

In Situ Observation of Corrosion at liq./sol. Interfaces

Masao KIMURA*¹ and Noriaki Ohta²

¹ Adv. Tech. Res. Lab., Nippon Steel Corp., Futtsu, Chiba, 293-8511, Japan

² Nippon Steel Technoresearch, Chiba 293-8500, Japan

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 specimen under a liquid film is investigated by x-ray scattering. The measured depth can be controlled easily in a range of nm - μm by changing the energy of incident beam and the incident angle of x-ray.

A part of the goniometer of the “EVA” system was shown in Fig.1. A 2θ - θ goniometer (“ χ -axis”) was mounted on the θ table of another 2θ - θ goniometer (“ θ -axis”). The combination of two diffractometer can be tilted against the x-ray beam from SR, which determines the angle of incidence into the surface.

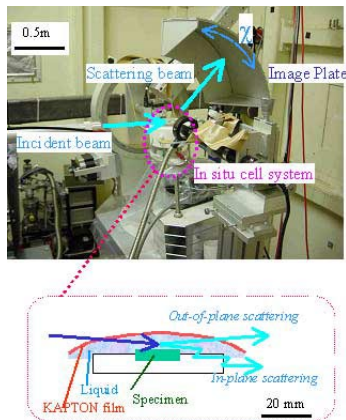


Figure 1 A part of the goniometer of the “EVA” system (above) and a sketch of *in situ* cell system (below).

A specimen of weathering steel was prepared. The composition is Fe-0.28Cu-0.55Cr-0.15Ni-0.49Mn-0.081P-0.51Si-0.10C (in mass%). Artificial sea water was used as liquid for corrosion, and the potential of the specimen was changed from -0.6 to 1.0 V vs. Ag/AgCl in a period of 1 hour.

During corrosion, the gap between the specimen surface and the KAPTON was kept about 5 mm in order to enhance of diffusion of ions and gas which were produced during corrosion. Then the gap was kept less than 0.1 mm so that the x-ray beam can penetrate into the specimen surface thorough the liquid.

Measurements were carried out at BL-3A at PF, KEK, Tsukuba, Japan. A typical parameters are: the energy of x-ray = 12.399 keV and the beam size = $1 \times 1 \text{ mm}^2$. Scattering intensities were measured using an image plate.

Results and Discussion

Figure 2 shows scattering intensities at (a) 23 min. and (b) 55 min. after the potential was applied. At the first stage of corrosion, colloidal products of FeOOH are expected to form. Diffuse intensities in Fig.1(a) correspond to the colloidal phase. As the corrosion progress, “Fe(O,OH)₆ network[3,4]” evolves and rusts were formed on the steel surface. Scattering from the rusts were clearly observed as shown by arrows in Fig.1(b).

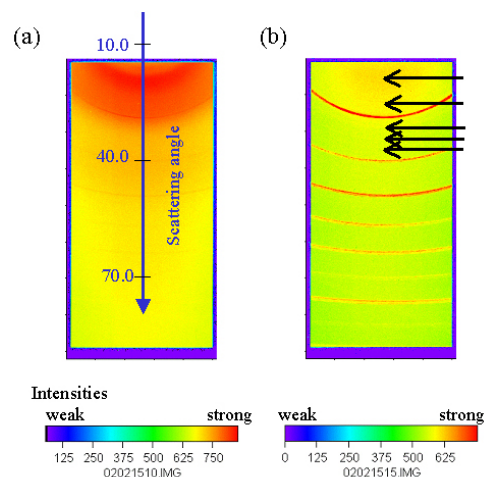


Figure 2 Scattering intensities measured by image plates at (a) 23 min. and (b) 55 min. after corrosion starts.

It has been shown that the evolution of “Fe(O,OH)₆ network” during corrosion was clearly observed by *in situ* technique using “EVA” system. Effects of additional elements on corrosion is being investigated by this approach.

We would like to thank Drs. T. Mori, M. Tanaka and T. Matsushita at PF for their great supports for experiments.

References

- [1] M. Kimura et al.: PF Activity Report 2001, 227 (2002).
- [2] M. Kimura et al.: J. App. Phys., **4**, 2034(2003).
- [3] M. Kimura et al: J. Japan Inst. Metals, **66**, 166 (2002).
- [4] M. Kimura et al: ISIJ International, **42**, 1534 (2002).

* kimura@re.nsc.co.jp