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

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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 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 form SR, which determines the angle of incidence into the surface.

A specimen of weathering steel was prepared. The composition is Fe-0.28Cu-0.55Cr-0.15Ni-0.49Mn-0.08P-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 = 1x1 mm². 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 form 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.

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References


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