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20. ABSTRACT. cont'd

properties of adsorbed molecules, ions and atoms at electrochemical interfaces. They have also been used to examine the adsorption isotherms and kinetics of adsorption-desorption.

The in-situ techniques, however, are insufficient to establish the type of adsorption sites or compositional questions. For this reason substantial use has also been made of ex-situ surface physics techniques including LEED, AES, XPS and secondary ion mass spectroscopy. Special hardware and techniques have been developed as part of the ONR contract research to facilitate the transfer of electrodes between the electro-chemical and ultrahigh vacuum environments and vice versa with the possibility for restructuring and contamination at a minimum. The report describes the application of these techniques to adsorption on single crystal electrodes.

FINAL REPORT

submitted to the

OFFICE OF NAVAL RESEARCH

for research on

ELECTROCHEMICAL INTERFACES AND ELECTRODE PROCESSES

Contractor:	Case Western Reserve University, Cleveland, Ohio 44106					
Department:	Case Center for Electrochemical Sciences and					
	the Chemistry Depar	tment				
Contract No.:		N00014-75-C-0953				
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Initiation Date	e:	1 January 1976				
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Project Directo	or:	Ernest Yeager, Hovorka Professor of Chemistry				

Date of Submission of Final Report: 15 August 1984



I. RESEARCH OBJECTIVES AND OVERVIEW

The objectives of the ONR sponsored research are two-fold:

 the understanding of the factors controlling electrosorption of various species on metal and semiconductor electrodes;

2. the relationship of the kinetics of various electrode processes

to the electrode properties and adsorption on electrodes. While electrochemical methods have high sensitivity to adsorption on electrodes (down to a small fraction of a monolayer), they lack sufficient atomic level specificity to identify uniquely the type of sites, the molecular configuration of the adsorbate, the nature of the bonding to the surface, the interaction between adsorbed species and the competitive aspects with respect to solvent adsorption. Consequently much of the emphasis on this ONR sponsored research has been in the use of complementary techniques to study adsorption on electrodes. The <u>in situ</u> techniques which have been used for this purpose as part of the ONR research and the systems which have been studied have included

- 1. ultraviolet-visible reflectance spectroscopy automatic ellipsometry
- 2. infrared attenuated reflectance spectroscopy
- 3. surface enhanced and resonant Raman
- 4. Mossbauer spectroscopy

These measurements have yielded critically needed information concerning the electronic and vibrational properties of electrosorbed species and anodic films as well as the adsorption isotherms and the kinetics of the adsorption-desorption and surface rearrangements. They are insufficient by themselves to pin down the type of adsorption sites or compositional questions. For this reason, we have also made substantial use of ex situ

- 2 -

techniques to complement the electrochemical and <u>in situ</u> spectroscopic measurements. These have included

1. LEED

- 2. Auger electron spectroscopy
- 3. X-ray photoelectron spectroscopy
- 4. secondary ion mass spectroscopy

Special apparatus and techniques have been developed as part of the ONR contract research to facilitate the transfer between the electrochemical and ultrahigh vacuum interface and vice versa with the possibility of restructuring minimized. The types and distribution of adsorption sites is dependent on the surface topography and particularly the crystallographic plane. LEED and ultrahigh vacuum techniques have proved essential for the preparation of single crystal surfaces and their characterization before and after the electrochemical measurements. While many groups are carrying out electrochemical measurements on single crystals, their work is in general not indicative of surfaces with known zone axes. To prepare a clean surface corresponding to a given surface plane requires in general vacuum-LEED techniques and very special transfer procedures. Furthermore, the surfaces usually reconstruct during the electrochemical measurements and post-electrochemical LEED examination is essential. The effort to carry out such "ultra" electrochemical experiments, however, is very demanding and time-consuming.

The electrochemical systems to which such <u>in situ</u> and <u>ex situ</u> methods have been applied in this ONR sponsored research have included

 underpotential deposited metals on gold: UV-visible, ellipsometry, LEED, AES (1-4,26)

- 3 -

- 2. electrosorbed hydrogen on platinum: LEED, AES, XPS (5-8,26).
- 3. adsorbed halogen ions on Au: UV-visible reflectance (including kinetics electroreflectance admittance) (9).
- adsorbed transition metal macrocyclic complexes on Ag, Au, Pt, basal plane of graphite, high area carbon: UV-visible reflectance Raman, Mossbauer (Fe, Co⁵⁷) (10-14,22,27).
- 5. adsorbed probe molecules on Ag, Pt, carbon, semiconductors: Raman (15-18

6. anodic films and passivation layers on Pt, Au, Fe^{*}, Fe-Cr^{*}, Ni, Ag: UV-visible reflectance, ellipsometry, infrared, Raman, Mossbauer, XPS, AES, SIMS (19-25,27,28).

This research for the most part is summarized in various technical reports and publications. More recent developments over the past year are summarized in the following sections of this report.

- II. RECENT DEVELOPMENTS IN CONTRACT RESEARCH
 - A. Upgrading of the thin-layer-electrochemical-cell-LEED-Auger-XPS system

The thin-layer-electrochemical cell-LEED system was designed and constructed in the latter 1970's as part of the ONR contract research. This system permits single crystal electrode surfaces to be prepared and characterized with LEED in ultra-high vacuum and then transferred into a thin layer electrochemical cell without removing the electrode from the vacuum chamber. The electrochemical measurements are carried out in the presence of ultrapure argon as a supporting gas. Following the electrochemical measurements, the electrode surface is again returned to the ultra-high vacuum and reexamined with LEED and AES. Most of the recent studies have been carried out with dilute aqueous HF since this electrolyte is not specifically adsorbed over a substantial range with single crystal surfaces such as gold

Some of the more recent measurements on iron and the iron-chromium alloys have been carried out as part of the ONR/SRO project at Case on the passivation of iron and ferrous alloys from Sept. 1979 - August 1982.

and platinum. The system was used to examine the electrosorption of hydrogen on single crystal platinum and underpotential deposition of lead on single crystal gold. The results have shown that each of the low index planes of these face-centered cubic single crystal metals have quite different adsorption characteristics. On the basis of these measurements it is clear that fundamental studies must be carried out on single crystal surfaces to be meaningful. On the other hand, the post-electrochemical LEED examination indicates that the surfaces often reconstruct in the electrochemical environment either giving new LEED patterns or becoming sufficiently disorganized so as not to give a pattern at low electron beam voltages where the diffraction arises principally from the surface layer. Only over relatively narrow potential ranges and then only on some crystal planes is it possible to maintain the original surface configuration. Furthermore, the AES measurements indicate that the surfaces are very easily contaminated and that even only a small fractional surface coverage can produce gross changes in the electrochemical properties.

On the basis of these earlier studies during 1978-81, the conclusion was reached that the study of electrochemical adsorption is only meaningful on single crystals for solid electrodes and that such electrochemical studies must be carried out with pre- and post-electrochemical LEED measurements with the most effective transfer possible. Consequently we have directed further effort to refining and upgrading this electrochemical-LEED-AES system. The changes made during the past one and a half years have included the following:

1. XPS-AES capability

The original system had only the limited capability afforded by the Varian RFD grid L^EED-AES system. A Vacuum Generators hemispherical electron analyzer (CLAM-100) has been added together with

- 5 -

a dual target X-ray source (A1,^{Mg}) and an electron gun. The available funds were insufficient to secure the signal processing computer system from Vacuum Generators. A surprisingly effective system, however, has been developed by our group using a Commodore Pet microprocessor together with a Nicolet signal average (1170) and associated peripheral equipment. This system now provides us a high performance XPS-AES capability. A diagram of the overall system as it now stands is shown in Fig. 1.

2. Increased pumping speed

The vacuum system consists of two principal vacuum chambers:

<u>Chamber A</u>: This chamber contains the LEED-AES-XPS system plus a sputtering gun and electron beam heater. The vacuum is usually in the 10^{-11} Torr range.

<u>Chamber B</u>: This chamber houses the thin layer electrochemical cell plus a quadrupole mass spectrometer. Ultra-pure argon is used in this chamber at ca. 0.3 atm when the thin-layer cell is formed. This chamber has heretofore been only pumped with a triode ion pump and after sufficient time reached the 10^{-10} Torr range. This chamber is connected to Chamber A with an isolation value between the two chambers and a magnetically operated transfer wand to convey the single crystal electrode between the two chambers.

After the electrochemical measurements it is important to purge out the support gas (argon) and volatilize the electrolyte (0.1M HF) as rapidly as possible. To accomplish such, a turbomolecular pump has been connected to Chamber B, which pulls the pressure down to 10^{-8} Torr in 90 - 120 s following the completion of the electrochemical experiments. The sample is then transferred into Chamber A through the low conduction connection between the two chambers. In 3 to 5 minutes, the sample is again in position for LEED or AES-XPS examination at a pressure of 10^{-10} Torr. This has greatly reduced the possibility of post-electrochemical contamination of single crystal surfaces such as Pt with CO and other materials released from the



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walls of the chamber or present in the argon or released from the triode pump while pumping out the large amount of argon in Chamber B. With this system it has been possible to reduce the carbon level on Pt single crystal surfaces in the post-electrochemical AES measurements to 5 to 10% of a monolayer. Even so, we are still seeking to lower the carbon surface contamination in the post-electrochemical measurements to a much lower level $\leq 2\%$.

3. Electrolyte handling system

The electrolyte handling system has been completely rebuilt to provide better control of the purity of the electrolytic solutions such as $0.1\underline{M}$ HF and better control of the volume delivered for the electrochemical measurements. The delivery system only exposes the electrolyte to Teflon. The portions of the Teflon delivery system outside the vacuum chamber are surrounded by an argon atmosphere to eliminate the possibility of 0_2 diffusing through the walls into the solution.

4. Thin-layer electrochemical cell

Heretofore the thin-layer cell has consisted of the single crystal working electrode and a combined counter-reference electrode of α -Pd-H. This counter-reference electrode, however, can prove erratic under some circumstances. Consequently a three-electrode system has also been developed in which a Teflon-surrounded α -Pd-H electrode with only the end exposed is used as a separate reference, placed between the working and counter electrodes. This arrangement is currently being tested and should result in a larger number of successful runs as well as more accurate voltammetry results.

The system for cleaning, charging and pretesting the α -Pd-H counter electrode has also been greatly upgraded, using a third small vacuum chamber

- 8 -

with its own ion pump.

B. Studies_of the electrosorption of hydrogen on single crystal platinum surfaces.

In 1977-79, hydrogen adsorption and desorption were examined on the low index planes of Pt in H_2SO_4 solutions using the earlier version of the thin-layer cell-LEED-Auger system. When the potential was cycled only in the 0.0 to 0.55 V vs. α -Pd-H, the voltammetry curves on the (111) and (110) planes each showed only one major adsorption-desorption peak corresponding to weakly bound hydrogen and with a total charge only a small fraction of that expected for a monolayer. The (100) surface also showed only one predominant adsorption-desorption peak at more anodic potentials corresponding to more strongly bound hydrogen also with the charge corresponding to a small fraction of a monolayer. Cycling the potential into the anodic film region (1.45 V) resulted in the growth of these peaks on each of the surfaces. This could be explained in two ways: (1) the surfaces restructured at more anodic potentials and (2) carbon or other contamination was oxidized off the surface by cycling out to the anodic potentials, thus freeing up the surface for H adsorption. Both probably occurred. The use of sulfuric acid as the electrolyte, however, interfered with the postelectrochemical LEED and AES examination since this electrolyte is difficult to volatilize and becomes highly oxidizing as water is preferentially lost.

Consequently, the decision was made to repeat the measurements with 0.1M HF as the electrolyte using the partially upgraded system. More emphasis was placed on the purity of the low index surfaces before introduction into the electrochemical environment and the question of carbon

- 9 -

monoxide contamination during the pump down following the electrochemical measurements. The voltammetry results on the Pt 111 and 110 (2 x 1) surfaces were rather similar. Cycling into the anodic range resulted in the growth of the principal hydrogen peak. The subsequent LEED patterns for electrodes not cycled into the anodic film region indicated the patterns to be similar to those initially obtained although the background was increased. Carbon levels for the initial surfaces were usually below detection and in the postelectrochemical AES measurements were less than 10% monolayer level. When the electrode was cycled into the anodic film region (1.45 V), the hydrogen peaks again grew but the LEED showed major restructuring with loss of most of the pattern at low electron beam voltages. The voltammetry results on the Pt 100 (5 x 20) in HF, however, were quite different (see Fig. 2). With potential sweeps in the hydrogen adsorption and so-called anion region, the cathodic peak appeared in the anodic sweep. This cathodic spike is attributed to the irreversible adsorption of hydrogen attending a structural rearrangement of the surface. The hydrogen can be desorbed very irreversibly at 0.8 V. Furthermore, potential window opening experments indicate that this hydrogen can be cathodically desorbed at potentials less than 0.15 V. The process is proposed to be

 $H(ads) + H^{+} \longrightarrow H - H^{+}(ads)$ → H₂(solution)

-10 -

Dr. P. Ross has just reported that F. Wagner and himself at Lawrence Berkeley have now also observed such a cathodic spike in the anodic sweep for Pt(100).





which may occur in one or two steps. These correspond to the Heyrovsky and Horiuti mechanisms for H_2 generation.

Highly irreversible structural rearrangements attending the adsorption of hydrogen on the $Pt(100 - 5 \times 20)$ surface from the gas phase have been reported by Norton <u>et al.</u> (28). The hydrogen is adsorbed at 120°K with the loss of the 5 x 20 overlayer and cannot be desorbed until 350-500°V. The cathodic peak in the anodic sweep at ca. 0.40 V in Fig. 2 is analogous to the low temperature adsorption from the gas phase and the desorption at the much more positive potential of 0.80 analogous to the high temperature desorption.

With multiple sweeps into the anodic film region (1.4 V) the cathodic spike is depressed and the voltammetry curve in the hydrogen region becomes similar to that found for $0.05\underline{M}$ H₂SO₄. Post-electrochemical LEED examination of the Pt (100) surface indicates loss of the 5 x 20 overlayer even if the potential is swept only in the limited range 0 - 0.5 V. With sweeps well into the anodic film region, the spot pattern becomes difficult to discern with a large amount of background diffuse scattering. With higher electron beam voltages the LEED pattern again exhibits well-defined spots. The surface after exposure to quite positive potentials has undergone substantial rearrangement.

These studies of hydrogen electrosorption on Pt in 0.1M HF were carried out before the installation of the Vacuum Generators Clam 100 hemispherical analyzer and the dual target x-ray source for XPS measurements. We are presently in the process of repeating a few of the measurements in 0.1MHF with post-electrochemical studies of the surfaces with XPS and AES to check more carefully on the presence of trace surface impurities.

- 12 -

C. Studies of underpotential deposition on single crystal metals

During the period 1976 - 79, the LEED-AES thin layer electrochemical cell system was used to study the underpotential deposition of lead on various gold single crystal surfaces. This work is summarized in published papers [15 and a technical report (51)]. During the last year of this contract, research has been initiated on a thorough LEED study of underpotential deposited layers on single crystal surfaces using this apparatus after the upgrading described earlier in this report. Underpotential deposition of lead on the low index surfaces of silver has been chosen for this study. The work has been continued under a follow on ONR contract. Research also has been initiated on the simultaneous underpotential deposition of two species, for example lead and copper on gold. The initial work is on polycrystalline gold but research under the continuation of this porject will involve single crystal gold substrates.

D. Studies of anodic film formation on platinum single crystal surfaces

When the platinum single crystal surfaces are exposed to potentials in the anodic film region and withdrawn at potentials cathodic to this region, the LEED patterns indicate major surface changes. Work has been initiated to pin down the nature of these changes from the LEED patterns as functions of electron beam voltage. The platinum surfaces are also being withdrawn from the electrochemical cell at different potentials in the anodic film region to investigate the nature of the film with LEED and to check on the state of the platinum and adsorbed oxygen with XPS. In preliminary experiments with electrodes withdrawn in the anodic film region we have found fluorine in the XPS but not if the electrode is withdrawn at less positive potentials. This warrants careful investigation. This work is on-going and promises much needed information to understand the electrocatalytic properties of platinum for such reactions as 0_2 electroreduction

-13-

E. Adsorbed transition metal macrocyclic complexes on electrode surfaces

Various transition metal macrocyclic complexes adsorbed on electrode surfaces exhibit high catalytic activity for various electrode reactions. The reduction of 0_2 on such surfaces has been of special interest. Some of these complexes such as the iron tetrasulfonated phthalocyanines (TSPc) have high activity for the overall 4-electron reduction of 0_2 to $0H^-$ or H_20 while others such as the CoTSPc promote two-electron reduction of 0_2 to peroxide (5,29,30). The former process is much preferred for such applications as fuel cells, metal-air batteries and certain industrial electrolytic processes.

The state of these adsorbed complexes on electrode surfaces and their interactions with 0_2 and reactive intermediates such as 0_2^- and $H0_2^-$ are not well understood. As part of the research sponsored by both DOE and ONR, the Raman and visible reflectance spectroscopy of adsorbed layers of Co and Fe-TSPc as well as transition metal-free H₂TSPc have been examined in acid, neutral and alkaline electrolytes (10-13). The Raman measurements have been carrie out on silver electrodes roughened by cycling the potential. The reflectance of the adsorbed Co and Fe-TSPc species were examined on the basal plane of stress-annealed graphite, platinum and gold (10,13). The Raman and UV-visible spec of the solution phase Co- and Fe-TSPc were also examined with and without 0_2 present in acid, neutral and alkaline electrolytes. Unfortunately fluorescence interfered with obtaining the Raman spectra of the H₂-TSPcNa₄, although the fluorescence was not pronounced for this species adsorbed on the electrode surface, probably because of quenching of the fluorescence by energy exchange with the metal (13).

The visible reflectance spectra of the adsorbed Co- and Fe-TSPc are similar to those of the solution and solid phase species. An easily detectable change occurs in the reflectance spectra with exposure of the

system to 0_2 at constant electrode potential. The Raman spectra for the adsorbed species have essentially the same frequencies as the solution phase and solid phase species. The relative intensities, however, are quite different and furthermore are strongly dependent on the electrode potential as well as laser excitation frequency. The peak heights fall into two distinct types of potential dependence. This suggests that the macrocyclic may be present on the surface in two orientations or configurations with the amount in each configuration dependent on potential. Unfortunately, the selection rules for molecules exhibiting both surface enhancement and intrinsic resonance or pre-resonance enhancement are not well identified and hence identification of the configuration on the surface is uncertain. The Raman also is not sensitive to the axial ligand using visible laser excitation (488.0, 514.5 or 632.8 nm) and it is necessary to carry out the Raman studies in the ultraviolet to pin down whether di-oxygen is bound in the axial position and if so in a superoxide or peroxide state. Further work is planned using ultraviolet Raman.

Some of the possible configurations for the Co- and Fe-TSPc adsorbed species are shown in Fig. 3. In view of the strong adsorption and similarity of the spectra to that of the solids, we favor configurations b and d.

Mossbauer effect spectroscopy provides a means for examining the Fe and Co macrocyclics including the Fe- and Co-Pc in high area carbon electrodes in porous carbon electrolyte structures (14,22,27). We have been successful in examining the Fe phthalocyanine and Co tetramethoxyphenyl porphyrin <u>in</u> <u>situ</u> in alkaline and acid electrolytes as well as <u>ex situ</u>. This is an exciting development since it is possible to gain fundamental insight into the behavior of the complexes within high area electrodes. These studies are in an early state but already quite useful information has been obtained. For the Fe

-15-



phthalocyanines, the concentration of the naturally present 57 Fe is sufficient with the FePc catalyzed electrode used as the absorber for the γ -rays from a 57 Co source. For the Co complexes, the species have been synthesized with 57 Co and the electrodes loaded with this species used as the γ -ray emitter. The relaxational properties of the intermediate 57 Fe produced from the 57 Co are such that the system behaves similarly to Fe.

For the FePc in a moderately high area Teflon bonded carbon electrode, two sets of doublets are observed: one associated with the bulk solid and the other with the adsorbed species. The quadrupole splitting for the adsorbed species (0.88 mm/s) is low and provides evidence that the adsorbed species is probably bound through an axial 0 ligand to the electrode surface. This would be compatible with configuration b in Fig. 3.

The Mossbauer effect spectroscopy has also provided some interesting information on the effects of the electrode treatment on the state of the FePc in the electrode (27). For example, with FePc on carbon treated at ~ 400 °C in H₂ before electrochemical examination, the Mossbauer measurements revealed the six doublets of metallic iron in a dispersed form. In helium under the same preparative conditions, there was no evidence of instability from the Mossbauer measurements. The Mossbauer measurements have also indicated that the amount of the macrocyclic complex in the adsorbed and bulk states in the electrode is quite sensitive to the preparative procedure.

-17-

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- 44. "Applications of <u>in situ</u> Mossbauer Effect Spectroscopy to the Study of Electrode-Electrolyte Interfaces," <u>D. Scherson</u>, S. Yao, E. Yeager, J. Eldridge, M. Kordesch and R. Hoffman, <u>ibid</u>.

<u> </u>

- 45. "Mechanistic Aspects of O₂ Electrocatalysis," by E. Yeager, D. Scherson and B. Simic-Glavaski, The Electrochemical Society National Meeting, San Francisco, May 1983. Extended Abstr. <u>83-1</u>, 706 (submitted for publication in Proceedings of the Symposium on Electrocatalysis).
- 46. "Oxygen Reduction Electrocatalysis: Fundamental Aspects," by
 E. Yeager, The Electrochemical Society, National Meeting, Washington, D.C. October 1983. Extended Abstr. 83-2, 387.

47. "Oxygen Interaction with Adsorbed Tatrasulfonated Phthalocyanines," by S. Zecevic, B. Simic-Glavaski and E. Yeager, loc. cit. Extended Abstr. 83-2, 482.

CONTRACT PERSONNEL IV.

- A. Senior Personnel
 - Ernest Yeager, Hovorka Professor of Chemistry and Director, 1. Case Center for Electrochemical Sciences: Project Director (1976 - 83)
 - B. D. Cahan, Professor of Chemistry: Co-principal investigator 2. (1976 - 83)
- Graduate Students (Ph.D theses based on ONR centered research) Β.
 - 1. C. Chen (1977-78) Ph.D 1979
 - 2. Arthur Homa (1978-82) Ph.D 1982
 - 3. Michael Hanson (1980-84) Ph.D thesis completion Oct. 1984
 - R. Allen Wilkinson (1981-84) Ph.D thesis completion, spring 1985 4.
 - G. McDougall (1982-84) Ph.D thesis completion, late 1985 5.

C. Postdoctoral appointments

- R. K. Sen (1976) 1.
- W. O'Grady (1976)
 B. Nikolic (1978)
- B. Simic-Glavaski (1977) 4.

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