A new system for *in situ* observation 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. *In situ* observation of reactions at the interface will give us a crucial information in order to understand the mechanism. In this study, a new system was developed for *in situ* observation of those reactions.

Design Concept

In order to perform *in situ* observation of electrochemical reactions at liquid/solid interfaces, the specimen under the film of water is investigated by x-ray scattering. The measured depth should be controlled easily in a range of nm-μm, because thickness of water and reaction-layer change as reactions progress. The electrochemical reactions is carried out in a special cell, where reaction parameters are controlled: the specimen potential, temp., pH, the gas potential inside the liquid., and so on.

The system was designed to satisfy the following conditions:

1) space for *in situ* cell
2) horizontal position of a specimen (the interface was easily retained during the reaction)
3) measuring both in- and out-of-plane scattering for surface-sensitive analysis
4) control of the angle of incidence of x-ray beam.

Results and Discussion

Based on the concept, a system ("EVA") was developed. The outline of the goniometer of the system was shown in Fig.1. A 2θ–θ goniometer ("χ-axis") was mounted on the table of another 2θ–θ goniometer ("θ-axis"). The combination of two diffractometer can be tilted against the x-ray beam form SR. The typical resolution of each axis is shown in Table1. Movement of each axis was controlled by PC through Ethernet.

Table 1 Resolution (deg.) of each axis

α : 2×10^{-5} ,	
χ2 : $<1\times10^{-3}$,	χ1 : $<8.4\times10^{-4}$
θ2 : $<1.4\times10^{-4}$,	θ1 : $<1.4\times10^{-4}$

This alignment enables to measure scattering intensities in a wide range of the reciprocal lattice, while controlling the incidence-angle (α_i) independently, which is called G-GIXS[1]. Two types of detector were used for measurement: an image plate (IP) and a NaI scintillation counter (SC) (Fig.2).

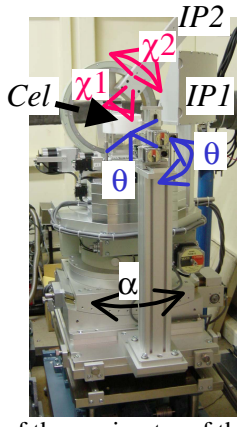

Fig.1 View of the goniomter of the EVA system

Then SC was used to scan the details of scattering intensities around Bragg peaks (Fig.2(b)). Two sets of slits, which limit the height and the width of the beam path, are positioned in front of the detector (Fig.2(b)). Resolution function was changed by altering the heights (h_{slit}), the width (w_{slit}), and the distance of two slits (d_{slit}=the distance S1-S2 in Fig.2(b)). The typical values are : h_{slit} w_{slit} and = 1-2 mm, d_{slit}= 310 mm; the resolution are $Q_{x\text{-}y} = 1\text{-}2\times10^{-2}$ and $Q_z = 2\text{-}4\times10^{-2}$ [r.l.u.].

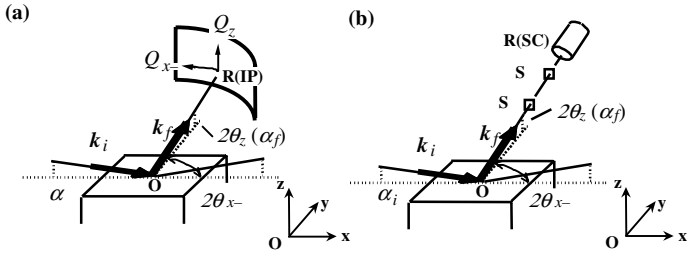

Fig.2 X-ray geometry for G-GIXS; (a) an image plate (IP) and (b) a NaI scintillation counter (SC) were used as a detector.

This system is now used for *in situ* observation of corrosion at metal surface with using a special cell at BL-3A at PF, KEK, Tsukuba, Japan.

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References

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