

Recent Progress in Electrochemical Surface Science with Atomic and Molecular Levels

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Until the mid 1980's, there had been only few *in situ* methods available for structural determination of an electrode surface in solution at atomic and monolayer levels. Nowadays, many powerful *in situ* techniques, such as electrochemical scanning tunneling microscopy (EC-STM), infrared reflection absorption spectroscopy (IRAS), surface-enhanced Raman scattering (SERS), and surface-enhanced infrared reflection absorption spectroscopy (SEIRAS), second harmonic generation (SHG), sum frequency generation (SFG), and surface X-ray scattering (SXS) have been widely employed to characterize the electrode surfaces under potential control with atomic and/or molecular resolution. The object of this review is to highlight some of the progress on *in situ* methods at solid-liquid interface with atomic and molecular levels. Several selected topics are focused on, specifically adsorbed anions on metal surface, electrocatalysis of the carbon oxide oxidation and dioxygen reduction, and direct observation of single crystal electrode surfaces.

Key Words: Solid-Liquid Interface, Single Crystal Electrode, Adsorption, Structure, Adsayer, Electrocatalysis, SHG, SFG, SXS, SEIRAS, STM

1 General Introduction

Kingo ITAYA & Soichiro YOSHIMOTO

Electrochemistry is important for industries concerned with products and processes such as batteries, fuel cells, electroplating, corrosion inhibition, electro-organic synthesis, and sensor devices.¹ Because electrode reactions take place at solid-liquid interface where the electrode is in contact with a solution, electrochemical interface plays an important role in controlling the electron transfer reaction. Understanding the nature of solid-liquid interface in detail not only leads to the elucidation of the electrode reaction mechanism but also provides information useful for designing new interfaces.

As a fundamental basis for all electrochemical studies, electrode-electrolyte interfaces must be prepared reproducibly, and methods must be established to observe these interfaces accurately. It is still difficult to elucidate electrochemical reactions on the atomic scale using polycrystalline electrodes. Well-defined single crystal surfaces must be exposed to solution to understand surface

structure-reactivity relationships on the atomic scale. Efforts have succeeded to produce extremely well-defined, atomically-flat surfaces of various electrodes made of noble metals, base metals, and semiconductors without either oxidation or contamination in solution.²⁻⁶

It was first realized in 1960 in the field of *electrochemical surface science* that the adsorption-desorption reaction of hydrogen on Pt single crystal electrodes is strongly dependent on their crystallographic orientations; this demonstrated the importance of the nature of metal surface at the atomic level.⁷⁻⁹ Clavilier was the first to develop a simple and important experimental method, the so-called *flame annealing and quenching method*,¹⁰ to expose well-defined electrode surfaces in aqueous electrolyte solutions without the use of ultrahigh vacuum (UHV) techniques. Nowadays, this technique has been extended by Hamelin for Au,¹¹ by Motoo and Furuya for Ir,¹² and by Itaya's group for Rh and Pd.¹³

Meanwhile, since the late 1980's, *in situ* analytical methods were developed all over the world. In the early stage, various experimental techniques, such as infrared absorption spectroscopy (IRAS),¹⁴⁻²⁰ ultra-violet (UV)-vis-

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