

In situ Structural Analyses of Electrochemically Deposited Pt Ultrathin Layers on Au Single Crystal Surfaces by RSXS

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Introduction.

Ultra-thin metal layers on the foreign metal substrates have been attracting interests because of their unique physical and chemical properties, particularly their high electro-catalytic activities. Such special catalytic activity is due to their unique surface atomic arrangements and/or induced surface electronic energy by them. Thus, it is of great importance to understand deposition process of ultra-thin metal layers on the foreign metal substrates with atomic dimensions. In this report, atomic arrangements of the Pt ultra-thin layers electrochemically deposited on the Au(111) single crystal surfaces investigated by electrochemical and resonance surface X-ray scattering (RSXS) measurements.

Experimentals.

After annealing and quenching of the Au(111) substrate, it was contact with the 0.1 M HClO₄ electrolyte solution containing 0.05 mM H₂PtCl₆. Potential was negatively scanned from open circuit potential (OCP) to (i) +0.77 V (vs. RHE), (ii) +0.90 V, and (iii) +0.92 V at a scan rate of 2 mV s⁻¹, the potential was kept at each potential for a certain period and then the Au(111) disk was removed from the electrochemical cell. After the sample was washed with conc. H₂SO₄ and ultrapure water, the disk was set to the conventional electrochemical cell and CVs were measured in 0.05 M H₂SO₄. The RSXS measurements were carried out at BL4C beamline at Photon Factory.

Results and Discussion.

When the overpotential was relatively smaller, the size of the formed nuclear was so small, the number of nuclei was very few, and then nucleus was hardly grown up. On the other hand, when the overpotential was relatively larger, the size of nuclear was too large and/or the number of nuclear was too many, nuclear-growth three-dimensionally took place, and then the rough three-dimensional Pt multilayer was formed on the Au(111) surface. In contrast with the above results, when the overpotential is an appropriate value between the above overpotentials, a size of nucleus and number of nuclei were appropriate, and then nuclear-growth two-dimensionally took place and then the monoatomically flat Pt layer was formed on the Au(111) surface. It was confirmed based on the *in situ* RSXS results that Pt layer electrochemically deposited on Au(111) by the present preparation condition is pseudomorphic. Thus, we found the electrochemical condition by which Pt is two-dimensionally formed on the Au(111) surface with Frank-van der Merwe (FM) mode, namely layer-by-layer mode.