

## *In situ* SXS Study of Electrodeposition Process on Electrodes

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Electrochemical metal deposition on metal and semiconductor is important not only for fundamental science but also for various industrial applications. Recently, we found that the electrochemical deposition of Pd and Pt layers on an Au(111) electrode surface proceeded epitaxially using STM.<sup>1)</sup> The spatial resolution of STM, however, is limited to the order of Å, while the bond length should be determined within  $\pm 0.05$  Å in order to fully understand the deposition process. SXS using SOR light, which can provide information with higher resolution, has recently been applied to electrode/electrolyte interfaces by several groups.

In this report, we investigated the structure of electrochemically deposited Pd layers on Au(111) by *in situ* SXS measurements.

Before mounting in the electrochemical cell, Au(111) substrate was annealed by H<sub>2</sub> flame. 1 ML and 8 ML of Pd were deposited electrochemically on Au(111) in 0.1 M H<sub>2</sub>SO<sub>4</sub> + 0.01 M PdCl<sub>4</sub><sup>-</sup>. *In situ* SXS measurements were carried out at BL14B1 in Spring-8.

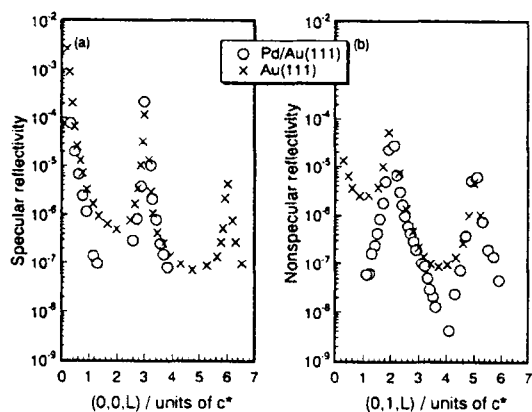


Fig. 1 Specular (a) and nonspecular (b) reflectivities of 1 ML of electrochemically deposited Pd on Au(111) (○) and H<sub>2</sub>O/Au(111) (×).

Figure 1 shows the specular (a) and nonspecular (b) reflectivities of 1 ML of Pd on Au(111). The shapes of these reflectivity

curves were different from those of Au(111)/pure water interface, which were reported before,<sup>2)</sup> showing that the deposited layers were commensurate with underlying Au(111) layer. This result suggests that the lateral atomic distance between Pd atoms at 1st layer on Au(111) is 2.885 Å, which is the same distance between the bulk Au atoms.

Figure 2 shows the in-plane diffraction along (H,0) direction at L = 0.6 of 1 and 8 ML of Pd on Au(111). At Pd(1 ML)/Au(111), only one large sharp peak was observed at H = 1.00. This peak position is exactly that of the Au(111) surface, showing that the lateral atomic distance between Pd atoms between 1st layer on Au(111) is 2.885 Å. On the other hand, a small peak at K = 1.04 corresponding to the atomic distance of 2.77 Å was observed of 8 ML of Pd in addition of that at K = 1.00. This result indicates that the lateral atomic distance of Pd atoms at 8th layer on Au(111) is 2.77 Å, which is the same distance between the bulk Pd atoms.

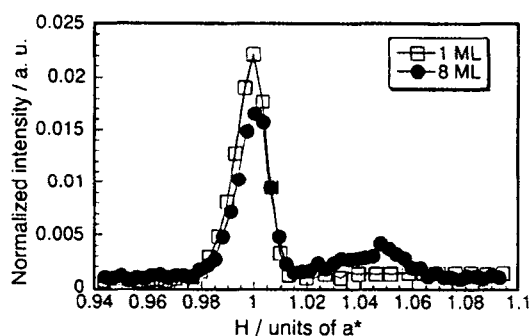


Fig. 2 In-plane diffraction of 1 (□) and 8 (●) ML of Pd on Au(111).

### References

- 1) H. Naohara, S. Ye, and K. Uosaki, *J. Phys. Chem. B*, **102** (1998) 4366; *Appl. Phys. A*, **66** (1998) S457; *Coll. Surf.*, in press (1998).
- 2) J. Wang et al., *Phys. Rev. B*, **46** (1992) 10321.