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X-RAY PHOTOELECTRON SPECTROSCOPIC STUDIES OF ELECTRODE SURFACES-ETC(U)

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X-RAY PHOTOELECTRON SPECTROSCOPIC STUDIES OF ELECTRODE SURFACES

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20 ABSTRACT (Continue on reverse side if necessary and identify by block number) Emphasis is on the development of x-ray photoelectron spectroscopy (XPS) in characterizing electrochemical reaction mechanisms. The advantages of XPS to the study of electrode surfaces are the depth sensitivity to most metals is quite high with the escape length of the emitted electrons occurring only through 10-20A of the sample surface. The measured binding energies are sensitive to the oxidation state of the metal atom. Underpotential deposition for silver and copper on platinum electrodes have been examined by both XPS and Auger			

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spectroscopy. A combination of XPS and secondary ion mass spectrometry (SIMS) was designed and connected with two satellite vacuum systems via a set of magnetically driven transfer device. This configuration allowed each technique to have its own chamber for electrode preparation. High energy ion beams were investigated for use in the ion implantation of various substrates aimed at creating new materials with unusual properties. Initial studies were focused on use of copper, silver, and gold ion beams directed toward silicon dioxide and graphite.

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I. Introduction

The basic objective of our AFOSR research has emphasized the development of x-ray photoelectron spectroscopy in characterizing electrochemical reaction mechanisms. During the last several years, we have focused our attention on developing XPS as a mechanistic aid in following electrode reactions, primarily those whose surfaces can be altered by the formation of oxide layers. In addition, the method has proved valuable in examining the surface chemistry of metal electrodes where monolayers of foreign metal ions can be deposited at underpotential. These studies are particularly relevant to electrocatalysis since trace metals interacting with an electrode surface can drastically alter the electrode characteristics. Finally, we have exploited the idea that the information gleaned about surfaces by XPS can be greatly enhanced by coupling this spectroscopy with other types such as secondary ion mass spectrometry (SIMS). Examples of the accomplishments relative to these specific objectives will not be discussed in more detail.

II. Results and Discussion

The major advantages of XPS to the study of electrode surfaces are that the depth sensitivity to most metals is quite high with the escape length of the emitted electrons occurring only through 10-20^o of the sample surface. In addition, the measured binding energies are sensitive to the oxidation state of the metal atom. Using our specially

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A. D. BLOSE
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designed apparatus for transferring the electrode into the vacuum system (1), we have successfully monitored the growth of PtO , $\text{Pt}(\text{OH})_2$ and PtO_2 films on platinum oxidized in acidic media. It was also possible to monitor the thickness of the overlayer by following the intensity of the various peaks. For example, the limiting thickness of the "oxide I" was found to be $8\text{\AA}$ (1). Higher acid concentrations also induced anion incorporation into the oxide layer which suggested that these components provided the mechanistic key to control the growth of oxide and to prevent multilayer PtO_2 growth on platinum electrodes.

The sensitivity of XPS makes it ideally suited to examining submonolayer quantities of impurities on electrodes. Certain metal ions, for example, are known to reduce below their thermodynamic potential, in the submonolayer regime. We have examined this underpotential deposition for Ag and Cu on Pt electrodes by both XPS and Auger spectroscopy(2). XPS chemical shifts of -0.65 and -0.95 eV vs. the bulk metal have been observed although no distinction was noted between the various underpotential states present in the cyclic voltammogram. The shifts were identical to vapor-deposited submonolayer films of Ag and Cu on Pt in the low coverage ($\theta \sim 0.1$) limit. A gradual shift with coverage to the bulk metal value for the vapor-deposited films and a constant shift with coverage for the underpotential deposit indicated that islanding was present only in the evaporated films. Measurement of the Cu and Ag Auger spectra gave

results independent of the surface work function when compared to the XPS spectra. Interpretation of this Auger parameter is, however, at present ambiguous. These results provide an excellent beginning in our understanding of the electronic structure of these important electrode surfaces.

Of special recent interest is the incorporation of additional surface analysis methods to our XPS spectrometer. The combination of XPS and SIMS seems particularly appropriate since many of the disadvantages of XPS are offset by the additional capability of SIMS. Its ability to detect hydrogen is such an example. Our instrument design philosophy is to connect two satellite vacuum systems via a set of magnetically driven transfer devices (3). This configuration allows each technique to have its own chamber and allows a separate chamber for electrode preparation. This combined approach has been illustrated in numerous applications (4-8) of the study of metal/oxygen systems.

A final accomplishment in the work is the investigation of high energy ion beams for use in the ion implantation of various substrates aimed at the possibility of creating new materials with unusual properties (9,10). Our initial studies have been focused on the use of Cu^+ , Ag^+ and Au^+ ion beams directed toward SiO_2 and graphite. For these systems, the XPS measurements reveal the implanted atoms have atomic-like electronic structures similar to the underpotential deposition procedure discussed earlier. The relevance of these investigations to electrochemical studies is preliminary, but this idea has considerable future potential, since ion implantation methods could be used to

prepare high purity doped electrodes or to prepare corrosion resistance surfaces by implantation of trace quantities of transition metals.

In conclusion, our work has focused on the development of modern surface analysis methods, particularly XPS and SIMS, for the characterization of metal or electrochemically modified surfaces. The chemical specificity and surface sensitivity of these methods clearly provides a new dimension to our ability to characterize these systems. It will be particularly interesting in the future to extend these systems to more complex surfaces such as alloys and metals covered with organic films, since the electrochemical properties of these assemblages are only barely known.

References

1. "X-ray Photoelectron Spectroscopic Study of Potentiostatic and Galvanostatic Oxidation of Pt Electrodes", J. S. Hammond and N. Winograd, J. Electroanal. Chem. 78, 55 (1977).
2. "X-ray Photoelectron Spectroscopic Study of Underpotential Deposition of Ag and Cu on Pt Electrodes", J. S. Hammond and N. Winograd, J. Electrochem. Soc. 124, 826 (1977).
3. "A Combined XPS-SIMS Instrument for Surface Studies", R. W. Hewitt, A. T. Shepard, W. E. Baitinger, N. Winograd and W. N. Delgass, Rev. Sci. Instr. 50, 5 (1979).
4. "Characterization of Metal Surfaces by SIMS and XPS", R. W. Hewitt, A. Shepard, W. E. Baitinger, G. L. Ott, W. N. Delgass and N. Winograd, Anal. Chem. 59, 1286 (1978).
5. "Detection of High Mass Cluster Ions Sputtered from Bi Surfaces", A. Shepard, R. W. Hewitt, G. S. Slusser, W. E. Baitinger, N. Winograd, R. G. Cooks, A. Varon, G. Devant, and W. N. Delgass, Chem. Phys. Lett. 44 371 (1976).
6. "Investigation of the Oxidation of Polycrystalline Lead by XPS and SIMS", R. W. Hewitt and N. Winograd, Surf. Sci. 78, 1 (1978).
7. "Chemisorption of Oxygen on Ni(100) by XPS and SIMS", T. Fleisch, W. N. Delgass, and N. Winograd, Surf. Sci. 78, 141 (1978).
8. "Oxidation of Polycrystalline Indium Studied by XPS and Static SIMS", R. W. Hewitt and N. Winograd, J. Applied Phys. in press.
9. "X-ray Photoelectron Spectroscopic Studies of Atom Implanted Solids: Ag and Au in SiO₂", V. Y. Young, R. A. Gibbs, K. S. Kim, and N. Winograd, Chem Phys Lett. 54, 378 (1978).

10. "X-ray Photoemission Studies of Atom Implanted Matrices: Cu, Ag, and Au in SiO₂", V. Y. Young, R. A. Gibbs, and N. Winograd, J. Chem. Phys. 70, 5714 (1979).

III. Lectures and Travel Related to the Grant

7

Nicholas Winograd

1. Gordon Research Conference, Electrochemical, Santa Barbara, California, January 17-23, 1976.
2. Electrochemical Society, Washington, D.C., May 2-5, 1976.
3. American Chemical Society, Midwest Regional Meeting, Chicago, Illinois, June 17-19, 1976.
4. Gordon Research Conference, Analytical, New Hampton, New Hampshire, August 9-13, 1976, "Recent Developments in Surface Analysis: A Multi-technique Approach". (invited lecturer)
5. University of Uppsala, Uppsala, Sweden, August 26, 1976, "Detection of High Mass Cluster Ions Sputtered from Bi Surfaces". (invited lecturer)
6. VII International Congress on Photobiology, Rome, Italy, August 29- September 3, 1976, "X-ray Photoelectron Spectroscopic Studies of the Thermal Stability of Chlorophyll a Monohydrate" and "In Vitro Solar Conversion After the Primary Light Reaction in Photosynthesis. Reversible Photogalvanic Effects of Chlorophyll-Quinhydrone Half-cell Reactions".
7. Society of Electron Spectroscopy, Naperville, Illinois, October 20, 1976.
8. University of Cincinnati, Cincinnati, Ohio, October 29, 1976, "A Multi-technique Approach to Characterization of Solid Surfaces". (invited lecturer)
9. Wayne State University, Detroit, Michigan, November 8, 1976, "A Multi-technique Approach to Characterization of Solid Surfaces". (invited lecturer)
10. University of Kentucky, Lexington, Kentucky, December 7, 1976, "Studies of Metal-oxygen Surfaces Using Electron and Particle Spectroscopy". (invited lecturer)

11. Colorado State University, Fort Collins, Colorado, January 12, 1977, "Studies of Metal-oxygen Surfaces Using Electron and Particle Spectroscopy". (invited lecturer)
12. Gordon Research Conference, Electrochemical, Santa Barbara, California, January 17-21, 1977.
13. Quantum Institute, University of California, Santa Barbara, California, January 19, 1977, "Studies of Metal-oxygen Surfaces Using Electron and Particle Spectroscopy". (invited lecturer).
14. Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Cleveland, Ohio, March 1-3, 1977, "X-ray Photoelectron Spectroscopy (ESCA) and Secondary Ion Mass Spectrometry (SIMS)". (invited lecturer).
15. Shell Development Company, Houston, Texas, March 8, 1977, "X-ray Photoelectron Spectroscopy (ESCA) and Secondary Ion Mass Spectrometry (SIMS)". (invited lecturer)
16. University of Houston, Houston, Texas, March 9, 1977, "X-ray Photoelectron Spectroscopy (ESCA) and Secondary Ion Mass Spectrometry (SIMS)". (invited lecturer)
17. University of Pittsburgh, Pittsburgh, Pennsylvania, March 17, 1977, "X-ray Photoelectron Spectroscopy (ESCA) and Secondary Ion Mass Spectrometry (SIMS)". (invited lecturer)
18. Society of Electron Spectroscopy, Purdue University, West Lafayette, Indiana, March 31, 1977, "X-ray Photoelectron Spectroscopy (ESCA) and Secondary Ion Mass Spectrometry (SIMS)". (invited lecturer)
19. The Chemical Society, Faraday Division, Electrochemistry, Southampton, England, April 19-21, 1977.
20. The University, Newcastle-Upon-Tyne, England, April 18, 1977, "Secondary Ion Mass Spectrometry and Surface Chemistry". (invited lecturer)

21. The Chemical Society, Faraday Division, University of Southampton, England, April 20, 1977, "X-ray Photoelectron Spectroscopic Studies of Electrode Surfaces". (invited lecturer)
22. Oxford, England, April 22, 1977, "Photogalvanic Effects and Photosynthesis". (invited lecturer)
23. VII Annual Symposium on the Analytical Chemistry of Pollutants, Lake Lanier, Georgia, April 26, 1977, "ESCA and SIMS: A Multi-technique Approach to Surface Analysis". (invited lecturer)
24. IV Biennial Air Force Electrochemistry Conference, The United States Air Force Academy, Colorado Springs, Colorado, April 28-29, 1977, "X-ray Photoelectron Spectroscopic Studies of Electrode Surfaces". (invited lecturer)
25. Japanese-American Seminar, U.S.-Japan Cooperative Science Program, Division of International Programs, National Science Foundation, San Francisco, California, May 16-19, 1977, "X-ray Photoelectron Spectroscopic Studies of Electrode Surfaces". (invited lecturer)
26. National American Chemical Society Meeting, Chicago, Illinois, August 29-30, 1977, "Characterization of Metal Surfaces by SIMS and XPS". (invited lecturer)
27. VII International Vacuum and III International Conference on Solid Surfaces, Vienna, Austria, September 12-16, 1977, "Quantitative Surface Studies with X-ray Photoelectron Spectroscopy (XPS) and Secondary Ion Mass Spectroscopy (SIMS)".
28. International Conference on SIMS and Ion Microprobes, Muenster, Germany, September 19-23, 1977, "Secondary Ion Mass Spectroscopic and X-ray Photoelectron Spectroscopic Studies of Metal-oxygen Surfaces". (invited lecturer)
29. Colloquium, Carnegie-Mellon University, Pittsburgh, Pennsylvania, September 22, 1977, "Characterization of Solid Surfaces Using Electron and Particle Spectroscopy". (invited lecturer)

30. Federation of Analytical Chemistry and Spectroscopy Societies, Detroit, Michigan, November 10, 1977, "XPS-SIMS: A New Approach to Surface Analysis". (invited lecturer)

31. IBM, San Jose, California, December 5, 1977, "Low Energy Ion Impact Phenomena on Single Crystal Surfaces". (invited lecturer)

32. E. I. DuPont de Nemours & Company, Inc., Wilmington, Delaware, January 18, 1978, "Surface Structures Using X-ray Photoelectron Spectroscopy and Secondary Ion Mass Spectrometry". (invited lecturer)

33. Gordon Research Conference, Electrochemical, Santa Barbara, California, January 23-27, 1978, "Studies of Electrode Surface Chemistry with Electron and Particle Spectroscopy". (invited lecturer)

34. Naval Postgraduate School, Monterey, California, March 17, 1978, "Ion Impact Phenomena on Single Crystal Surfaces". (invited lecturer)

35. Shell Research & Development, Houston, Texas, April 7, 1978, "Ion Impact Phenomena on Clean and Reacted Single Crystal Surfaces". (invited lecturer)

36. Surface Science and Catalysis Seminar, University of California and Lawrence Berkeley Laboratory, Berkeley, California, April 26, 1978, "Ion Impact Phenomena on Clean and Reacted Single Crystal Surfaces". (invited lecturer)

37. 1978 IEEE International Conference on Plasma Science, Monterey, California, May 15-17, 1978, "Ion Impact Phenomena on Clean and Reacted Single Crystal Surfaces", with B.J. Garrison.

38. American Society of Mass Spectrometry, St. Louis, Missouri, May 15, 1978, "Ion Impact Phenomena on Clean and Reacted Single Crystal Surfaces", with D. E. Harrison, Jr., and B. J. Garrison.

39. American Institute of Chemical Engineers Meeting, Philadelphia, Pennsylvania, June 5, 1978, "X-ray Photoelectron Spectroscopy and Secondary Ion Mass Spectroscopy of Alloy Catalysis", with W. N. Delgass.

40. 38th Annual Conference on Physical Electronics, Gatlinburg, Tennessee, June 21-24, 1978, "Structure-sensitive Factors in Molecular Cluster Emission by Ion Bombardment of Reacted Single Crystal Surfaces".

41. 4th Pennsylvania State Read Conference on Electrodeposition, University Park, Pennsylvania, August 7-11, 1978, "ESCA and Its Use in the Study of Electrodeposition". (invited lecturer)

42. 176th American Chemical Society Meeting, Miami, Florida, September 10-15, 1978, "XPS/SIMS Investigations of Electrode Surfaces in Chemistry" and "SIMS/XPS Studies of Clean and Reacted Metal Surfaces". (invited lecturer)

43. Foster Lecture Colloquium, Department of Chemistry, University of New York at Buffalo, Buffalo, New York, November 1, 1978, "Close Encounters of Another Kind: Ion Solid Interactions". (invited lecturer).

44. 25th National Vacuum Symposium, American Vacuum Society, San Francisco, California, November 27-30, 1978, "Structure-sensitive Factors in Molecular Cluster Ejection by Ion Bombardment of Ni Single Crystals Reacted with CO and O₂". (invited lecturer)

45. Pennsylvania State University, University Park, Pennsylvania, January 14, 1979, "Understanding the Role of Solid Surfaces in Chemical Processes: Analysis by XPS and SIMS". (invited lecturer)

46. Department of Chemistry, Northwestern University, Evanston, Illinois, February 9, 1979, "Surface Structure Determination by Ion Bombardment of Single Crystals". (invited lecturer)

47. Colloquium, Upjohn, Kalamazoo, Michigan, February 20, 1979, "Surface Analysis by ESCA". (invited lecturer)

48. National Bureau of Standards, Washington, D.C., February 26, 1979, "Determination of Surface Structure with Ion Beams". (invited lecturer)

49. National Science Foundation, Washington, D.C., February 27, 1979, "Determination of Surface Structure with Ion Beams". (invited lecturer)

50. American Physical Society, Chicago, Illinois, March 19-23, 1979,¹²
"Structure Determination by Ion Bombardment of Single Crystals". (invited
lecturer)

51. 177th American Chemical Society Meeting, Honolulu, Hawaii, April 1-6,
1979, "Surface Structure Determinations by Ion Bombardment of Single Crystals".
(invited lecturer)

52. General Motors Technical Center, Research Laboratory, Warren, Michigan,
April 16, 1979, "Surface Structure by Ion Bombardment of Single Crystals".
(invited lecturer)

53. American Chemical Society, Midwest Regional Meeting, Columbus, Ohio,
May 7-9, 1979, "SIMS and the Surface Analysis Problem". (invited lecturer)

54. Physical Electronics Conference, College Park, Maryland, June 19,
1979, "Surface Structure from Angle-Resolved SIMS".

55. Second International Conference on Secondary Ion Mass Spectrometry,
Stanford, California, August 27-30, 1979.

56. International Conference on Non-traditional Approaches to the Study
of the Solid-Electrolyte Interface, Snowmass, Colorado, September 24-27, 1979.

57. Expo-Chem, Houston, Texas, October 22-25, 1979, "Surface Analysis
with Ion Beams". (invited lecturer)

58. Analytical Chemistry Seminar, Texas A & M, College Station, Texas,
October 26, 1979, "Determination of Surface Structure with Ion Beams".

59. Department of Chemistry Colloquium, University of Arizona, Tucson,
Arizona, November 19, 1979.

IV. PUBLICATIONS

1. Detection of High Mass Cluster Ions Sputtered from Bi Surfaces, A. Shepard, R. W. Hewitt, G. S. Slusser, W. E. Baitinger, N. Winograd, R. G. Cooks, A. Varon, G. Devant, and W. N. Delgass, Chem. Phys. Lett., 44, 371 (1976).
2. X-ray Photoelectron Spectroscopic Study of Potentiostatic and Galvanostatic Oxidation of Pt Electrodes, J. S. Hammond, and N. Winograd, J. Electroanal. Chem., 78, 55 (1977).
3. X-ray Photoelectron Spectroscopic Study of Underpotential Deposition of Ag and Cu on Pt Electrodes, J. S. Hammond and N. Winograd, J. Electrochem. Soc., 124, 826 (1977)
4. X-ray Photoelectron Spectroscopic Studies of Atom Implanted Solids: Ag and Au in SiO₂, V. Y. Young, R. A. Gibbs, K. S. Kim and N. Winograd, Chem. Phys. Lett., 54, 378 (1978).
5. Stoichiometric Determination of Chlorophyll a Water Aggregates and Photosynthesis. Symbiotic Roles of the Mg Atom and the Ring V Cyclopentanone Group in the Structural and Photochemical Properties of Chlorophyll a Monohydrate and Dihydrate, J. C. Brace, F. K. Fong, D. H. Karweik, V. Koester, A. Shepard, and N. Winograd. J. Am. Chem. Soc., 50, 1286 (1978).
6. Characterization of Metal Surfaces by SIMS and XPS, R. W. Hewitt, A. Shepard, W. E. Baitinger, G. L. Ott, W. N. Delgass and N. Winograd, Anal. Chem., 59, 1286 (1978).
7. Low Energy Ion Impact Phenomena on Single Crystal Surfaces, D. E. Harrison, Jr., P. W. Kelly, B. J. Garrison and N. Winograd, Surface Sci., 76, 311 (1978).
8. Formation of Small Metal Clusters By Ion Bombardment of Single Crystal Surfaces, B. J. Garrison, N. Winograd, and D. E. Harrison, Jr.,

J. Chem. Phys., 69, 1440 (1978).

9. Angular Distributions of Ejected Particles from Ion Bombarded Clean and Reacted Single Crystal Surfaces, N. Winograd, B. J. Garrison, and D. E. Harrison, Jr., Phys. Rev. Lett., 41, 1120 (1978).

10. Chemisorption of Oxygen on Ni(100) by XPS and SIMS, T. Fleisch, W. N. Delgass, and N. Winograd, Surface Sci., 78, 141 (1978).

11. Investigation of the Oxidation of Polycrystalline Lead by XPS and SIMS, R. W. Hewitt and N. Winograd, Surface Sci., 78, 1 (1978).

12. Atomic and Molecular Ejection from Ion Bombarded Reacted Single Crystal Surfaces. Oxygen on Copper(100), B. J. Garrison, N. Winograd, and D. E. Harrison, Jr., Phys. Rev. b, 18, 6000 (1978).

13. Chemisorption of CO on Ni(100) by SIMS and XPS, T. Fleisch, G. L. Ott, W. N. Delgass, and N. Winograd, Surface Sci., 81, 1 (1979).

14. Surface Segregation of PdAg Alloys Induced by Argon Ion Bombardment, G. Slusser and N. Winograd, Surface Sci., 84, 211 (1979).

15. Ejection of Molecular Clusters from Ion Bombarded Surfaces, B. J. Garrison, N. Winograd and D. E. Harrison, Jr., J. Vac. Sci. Tech., 16, 789 (1979).

16. Particle Ejection from Ion Bombarded Clean and Reacted Single Crystal Surfaces, N. Winograd, B. J. Garrison, T. Fleisch, W. N. Delgass and D. E. Harrison, Jr., J. Vac. Sci. Tech., 16, 629 (1979).

17. X-ray Photoelectron Spectra of N-Methyltetraphenylporphrin: The Free Base, Diacid Cation and Complexes, D. K. Lavalley, J. G. Brace, and N. Winograd, Inorg. Chem., 18, 1776 (1979).

18. X-ray Photoemission Studies of Atom Implanted Matrices: Cu, Ag, and Au in SiO₂, V. Y. Young, R. A. Gibbs, and N. Winograd, J. Chem. Phys., 70, 5714 (1979).

19. Classical Trajectory Calculations of the Energy Distribution of Ejected Atoms from Ion Bombarded Single Crystals, B. J. Garrison, N. Winograd, and D. E. Harrison, Jr., Surface Sci., 87, 101 (1979).
20. Surface Structure from Angle-resolved SIMS. Oxygen on Cu(001), S. P. Holland, B. J. Garrison, and N. Winograd, Phys. Rev. Lett., 43, 220 (1979).
21. Evidence for a Recombination Mechanism of Cluster Formation from Ion Bombarded Surfaces, N. Winograd, K. E. Foley, B. J. Garrison, and D. E. Harrison, Jr., Phys. Lett., 73A, 253 (1979).
22. A Combined XPS-SIMS Instrument for Surface Studies, R. W. Hewitt, A. T. Shepard, W. E. Baitinger, N. Winograd, and W. N. Delgass, Rev. Sci. Instr., 50, 5 (1979).
23. One Photon and Two Photon processes in Chlorophyll a Water Splitting Light Reactions. Reversible and Irreversible Photochemical Pathways, L. M. Fetterman, F. K. Fong, and N. Winograd, Photochem. and Photobiol., submitted.
24. Mechanisms of CO Ejection from Ion Bombarded Single Crystal Surfaces N. Winograd, B. J. Garrison and D. E. Harrison, Jr., Phys. Rev. B, submitted.
25. Detection of Reversibly Bound Adsorbates by SIMS, CO on Ni(001) and Polycrystalline Iron, T. Fleisch, G. L. Ott, W. N. Delgass and N. Winograd, Surface Sci., submitted.
26. Oxidation of Polycrystalline Indium Studies by XPS and Static SIMS, R. W. Hewitt and N. Winograd, J. Applied Phys., submitted.

V. HONORS AND AWARDS

Alfred P. Sloan Foundation Fellow	1974-1978
J. S. Guggenheim Memorial Foundation Fellow	1977-1978

VI. RESEARCH PERSONNEL

Principal Investigator:	Professor Nicholas Winograd
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Postdoctoral Associates:	Dr. Theo Fleisch
	Dr. Kwang Kim
	Dr. Vaniecia Young

Research Assistants:	Stephen Gaarenstroom
	Richard Gibbs
	Richard Hewitt
	John Holland
	John Brace
	John Hammond