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OVERVIEW OF RESEARCH PROGRAM

High resolution Electron Energy Loss Spectroscopy (EELS) is an important new spectroscopic technique which can be utilized to study chemical processes at surfaces. The fundamental principle which forms the basis for this spectroscopy is very simple: Vibrational energies of matter, in particular, solids and species adsorbed on surfaces, are quantized. Therefore, energy exchange involving vibrational levels occurs in quantized units of energy. Electron energy loss spectroscopy determines these quantized vibrational levels by measuring the energy difference (either loss or gain) between electrons incident and scattered at a surface.

One of the most useful aspects of EELS is that it is sensitive to molecular parameters. Unlike Auger spectroscopy, for example, which is an atomic spectroscopy, EELS spectra reflects the molecular composition of chemisorbed species, and in the case of atomic adsorption (ie dissociative adsorption of gas phase molecules), the vibrational levels can contain information about the local adsorption site. The ability of EELS to provide molecular information at surfaces accounts for the broad range of potential applications anticipated for the spectroscopy. This also accounts for the explosive rate of growth of EELS investigations which is now becoming apparent.

The objective of the current research project was to utilize EELS in conjunction with other standard surface science probes including low energy electron diffraction (LEED) and Auger electron spectroscopy to investigate chemical processes which occur at surfaces of metals having important technological applications. Metals falling into this category from the point of view of the Air Force clearly include aluminum and titanium, but also include such metals as nickel, copper and tungsten for example. EELS is a very new technique and nearly any metal-adsorbate system one chooses to investigate in any detail using EELS represents a legitimate scientific project. We have directed most of our efforts during the last three years toward the study of three systems: O/Al(111); O/Ni(100) and H/W(110). These studies have yielded important results which have led to a better understanding of the surface chemistry of these systems. In addition, our results have expanded the understanding of EELS mechanisms and opened up new applications. These results are summarized in this document.

INSTRUMENT DEVELOPMENT

The first 12-16 months of our project were dominated by efforts to set up our spectrometer and characterize its performance. Our EELS spectrometer incorporates several of the more common but very important surface diagnostic capabilities including LEED, Auger analysis and mass spectroscopy in addition to a state-of-the-art EELS spectrometer. Considerable design work and custom machine shop work were required to properly integrate all of these techniques into a common vacuum chamber without compromising performance of any one of the techniques. Our first interim progress report summarized the performance of our EELS optics. Our instrument achieves performance equal to the best which has been reported. Important instrument characteristics include:

Resolution: 4meV FWHM (direct beam geometry @ I=1x10⁻¹¹ A) Energy Stability: 1meV or greater over a 10 hour period

We have interfaced our EELS and Auger spectrometer with a computer based signal averager which permits signal averaging of weak signals and direct transmission of data to a digital computer for curve fitting and analysis.

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SCIENTIFIC WORK

A broad range of metal surfaces with different adsorbates have been investigated. A brief description of most of these has been given in the two previous (interim) scientific reports. In this (final) report, only the systems which have produced significant scientific results are described in detail. Selected reprints are included in the appendix of this report. In this section, a concise summary of each specific result is given with an indication of its importance and how it ties in with other work.

A. The Initial Stage of Oxide Formation on Al(111).

Surface processes which occur during the initial stage of oxidation of aluminum have been investigated using a variety of techniques and appear to be rather complicated. This fact was clear when we begin our EELS investigation of O/Al(111). Our EELS study of O/Al(111) established clearly for the first time that the initial stage of oxide formation on aluminum involved simultaneous formation of underlayers and overlayers. This result helped explain many of the difficulties encountered in previous studies. (see ref. 1 and appendix A). B. Application of EELS to Underlayer Atoms

The observation of vibrational loss peaks due to subsurface atoms demonstrated the first application of EELS to diffusion processes near surfaces. The dipole scattering mechanism clearly describes scattering from subsurface atoms (see ref. 1 and appendix A) and lattice dynamical calculations later verified the peak assignments to subsurface atoms (see ref. 2 and appendix B). C. Application of Lattice Dynamical Models to EELS

In order to understand our EELS spectra for 0/Al(111) in more detail and to verify the peak assignments, we carried out extensive lattice dynamical calculations for various configurations of oxygen overlayers and underlayers at Al(111) surfaces. These calculations demonstrated clearly the important role

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the substrate lattice dynamics play in EELS spectra for metal/adsorbate complexes having substrate atoms with mass comparable to the adsorbed species. These calculations verified peak assignments (surface and subsurface modes) for O/Al(111) and permitted an accurate estimate of the preferred high symmetry sites for surface and subsurface atoms (see ref. 2 and 3 and appendix B and C). D. Impact Scattering Studies

Several distinct scattering regimes can be identified in electron scattering from vibrational modes at surfaces. Dipole scattering events typically dominate spectra taken in specular scattering geometry where $\Delta q_{\mu}=0$. When the scattering angle is significantly different from the incident angle, or out of plane scattering is studied, $\Delta q_{\mu} \neq 0$ and the dipole scattering mechanism is ineffective. A new mechanism called impact scattering begins to play an important role in inelastic losses which occur. The same mechanism accounts for the thermal diffuse scattering in LEED.

Impact scattering has some extremely important potential applications in surface studies because of selection rules (associated with the scattering processes) which have direct relationships to the mode symmetries and hence the local site symmetry. We conducted an experiment aimed at observing the effects predicted by the symmetry relationships. The experiment involved studying the azimuthal dependence of EELS spectra from H on W(110). This surface has two inequivalent mirror planes compared with two equivalent mirror planes for W(100). One therefore expects to observe a different behavior in the cross-section as a function of azimuthal angle. More specifically the asymmetric parallel vibration of a bridge bonded H atom on W(100) has two equivalent directions separated by a 90° azimuthal angle. For W(110) one can see possibilities for a 180° angle.

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Our experiment did not establish conclusively the adsorption site of H/W(110) or verify explicitly the selection rules. The experiment did produce a striking azimuthal pattern and did definitely show that impact scattering and dipole scattering are qualitatively different in terms of azimuthal dependence of the scattering cross-section. This work was reported at the Asilomar Conference (see ref. 4 and appendix D).

The experiments and calculations briefly summarized in this section are described in detail in the attached appendix (A-D).

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PUBLICATIONS AFOSR-80-0154

- A Journal Articles (Reprints are attached)
- J. L. Erskine and R. L. Strong, "High Resolution Electron-Energy-Loss Spectroscopy Study of the Oxidation of Al(111)," Phys. Rev. B25, 5547 (1982).
- R. L. Strong, B. Firey, F. W. deWette and J. L. Erskine, "Surface-Site Determination Using Electron-Energy-Loss-Spectroscopy and Lattice Dynamical Models," Phys. Rev. B26, 3483 (1982).
- R. L. Strong, B. Firey, F. W. deWette and J. L. Erskine, "Adsorbate Structure Modeling Based on Electron-Energy-Loss-Spectroscopy and Lattice Dynamical Calculations: Application to O/A1(111)," J. Electron Spectroscopy Rel. Phenom. 29, 187 (1983).
- B. M. Davies and J. L. Erskine, "Azimuthal Dependence of Impact Scattering in Electron-Energy-Loss-Spectroscopy," J. Electron Spectroscopy Rel. Phenom. 29, 323 (1983).
- B Conferences/Workshops/Seminars/Interactions

Papers at Meetings

- R. L. Strong and J. L. Erskine, "Off-Specular EELS Studies of Adsorbates on Ni(100). Bull. American Phys. Soc. 27, 374 (1982). (March Meeting of the American Physical Society, Dallas, 1982).
- 2. See publications 3 and 4. These papers were presented at the Third International Conference on Vibrations at Surfaces, Asilomar, California, September 1-4, 1982.
- 3. J. L. Erskine, "High-Resolution Electron Energy Loss Studies of Subsurface Atoms," AFOSR/FJSR Molecular Dynamics and Surface Chemistry Conference, USAF Academy, Colorado Springs, Co., December, 1982.

Seminars

 J. L. Erskine, "Electron Energy Loss Spectroscopy of Adsorbates at Metal Surfaces," Surface Science Seminar, Cornell University, Ithaca, New York, February 24, 1982.

Interactions with Other Investigators

- 1. Meeting with Dr. S. Y. Tong, Department of Physics, University of Wisconsin at Milwaukee regarding our work on impact scattering. Dr. Tong is a leading authority on multiple scattering.
- 2. Meeting with Dr. D. L. Mills, Department of Physics, University of California, Irvine regarding our work on subsurface atoms at Al(111).
- 3. Meeting with Dr. W. Ho, Department of Physics, Cornell University, Ithaca, N.Y. regarding our work on impact scattering.