

## In Situ Observation of Corrosion at liq./sol. Interfaces (II)

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### Introduction

Electrochemical reactions are widely found in applicative as well as fundamental fields, such as corrosion, battery, catalysis and so on. One of its characteristics is that they occur at liquid/solid interfaces. A new system (“EVA”[1]) was developed for *in situ* observation of reactions at the interface by G-GIXS[2]. In this study, a new system was utilized for *in situ* observation of steel corrosion.

### Experiments

In order to perform *in situ* observation of electrochemical reactions at liquid/solid interfaces, a of new system (“EVA”) has been developed. A part of the goniometer of the “EVA” system was shown in Fig.1. A specimen surface under a liquid film can be investigated by X-ray scattering. Changing the energy of incident beam and the incident angle of x-ray can control the measured depth easily.

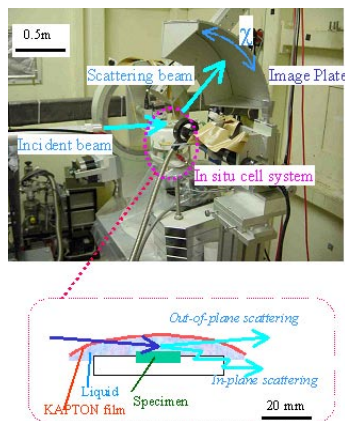


Figure 1 A part of the goniometer of the “EVA” system (above) and a sketch of *in situ* cell system (below).

*In situ* measurements were performed for a block specimen of the advanced weathering steel (Fe-0.4Cu-2.0Ni, in mass%) [3]. The surface of the specimen was covered with a thickness of ca. 20  $\mu\text{m}$  of diluted artificial seawater and then dried in air at 300 K. One wet-dry cycle was about 90 min. X-ray diffraction patterns were measured with  $E=12.399$  keV using an image plate at BL-3A at PF, KEK, Tsukuba, Japan.

### Results and Discussion

Figure 2 shows scattering intensities at (a)  $t=0$ , (b)  $t=33$ , and (c)  $t=91$  min. after the seawater covered the surface. The angle of incidence was kept at 1.0 degree. At  $t=0$

min. surface was covered by the film of sea water and the diffraction intensities from the substrate (bcc iron) were observed. At  $t=33$  min. the surface was still wet but the thickness of sea-water film was less than 20  $\mu\text{m}$ . Formation of  $\text{Fe}(\text{OH})_2$  was clearly observed. At  $t=91$  min. the surface was almost dry. Diffraction peaks show the formation of  $\gamma\text{-FeOOH}$  and  $\text{Fe}_3\text{O}_4$ .

These results show early stage of reactions of the advanced weathering steel. When the surface gets wet, dissolved ferrous ions form  $\text{Fe}(\text{OH})_2$ . In the transition from a wet to a dry state,  $\text{Fe}(\text{OH})_2$  transforms into  $\gamma\text{-FeOOH}$  and  $\text{Fe}_3\text{O}_4$  through oxidation by oxygen in air and seawater.  $\text{Fe}_3\text{O}_4$  remained in the following dry cycle in the case of advanced weathering steel, whereas  $\text{Fe}_3\text{O}_4$  was easily oxidized to form  $\alpha$ - and/or  $\gamma\text{-FeOOH}$  in a dry cycle in the case of conventional weathering steel[4]. This is the unique feature is deeply relating with high corrosion resistance of the advanced weathering steel [5].

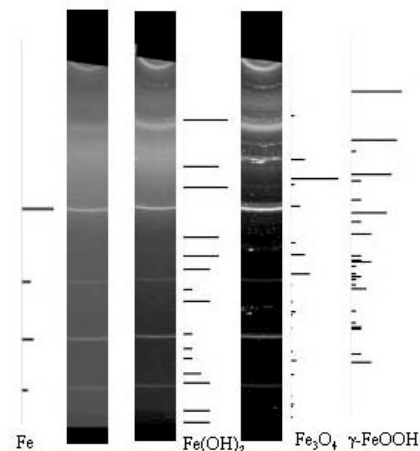


Figure 2 Scattering intensities measured by image plates at  $t=0$  (left),  $t=33$  (middle), and  $t=91$  (right) min.

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### References

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