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Temperature Distribution and NO_x Emission Profile in the Sinter Beds Visualized by in-situ Measurement Techniques

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Abstract

All the chemical reactions in the sintering process of the steel industry proceed in a packed bed together with the combustion of coke particles. Although detailed information is desired for pursuing higher productivity and lower environmental load of the process, it is difficult to monitor the inside of the packed bed during combustion. This report demonstrates the experimentally determined temperature distribution and NO_x emission profile during combustion in the packed bed. The in-situ temperature measurement was performed by scanning a sheathed thermocouple in the sinter bed. The information on the temperature variation during sintering showed a clear correlation between the quality of the sinter and the sinter heat pattern for each layer. The time-course profile of NO_x emission was also determined by analyzing the results of in-situ gas measurement during packed bed combustion. Both the emission of NO and the conversion rate were higher in the lower-temperature region in the combustion layer, corresponding to the beginning of coke combustion. The presented in-situ measurement techniques have enabled unprecedented detailed discussions with the data on the temperature and gas composition inside the sinter bed during sintering.

1. Introduction

The steel industry in Japan produces approximately 100 million tons of crude steel every year. Among them, approximately 80 million tons are manufactured in large-scale reactors called blast furnaces.¹⁾ Raw materials such as iron oxides and carbon are charged into blast furnaces to produce metallic iron by reducing the iron oxides. At this time, if powdery particles with a small diameter are charged as raw materials, they block the blast furnaces and hinder the gas flow, which delays the reaction.²⁾ This problem can be circumvented by purchasing and using iron ore that is massive in size. However, most of the actually imported iron ore is small in size. Therefore, iron ore particles need to be agglomerated into an appropriate size by sintering before charge into blast furnaces. Such agglomerated iron ore is called sintered ore and the processes for manufacturing the sintered ore are called the sintering process.

Figure 1 illustrates an outline of the sintering process. Raw materials, such as iron ore, limestone, and coke, are charged into connected pallets and the top of the layer is ignited with a burner while the materials are moving toward the right side in the figure.^{3, 4)} The

ignition front propagates downward along the air flow going down inside the packed bed and forms the sinter bed. To produce 1 ton of pig iron, 1.1 to 1.2 tons of sintered ore are used⁵⁾ and approximately 100 million tons of sintered ore are used in Japan per year. The obtained sinter bed is broken into appropriate sizes and charged into blast furnaces.



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The size of sinter is not the only qualification required for using it in blast furnaces. The chemical states of the sintered ore change the reduction reaction rate in blast furnaces significantly. A lot of research and development has been performed to improve the quality of the sintered ore. Such studies have been conducted as follows: how to charge raw materials,^{6,7)} the composition and composition ratio of the raw materials,^{8,9)} and the grain size distribution are controlled; a burner is ignited, and the quality of the obtained sintered ore is evaluated after the sintering reaction completes. However, monitoring the sintering reaction from the outside of the test equipment is difficult because it proceeds in packed beds.

The temperature exceeds 1 300°C at maximum during the reaction and the combustion propagates downward as time passes. Due to the unsteady reaction condition and continuous movements of the combustion layer, the application of general measurement methods is difficult. In addition, iron oxides in sintered layers absorb electromagnetic waves ranging from microwaves to X-rays intensively. This characteristic of the iron ore makes spectroscopic analyses inapplicable to the sinter bed. This paper summarizes the new in-situ measurement techniques to overcome these problems. Outlines of the experimental setup and some results are presented relating to temperature distribution¹⁰⁾ in sintered layers and analysis results of the combustion in the sintered layers.³⁾ The new techniques would accelerate the development of technologies for controlling the sintering reaction.

2. Main Disclosure

2.1 Visualization of temperature distribution in sinter bed¹⁰⁾

2.1.1 Background

Researchers have been studying how to improve the yield and quality of sintered ore focusing on many parameters. In recent years, changes in the temperature distribution in sintered layers over time (heat patterns) have been gaining attention.^{11, 12)} After igniting the surface of coke with a burner, while the ignition front moves downward, the temperature of the combustion layer gradually changes. The temperature of the upper layer tends to be low while that of the lower layer tends to be high. As a result, the yield for the upper layer tends to be low while that for the lower layer is retained high. However, the lower layer is exposed to excessively high temperatures, so phases that are not appropriate for reaction in blast furnaces tend to appear. To avoid such a phenomenon, a number of technologies have been developed and applied to intentionally vary the quantity and size of coke particles in the vertical direction of the raw material layer.¹³⁻¹⁶ Variation in iron ore type and particle size in addition to coke have also been studied.¹⁷⁾

To study the influence of various measures on heat patterns in sintered layers, several thermocouples are inserted from the side of equipment at intervals of 10 cm or more in sintering reaction tests to measure temperature changes at each point. Inserting more thermocouples enables studying heat patterns more closely. However, the raw material layer at the positions at which the thermocouples are inserted from the side collapses and the permeability is also affected. In addition, air unavoidably flows into the pot through the holes on the side wall of the test equipment. Therefore, when thermocouples are inserted from the side, measurements are limited to several points. Due to the nature of the grain mixture, the sinter bed then becomes unavoidably uneven. The particle-size distribution and chemical composition vary depending on the position in the sinter bed. It is desired for further detailed discussion to monitor temperatures at more spots and evaluate heat patterns considering unevenness between the positions.

Other industries also desire to study temperature distribution in reaction fields closely. There are some reports on the temperature distribution in catalyst layers achieved at high spatial resolution in sub-mm in catalytic reaction tests on a laboratory scale.^{18, 19} In such studies, thermocouples were scanned in the flow direction in the catalyst layers to study the position dependence of the temperature. This study succeeded in applying the approach of scanning thermocouples in packed beds to sintered layers by modifying the approach appropriately.¹⁰ Measurement was difficult in the case of the sintering reaction because the maximum temperature in layers is higher in the case of the sintering reaction than that in the catalytic reaction. In addition, oxide melt was formed, and the combustion layer moved over time. This section describes detailed measurement procedures along with some measurement results.

2.1.2 Experimental

An outline of the experimental procedures is described below. More detailed procedures are shown in a previous study report.¹⁰⁾ As shown in Fig. 2, a sinter pot with an inner diameter of 100 mm and a depth of 440 mm was used. Glass-wool insulation sheets were placed on its inner wall. An alumina tube (TRIO Ceramics Co., Ltd., PTO series), which has an inner diameter of 1.2 mm and an outer diameter of 1.8 mm, was inserted along the central axis of the sinter pot. Raw materials with standard composition were charged into the sinter pot. The surface was ignited with a burner for 60 s and then a high temperature tolerant type-K sheathed thermocouple (Okazaki Manufacturing Company, HOSKINS2300 series), which has an outer diameter of 1.0 mm, was inserted into the alumina tube. An electric linear slider (Oriental Motor Co., Ltd., EAS series) was moved in the vertical direction to measure the temperature and record it with a data logger. The negative pressure of the window box at the bottom of the sinter pot was maintained at 350 mmH₂O throughout the sintering to flow the air to promote the sintering reaction. The temperature measurement with the thermocouple was continued until the sintering reaction completed. After having completed the sintering process, the obtained sinter ore was divided into seven equal parts from the top to the bottom. Each portion was subjected to a shutter test and the particle size distribution was measured.

2.1.3 Results and discussion

This study succeeded in visualizing the temperature distribution in sintered layers and its time variation by scanning the thermocou-



Fig. 2 Schematic of the pot test equipment for in-situ temperature measurement

ple in the vertical direction in the sintered layers to measure the temperature and analyze the data. This section describes the analysis procedures along with part of the discussion based on the acquired data.

Figure 3 shows the position of the thermocouple tip at each sintering time. The thermocouple was moved in the vertical direction to measure the temperature in the vicinity of the combustion layer. As the combustion layer moved downward, the scanned position was also moved downward. There are mainly two reasons why the temperature only near the combustion layer was intensively measured.

The first reason is that the temperature of the other sections is not so important. Other measurements have confirmed that the temperature of the sections before ignition is constant around 70°C. After the combustion completes, the temperature decreased lower than 400°C within a few minutes. The temperature at that section negligibly affects the quality of the sintered ore. The other reason is to improve the time resolution of the measurement. Shortening the travel distance of the thermocouple increases the number of times of reciprocation per unit time, which enables frequent temperature measurement of the combustion layer. **Figure 4** shows the temperature measured with the scanned thermocouple. Many sharp peaks are observed in the figure thanks to many reciprocation operations near the combustion layer. Every upward or downward scan through the combustion layer appears as a peak in the figure.

The results in Figs. 3 and 4 show the correlation between 3D



Fig. 3 Time variation of the thermocouple-tip position during sintering



Fig. 4 Time variation of the monitored temperature during sintering

data, i.e., the time, position, and temperature. **Figure 5** is a 3D graph of this data created with MATLAB. The intervals between the data points are 10 s along the time axis and 2 mm along the place axis. Each data point was obtained by interpolating the obtained data. Figure 5 shows that the lower layers have a longer high-temperature hold time. Particularly, the layers below the combustion layer have higher peak temperatures than the upper and medium layers. These results are consistent with the widely accepted tendency in the sinter bed.^{7, 11, 12} Thus, this measurement method can be used to measure the time variation of temperature distribution in the depth direction of sintered layers at high spatial resolution.

The obtained data allow a more detailed discussion. This paper shows only the relation between the yield of sintered layers and temperature changes on account of limited space. Previous studies have shown that the microstructure of sintered ore is strongly correlated with the high-temperature holding time during sintering reaction.²⁰⁻²²⁾ Generally, the longer the high-temperature holding time, the higher the yield tends to be. The time period during which each layer was retained at 1 100°C or higher was estimated based on Fig. 5 and the result was compared to the measured yield of the corresponding layer. **Figure 6** shows the results. As the yield index for



Fig. 5 Time variation of the temperature distribution



Fig. 6 Average holding time of temperature above 1100°C, and the grain size distribution after the shutter test for each portion of the sinter

evaluation, the ratio of sintered ore particles remaining on a 5-mm mesh was used.

At a depth of 126 mm from the surface, particles with a ratio of 5-mm or larger increased as the holding time of temperature equal to or higher than 1100°C increased. However, in the case of the lower layers from 128 to 436 mm, the relation between the high-temperature holding time and yield is weaker. In the case of the lowest layer of 376 to 436 mm, the ratio of large sintered ore particles (16 mm or more) increases. Thus, studying temperature changes in sintered layers has made it possible to link the quality of sintered ore to temperature changes in the sintered layers. Performing similar measurements while changing the segregation conditions of raw materials and ignition conditions may enable quantifying the degree of effects these conditions will exert and at which depth.

2.2 Visualization of burning profile through analysis of gas in sintered layers³⁾

2.2.1 Background

Japan uses approximately 100 million tons of sintered ore every year. Such enormous production consumes huge energy and emits enormous flue gas. Previous studies have shown that NO_x emissions are particularly high.^{23,24)} Approximately 35% of NO_x released from an entire integrated steelworks comes from the sintering process.²⁵⁾ Reducing environmental stress from the sintering process is also an important issue. Therefore, a lot of research and development has been conducted to reduce NO_x emissions for an extended period of time.

Many steelworks use gas treatment equipment to remove NO_x in exhaust gas from the sintering process. NH_3 reacts with NO_x in exhaust gas to reduce NO_x to N_2 on activated carbon or catalysts in the equipment. In order to reduce NO_x emissions within the sintering process, coal type selection and coke pretreatment procedures have been developed and put to practical use.

Further studies would reduce NO_x emissions in the sintering process. However, such studies are very difficult.

One reason is the unsteady burning profile in sinter beds. NO_x emission from burned solid fuel has been studied for many years. Previous studies have shown that the NO_x emission is under the significant influence of the combustion temperature, ^{26, 27)} fuel particle size, ^{27, 28)} burning atmosphere, ²⁶⁾ and compound type that comes into contact with fuel.^{29–31)} In the sintering process, all the aforementioned conditions change simultaneously as time passes. The combustion conditions change from moment to moment. No technique directly identifies when NO_x is released from where and to what extent just by monitoring the gas composition. Therefore, it used to be impossible to discuss which measure contributed to which part of NO_x emissions reduction and to what extent. This has hindered the promotion of studies.

I have been working to visualize NO_x emission profiles as a result of combustion in sintered layers to accelerate the development of NO_x emissions reduction technology. Coke that was widely used in the sintering process was used in an experiment under general operation conditions to discuss NO_x emission profiles. This chapter describes the results.³⁾

2.2.2 Experimental

An outline of the experimental procedures is described below. More detailed experimental conditions and measurement procedures are described in a previous report.³⁾ The blending ratio of raw materials was standard and 3.8 wt% of coke was added as fuel. **Table 1** lists the chemical composition of the coke used. The content of the ash was determined in accordance with JIS M 8812, those of C, H,

Table 1 Ultimate and proximate analysis results of coke

	C	Н	0*	Ν	S	Ash
Ratio (dry basis, wt%)	85.5	0.3	2.7	1.1	0.6	9.8

* Amount of oxygen was calculated by subtracting other values from 100%.



Fig. 7 Schematic of in-situ gas measurement during combustion

and N were determined in accordance with JIS M 8819, and that of S was determined in accordance with JIS M 8813. The content of O was calculated using the difference.

Figure 7 illustrates an outline of the experiment. A sinter pot with an inner diameter of 100 mm and a depth of 440 mm was used. Glass-wool insulation sheets were placed on its inner wall. The aforementioned raw materials were charged into the sinter pot. From a hole on the side of the sinter pot (120 mm from the surface), a high temperature tolerant type-K sheathed thermocouple (Okazaki Manufacturing, HOSKINS 2300 series), which has an outer diameter of 1.6 mm, for temperature measurement and an alumina tube for sampling gas (TRIO Ceramics, PTO series), which has an inner diameter of 1.2 mm and an outer diameter of 1.8 mm, were inserted. At this time, the end of the thermocouple was almost aligned with that of the alumina tube for sampling gas. Both were set near the central axis of the raw material layer. The gas sampling tube was connected to the mass flow controller, diaphragm pump, and nondispersive infrared gas analyzer (Yokogawa Electric Corporation, IR200) via a cold trap, quadrupole mass spectrometer (Pfeiffer Vacuum GmbH, OmniStar GSD 320), and filter as shown in Fig. 7.

Sampling of the gas was started using the gas sampling tube at 0.5 L/min. Then, the surface was ignited with a burner for 60 s to initiate sintering reaction at a constant air flow rate of 150 L/min. The sampled gas was dehumidified at the cold trap and then analyzed with the quadrupole mass spectrometer and infrared gas analyzer. The quadrupole mass spectrometer was used to obtain the following m/z values: 18 (H₂O⁺), 28 (N₂⁺), 30 (NO⁺), 32 (O₂⁺), 40 (Ar⁺), 44 (CO₂⁺), and 64 (SO₂⁺). The infrared gas analyzer was used to obtain the concentrations of CO and CO₂. Both CO₂ concentrations were compared to evaluate whether the measurement results were valid.

2.2.3 Results and discussion

Figure 8 shows the analysis results of the gas sampled from the side during the sintering reaction. The figure shows that the temperature at the measurement point and the sampled gas composition change as time passes. At the measurement time point of 530 s, the

 CO_2 concentration is high at approximately 10% while it is almost zero at 680 s. This change in the gas composition over time can be explained based on the relative position of the sampling position and combustion layer. As shown in **Fig. 9**, at around 550 s, the sampling position is located at the lower end of the combustion layer. Therefore, the gas emitted from the combustion layer was sampled. At around 650 s, the sampling position is located near the upper end of the combustion layer. As the air about to flow into the combustion layer was sampled at this time, the composition of the sampled gas was almost the same as that of the air. Between 550 s and 650 s, the sample has an intermediate composition.

As described above, the composition of the sampled gas is determined based on the relative position between the combustion layer and sampling position. The measurement results at the measurement time point of 550 s correspond to the gas composition in the lower section of the combustion layer. The results at 650 s correspond to the gas composition in the upper section of the combustion layer. Considering that the combustion layer moves downward at a nearly constant rate, the horizontal axis in Fig. 8 can be converted to the relative sampling positions in the combustion layer. In this test, the



<u>Sampling</u> <u>position</u> <u>in</u> Bottom Middle Top <u>Layer</u>

Fig. 9 Time variation of the sampling position in the combustion layer

travel rate of the combustion layer was calculated as 16.3 mm/min. **Figure 10** shows the gas composition distribution at relative positions in the combustion layer acquired using this value.

The values of each gas concentration were averaged over 3.75 mm, because it was estimated that the gas was mixed and agitated by a vortex in the sintered layers. This representation of the gas composition in the relative position enabled the discussion of the gas composition from the viewpoint of gas flow. **Figure 11** illustrates this gas flow. The air flows in from the upstream side (right side in Figs. 10 and 11) of the combustion layer and the coke is combusted in the combustion layer using O_2 in the air, which releases CO_2 and NO; therefore, the concentrations of CO_2 and NO increase towards the downstream side (left side in Figs. 10 and 11).

The dependence of the gas composition on the relative position enables the discussion of the gas composition from the viewpoint of gas flow. Specifically, the gas generated at each position mixes into the downstream gas and the amount of gas consumed at each position becomes less in the downstream gas. The gas emission amount and consumption at each position were calculated by the differentiation of the gas composition with the position. The molar flow rate of inert gas can be considered as constant during combustion. Since the experiment was performed at a constant air flow rate into the sinter bed, the molar flow rate of N₂, which is inert gas in the air, can be regarded almost equal. Therefore, the equations below hold

$$F_{N_2} \times \frac{d \frac{C_{CO_2}(x)}{C_{N_2}(x)}}{dx} = A_{CO_2}(x), \qquad (1)$$

$$F_{N_2} \times \frac{d \frac{C_{O_2}(x)}{C_{N_2}(x)}}{dx} = A_{O_2}(x), \qquad (2)$$

$$r_{N2} \times \frac{d \frac{C_{NO}(x)}{C_{N2}(x)}}{dx} = A_{NO}(x),$$
 (3)

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$$F_{N_2} \times \frac{d \frac{C_{CO}(x)}{C_{N_2}(x)}}{dx} = A_{CO}(x).$$
 (4)



Fig. 10 Gas composition at each relative position inside the combustion layer; a positive value corresponds to the upward direction

Where, x is the relative position, F_{N_2} is the molar flow rate of N₂, $C_{\chi}(x)$ is the molar concentration of gas type X, and $A_{\chi}(x)$ is the emission amount of gas type X per unit thickness.

From the equations above, the gas emission amount at each position (**Fig. 12**) was calculated by numerical differentiation of the gas concentration with the position (Fig. 10). Figure 12 shows that around 580 s, the temperature in the layers increases and a large quantity of O_2 is consumed by the combustion of the coke, which releases CO_2 and NO. Around 630 s at which the temperature of the sintered layers becomes stable, the coke has completely burned and thereby both gas consumption and emission are small. This indicates that the combustion of coke proceeds more in the front section (lower layer side) in the combustion layer, in particular. The NO emission amount is also high at that position.

The quantity of gas released at the position was calculated by differentiating the gas concentration at each position in the combustion layer. This confirms that the NO emission amount is particularly large in the lower layer side in the combustion layer. To discuss techniques to reduce the NO emission amount, another index called the NO_x conversion rate is required.^{27, 32}) The NO_x conversion rate



Fig. 11 Schematic of gas composition change inside the combustion layers



Fig. 12 O₂ consumption and NO emission at each position during combustion and the reaction time

refers to the ratio of N contained in coke that is converted to NO_x . The ratio of contained N varies depending on the carbon material used as fuel. Using coal containing a large quantity of N naturally emits more NO.

Even when the same amount of N is contained, the NO emission amount can be reduced if the NO_x conversion rate is low. Therefore, the NO_x conversion rate is kept low in order to reduce the NO emission amount from the sintering process. Further analysis was performed to estimate the NO_x conversion rate at positions in the combustion layer. On the assumption that N is almost evenly present in coke particles and the combustion propagates from the outside of the coke particles according to the unreacted nucleus model, the NO_x conversion rate at the positions can be estimated from the quantity of O₂ consumed during the combustion of the coke and NO emissions using Equation (5) shown below. Where, M_c and M_N refer to the molar ratios of C and N in the coke, respectively and A_x is the emission amount of gas type X at a focused point.

$$\eta(\%) = \frac{M_C}{M_N} \times \frac{A_{NO}}{A_{O2} \times \frac{A_{CO2} + A_{CO}}{A_{CO2} + \frac{1}{2}A_{CO}}} \times 100$$
(5)

Figure 12 also shows the NO_x conversion rate value at each point in the combustion layer calculated using Equation (5). Comparing them to the emission amount of each gas type in the graph shows that the NO_x conversion rate is high at the positions with large CO₂ and NO emission amounts. This confirms that both the NO emission amount and NO_x conversion rate are high on the lower layer side in the combustion layer.

Although many previous reports showed the composition of flue gas emitted as a result of combustion in sintered layers, this report is the first to show the NO_x conversion rate distribution in the combustion layer. This technique has clarified the NO_x emission profile in sintered layers, which has enabled discussion on which part various measures implemented to reduce NO_x emissions are effective. The developed method will visualize the influence of various techniques on the NO_x emission amount and use the acquired knowledge to contribute to developing techniques to further reduce the NO_x emission amount.

3. Conclusion

In the sintering process, the sintering reaction proceeds in the sinter bed charged in movable pallets. Observation of the reaction due to combustion in packed beds is difficult from the outside, which hinders development for further increasing the sinter productivity and reducing environmental stress. This study visualized the temperature distribution in sintered layers during the sintering reaction as well as NO_x emission due to combustion. Regarding the temperature measurement, a thermocouple was vertically scanned in the sinter bed and the obtained temperature data was analyzed. This analysis enabled visualizing the time variation of the temperature distribution in the packed bed. Regarding NO_x emission during combustion, the sampled gas from the sinter bed was analyzed to clarify the time variation of the gas composition during combustion. This confirmed that both the NO_x emission amount and NO_x conversion rate are high on the lower layer side in the combustion layer. Nippon Steel Corporation will apply these analysis techniques to perform tests under various operation conditions to verify the effects of existing technologies and use them for technology development in the future.

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