

Surface Science Division

Room 308 - Session SS1+NS-MoA

Novel Surface Probes

Moderator: J.T. Yates, Jr., University of Pittsburgh

2:00pm SS1+NS-MoA1 Momentum Resolved ESDIAD, A New Technique, Probing the Low Frequency Motion of Adsorbed Molecules on Single Crystal Surfaces@footnote 1@, *J.W. Ahner, D. Mocuta, J.T. Yates, Jr.*, University of Pittsburgh

A new technique, Momentum Resolved ESDIAD (Electron Stimulated Desorption Ion Angular Distribution), provides a method for taking snapshots of the zero-point position and lateral momentum of particles adsorbed on crystalline surfaces. By employing state of the art electronics and computer technology it is possible to record for each desorbing particle the desorption direction together with the flight time. Highly momentum and directional resolved images are obtained, with time-of-flight resolution in the picosecond range and data acquisition rates up to 100 kHz. This enables us to deconvolute spatial and momentum contributions to the ESDIAD pattern and to map the low frequency motion of the adsorbed particles. These maps reflect the adsorbate interactions with the substrate and with neighboring species on the substrate. For selected examples we will present data 'movies' demonstrating how these unique maps of the dynamical behavior of adsorbed species are used in several ways to probe the lowest energy states, as well as to measure the momentum distribution when the particle gains thermal energy. One major opportunity involves dissimilar chemisorbed species which, when imaged together in momentum and real space, give new insights into the first stages of interaction between the species, leading ultimately to a chemical reaction. In addition we present lateral momentum distribution studies for an adsorbed molecule with a rotational symmetry axis showing the rotation of the molecule on its adsorption site about this axis. Such information can be used as a basis for thinking about anisotropies in lateral motion of particles on surfaces. @FootnoteText@ @footnote 1@work supported by DOE/BES.

2:20pm SS1+NS-MoA2 UV Spectroscopy of CO and Benzene on Pt(110). *N. Chen, I. Lee, R. Masel*, University of Illinois, Urbana

Recently there has been some controversy about the role of d-backbonding in the adsorption of gases on transition metals. People have suggested that the antibonding orbitals should shift, but without any direct measurements, the theory remains controversial. In this paper we use a standard HREELS spectrometer, with modified electronics to measure the equivalent of a UV spectrum for two different systems: CO on Pt(110) and benzene on Pt(110). In the CO case, the UV spectrum shows peaks at 5.41, 5.58 and 7.91 eV independent of coverage. By comparison, gas phase CO shows peaks at 6.04, 6.92, 7.58, and 7.94 eV. The large shifts are indicative of the antibonding orbitals being stabilized, as one would expect from the Blyholder model and recent calculations of Ilias et al, Surface Science 376, (1997) 279 but not with the calculations of Ohsishi and Watarri Phys Rev B 49(1994)14619. In the benzene case we observe two different spectra: a first monolayer spectrum with a broad peak center at 4.71 eV, and a multilayer spectrum with peaks at 3.78, 4.73, 6.11 and 6.82 eV. The multilayer spectrum matches the spectrum of condensed benzene, but the first monolayer spectrum is quite different. Again these results suggest that there is a substantial stabilization of the antibonding orbitals of adsorbed benzene. Together these results show that UV spectroscopy provides useful information about adsorbates on surfaces.

2:40pm SS1+NS-MoA3 Calorimetric Measurements of Metal Adsorption and Adhesion Energies on Clean, Single-Crystalline Surfaces. *C.T. Campbell, J.T. Stuckless, D.J. Bald, D.E. Starr, J.E. Musgrove*, University of Washington

The adsorption and adhesion energies of metals on solids are important in many materials and chemistry applications including oxide-supported metal catalysts, bimetallic catalysts, epitaxial thin film growth, metal-ceramic interfaces in microelectronics, metalization of polymers, composite materials and metal adsorption on minerals in soils. The heats of adsorption of metals have been measured calorimetrically for the first time on clean, single-crystalline surfaces. A pulse of metal vapor from a chopped atomic beam adsorbs onto an ultrathin single crystal's surface in ultrahigh vacuum, causing a transient temperature rise. This heat input is detected by a pyroelectric polymer ribbon, which is gently touched to the back of the crystal during calorimetry. The sticking probability is measured by detecting

the reflected fraction mass spectrometrically with a line-of-sight modification of the King and Well's method. The differential heat of adsorption is thus measured as a detailed function of coverage up through multilayer coverages. The integral heat of adsorption also provides the adhesion energy of the metal film, if the surface free energy of the clean metal surface is known. Adsorption and adhesion energies for metals (Pb or Cu) on the clean Mo(100) surface, on well-defined surface oxides of Mo(100) and W(100), and on clean and hydroxylated MgO(100) thin films will be reported. By comparing a variety of surfaces in Pb and Cu adsorption, an interesting correlation between the growth morphology of thin metal films and the initial heat of adsorption of the metal is revealed. The sticking probability also correlates with the heat of adsorption of the metal.

3:20pm SS1+NS-MoA5 Multispectral Image Classifications of Si(001) Surface Electronic Structure. *K.M. Horn, B.S. Swartzentruber, G.C. Osbourn*, Sandia National Laboratories

We have imaged the electronic structure of Si(001) surfaces by applying multispectral image analysis techniques to multi-bias STM conductance data. Atomic surfaces are first characterized by recording conductance spectra, $C(V)$, at each point in a 2D scan of the surface. The resulting 3D data set, $(x, y, C(V))$, is then converted into a series of bias-dependent conductance images. These images are analyzed to produce a single, color-coded, classed image that reflects the surface's electronic structure. The image analysis is performed by a computed grouping algorithm that identifies pixels sharing common conductance characteristics. The resulting classed images distinguish features not clearly resolved in a topographic image, and reveal stark electronic differences between topographically similar features. We first demonstrate the reliability of this classification technique on simple Si(001) features. Classed surfaces are then presented for various surface defects and Si and Ge structures that have been deposited on the Si(001) surface. These electronic structure images reveal features that are not readily visible or distinguished in a constant-current topograph. Direct comparison of the conductance spectra from these features confirms the classification result. This computer-based data reduction technique may prove useful in defect detection, validating surface models, and in understanding more complicated systems in which atomistic models are derived from a limited number of single-bias topographic images. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S.DOE under contract DE-AC04-94AL85000.

3:40pm SS1+NS-MoA6 A Simple Nulling Technique for Measuring Complex-Valued Nonlinear-Optical Susceptibilities of Interfaces. *J.R. Dennis, V. Vogel*, University of Washington

For studies of isotropic interfaces by nonlinear optics, a general technique is presented to measure the complete second-order surface susceptibility, up to an overall phase factor. The measurement and data analysis are simple and rapid, with no use of a variable reference phase, and the susceptibility is overdetermined, allowing consistency checks. The technique involves measuring the complete polarization state of the nonlinear-optical signal by nulling the signal, for several linear polarizations of the input beam. Measurements of second harmonic generation from Langmuir monolayers of the liquid crystal 4'-n-octyl-4-cyano-biphenyl (8CB) have been made with this technique, giving results which agree with previous data, and also revealing a small phase shift between some susceptibility components. This phase shift may be explained by introducing a complex dielectric constant for the monolayer at the second harmonic frequency. Data for the free surface of isotropic 8CB have also been analyzed with this technique. The technique is particularly well-suited to testing or fitting different models of Fresnel factors and local-field factors.

4:00pm SS1+NS-MoA7 Characterization of Near-Field Probes for Enhanced Raman Spectroscopy. *C.E. Jordan, L.J. Richter, R.R. Cavanagh, S.J. Stranick*, National Institute of Standards and Technology

Near-field Raman spectroscopy can potentially obtain chemical specificity with the subwavelength resolution of a near-field scanning optical microscope (NSOM). Signals from a single crystal diamond sample have been evaluated to assess the limits of this technique. Three different types of illumination mode fiber optic probes have been used in the near-field Raman experiments: an uncoated probe; a probe coated with a smooth layer of aluminum which has an aperture that is less than 100 nm in diameter; and a probe coated with a smooth layer of aluminum and then overcoated with a rough layer of silver. In order to discriminate between the enhanced contribution to the Raman signal observed in the near-field

of the probe and bulk scattering, the Raman intensity from each type of probe is measured as a function of the probe sample separation. Very little change in the Raman intensity is observed as a function of probe sample separation when an uncoated fiber optic probe is used. For an optical probe coated with a smooth layer of aluminum the Raman intensity is about a factor of seven higher when the sample is in the near-field of the probe compared to the intensity measured when the sample is farther than 100 nm away from the probe. Probes that are coated with a rough silver film show a greater enhancement in the near-field Raman intensity than is observed for probes coated only with aluminum.

4:20pm **SS1+NS-MoA8 Element Specific Real-Space Imaging Surface Crystallography**, *L. Houssiau, J.W. Rabalais*, University of Houston

Scattering and recoiling imaging spectrometry (SARIS) extends the technique of time-of-flight scattering and recoiling spectrometry (TOF-SARS) to include both spatial and time resolution of scattered and recoiled particles. SARIS uses a time-resolving, position sensitive, microchannel plate (MCP) detector, resistive anode encoder (RAE), time-to-digital converter (TDC), and a triple axis UHV goniometer to measure the velocity-resolved spatial distribution patterns of scattered and recoiled particles produced by a keV beam of pulsed ions from on a crystalline surface. The images combine the advantage of atomic scale microscopy and spatial averaging simultaneously since they are created from a macroscopic surface area but they are directly related to the short-range ($< 10 \text{ \AA}$) atomic arrangements in the surface. The non-planar scattering features in the images are not normally observed in conventional ion scattering experiments using small-area detectors. The technique is applied to carry out real space imaging of Ni(110) and oxygen chemisorbed Ni(110) with 4 keV He⁺ ions. A mapping of the entire hemisphere where the ions are reflected was made possible by collecting several images at different angles and merging them together. These maps reveal the blocking cones of surface atoms, which gives a real space image of the crystal surface. After oxygen exposure, the images are modified and reveal the O chemisorption sites. The features of these images can be accurately reproduced by classical ion trajectory simulations using the scattering and recoiling imaging code (SARIC).

4:40pm **SS1+NS-MoA9 An Axial Resonant Force Probe for Atomic Force Microscopy**, *J.A. Harley, T.W. Kenny*, Stanford University

A resonant force probe has been constructed which exhibits high force sensitivity from a stiff transducer with a stationary tip. The resonant beam is mounted vertically relative to the surface, as in shear force microscopy, but a tether has been added near the tip. The tether forces the beam to oscillate in a pinned-pinned mode while the tip remains stationary, but does not interfere with axial forces. This configuration has several advantages over current force measurement techniques. First, since the beam is perpendicular to the surface, the probe is not susceptible to force gradient instabilities. Second, the stationary tip provides high spatial resolution in the force measurements. Typical oscillating cantilevers average forces over the oscillation amplitude. Third, since the oscillations can be large, the resonant detection method is not as demanding on the secondary detector, so the force sensitivity in a piezoresistive sensor could approach that of optical lever techniques. Finally, the oscillator could potentially be encapsulated, allowing a high Q resonator in a liquid environment. The beam was constructed out of single crystal silicon, and measures $0.2 \times 3 \times 200 \mu\text{m}$, with an implanted piezoresistor to detect the oscillations. The axial spring constant is over 200N/m. Resonant frequency shifts of 30kHz/ μN are detected using a phase-lock loop circuit. In air, the oscillator is heavily damped (Q of 15) but still demonstrates 10nN force resolution in a 1kHz bandwidth. In a moderate vacuum the resonance quality improves to 1200, and 10pN force resolution is expected. The design, analysis, and theoretical limitations of these sensors will be discussed.

5:00pm **SS1+NS-MoA10 Super Transmission and Resolution Energy Analyzer and Mass-Analyzer System (STREAMS)**, *K. Siegbahn, R. Maripuu*, ESCA LASER Lab Institute for Materials Science, Sweden; *N. Kholine*, Russian Academy of Sciences, Russia; *U. Golikov*, State Technical University, Russia; *M. Larin*, Joint Stock Co. CRYOVACS, Russia

A new type of instrument for scientific and technological research is proposed. Its main peculiarity and advantage are the capability to separate charged particles in accordance with their energies and masses on high level of resolution and sensitivity. A basis of the spectrometer is electrostatic axially symmetrical field structure with matched radial and axial potential gradient. The electron optical system can function either in dispersion or time-of-flight mode of operation. The charge particles follow

the same trajectories in this field independently of the mode of operation. Electron spectroscopy or mass-spectroscopy information from the same point of the analyzed sample can be received by switching over to the appropriate potentials of the power source and the detector system. So one and the same instrument can function as a high performance electron spectrometer or a mass-spectrometer. Relative energy resolution better than 0.05% in the energy range of 20-3000 eV and mass resolution more than 5000 in the mass range 1-500 a.e.m. are easily realized for acceptance solid angle of the spectrometer equals to at least 30% out of hemisphere. The diameter of the analyzer is 200 mm, its length is 600 mm. An ultra high vacuum is ensured in the spectrometer at the level 10^{-11} mbar by oil free pumping system with highly economical cryo condensation-absorption pump cooled by liquid helium. Almost all the spectrometer is made of nonmagnetic materials and first of all from titanium. The surfaces faced into vacuum have special plating with very low absorption-desorption capacity. So ultra high vacuum is achieved without baking out the spectrometer.

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