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In Situ Structural Study of the Electrochemical Deposited Pd Layers on Au(111) and Au(100) by Surface X-ray Scattering

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Introduction

The electrochemical reactivity is strongly affected by the structure of the electrode surface. Structural studies of the electrode surface have been progressed quite significantly in the last decade because of the development of STM and because the preparation of the single crystal became easier. We have investigated the electrochemical deposition of Pd on Au(111) and Au(100) by *in situ* STM, EQCM, and *ex situ* XRD and reported that the electrodeposition of Pd proceeds epitaxially.[1] More detailed information, such as an adsorption site and a distance between each atomic layers, is not available because STM can provide only information of the surface structure and the lateral resolution of STM is not high enough. Surface X-ray scattering (SXS) can determine not only lateral but also vertical atomic distance with much higher resolution, and therefore, the above information can be obtained by this technique. In this report, we investigate the structure of the electrochemically deposited Pd layers on Au(111)[2] and Au(100) by *in situ* SXS.

Experimental

In situ SXS experiments were carried out at the bending magnet beamline BL4C at Photon Factory. Electrochemical deposition of Pd was carried out by scanning the potential from +0.90 V to the negative to +0.56 V and +0.58 V at Au(111) and Au(100), respectively, keeping the potential for a certain period, back to +0.60 V and +0.63 V at Au(111) and Au(100), respectively, where neither cathodic nor anodic current was observed, and then, keeping the potential at this value during SXS measurements. The surface coverage of Pd was determined by cathodic charge passed during the above potential scan. Incident X-ray energy of 14.952 keV was selected by using Si(311) double crystal monochromator.

Results and Discussion

Figure 1 shows the specular ((00) rod) and non-specular ((01) rod) reflectivities from the 1 ML Pd on (A) Au(111) and (B) Au(100). Large dips in both figures were observed between the Bragg peaks, indicating pseudomorphic growth of Pd onto both Au(111) and Au(100).

When electrochemical deposition of Pd on the Au(111) substrate proceeds, the structure of deposited Pd layer changed. Only the first deposited Pd layer forms pseudomorphic and smooth layers and the bulk Pd layer started to grow with a slightly roughening along with a (111) direction from the second - fourth layer of Pd on the Au(111) substrate.

Contrary to the case of Au(111), on the Au(100) substrate, pseudomorphic and uniform Pd overlayers formed up to at least 14 ML and above 30 ML at least, the bulk Pd layer started to grow along with a (100) direction.

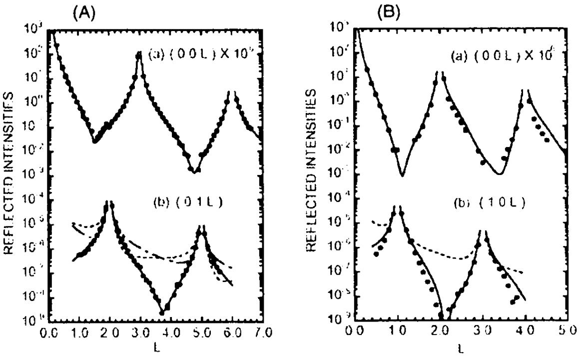

Fig. 1 The specular (a) and non-specular (b) rod profiles for 1 ML Pd on (A) Au(111) and (B) Au(100). The solid lines are the best fit curves obtained from a pseudomorphic growth model. The dashed and dotted lines in the non-specular rod profiles were calculated for the Pd adsorption to the atop and hcp sites, respectively, with the same structural parameters as the best fit model.

References

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