

In situ observation of reduction of iron oxides at high temperatures

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Introduction

Sintered iron ores with lime stone are used as raw material for a blast furnace. The required properties for sintered ores, a mixture of Fe₂O₃, Fe₃O₄, and calcium ferrite (FeCa_xO_y), are the mechanical strength and the reactivity with reduction gas. The reduction properties are evaluated by heating the sintered ore with a gas of CO/N₂ = 30/70(vol.% ratio) at $T = 1173$ K and the amount of reduced iron was measured by the compositional analysis (JIS M 8713). The reaction of reduction has been measured with quenched specimens using the techniques as described. But the time resolution of the analysis is as long as *ca.* min., and the quenching method results in difficulty in quantitative analysis of reaction rates.

Experiments

A special reaction cell for *in situ* XAFS measurements[1] was used. The cell is made of stainless steel with Kapton®-film windows which an X-ray beam can pass through. A specimen powder was mixed with a powder of hexagonal boron nitride (BN) for 40 min. and the mixture was pressed into a shape of tablet by a pressure of *ca.* a few tenths MPa. A typical size of the tablet was 7 mm in diameter and 2-3 mm in thickness. The specimen can be heated up to *ca.* 1223 K by rod heaters located above the specimen in a flow of gas. The temperature of the specimen was monitored with a thermo-couple located above the specimen holder.

In situ XAFS measurements were carried out with a transparent geometry. The specimen was heated up to a specific temperature in a flow-gas of 20 vol.%O₂/He. The flow-gas was switched into a various concentrations of H₂-He, and the change of XAFS spectra were measured with QXAFS technique [2] with an interval of 15-60 sec. Experiments were conducted at a bending beam-line of BL-9A and 12C at PF, KEK, Tsukuba, Japan.

Results and Discussion

Fe₂O₃ and CaFe₂O₄ powders were heated up to $T = 1173$ K in a flow-gas of 20 vol.%O₂/He. The flow-gas was switched into a vol.%H₂/He. XAFS spectra were measured with an interval of 15 sec. Fractions of Fe(0)

and Fe(II) were determined by analysis of each XAFS spectrum. Figure 1 shows the change of fraction of Fe(0) during the reduction.

It has been shown that the reduction speed of Fe₂O₃ is *ca.* 1.5 times as high as that of CaFe₂O₄ [3]. In the CaFe₂O₄ crystal, calcium atoms are located at the 3rd neighboring site of iron atoms. Thus, it is easily expected that the existence of calcium atoms reduces the speed of Fe-reduction where oxygen atoms located at the 2nd neighboring sites react with hydrogen gas.

Further experiments are expected give fundamental information on the mechanism of reduction in the iron oxide system.

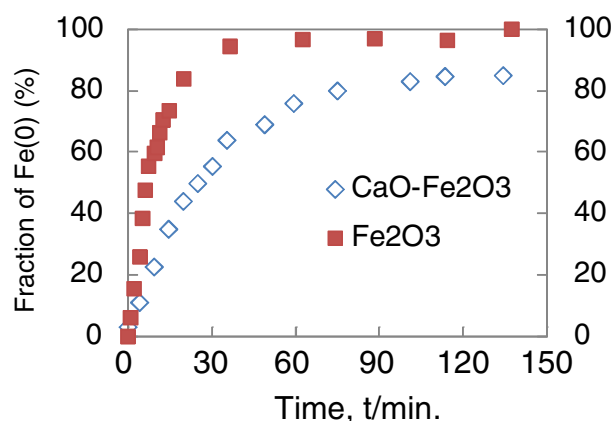


Fig.1 Relation between the fraction of Fe(0) and the time after flowing the reduction gas.

References

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