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Full Length Article

Existence state and structures of extracted coke and accompanied samples from tuyere zone of a large-scale blast furnace

Kejiang Li*, Jianliang Zhang, Minmin Sun, Chunhe Jiang, Ziming Wang, Jianbo Zhong, Zhengjian Liu

School of Metallurgical and Ecological Engineering, University of Science and Technology Beijing, Beijing 100083, PR China

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Keywords: Tuyere sample Blast furnace Microstructure Graphitization	An in-depth understanding about the existence state and structure of materials in the high-temperature zone of a blast furnace is critical to optimize both the ironmaking blast furnace and many other metallurgical processes based on carbothermic reduction. In the present study, coke as well as other accompanied samples (slag, metal and fines) were extracted from an industrial large-scale blast furnace (BF) tuyere zone and were comprehensively characterized to evaluate their existence state and structure. It was found that the weight percentage of slag and metal in the total extracted samples increases first and then keeps at a relative stable level, while the average coke particle size decreases firstly and then keeps at a relatively small size when the position is closer to the blast furnace center. This indicates that smelting and separation of slag and iron as well as the main coke degradation process occur mainly in the center part of BF, and the state in the region (deadman) is relatively stable with similar permeability. Tuyere cokes with various sizes are all extensively reacted with highly developed pores. Due to the improvement of coke pore size, blast furnace melts (bosh slag or molten coke/coal ash) can migrate into the coke matrix through those connected open pores. Coke carrying slag in its inner pores may enter the iron bath of the BF hearth, which might affect coke dissolution into hot metal and degrade the refractory of the hearth bottom. Alkalis content increase significantly when the distance to the tuyere entrance increases, indicating the alkali vapors are mainly recycled and enriched in the center part of blast furnace. The graphitization of coke in the high temperature zone start from the coke surface and the graphitization process may lead to the formation of coke fines.

1. Introduction

The blast furnace (BF) ironmaking process is continuing to evolve under the twin pressures of cost escalation and reductions in greenhouse gas emissions. Tuyere zone, the highest temperature zone inside BF where energy and reducing gas are produced by the coke/coal combustion, is essentially important for the whole blast furnace ironmaking process. Coke, the only solid material remaining throughout the lower high temperature zone of BF, degrades significantly in or near the tuyere zone. By blast furnace dissection study, it was found that mean size of coke decreased rapidly with increasing temperature in the lower regions corresponding to the lower shaft to just above the tuyere level (1400–1600 °C); coke fines were generated due to significant abrasion and impact forces [1]. Researches from Kawasaki Steel and Sumitomo metals showed a similar behavior indicating a $\sim 25\%$ reduction in coke mean size below tuyere level along with $\sim 8.5\%$ reduction in coke strength. Fine generation increased rapidly $\sim 1-3$ m above the tuyere level. Since coke provides the mechanical support for the burden above it and ensures the permeability of the materials column inside a BF [2], this irreplaceable function make coke behavior in the high temperature zones a key factor that influences the operational efficiency as well as campaign life of blast furnace [3,4]. An indepth understanding about coke reaction behavior in the high-temperature zone of a blast furnace is critical to optimize both the ironmaking blast furnace and many other metallurgical processes based on carbothermic reduction if these processes are to be optimized with respect to their carbon/coke consumption.

Direct observation of the coke behavior inside the tuyere zone is impossible due to the harsh environment and limited access to the lower high temperature zone of an operating blast furnace. Recently, useful information about various important inner phenomena in the tuyere zone is obtained by analyzing samples extracted from an operating blast furnace using tuyere drilling technique [5,6]. Using this method, the change of coke characteristics, i.e., carbon structural order

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^{*} Corresponding author at: No.30 Xueyuan Rd, Haidian District, Beijing 100083, PR China. *E-mail address*: likejiang@ustb.edu.cn (K. Li).

Table 1

Blast Furnace operating data during the drilling period.

Item	Value	Item	Value
Coke (kg/thm) Coke nut (kg/thm) Coal (kg/thm) Slag rate (kg/thm) Top dust (kg/thm) Hot blast (Nm ³ /min)	313.36 25.89 165.13 288.73 21.3 8333	Hot metal (t/m ³ /d) Hot metal carbon (%) Hot metal silicon (%) Hot metal sulphur (%) Top temperature (°C) Blast temperature (°C)	2.24 4.44 0.26 0.04 159.10 1213.7
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[5], mineral transformation [7], reactivity and strength [6], and formation/accumulation of new phases [2,8–10] are somewhat understood. However, except solid coke, slag, metal and fines also exist in liquid state in the tuyere level, and they can also be extracted with coke samples simultaneously during the tuyere drilling process. The samples in which coke, slag and metal coexist could provide meaningful information about the interactions between them as well as the whole state in this high temperature region.

In the present study, tuyere samples (coke, slag, metal and fines) were extracted from a large-scale blast furnace, and those samples were carefully separated and comprehensively characterized to reveal their existence state and structure. The weigh percentage evolution of slag and metal in the extracted samples is analyzed firstly. And then, the coke particle size distribution is presented, while the morphology and state of samples with different particle size are analyzed comprehensively by scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectrometer (EDS). Finally, coke graphitization process was discussed based on the X-ray diffraction data of tuyere coke samples. Results in the present study provide evidence to understanding the state of BF high temperature zone.

2. Experiment

The typical drilling process with detailed procedures has been described in earlier publications [11,12]. In the present study, two batches of coke samples were obtained by tuyere drilling of a large blast furnace with its volume larger than 5000 m³. Detailed operation data during the drill period is shown in Table 1. High quality coke is used during this period, with the detailed quality indexes shown in Table 2. This furnace is equipped with 4 tap holes and 42 tuyeres. The hearth diameter is 15 m. Fig. 1 (a) shows the mobile drill used for tuyere coke sampling. In the drilling procedure, a steel tube is inserted from one of the opened tuyere during the stoppage. The actual length of drill penetration in the furnace is limited by the physical conditions of tuyere regions. In the present case, the probe can extend up to 8 m from the tuyere entrance, 0.5 m beyond the center point of the furnace (7.5 m). The actual penetrated length is 6 m, while the actual core length is only 2 m, which is attributed to compression of samples during the drilling. The amount of fines generated due to the drilling procedure is considered to be ignorable compared to the total fines collected from the blast furnace. Various tuyere regions of the blast furnace is presented by the core samples. The actual length of each region in the core varies due to the different working state of the blast furnace. On the basis of past experience [5] and for convenience, the core in this study in divided into four sections with typical tuyere region lengths even though the actual length of each zone in the blast furnace is slightly different (Fig. 1 (a)). The highest temperature zone of a blast furnace is located in the raceway. The "birds nest" region is usually associated with accumulation of hot metal, and this is obvious in Fig. 1 (b) C and C'. The "deadman" region is usually packed with finest materials which tends

Table	2
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CRI, CSR, M40 and M10 values and proximate analysis of charged coke during the tuyere period.

Items	Values (%)
CRI	20.07
CSR	69.49
M40	90.92
M10	5.70
Moisture, ad	0.31
Ash, ad	11.71
Volatile matter, ad	1.23
Fixed carbon, ad	86.05

to stop gas from up flowing, as shown in Fig. 1 (b) D and D'. It is interesting to observe that metal-slag-coke fine compound (MSC) is obvious in the birdnest and deadman regions. Those MSC is thought to decrease the permeability of gas and liquid in the high temperature zone of blast furnace [13,14].

After the drilling process, extracted samples in the steel tube were separated at intervals of 0.5 m. Samples in each interval were sieved by different screens with various screen sizes (20, 10, 5, 1, 0.074 mm) to obtain the particle size distribution. Slag and metal are usually combined together in the extracted samples. They were selected and separated from the samples by hand since no standard method is available. The slag and metal (S&M) rate represents their weight percentage in the total mass of the extracted samples. Coke samples larger than 20 mm represent coke lump which is not greatly reacted and located mainly near the tuyere entrance, while coke samples less than 5 mm represent coke particle that is greatly reacted and located mainly in the center region of blast furnace (far from the tuyere entrance). To reveal the reaction state of tuyere coke samples before and after the high temperature reaction process in the tuyere level, samples larger than 20 mm and less than 5 mm were mounted by resin and were ground and polished similar to the previous study [2,13]. The samples were coated with carbon and then examined with a Quanta 250 Environmental scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectrometer (EDS) for chemical analysis and element mapping. Selected samples were also grinded to powder (less than 74 µm) for Xray Diffraction (XRD) examination. The analysis was conducted using a Rigaku diffractometer (DMAX-RB 12 kW; Rigaku Corporation, Tokyo, Japan) with Cu Ka radiation; the scanning angles were in the range from 10 deg to 90 deg (2 θ) at a scan rate of 10 deg/min.

3. Results and discussion

3.1. Evolution of slag and metal rate

The evolution of S&M rate with the position in the tuyere level is shown in Fig. 2. It can be seen that the S&M rate obtained from the two batches samples increases when the position is closer to the furnace center. The S&M values differ obviously with each other. This may be caused by the different inner-state of the furnace during the two extraction procedures. As shown in Fig. 1, metal drops are more obvious in the second batch samples than that in the first batch samples. In addition, the method to separate slag and metal by hand may also cause some unavoidable errors. Zhu et al. [15] analyzed the slag and metal rate of tuyere samples for a large blast furnace (5500 m³), and it was found that S&M ratio keeps increasing when the distance to the tuyere entrance is less than 2.5 m and keeps relative constant when distance is above 2.5 m. This indicates that smelting and separation of slag and

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