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Application News

Electron Probe Microanalyzer EPMA-8050G

Analysis of Sintered Ore for Steel Manufacturing after Hydrogen Reduction

Satoshi Yoshimi, Hiroshi Hayashi

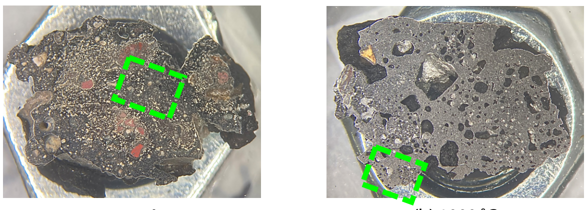
User Benefits

- The mineral structure of sintered ore can be investigated by quantitative mapping of Al, Mg, Si, Ca, and Fe.
- This technique is useful in research on the composition of the calcium ferrite phase due to differences in the reduction reaction depending on the furnace temperature.
- Phase analysis by quantitative mapping of O and Fe is useful in research on the species of iron oxides.

■ Introduction

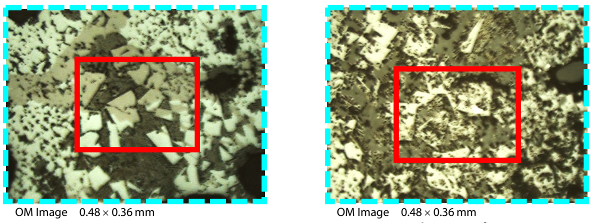
Reduction of carbon dioxide (CO_2) emissions is being promoted with the aim of achieving carbon neutrality in 2050. In the hydrogen reduction ironmaking process, CO_2 emissions are decreased by reducing iron ore by using hydrogen (H_2) in place of coke (C) to form water (H_2O). In Japan, research on a hydrogen reduction technology using hydrogen in the blast furnace (COURSE50) is now underway, including study of reduction of the blast furnace heat requirement and optimization of the raw material reaction conditions, as well as circulating use of gas with a regenerated reduction capacity, by promoting hydrogen reduction, which is a smaller endothermic reaction than direct reduction by coke. The target of the COURSE50 project is a CO_2 reduction of 10 % or more, and development of Super COURSE50 has also begun with the aim of injecting a larger amount of hydrogen into the blast furnace.

This article introduces an example of analysis of sintered iron ore after hydrogen reduction, in which an EPMA™ (EPMA-8050G) electron probe microanalyzer was used.



(a) 600 ˚C (b) 1000 ˚C

Fig. 1 External Appearance of Sintered Ore after Reduction



(a) 600 ˚C (b) 1000 ˚C

Fig. 2 Optical Microscope Images of Sintered Ore after Reduction

■ Sintered Ore Structure after Hydrogen Reduction

One issue when attempting to expand hydrogen reduction by the blast furnace process is maintaining a high temperature in the furnace. Because the reduction reaction using hydrogen is an endothermic reaction that absorbs heat, the temperature distribution in the blast furnace changes, and low temperature reduction powdering of the burden materials becomes remarkable due to expansion of the low temperature region. Heating is necessary to suppress this powdering, which results in an increase in coke use.

Fig. 1 shows the external appearance of sintered ore heated to (a) 600 ˚C and (b) 1000 ˚C using an experimental apparatus simulating the conditions in a blast furnace.

Fig. 2 shows EPMA optical microscope images (OM-images) of the sintered ore heated to 600 ˚C and 1000 ˚C.

The elemental distribution images in Fig. 3 and Fig. 4 are the results of a mapping analysis of the red regions in the OM-images, in which each element was converted to the wt% concentration of the simple oxide (in the case of Fe, Fe_2O_3). (See Related Application News 1.)

Fig. 3 is the sintered ore that was reduced to the low temperature of 600 ˚C under a high-hydrogen blast furnace condition. Since the reduction reaction was incomplete, the region of high contrast in the COMPO image is iron oxide. It can be understood that calcium oxide (CaO), which is the basic component of calcium ferrite, shows a correlation with silica (SiO_2), while the basic component magnesium oxide (MgO) shows an inverse relationship with SiO_2. Furthermore, iron oxide decreases as the alumina (Al_2O_3) in the calcium ferrite increases.

Fig. 4 is the sintered ore reduced to the high temperature of 1000 ˚C under the high-hydrogen blast furnace condition. Here, the calcium ferrite around the rhomboidalized secondary hematite with a melt-type microstructure has decomposed and formed calciowustite (FeO-CaO), which has a high concentration of Fe, and slag (or olivine). This indicates that the calcium ferrite was reduced. In addition, the formation of metallic iron (Fe) in grains of iron oxide can be observed. From this, it is clear that reduction of the iron oxide has progressed.

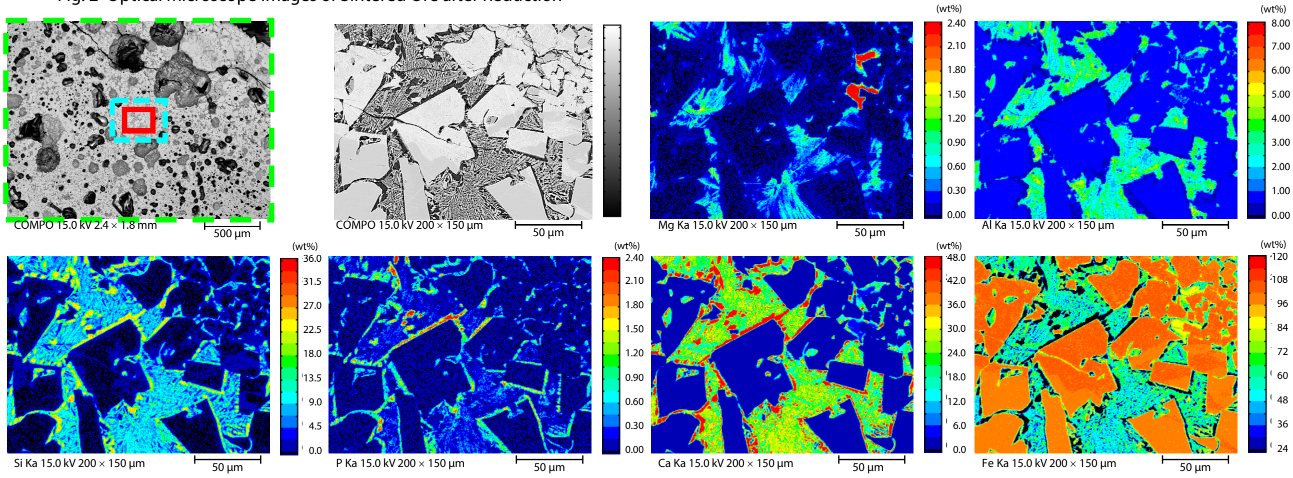


Fig. 3 Mapping of Sintered Ore after 600 ˚C Reduction

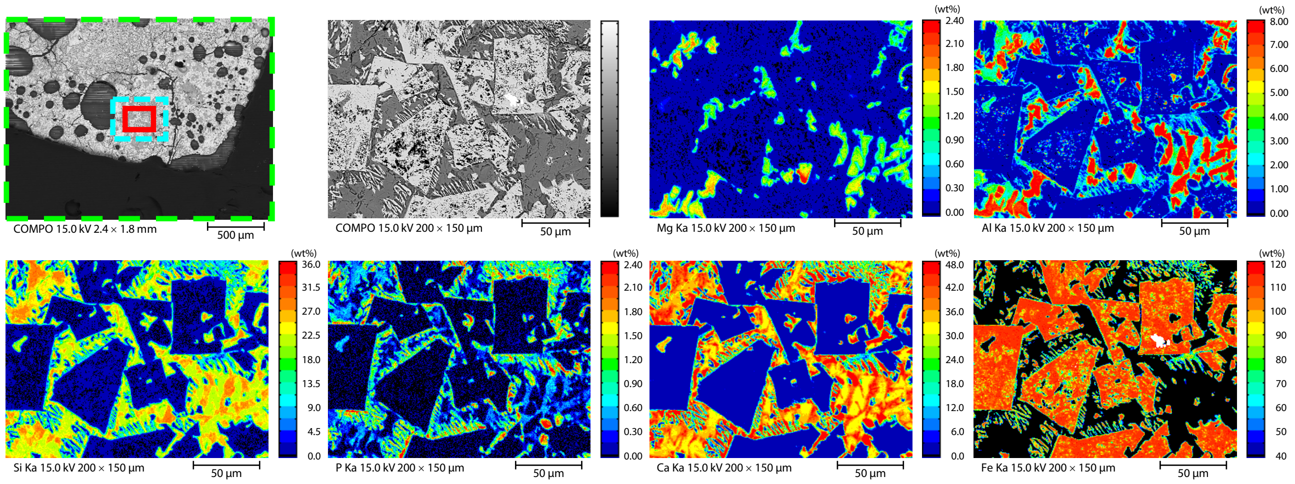
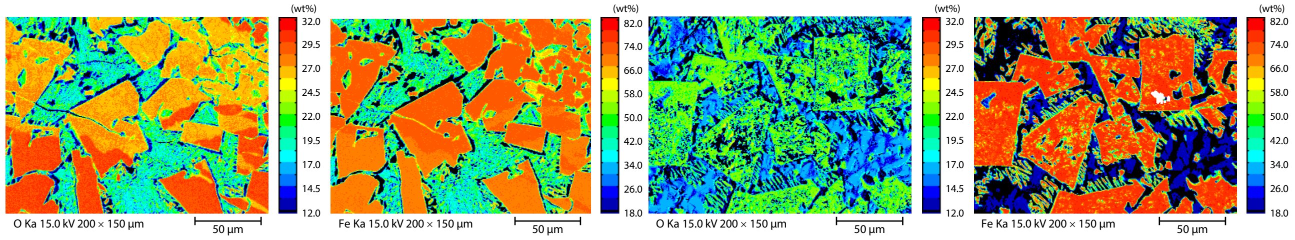
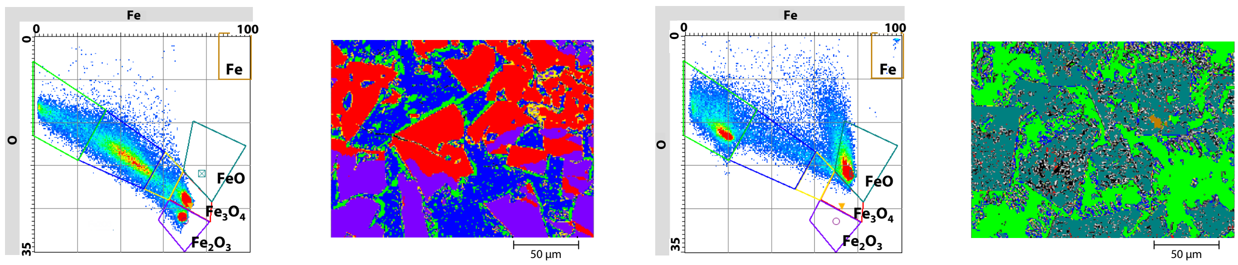


Fig. 4 Mapping of Sintered Ore after 1000 ˚C Reduction



(a) 600 ˚C (b) 1000 ˚C

Fig. 5 Mapping of O and Fe of Sintered Ore after Reduction



(a) 600 ˚C: Fe-O Scatter Diagram (b) 600 ˚C: Phase Diagram (c) 1000 ˚C: Fe-O Scatter Diagram (d) 1000 ˚C: Phase Diagram

Fig. 6 Phase Analysis of Sintered Ore after Reduction

■ Phase Analysis of Sintered Ore after Hydrogen Reduction

In order to identify differences in the oxidation valence of the oxides, the element distribution images in Fig. 5 show oxygen (O) and iron (Fe) by the weight concentrations of the simple elements.

Fig. 6 (a) and (c) are Fe-O scatter diagrams showing the positions of compounds by the theoretical concentration of iron oxide. In Fig. 6 (a), the symbols ○ and ▼ indicate hematite (Fe_2O_3) and magnetite (Fe_3O_4), respectively, and in Fig. 6 (c), clusters of dots can be seen in the regions showing ⊠ wustite (FeO) and iron (Fe) compounds. Fig. 6 (b) and (d) are phases diagrams in which filters were set by the Fe-O scatter diagrams (see Related Application News 1). In the phase diagrams, purple regions are hematite, red regions are magnetite, green is wustite, brown is iron, yellow and blue regions are calcium ferrite, and yellow-green regions are slag. Although the sintered ore contains hematite and magnetite before reduction, it can be understood that the species of iron oxides formed by the reduction reaction differ depending on differences in the furnace temperature, and metallic iron (Fe) has formed in the sintered ore subjected to hydrogen reduction at the high temperature of 1000 ˚C

■ Conclusion

In this experiment, it was possible to identify the species of iron oxides and calcium ferrite by a quantitative mapping analysis and phase analysis of sintered ore after hydrogen reduction, in which the EPMA-8050G was used, thus demonstrating that this technique can be used in structural analyses of sintered ore and research and development related to ore reduction. This technique is also expected to be used in research and development preconditioned on the use of low-grade iron ores.

<Acknowledgement>

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<Related Applications>

1. Analysis of Sintered Ore for Steel Making, [Application News No.01-00584-EN]

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