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Department of Chemistry
University of Texas at Austin
Austin, Texas 78712
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Final Technical Report
AFOSR - 87 - 0070

Surface Analysis System and Surface Raman Spectroscopy

Alan Campion
The University of Texas at Austin
Austin, TX 78712

5 August 1988

Abstract

A multipurpose surface analysis chamber equipped with X-ray photoelectron spectroscopy, ion scattering spectroscopy, secondary ion mass spectrometry and Auger electron spectroscopy has been constructed and installed. The Leybold LHS-12 system which was partially funded from this grant, comprises both high pressure and ultrahigh vacuum sample preparation and reaction chambers, thermal evaporation and radiofrequency sputtering sources and a rapid entry load lock. This system is fully operational, meeting all specifications, and is being used in a wide variety of surface science and other applications. These include the XPS study of the decomposition of alkyl halides in support of our surface Raman spectroscopy efforts, an XPS study of the X-ray induced degradation of thin polymer films and a study of the role of peroxide ions in the mechanism of high temperature superconductivity.

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This report covers the design, construction and installation of a multipurpose surface analysis system in support of our AFOSR - sponsored research in the surface chemistry leading to the preparation of electronic materials, with emphasis on laser control of the process. The instrument was requested to provide information which is complementary to that obtained by in-situ methods under development in this laboratory, chiefly surface Raman spectroscopy. The overall goal of the proposed research was to understand the microscopic mechanisms of surface mediated chemical reactions. The short range goal of the limited duration AFOSR grant was to develop further our capabilities in unenhanced surface Raman spectroscopy, specifically to improve sensitivity. A description of the instrument acquired follows as well as a discussion of its relationship to AFOSR and other supported research.

The instrument comprises a Leybold-Heraeus LHS-12 surface analysis system with both high pressure and ultrahigh vacuum sample preparation and reaction chambers. The ultrahigh vacuum sample preparation chamber is equipped with facilities for thermal evaporation sources and radiofrequency sputtering sources for materials deposition. Samples may be transferred under vacuum from either of these materials preparation chambers into the main surface analysis chamber. There is also a rapid entry load lock for quick turnaround of standard surface analyses of materials requiring no preparation.

The main surface analysis system comprises X-ray photoelectron spectroscopy, ion scattering spectroscopy, secondary ion mass spectrometry and Auger electron spectroscopy. Specifications which have been met include:

X-ray Photoelectron Spectroscopy (XPS)

EA-11/100 Analyzer

- 1) 80,000 cps at 0.9 eV FWHM resolution. 300 W Mg Anode on the Ag 3 d_{5/2} line.

Auger Electron Spectroscopy (AES)

Specifications taken on Cu

- 1) 0.3% energy resolution.
- 2) S/N > 100 for 10nA primary beam current in pulse counting mode.
S/N > 300 with analog detection at 100nA primary current.

Ion Scattering Spectroscopy (ISS)

- 1) 200,000 cps from 2 keV He⁺ ions at 1 μA beam current from Ag.

Secondary Ion Mass Spectrometry (SIMS)

- 1) 6×10^4 cps from oxidized molybdenum. 1nA, 5 keV Ar⁺ primary ions.

The system has been used for a variety of surface science and other studies, supported both by AFOSR and other agencies. Manuscripts describing this work are under preparation.

In support of our efforts in Raman spectroscopy we have used XPS to look at thermal reactions of methylene chloride on metal surfaces as a source of surface methylene radicals. These reactive intermediates have very low Raman cross sections which we want to use to quantify our developmental efforts in surface Raman spectroscopy. In a second project XPS has been used to study X-ray damage to thin polyvinyl alcohol films. In a third project area the oxygen enhancement of the SIMS yield is being studied by correlation of work function and yield for low doses of oxygen on metal surfaces. Finally we have just completed an XPS study of the role of peroxide ions in the super conducting mechanism in the YBa₂Cu₃O_{7-δ} family of high temperature superconductors.

In summary, this instrument represents an invaluable addition to our capabilities in surface science. It offers extremely high performance and reliability and has provided tremendous throughput.