In situ observation of sintering in CaO-Fe$_2$O$_3$ system at high temperatures (II) 
“The first “Continuous cooling transformation (CCT)” concept for iron ore sintering

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1 Introduction

Phase equilibrium of Fe$_2$O$_3$-CaO system has a great importance in the process of iron making. Sintered iron ores with lime stone are used as raw material for a blast furnace. The process of sintering proceeds at a temperature higher than 1773 K and the sintered ores are cooled down before the thermal equilibrium attained. The required properties for sintered ores, such as the mechanical strength and the reactivity with reduction gas, are largely affected by the types of coexisting phases and their fractions, and its microstructure. Thus in situ observation of the change of structure during sintering processes is of a great importance.

2 Experiment

A special reaction cell for in situ X-ray diffraction was developed [1]. Powder specimens are heated in various gas up to $T = 1773$ K. The reaction cell was mounted on a special goniometer [2] which can maintain the specimen in a near-horizontal position while scanning a detector in both an in-plane and out-of-plane directions [3]. An area detector, PILATUS® (PIXeli Apparatus for the SLS, DECTRES and Rigaku), was used in order to measure a part of diffraction Debye-ring in a short period.

Powder specimens, a mixture of Fe$_2$O$_3$ and CaO with various ratios, were mounted in the center of the reaction cell. They were heated in air up to $T = 1773$ K, and the change in the diffraction patterns were measured using an X-ray beam with a size of $1 \times 1$ mm$^2$ and $\lambda = 0.178897$ nm. Experiments were conducted at a bending beam-line of BL-6C at PF, KEK, Tsukuba, Japan.

In situ and real-time observation of microstructures were performed using an in situ laser microscope.

3 Results and Discussion

The specimens Fe$_2$O$_3$: CaO = 64:36, 80:20, and 90:10 (mass%) were heated up from 300 to 1773 K at $5.0 \times 10^{-1}$ K/s, and cooled down to 300 K with different cooling rates: $-3.3$ K/s, $-8.3 \times 10^{-1}$ K/s, and $-8.3 \times 10^{-2}$ K/s, for simulation of industrial processes. Reaction schemes are basically the same as expected from the quasibinary phase diagram, but in situ observation both of crystal structure and microstructure successfully revealed the effects of heating and cooling rates on the sintering reaction in the CaO-Fe$_2$O$_3$ system with special attention to overheating and overcooling phenomena. The first continuous cooling transformation (CCT) concept for iron ore sintering was proposed to understand overheating phenomena when the molten oxide cooled down to room temperature and magnetite (Fe$_3$O$_4$), hematite (Fe$_2$O$_3$), and various types of calcium ferrite were formed. Figure 1 shows the first CCT diagram for sintering of specimen with Fe$_2$O$_3$: CaO = 90:10 (mass%). Solid lines show the time-temperature curves for cooling rates: (i) $-3.3$ K/s, (ii) $8.3 \times 10^{-1}$ K/s, and (iii) $-8.3 \times 10^{-2}$ K/s.

The CCT diagram for sintering provides crucial and fundamental information on the sintering accompanying solidification, precipitation, and formation of calcium ferrites from the molten oxide, and can be used as a guideline for controlling sintering processes [4].

![CCT Diagram](http://example.com/cct_diagram.png)

Figure 1 the first CCT diagram for sintering of specimen with Fe$_2$O$_3$: CaO = 90:10 (mass%).

References


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