as 450 °C. Spraying CaCl_2 solution onto the burden surface to hinder the reducing gas diffusion is a common method for reduction disintegration prevention [24]. However, the Cl^- ions introduced by CaCl_2 also bring series of troubles to BF operation and environmental pollution [25].

It can be noticed that only few researchers studied disintegration of iron ore pellets during low temperature reduction with H_2 -CO mixtures, especially for the gas-based shaft furnace conditions. So, in the present work disintegration behavior of iron ore pellets in H_2 -CO mixtures was studied by simulating the upper part of shaft furnace. And mechanisms were also revealed, which was hoped to provide fine guidance to the practical operation of shaft furnace direct reduction process.

2 Experimental

2.1 Materials

Iron ore pellet sample supplied by a domestic pelletizing plant (China) was used as furnace burden in this work. The elemental composition of iron ore pellets was shown in Table 1. And the charged pellets were in the size range of 12.5-16 mm with average crushing strength of 2973 N/pellet.

Table 1 Chemical composition of iron ore pellet

Fig.1 presented the morphology of iron ore pellets used in this research. Image analysis showed that hematite was the major phase in the pellet. Plenty of plate-like hematite crystallized well and pores distributed in the connected hematite crystals uniformly.

Fig.1 Morphology of iron ore pellet sample, a-optical microscope image of cross section,

b-SEM micrographs of fractured surfaces, H=hematite, P=pore

High purity (99.99%) H_2 , CO, CO_2 and N_2 were used as experimental gas. The reducing gases in the upper part of shaft furnace (vol.% (H_2+CO)=70%, vol.% (CO_2+N_2)=30%) were mixtures of H_2 , CO, CO_2 and N_2 with various proportions by simulating coal gasification gas (No.2), Midrex gas (No.3) and HYL-III gas (No.4) respectively as shown in Table 2 [12, 13, 26].

Table 2 Composition of reducing gases

2.2 Methods

The schematic diagram of experimental apparatus and gas flow system used in this study were presented in Fig. 2. Iron ore pellets were reduced at 450–600 °C under load of 0.4 kg/cm² by simulating upper shaft furnace conditions [27, 28]. For each reduction experiment, iron ore pellets were heated to the required temperature and then reduced for 60 min in reducing gases (listed in Table 2) at flow rate of 0.6m³/h. Subsequently, the reduced samples were cooled to room temperature in atmosphere of nitrogen. The reduction degree (RD) was evaluated as Eq. (1).

$$RD = (W_0 - W_1) / W_2 \times 100\% \quad (1)$$

Where W_0 and W_1 were the weights of the sample before and after reduction respectively and W_2 was the total possible weight loss due to the oxygen removal in the pellet.

Fig.2 Schematic diagram of the reduction apparatus,

1-load, 2-electric furnace, 3-graphite crucible, 4-pellets, 5-thermocouple,

6-graphite pusher, 7-gas mixer, 8-flowmeter, 9-cylinder

Disintegration tests were conducted on the reduced samples based on Chinese industrial standards (GB/T13242-91), using a tumbling drum with an inner diameter of 130 mm and length of 200mm, at a rotation speed of 30 rpm for 10 min. Then, the

sample was sieved with a 3.15 mm mesh. Finally, the reduction disintegration index (RDI) value was calculated by Eq. (2).

$$RDI_{<3.15mm} = (1 - W_3 / W_4) \times 100\% \quad (2)$$

Where W_3 was the weight of particles larger than 3.15 mm and W_4 was the weight of reduced sample before drum test.

The morphology (cross section) of reduced sample was observed by electron microscopy (Leica DMRXP). Cracks in every vision field were distinguished and counted by the equipped image analysis software (Qwin). The crack distribution (CD) was defined by Eq. (3). And Scanning Electron Microscope (FEI Quanta-200) was also applied to analyze the microstructure transformation of reduced samples.

$$CD = A_c / A_t \times 100\% \quad (3)$$

Where A_c was the area occupied by cracks in the vision field and A_t was the total area.

3 Results and discussion

3.1 Effect of temperature

The reduction disintegration indexes (RDIs) of pellets reduced at different temperatures was given in Fig.3. It can be seen from the line chart, the iron ore pellets adopted in this research showed good quality for anti-disintegration at low temperatures. The RDI of pellet fluctuated within a range of 0.4% to 3.2%. In general, RDI increased steadily with temperature, peaking at 550 °C, then presented a downward trend to 600 °C. However it cannot be ignored that the RDI difference

among pellets reduced by CO, H₂ and H₂-CO mixtures as displayed in Fig. 3. And it would be discussed deeply in the section 3.2.

Fig.3 Effect of temperature on reduction disintegration index,

1-CO, 2-H₂/CO=0.8, 3-H₂/CO=1.6, 4-H₂/CO=2.6, 5-H₂

The lattice structure transformation of Fe₂O₃ to Fe₃O₄ and consequent stress generation was considered to be the root cause of reduction disintegration at low temperature. Temperature rise, on one hand, intensified this stress because of the more reduction of Fe₂O₃ and therefore worsen the RDI. Fig. 4 showed this positive correlation between RDI value and reduction degree of pellets in our experiment obviously. On the other, the plasticity which determined the stress bearing property of pellets also increased with temperature. So, the disintegration of pellets displayed a weakening trend as temperature further rise to 600 °C not surprisingly (shown in Fig. 3). Wu *et al.* [9] studied the low temperature reduction disintegration properties of sinter, pellet and lump ore for BF process also concluded that RDIs deteriorated seriously at 550 °C.

Fig.4 Relationship between RDI value and reduction degree

Microstructure (cross section) of reduced samples at 500 °C, 550 °C and 600 °C (reduced by H₂/CO=0.8) were characterized, as shown in Fig. 5, to reveal the intrinsic influence of temperature.

**Fig.5 SEM images of samples reduced at low temperatures, a&b-500°C, c&d-550°C,
e&f-600°C**

As can be seen from Fig.5 (a) and (b), pellet reduced at 500 °C retained nearly intact structure. The plate-like magnetite (bright) formed by reduction at 500 °C connected fully without obvious defect. It was this structure feature ensured its strength and therefore nice anti-disintegration property. However, the situation differed widely in the case of 550 °C. Cracks were found in the reduced sample as shown in Fig.5 (c). Cracks generated initially from the pores (dark) and then extended to the magnetite zone. Due to the presence of pores, reducing gas was more likely to contact the surrounding hematite through them and produced cracks by reduction. Then reducing gas continued to diffuse along the crack and react with hematite, which promoted the expansion of crack and severe disintegration eventually. At the same time, fractured magnetite grains with fine cracks also formed in large quantities (Fig.5 (d)) for the lattice transformation stress. It further intensified the disintegration of pellets during reduction at 550 °C. However, no extensive crack had been found in pellets reduced at 600 °C as presented in Fig.5 (e). Maybe the temperature rise enhanced plasticity of binder and slag phase in pellet, which avoided severe structure failure. But magnetite grains with fine cracks still existed (shown in Fig.5 (f)) as that of 550 °C. So, the coarse cracks and fractured magnetite grains formed during reduction were the main cause for severe disintegration at 550 °C. The disappearance of coarse

crack at 600 °C eased the disintegration of pellets effectively.

3.2 Effect of reducing gas

Fig.6 showed the effect of reducing gas on reduction disintegration indexes (RDIs) of pellets. As demonstrated in the line chart, RDI increased steadily with the H_2 proportion in reducing gas, reaching a peak of 1.9%~3.1% (various with temperature) at $H_2/(H_2+CO)=0.44$ ($H_2/CO=0.8$), then presented a downward trend to $H_2/(H_2+CO)=1.0$ (H_2 atmosphere). The RDI of pellets reduced by CO or H_2 reached as low as 0.5%.

Fig.6 Effect of reducing gas on reduction disintegration index,

1-450 °C, 2-500 °C, 3-550 °C, 4-600 °C

To understand the difference, reduction degree (RD) change of pellets with gas composition was examined as shown in Fig.7. To be specific, RD of pellet reduced by CO was 13.2% and this value reached the maximum of 20.5% at $H_2/CO=0.8$. Then RD gradually dropped down to 13.8% with the increase of H_2 proportion in reducing gas. On one hand, due to the smaller molecular diameter the diffusion coefficient of H_2 in pellet particles was three times higher than that of CO at 500 °C [29]. Thus H_2 proportion increase could enhance the reduction process. On the other, excessive H_2 proportion was unfavorable for the low temperature reduction of iron oxide because of its endothermic reaction characteristic [30]. The RD of pellet influenced by

reducing gas corresponded well with its RDI variation tendency. That is, the higher RD of pellet led to a higher RDI for its more stress generation during reduction. This finding was in accordance with the reports of Taichi Murakami *et al.* [7].

Fig.7 Effect of reducing gas on the reduction degree of samples

Microstructure (cross section) of samples reduced by CO, $H_2/CO=0.8$, $H_2/CO=1.6$, $H_2/CO=2.6$, H_2 (at 550 °C) were characterized respectively, as shown in Fig. 8, to reveal the intrinsic influence of reducing gas.

**Fig.8 SEM images of samples reduced with different reducing gases, a-CO, b- $H_2/CO=0.8$,
c- $H_2/CO=1.6$, d- $H_2/CO=2.6$, e- H_2**

As presented in Fig. 8 (a), pellet that reduced by CO mainly kept the original structure except for a small amount of local failure. And pellet showed a low RDI value during reduction. However, the case differed obviously as pellets reduced by $H_2/CO=0.8$ or 1.6. From Fig. 8 (b), coarse extensive cracks formed widely in pellet for the violent reduction therefore led to fragile structure and high RDI. Instead of coarse crack, numerous of criss-cross fine cracks produced by lattice transformation stress were found as pellet reduced in atmosphere of $H_2/CO=1.6$ (shown in Fig. 8 (c)). Cracks lessened and disappeared eventually with the further increase of H_2 ratio in reducing gas as given in Fig. 8 (d) and (e). Besides RD difference discussed above (in Fig. 7), the reduction feature of H_2 was considered to be another cause for this

phenomenon. The strain level in magnetite crystal formed by H_2 reduction was lower than that of CO because of the relatively uniform reduction of hematite [12, 13]. Therefore, there was no stress concentration at surface and less structure failure generation in pellet, unlike during CO reduction.

3.3 Effect of gangue

In consideration of the possible influence of gangue on pellet disintegration, magnetite powder (analytical pure) was mixed with different proportions of CaO, MgO, SiO_2 and Al_2O_3 (analytical pure) respectively based on Table 1. Then briquetting and oxidation roasting were carried out to prepare pellet according to our previous work [31]. Finally, the pellets were reduced at 550 °C with $H_2/CO=0.8$ atmosphere to investigate their RDIs as presented in Fig.9.

Fig.9 Effect of gangue on the reduction disintegration index, a- SiO_2 , b-CaO, c-MgO, d- Al_2O_3

It can be seen from the line chart that all the four usual gangue mentioned above could, to some extent, weaken the disintegration phenomenon. The disintegration of prepared pellets without any gangue composition was extremely serious ($RDI=20.4\%$). From Fig.9 the RDIs declined sharply with SiO_2 or CaO addition. As the content of SiO_2 and CaO increased to 5.3% and 1.6%, the RDIs of prepared pellets decreased to 9.8% and 5.2% respectively. However, there was only a minor drop of RDIs with MgO or Al_2O_3 addition. The RDIs of prepared pellets with 1.0% MgO and 1.6%

Al_2O_3 addition was still as high as 17.2% and 15.4% respectively. So, addition of CaO or SiO_2 could weaken the pellet disintegration remarkably but effect of MgO or Al_2O_3 was inferior to the former.

Fig.10 Crack distribution in reduced compacts with Al_2O_3 (left) and CaO (right) addition

Cross section of reduced pellets with equal Al_2O_3 and CaO addition (1.6%) were illustrated in Fig.10 to reveal this difference. Cross section of reduced pellets with equal Al_2O_3 and CaO addition (1.6%) were illustrated in Fig.10 to reveal this difference. As can be seen, not only coarse cracks but also vast criss-cross fine cracks widely distributed in Al_2O_3 doped pellet. During reduction process, the coarse cracks broke integrated structure of pellet and the fine ones aggravated powder generation. However, few fine cracks could be observed in CaO doped pellet except for several coarse ones which probably ensured its less powder formation. This crack distribution difference also existed between MgO and SiO_2 doped pellets. An electron microscopy equipped with image analysis system was employed to find the relation between gangue and crack distribution character. The area percentage occupied by cracks on cross section surface was given in Fig.11.

Fig.11 Effect of gangue on the crack distribution, a- SiO_2 , b- CaO , c- MgO , d- Al_2O_3

As illustrated in Fig.11, the crack density of pellets formed during reduction varied with gangue addition apparently. The pellet prepared with no gangue addition

showed high density of crack distribution (about $42.7\% \cdot \text{cm}^{-2}$) after reduction. SiO_2 and CaO showed nice inhibition effect on crack formation. The crack distribution density of reduced pellets decreased to 10.4% and 13.6% respectively as SiO_2 and CaO were doped in pure magnetite. However, this effect was not so obvious in MgO or Al_2O_3 doped pellets. The crack distribution density dropped slightly to 24.5% and 33.3% respectively. This variation tendency coincided well with their RDIs change as compared with Fig.9. That is, the more cracks formed during reduction process the higher RDI value of pellet would generate. The possible products formed by different gangue and magnetite during pellets preparation may be the cause for their crack diversity. The fayalite ($2\text{FeO} \cdot \text{SiO}_2$) and calcium ferrite ($\text{CaO} \cdot \text{Fe}_2\text{O}_3$) products with low melting point [32, 33] formed in SiO_2 and CaO doped pellets may act as binder phase, which ensured their low disintegration during subsequent reduction. While such situation was almost absent in the case of MgO or Al_2O_3 addition.

4 Conclusions

In the present paper, disintegration behavior of iron ore pellet in simulated shaft furnace conditions was investigated in details. Disintegration was found to be most severe as pellet reduced by $\text{H}_2/\text{CO}=0.8$ at $550\text{ }^\circ\text{C}$. Lattice transition ($\text{Fe}_2\text{O}_3 \rightarrow \text{Fe}_3\text{O}_4$) stress and crack generation brought about by the low temperature reduction were considered to be the main reason for pellet structure failure. The higher reduction degree of pellet reached the more failure and therefore disintegration would be. Gangue minerals in pellet were proved to ease the disintegration in various degrees.

SiO₂ or CaO could remarkably lower RDI for the low melting point phase (2FeO·SiO₂ or CaO·Fe₂O₃) improved stress resistance of pellet. However, MgO or Al₂O₃ could not play a similar role.

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