

***In situ* observation of reduction kinetics in iron-ore sinters**Masao KIMURA^{1,2*}, Reiko Murao³, Yasuhiro Niwa¹, and Ken'ichi Kimijima¹¹ Photon Factory, Institute of Materials Structure Science,

High Energy Accelerator Research Organization, Tsukuba, 305-0801, Japan

² Dept. Mater. Structure Sci., School of High Energy Accelerator Sci., SOKENDAI
(The Graduate University for Advanced Studies) Tsukuba, Ibaraki 305-0801, Japan³ Adv. Tech. Res. Lab., Nippon Steel & Sumitomo Metal Co., Chiba 293-8511, Japan**1 Introduction**

Iron oxides and calcium-ferrites (CFs: Fe-Ca-O) as the bonding layer are major phases in iron-ore sintered ore. The amounts and types of oxides and CF phases and their microstructures affect properties of the sintered ore, such as strength, reduction-ability and reduction-disintegration. In this study, the reduction kinetics of each oxide and CF phase was quantitatively investigated using a powder CF by *in situ* XAFS (X-ray absorption fine structures) measurements at temperatures up to 1173 K.

2 Experiment

In-situ quick XAFS measurements were conducted at BL9A and BL9C, Photon Factory, IMSS, KEK. Details of the experimental set-up for high temperature XAFS measurement was well described in the previous report¹. Sample was diluted with BN to set $\Delta\mu t = 1.0$ -1.5. Fe *K*-edge and Ca *K*-edge XANES spectra were corrected at 1173K under He-20vol%H₂ or He-20vol%CO atmosphere with transmitting methods with a typical time interval of 30 s.

3 Results and Discussion

Figure 1 shows a typical example for Fe₂O₃ with He-20vol%H₂ as reductive gas. Time-evolution of XANES spectra around Fe *K*-edge ($t = 8$ to 20 s) (Fig.1 (a)) exhibits isosbestic points at $E = 7122$ and 7143 eV (shown by "1" and "3" in Fig.1) and implies the existence of two chemical states: Fe^{III} and Fe⁰. This was confirmed by fitting each spectrum by the linear combination of that of Fe^{III} and Fe⁰. As a simple indicator, the edge energy showed the progress of reduction reaction (Fig.2).

The same measurements were carried out to evaluate the reaction speed of Fe₂O₃, Fe₃O₄, FeO, and CaFe₂O₄ in powder for H₂ and CO gas flow. The reaction rate $k_{\text{III-II}}$ from Fe^{III} to Fe^{II} was 1-2 min⁻¹ and ten times higher than that of $k_{\text{II-0}}$ from Fe^{II} to Fe⁰ (*ca.* 0.2 min⁻¹) for H₂ gas flow. In the case of CO gas, reaction rates of both reactions became smaller with a factor of 3-5 times.

Comparison of XAS around Fe *K* and Ca *K*-edges showed the reduction accompanied the decomposition of CFs. The reaction rates of each reaction differed among CFs, which can be explained by the Ca/Fe ratios.

It was shown that (a) Ca/Fe ratios of CF phases, (b) the type of reductive gas, and (c) the location of CFs relative to pores are significant factors in the reduction process of iron sinters.

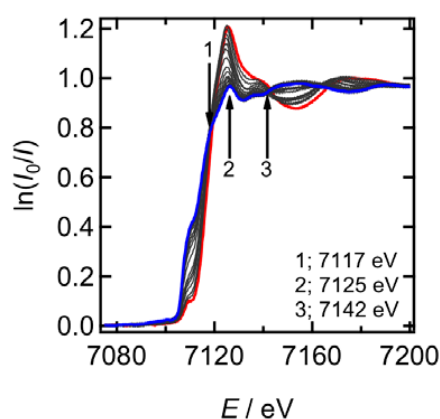


Figure 1 Time evolution of XAFS spectra for Fe₂O₃ with He-20vol%H₂ as reductive gas.

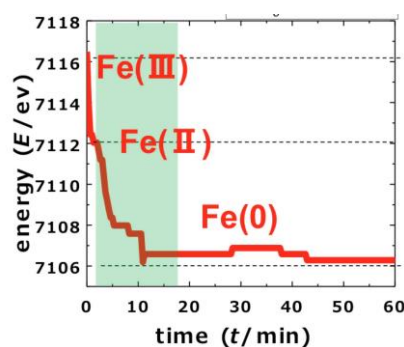


Figure 2 The change of absorbing edge corresponding to the chemical states obtained from the time evolution of XANES spectra as shown in Fig.1.

References

[1] M. Kimura, *et al.*: *Journal of Physics: Conference Series*, 430, (2013), 012074.

* masao.kimura@kek.jp