Materials Science

6C/2010C002

Development of *in situ* observation of X-ray diffraction at high temperatures

Masao KIMURA[1], Tohru TAKAYA[1], Noriaki OHTA[2]
[1] Adv. Tech. Res. Lab., Nippon Steel Corp., Futtsu, Chiba, 293-8511, Japan
[2] Nippon Steel Technoresearch, Chiba 293-8500, Japan

Introduction

Phase equilibrium of Fe_2O_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.

Experiments

A special reaction cell for *in situ* X-ray diffraction was developed [1]. Powder specimens are heated in various gas up to T = 1473 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® (PIxeL 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 FeOOH, Fe_2O_3 and CaO with various ratios, were mounted in the center of the reaction cell. They were heated in air up to to T = 1773 K, and the change in the diffraction patterns were measured using an X-ray beam with a size of 1 X 1 mm^2 and E = 6932 eV. Experiments were conducted at a bending beam-line of BL-6C at PF, KEK, Tsukuba, Japan.

Results and Discussion

A mixture of FeOOH (74 mass%), Fe_2O_3 (18 mass %), and $CaCO_3$ (8 mass %) was heated with a rate of 20 K/min. up to T = 1773 K, and was kept for 10 min, at T = 1773 K, and then cooled down to a room temperature in air. Diffraction patters were measured with an exposure time of 40 sec. Figure 1 shows a typical example of diffraction pattern measured. Debye-rings of calcium ferrite ($FeCa_xO_y$) as well as Fe_2O_3 were clearly observed.

Detailed analysis of the diffraction patterns showed the following reaction.

(1) *On heating: ca.* 523 K < T < 623 K
Dehydration of goethite forms hematite:
$2FeOOH \rightarrow Fe_2O_3+H_2O$.

(2) *On heating:* T > *ca.* 1673 K
Formation of liquid of Fe_3O_4-CaO

(3) *On cooling:* T < *ca.* 1450K
Formation of calcium ferrite ($FeCa_xO_y$).

Further experiments are expected give crucial information on the sintering process of iron ores.

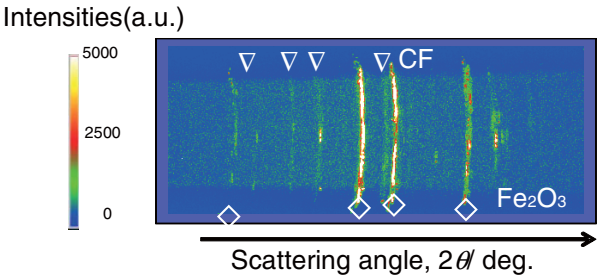

Fig.1 Diffraction pattern of the specimen at T = 1373 K. Each diffraction is assigned to Fe_2O_3 (diamonds) and calcium ferrite (triangles).

References

[1] Kimura, M., & Takayama, T. (2010). *Development of rapid X-ray diffraction system at high temperatures for observation of sintering.* Paper presented at the The 160th fall meeting of ISIJ, Sapporo, Japan.
[2] Kimura, M., Kihira, H., Ohta, N., et al.: Corros. Sci., 47, 2499-2509 (2005).
[3] Kimura, M., Acosta, A., Fujioka, H., et al.: J. Appl. Phys., 93(4), 2034-2040 (2003).