

# Electrochemical Surface Science

**Molecular Phenomena  
at Electrode Surfaces**

ACS SYMPOSIUM SERIES

# **Electrochemical Surface Science**

## **Molecular Phenomena at Electrode Surfaces**

**Manuel P. Soriaga, EDITOR**  
*Texas A&M University*

Developed from a symposium sponsored  
by the Division of Colloid and Surface Chemistry  
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## Foreword

The ACS SYMPOSIUM SERIES was founded in 1974 to provide a medium for publishing symposia quickly in book form. The format of the Series parallels that of the continuing ADVANCES IN CHEMISTRY SERIES except that, in order to save time, the papers are not typeset but are reproduced as they are submitted by the authors in camera-ready form. Papers are reviewed under the supervision of the Editors with the assistance of the Series Advisory Board and are selected to maintain the integrity of the symposia; however, verbatim reproductions of previously published papers are not accepted. Both reviews and reports of research are acceptable, because symposia may embrace both types of presentation.

## Preface

DEVELOPMENT OF SOPHISTICATED SURFACE ANALYTICAL TECHNIQUES over the past two decades has revived interest in the study of phenomena that occur at the electrode-solution interface. As a consequence of this renewed activity, electrochemical surface science is experiencing a rapid growth in empirical information. The symposium on which this book was based brought together established and up-and-coming researchers from the three interrelated disciplines of electrochemistry, surface science, and metal-cluster chemistry to help provide a better focus on the current status and future directions of research in electrochemistry. The symposium was part of the continuing series on Photochemical and Electrochemical Surface Science sponsored by the Division of Colloid and Surface Chemistry of the American Chemical Society.

This volume contains 36 chapters, the first of which is an overview chapter. The eleven following chapters describe the use of ultrahigh vacuum surface spectroscopic methods in the study of the electrode-solution interface. The next eight chapters report recent advances in the adaptation of new experimental techniques, such as scanning tunneling microscopy, to electrochemical problems. The seven following chapters discuss critical aspects of in situ vibrational spectroscopy. The last nine chapters describe various electrode processes such as the electrochemical reactivity of polymer deposits,  $\text{CO}_2$  methanation, and surface organometallic chemistry. The broad but balanced range of important topics discussed by experts here should make this volume invaluable to students and active researchers in electrochemical surface science.

### Acknowledgments

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# Chapter 1

## Electrochemistry: The Senior but Underused Area of Surface Science

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Electrochemistry ought to be regarded as a vintage area of interfacial science. It has long been known that the application of an external potential at a (conducting) solid-solution interface enhances the reactivities of adjacent chemical species. Catalytic reactions activated by control of potential at the electrode-solution interface thus serves as an alternative to activation by control of temperature at gas-solid interfaces. In fact, on the singular basis of catalytic selectivity, the electrochemical route towards synthesis and catalysis should be favored over the thermal approach because the application of elevated temperatures activates other reaction pathways. For a two-electron reaction, for example, a potential change of only half a volt can lead to a  $10^8$ -fold increase in reaction rate. Assuming an activation energy of  $75 \text{ kJ mole}^{-1}$ , this enormous rate increase can be effected only at temperatures above 600 K. Such drastic conditions invariably lead to a wide spectrum of thermal excitations which could result in undesirable side-reactions such as indiscriminate bond-breaking.

In addition to the universal concern for catalytic selectivity, the following reasons could be advanced to argue why an electrochemical scheme would be preferred over a thermal approach: (i) There are experimental parameters (pH, solvent, electrolyte, potential) unique only to the electrode-solution interface which can be manipulated to dictate a certain reaction pathway. (ii) The presence of solvent and supporting electrolyte may sufficiently passivate the electrode surface to minimize catalytic fragmentation of starting materials. (iii) Catalyst poisons due to reagent decomposition may form less readily at ambient temperatures. (iv) The chemical behavior of surface intermediates formed in electrolytic solutions can be closely modelled after analogous well-characterized molecular or cluster complexes (1-8). (v) For hydrogenation (or oxidation) reactions, the aqueous solvent functions as a convenient source of hydrogen (or

oxygen). (vi) Because electrocatalysis employs a reducing and an oxidizing electrode, there is promise for implementation of the concept of paired electrosynthesis (9).

It is true that electrochemistry has enjoyed tremendous successes in the fields of chemical analysis (sensors) and energy conversion (batteries). It is also a fact that the use of electrochemistry has become widespread in modern-day inorganic chemistry where redox potentials of newly synthesized materials are determined almost as routinely as spectroscopic and crystallographic data. However, despite its historical significance and despite all the apparent advantages enumerated above, electrochemistry remains relegated to the background insofar as large-scale catalytic synthesis is concerned. Technological and engineering complexities, such as slow mass transport in solution and the cost of electricity, are important factors for the persistent neglect of the electrochemical approach. Another equally critical reason is the absence of atomic-level descriptions of processes which occur at the electrode-solution interface. This lack of fundamental information can be attributed to the fact that, until these past two decades, conventional electrochemical methods (10), such as voltammetric, amperometric, coulometric, impedance, and transient measurements, were the only tools readily available for the study of electrode processes; these techniques are severely restricted in the type of information they provide since the results obtained from them are manifestations of only the macroscopic properties of the electrode-solution interface.

The recognition of the lack of molecular-level information on electrocatalytic phenomena and the resolve to have this weakness rectified is now firmly established within the electrochemical and surface science communities. This is evidenced by recent international conferences and workshops devoted solely to electrochemical problems (11-4). It is also well-realized that electrochemistry cannot advance further unless the practitioners in this field begin to view electrode-solution processes from a molecular-level perspective.

The central issue which has to be addressed in any comprehensive study of electrode-surface phenomena is the determination of an unambiguous correlation between interfacial composition, interfacial structure, and interfacial reactivity. This principal concern is of course identical to the goal of fundamental studies in heterogeneous catalysis at gas-solid interfaces. However, electrochemical systems are far more complicated since a full treatment of the electrode-solution interface must incorporate not only the compact (inner) layer but also the boundary (outer) layer of the electrical double-layer. The effect of the outer layer on electrode reactions has been neglected in most surface electrochemical studies but in certain situations, such as in conducting polymers and

biological systems, processes within the boundary layer can exert non-trivial influences.

In the surface electrochemist's pursuit of fundamental structure-composition-reactivity correlations, lessons can be and have been learned from the successes achieved in both heterogeneous and homogeneous catalysis. For example, extensive work on fuel cell electrodes (15-24) has accomplished much to show that concepts developed in the study of oxygen reduction by Pt particles in gas-solid reactions are applicable to the electrochemistry of  $H_2$  and  $O_2$ . The essential ingredients in experimental strategies which have contributed to the molecular-level understanding of a wide variety of homogeneous catalytic processes include (25-6): (i) the use of pure and well-characterized starting materials, (ii) structural and compositional analysis of homogeneous catalysts, (iii) structural and compositional analysis of important intermediates, (iv) detailed kinetic measurements including the concentration of catalyst precursors, and (v) quantitative analysis of reaction product distributions. These elements, which have now become routine in model studies of heterogeneous catalysis at gas-solid interfaces, are just beginning to be appreciated in surface electrochemical investigations.

Studies of the physics and chemistry at the gas-solid interface have enjoyed tremendous advances from the systematic application of modern surface spectroscopic methods (27), such as low-energy electron diffraction for surface crystallographic determinations, Auger electron spectroscopy for surface elemental analysis, X-ray photoelectron spectroscopy for surface bonding studies, high-resolution electron-energy loss spectroscopy for surface vibrational information, and thermal desorption mass spectrometry for adsorption enthalpy measurements. These powerful techniques have provided revealing glimpses of gas-solid interfacial processes at the atomic level. Unfortunately, they cannot be employed to probe the electrode-electrolyte interface under reaction conditions since surface characterization has to be performed outside the electrochemical cell. Doubts have been raised concerning the validity of correlating structural information acquired in the absence of an electrochemical environment with reactivity data obtained under potential control. To help mollify these concerns, it has been pointed out that fruitful structure-reactivity correlations have been accomplished in homogeneous systems (25-6) despite the fact that structural determinations are done in the solid state while the reactivity measurements are performed in solution. Results from recent studies of emersed electrodes (12,28-9) have also dispelled most of the earlier doubts. A recent workshop organized by the National Research Council has recommended that the use of high-vacuum surface physics methods should be continued (12). The utilization of such techniques in surface electrochemistry was initially restricted to only a

handful of laboratories (30-2) but the number has now increased. It is particularly encouraging to note that surface science laboratories which previously devoted all efforts to gas-solid interfaces now apportion significant time to the study electrocatalytic phenomena (33-4).

Optical spectroscopic methods not restricted to high-vacuum conditions, such as infrared reflection-absorption spectroscopy (35), ellipsometry (36), and surface-enhanced Raman spectroscopy (37-8), are available for examining intermediates at the electrochemical double-layer under reaction conditions. However, none of these can determine the complete structure and composition of the electrocatalyst-adsorbate interfacial aggregate. The capabilities of laser [as in second-harmonic generation (39-40)] and synchrotron radiation [as in near-edge and extended X-ray absorption fine structure (41)] in elucidating interfacial structures under electrochemical conditions have recently been demonstrated; research in these areas have been intensified. Present vigorous activities in surface electrochemical studies have included the adaptation of new technologies, such as scanning tunneling microscopy (42-3), and the revival of conventional methods, such as Mossbauer spectroscopy (44), nuclear magnetic resonance, and X-ray diffraction (45).

In view of the complexity of heterogeneous systems, none of the above techniques will be able to supply, by itself, a complete atomic-level description of surface phenomena. A multi-technique approach has been perceived by many as most appropriate for fundamental studies in electrochemical surface science (30-2). Since none of the existing electrochemical laboratories are adequately equipped to perform a comprehensive experimental study, collaborative efforts between research groups of different expertise are burgeoning. Easier access to national or central facilities are also being contemplated for experiments which cannot be performed elsewhere. The judicious combination of the available methods in conjunction with the appropriate electrochemical measurements are permitting studies of electrocatalyst surface phenomena unparalleled in molecular detail.

In any scientific endeavor, mere accumulation of empirical information is not enough to guarantee major advances. Efforts have to be exerted to formulate fundamental concepts from the available data. For example, it is no longer sufficient to describe how an adsorbed species is bound to the surface simply in terms of its interfacial orientation. It is essential to proceed beyond such phenomenological illustrations and actually examine the nature of the surface-adsorbate chemical bond. It is in this aspect that concepts from the related areas of coordination and organometallic chemistry have been most meaningful. For example, in the so-called surface-cluster analogy (1-4), bonding in chemisorption systems is modelled after bonding in molecular or cluster complexes. Skepticism to the surface-cluster analogy has been raised



because of the widespread knowledge that while metal complexes can be described by discrete orbitals, metal surfaces are usually characterized by band structure. However, what is not known to as many is the fact that the surface band structure can actually be deconvoluted into components of given symmetries with respect to a given site (3-4). In this manner, the surface-adsorbate bond can be described in terms of the overlap of symmetry-adapted surface bands and adsorbate orbitals; this description is exactly the same as that of the metal-ligand bond in monometal or small cluster complexes.

With the experimental and theoretical strategies available today, research in electrochemical surface science has been revived. There is great optimism that much of the mysteries surrounding electrocatalysis will soon be unravelled in molecular detail hitherto unachievable.

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