

Wednesday Morning Poster Sessions

Surface Science

Room: Exhibit Hall B2 - Session SS-WeP

Surface Science Poster Session

SS-WeP1 Study of Polymethylmethacrylate Removers for Electron Beam Lithography through Quantitative Surface Roughness Characterization by AFM, Q. Hang, D. Hill, G.H. Bernstein, University of Notre Dame

As nanotechnology approaches molecular scales, issues of surface contamination by unremoved resists will play an important role in device fabrication. Electron beam lithography (EBL) of polymethylmethacrylate (PMMA) resist is still among the most widely used nanofabrication techniques, so it is relevant to study its residual contamination on both exposed and unexposed surfaces using a variety of resist removers. Besides preventing good metal adhesion, the resulting contaminant-induced surface roughness reduces the ability to characterize deposited molecular patterns by atomic force microscopy (AFM). We are aware of no systematic, quantitative study of surface roughness after removal of PMMA by different solvents toward determining the least amount of residual resist. The ideal stripper exhibits high affinity for both the polymer (as quantified by the lowest Flory-Huggins interaction parameter) and the substrate, the latter aimed at reducing the solid-liquid surface energy. We characterized the effectiveness of several different strippers: acetone, dichloromethane (DCM), a mixture of acetone and DCM (volume ratio 1:1), 1,2-dichloroethane (DCE), a mixture of acetone and DCE (volume ratio 1:1), and the commercial PMMA remover NanoTM Acryl Strip (MicroChem), on two different molecular weights of PMMA. Environmental AFM was used to investigate SiO₂ surfaces before and after PMMA was applied and removed by those strippers. The effects of electron beam exposure of the PMMA will also be presented. Power spectral density and root mean square surface roughness analyses showed that DCM and DEC are the best PMMA removers, and can produce the same surface roughness as the original SiO₂ surface (i.e. no contamination). Estimates of the polymer-solvent, Flory-Huggins, interaction parameters and surface-solvent interfacial energy (from contact angle measurements) satisfactorily predict the effectiveness of the solvents.

SS-WeP2 Functionalization and Patterning of C-H Containing Surfaces Using Oxalyl Chloride, G. Hussein, E.T. Sevy, M.C. Asplund, M.R. Linford, Brigham Young University

Functionalized and patterned surfaces are of great utility in a variety of areas of science and technology. One particularly useful functional group is the acid chloride, which readily reacts with amines and alcohols. Here we describe a facile gas-phase method of patterning acid chloride groups onto C-H containing surfaces. Basically, a surface that contains C-H groups, e.g., alkylated silicon or polyethylene, is introduced into a flow-through cell that has a quartz window. A mixture of N₂ carrier gas with a low concentration of oxalyl chloride then flows through the cell at room temperature. The surface is next illuminated with 365 nm light, which is known to cause dissociation of oxalyl chloride. Surface reactions then occur that introduce the -COCl group onto the surface where the surface was illuminated and nowhere else. In particular, clean silicon surfaces are first alkylated with dimethyloctadecylchlorosilane. Acid chloride groups are then introduced onto the surface as mentioned above, followed by characterization using XPS, FTIR, ellipsometry and contact angle goniometry. XPS confirms the presence of chemically shifted carbon. FTIR confirms the presence of carbonyl groups. Optical ellipsometry shows an increase in film thickness. Contact angle goniometry shows a decrease in water contact angles. The main advantage of this work is that silicon surfaces can be easily derivatized with an acid chloride in a one-step-photochemical-gas-phase reaction. We are in the process of extending this work to perform photolithographic patterning of surfaces. The resulting functional groups can be used to attach molecules, including biomolecules such as DNA and peptides.

SS-WeP3 Functional Group Effects on the Adsorption of Organics on Silicon Surfaces, S.M. Casey, L. Zhang, A.J. Carman, University of Nevada

Computational studies have been undertaken using density functional theory and cluster models of the Si(100) surface in order to compare the adsorption pathways for small organic molecules that contain different functional groups. The efficiency of nitrogen-addition via the amine group can thus be compared to the efficiency of [2+2] cycloaddition via the alkene group or oxygen-addition via the alcohol group in this manner. By variation

of the functional group, the relative reactivity of these groups with the silicon surface dimers can be surmised. Both nitrogen-addition and oxygen-addition appear to be favored adsorption pathways compared to the [2+2] cycloaddition pathway. A clear preference between the two addition channels is more difficult to determine, however. The results from these computations compare favorably with results from experimental studies of the adsorption of allyl amine, propanamine, allyl alcohol, propanol, propene, and 3-amino-1-propanol on this surface.

SS-WeP4 The Formation and Segregation of Carbon at a Ni(111) Surface, H. Nakano, J. Ogawa, H. Hirashima, J. Nakamura, University of Tsukuba, Japan

The formation and segregation of carbon at Ni surfaces are important as initial processes for synthesis of carbon nanotube by Ni catalysts. We have studied the carbon formation by the Boudouard reaction ($2\text{CO} \rightarrow \text{C} + \text{CO}_2$) and the decomposition of ethylene ($\text{C}_2\text{H}_4 \rightarrow 2\text{C} + 2\text{H}_2$) using scanning tunneling microscopy (STM), Auger electron spectroscopy (AES) and low energy electron diffraction (LEED). It was found that the behavior of the carbon deposition was very different between the Boudouard reaction and the ethylene decomposition. As for the Boudouard reaction, the carbon was formed at step edges on Ni(111), which immediately diffused into the bulk at 400 - 500 K. The carbon dissolved in the bulk then segregated to the subsurface at low temperatures of 300 - 400 K. The segregation rate was greater at lower temperatures. A single domain of carbide islands located along the step edges was clearly observed by STM, which was explained by the growth of the island initiated at the step edge. On the other hand, no such segregation was found for the carbon formation by the decomposition of ethylene. The decomposition took place on the terrace leading to an isolated unit or carbide short strings without forming islands. The difference in the decomposition behavior was ascribed to that in the site of carbon formation. That is, CO should dissociate at the step edge on Ni(111), while ethylene should decompose to carbon at the terrace site. We have also examined the effect of sulfur upon the carbon formation on Ni(111). Interestingly, no significant poisoning effect was observed upon the formation rate of carbon for both Boudouard reaction and ethylene decomposition.

SS-WeP5 Surface Characterization of Dendrimer-Encapsulated Pt Nanoparticles, B.T. Long, F. Parsons, J. Gao, C. Murphy, D.A. Chen, University of South Carolina

The ability to prepare metal nanoparticles with narrow size distributions has many potential applications in the field of heterogeneous catalysis. Platinum nanoparticles with uniform size distributions can be prepared from solution in the presence of poly(amidoamine) generation 4 starburst dendrimers. The resulting Pt nanoparticles have diameters of ~1.5 nm and are encapsulated by the dendrimers. These dendrimer-encapsulated metal nanoparticles have been spin-coated on mica and Au surfaces and imaged by tapping mode AFM and STM. At higher concentrations, the surface is covered by a single-layer dendrimer film while at lower concentrations, dendrimer aggregates are observed. Our goal is to remove the dendrimers from the Pt particles, which can then be used for catalytic studies. Thermal gravimetric analysis experiments have shown that the dendrimer begins to decompose at approximately 200°C, but decomposition is not complete at temperatures below 400°C. Ultrahigh vacuum studies will be used to further address the decomposition of the dendrimers. Specifically, the species that remain on the surface at various stages of dendrimer decomposition will be characterized by X-ray photoelectron spectroscopy. Gaseous products that desorb from the surface during decomposition will be monitored by temperature programmed desorption.

SS-WeP6 Dimethyl Methylphosphonate Reaction on Metal Nanoparticles Deposited on a TiO₂(110)-(1x2) Surface, J. Zhou, K. Varazo, D.A. Chen, University of South Carolina

The thermal decomposition of dimethyl methylphosphonate (DMMP) on supported copper and nickel nanoparticles has been investigated under UHV conditions by scanning tunneling microscopy (STM), X-ray photoelectron spectroscopy (XPS) and temperature programmed desorption (TPD). Our STM studies have shown that Cu particles grown on a TiO₂(110)-(1x2) surface at room temperature have a uniform particle size distribution. By annealing the surface to higher temperatures, the particle size can be increased while maintaining narrow size distributions. XPS studies of DMMP adsorbed on small Cu nanoparticles (35 Å diameter) indicate that molecularly adsorbed DMMP is the main species at room temperature. Heating to 350 K results in P-C and P-OCH₃ bond scission and the formation of two distinct phosphorous-containing species on the surface. Between 350 K and 700 K, the molecular DMMP continues to

decompose, producing methane and hydrogen as the major gaseous products as observed by TPD. Upon heating to 800 K, all of the carbon is removed from the surface, but 30% of the original phosphorous signal is still observed up to 1000 K. DMMP reaction on the titania surface has also been studied. The surface chemistry of DMMP on larger Cu particles (70-100 Å diameter) will be investigated in order to understand if the size of the Cu particles affects DMMP chemistry. Furthermore, DMMP decomposition on Ni particles of various sizes will also be investigated.

SS-WeP7 S K-edge NEXAFS and S 1s XPS Studies of L-cysteine on Transition Metals. *Y. Matsumura, S. Yagi, Y. Nakano*, Nagoya University, Japan, *E. Ikenaga, S.A. Sardar, J.A. Syed*, Hiroshima University, Japan, *K. Soda*, Nagoya University, Japan, *E. Hashimoto, K. Tanaka, M. Taniguchi*, Hiroshima University, Japan

We have paid attention to L-cysteine [$\text{HSCH}_2\text{CH}(\text{NH}_2)\text{COOH}$] and investigated the adsorption behavior on polycrystalline Cu, Mo and Ni substrates by S K-edge Near Edge X-ray Absorption Fine Structure (NEXAFS) and S 1s X-ray Photoelectron Spectroscopy (XPS) techniques. The sample was prepared by adsorbing L-cysteine molecules on clean metal surfaces in an aqueous solution at room temperature. Curve fitting of the S K-edge NEXAFS spectra reveals that there is a peak at around 2470 eV. The NEXAFS spectra clarify L-cysteine molecule dissociates for Ni. In the S 1s XPS spectra a sharp peak and a shoulder structure appear at around 2473 eV and 2470 eV, respectively. The peak deconvolution of the XPS spectra reveals the presence of three S chemical states, which are L-cysteine molecule, thiolate and atomic sulfur. The result of NEXAFS is consistent with that of XPS qualitatively. Keywords: NEXAFS, XPS, L-cysteine, adsorption behavior, Cu, Mo, Ni.

SS-WeP8 Structural, Electronic and Catalytic Properties of Clean and Overlayer-Covered Ir(210). *I. Ermanowski, W. Chen, M.J. Gladys, J.S. Quinton, T.E. Madey, Rutgers*, The State University of New Jersey, *M.D. Ulrich, J.E. Rowe*, North Carolina State University

We report results on the structure, morphology, electronic and catalytic properties of the Ir(210) surface - clean as well as covered with gaseous and metallic overlayers. The atomically rough Ir(210) surface is morphologically unstable: When Ir(210) is covered with more than 0.6 ML of oxygen and annealed, pyramidal facets exposing {110} and {311} surfaces develop on the initially planar surface. We have used a variety of methods to characterize this surface, including LEED, STM and high resolution soft X-ray photoelectron spectroscopy (HRSXPS) using synchrotron radiation. To prepare an oxygen-free faceted surface, we use catalytic CO oxidation at ~500 K to react the oxygen off and "freeze" the surface in its pre-prepared faceted state. HRSXPS has been employed to investigate core-level features of all the surfaces mentioned. The Ir 4f_{7/2} core levels are fitted with Doniach-Sunjić lineshapes. Surface and bulk peak identifications are supported by measurements at different photon energies (different electron escape depths) and variable photoemission angles. All of the surface components (first, second and third layer peaks) are identified with core-level shifts positioned at higher binding energies with respect to the bulk. This result is in contrast to previous reports of binding energy inversion on the Ir(100) surface. Using TPD we find evidence for structural sensitivity in a surface reaction, i.e. differences in the chemical reactivity of on the planar and faceted surfaces. Overlayers of Pd and Pt exhibit structural changes upon annealing, and HRSXPS measurements show changes in the core-level properties of the overlayers. Supported by US DOE and ARO.

SS-WeP9 Surface Stress Induced during the Formation of Alkanethiol Self-Assembled Monolayers on Gold-Coated Cantilevers. *M. Godin, O. Laroche, V. Tabard-Cossa, B. Seivewright*, McGill University, Canada, *P. Williams*, Acadia University, Canada, *B. Lennox, P. Grütter*, McGill University, Canada

Functionalized alkanethiol self-assembled monolayers (SAM) have proven to be quite versatile in producing sensing layers that are both selective and reactive to specific target molecules during chemical or biochemical sensing. By self-assembling these sensing layers onto the surface of standard atomic force microscope (AFM) cantilevers, we can design micromechanical sensors that are tailor-made to respond to very specific chemical signals. The surface stress induced at the sensing layer during chemical sensing is directly measured as a deflection of the cantilever.¹ We have developed a new method² of obtaining quantitative surface stress measurements from cantilever deflection signals that does not require the knowledge of the cantilever's Young's modulus, which is often difficult to estimate when different sensing layers are used or for commonly used SiN_x. We systematically investigated the self-assembly process of alkanethiol monolayers on gold in order to model the surface stress buildup during chemical sensing, ultimately aiming to optimize the sensor's response. In particular, we studied the kinetics of alkanethiol SAM formation by

combining real-time, in-situ, surface stress and thickness measurements. A differential micromechanical sensor was used to measure the surface stress evolution during SAM formation on gold-coated cantilevers from the vapor phase, while thickness measurements were performed by ellipsometry with sub-monolayer sensitivity. Results clearly demonstrate that analyte introduction and cell geometry play a determinant role in the structural development of the SAM, which not only affects the final stress values achieved, but also the kinetics during SAM buildup. Such considerations are crucial when optimizing sensor response through modeling or when comparing results achieved with different sensor systems.

¹R. Berger et al. Science 276, 2021 (2000)

²M. Godin et al. APL 79, 551 (2001).

SS-WeP10 Characterization of Functionalized Thiol-SA-Layers on Au using TP-SIMS and Polyatomic ToF-SIMS. *M. Schröder, J.C. Feldner, S. Sohn, H.F. Arlinghaus*, Westfälische Wilhelms-Universität, Germany

Functionalized monolayer substrates provide the basis for biosensor chips and other biorecognition systems. Several factors, such as type of SA layer building molecules, their functional headgroups and the different parameters concerning the immobilization process, influence the physical, chemical and biochemical properties of these substrate surfaces. We investigated different thiol classes, such as alkanethiols, C_nH_{2n+2}S, alkanethiol derivatives and aromatic thiols, with different functional headgroups, e.g. NH₂, COOH or OH, immobilized onto Au-substrates. Thiols with functional headgroups could be linked to larger biomolecules such as DNA and proteins in order to design biosensor-chips for diagnostics. To link NH₂-terminated molecules to carboxylated surfaces, we used 1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide (EDC) to catalyze the formation of an amide bond. Another possibility for linking such molecules to a surface is to bind an NH₂-terminated thiol to the gold and use di-(n-succinimidyl)-carbonate (DSC) as a crosslinking mediator. The different reaction steps were monitored using time-of-flight secondary ion mass spectrometry (ToF-SIMS). We carried out ToF-SIMS measurements on SA-layers of different alkanethiols to investigate the time, concentration, and chain length dependence of the layer formation. Also, the influence of primary ion mass and of primary ion constituents on the secondary ion yield was investigated. With temperature-programmed SIMS (TP-SIMS), we were able to compare the desorption temperatures of different thiols, e.g. alkanethiols and their derivatives, and to investigate the influence of functional headgroups on the surface binding energy. From the obtained data it can be concluded that ToF-SIMS and TP-SIMS are useful techniques for investigating physical properties of SA-layers and for controlling crosslinking reactions to optimize e.g. binding conditions of DNA or PNA.

SS-WeP11 Low Temperature STM Investigation of Halosubstituted Biphenyls on Copper Surfaces. *L. Bartels, B.V. Rao, Q. Zhang*, University of California at Riverside

4,4'-Di-bromo-biphenyl was adsorbed on a Cu(111) single crystal surface in ultra-high vacuum at 25K. The preferred adsorption site and the mobility of the species were investigated. 4,4'-Di-bromo-biphenyl was found to adsorb primarily at step edges on the surface with the long axis of the molecule aligned parallel to the step edge. High-resolution images reveal the individual, substituted benzene rings. Their separation is in good agreement with the value expected from the literature. In contrast, adsorption at nitrogen temperatures does not permit STM investigation of the unreacted species on the surface. STM images are poor and high fluctuations in the tunneling current point to a mobility of the adsorbate beyond the time-scale of the feedback loop. Retracting the tip several nanometers from the sample, the tunneling current does not vanish completely in a reliable fashion. This points to the spontaneous polymerization of 4,4'-di-bromo-biphenyl to long tethers on the surface.

SS-WeP12 Adsorption Behavior of L-cysteine on Transition Metals (Ni, Cu) in Aqueous or Methanol Solution Studied by S K-edge NEXAFS and XPS. *S. Yagi, Y. Matsumura, Y. Nakano*, Nagoya University, Japan, *E. Ikenaga, S.A. Sardar, J.A. Syed*, Hiroshima University, Japan, *K. Soda*, Nagoya University, Japan, *E. Hashimoto, K. Tanaka, M. Taniguchi*, Hiroshima University, Japan

Adsorption behavior of a sulfur-containing amino acid molecule on transition metal surface has been interested in a bio-catalytic and surface reaction fields. In this study, we have studied the adsorption behavior of the L-cysteine amino acid [$\text{HSCH}_2\text{CH}(\text{NH}_2)\text{COOH}$] on polycrystalline Ni and Cu surfaces by sulfur K-edge Near Edge X-ray Absorption Fine Structure (NEXAFS) and sulfur 1s X-ray Photoelectron Spectroscopy (XPS) techniques. The sample was prepared by scratching the substrate in the aqueous or methanol solution of the L-cysteine at room temperature. In the NEXAFS spectra, a significant difference of the first peak (1s-to-σ*(S-C)) shape was obtained between two kinds of Ni samples, which were prepared

in the aqueous and methanol solutions. Curve-fitting results of the NEXAFS spectra indicates that the L-cysteine on Ni in the aqueous solution dissociates. On the other hand, the L-cysteine in the methanol solution on Ni does not dissociate. Keywords: NEXAFS, XPS, L-cysteine, adsorption behavior, Ni, Cu, aqueous solution, methanol solution.

SS-WeP13 LEED and HREELS Study of Methane Physisorbed on Ag(111), M. Sakurai, T. Nanba, Kobe University, Japan

The structure of methane monolayer physisorbed on a Ag(111) surface was observed by LEED. The picture was recorded with suppressed electron current using cooled CCD camera in order to minimize the electron stimulated desorption of adsorbed methane. We have observed that the anomalous feature in the IV curve of very low energy electron scattered from this system at 40K depends on the ambient pressure of methane, with which the physisorbed layer is in equilibrium. By the present measurement, the LEED pattern indicated that the lattice of methane molecules forms hexagonal structure with rotationally commensurate direction with the substrate lattice. The LEED spots were somewhat obscure and had broadened nature in azimuthal direction at the equilibrium conditions; however, they became sharp ones when the substrate was cooled to 25K. We have also performed vibrational spectroscopy of adsorbed methane molecules by HREELS and anomaly in the vibrational excitation cross section (i.e. inelastic IV curve) has been observed. The relation between the IV curve and structure will be discussed.

SS-WeP14 Adsorption and Reaction of NO, CO and O₂ on Cu(100) at Low Temperatures Studied by Infrared Reflection Absorption Spectroscopy, C.-W. Yi, Texas A&M University, C.M. Kim, Kyungpook National University, Korea, D.W. Goodman, Texas A&M University

The adsorption and reaction of NO, CO, and O₂ on Cu (100) have been investigated in the temperature range 21 - 200 K using infrared reflection absorption spectroscopy (IRAS). NO forms dimers within the monolayer and multilayer regime at 21 K. The dimer, in turn, converts to N₂O above 60 K. A strong lateral interaction between coadsorbed NO and CO at 21 K results in the NO and CO molecules tilting from the surface normal by approximately 45 degree. CO interacts strongly with chemisorbed dioxygen on Cu(100) within the temperature range 50 - 100 K resulting in the vibrational frequency of CO being blue-shifted by 25 cm⁻¹. These studies provide new insights into the molecular interactions that are precursors to reactions between CO and NO/O₂.

SS-WeP15 Artificial Control of Reaction Selectivity in Methanol Oxidation and Ethanol Decomposition on Pt, Pd and Ag Catalysts by Dynamic Lattice Displacement of Thickness Extension Mode Resonance Oscillation, Y. Yukawa, N. Saito, H. Nishiyama, Y. Inoue, Nagaoka University of Technology, Japan

Selectivity is a very important factor in heterogeneous catalysis, and it is highly desirable to precisely control it in an artificial manner. We have employed the thickness extension mode resonance oscillation (TERO) of acoustic waves. In the present work, the TERO effects on the selectivity of methanol oxidation and ethanol decomposition on Pt, Ag and Pd catalysts were studied. To change the resonance frequencies of the TERO, four z-cut LiNbO₃ single crystals with different thickness of 0.3, 0.5, 1.0 and 2.0 mm were used: the first resonance frequencies were 11.2 MHz for 0.3 mm thick crystal, 7.3 MHz for 0.5 mm, 3.6 MHz for 1.0 mm and 1.8 MHz for 2.0 mm. A Pt, Ag or Pd film was deposited at 100 nm on the crystals. For methanol oxidation on Pt, the major products were CO₂, HCHO and HCOOCH₃. The TERO of 3.6 MHz accelerated the production of CO₂ and HCHO, but little HCOOCH₃. With increasing rf power, the selectivity for HCHO production increased from 9% without TERO, reached a maximum level of 18% at 0.5 W, and decreased. For the same reaction on Pd, the selectivity increased monotonously with increasing rf power. For ethanol decomposition on a Ag catalyst, the TERO accelerated the ethylene production without affecting acetaldehyde production. With increasing resonance frequencies, the selectivity for ethylene production increased, passed through a maximum at 7.3 MHz and decreased. Laser Doppler measurements showed that the magnitudes of standing waves (lattice displacement) caused by the TERO attenuated monotonously with frequency, whereas the number of the standing waves per area increased remarkably. The dependence of selectivity on the resonance frequency is discussed based on the contribution of the magnitudes and density of lattice displacement.

SS-WeP16 Oxygen Adsorption on Cu-9%Al(111) Studied by LEED and AES, M. Yoshitake, S. Bera, Y. Yamauchi, W. Song, National Institute for Materials Science, Japan

Cu-based alloys have been used for electric cables for long time. In the field of microelectronics, Al had been used for electrical wiring. However, it became clear that electro-migration occurs in Al that causes breaking of

wires in minute wirings. Due to this problem, Cu wiring is used in most-advanced microprocessors. Cu metal is more corrosive than Al and Cu-based alloys with a small amount of Al is expected to solve problems both on electro-migration and corrosion. The initial stage of corrosion is oxygen adsorption. We studied surface segregation of Al on Cu-9%Al(111) and oxygen adsorption on the surface with/without Al segregation in UHV by LEED and AES. It was found that Al segregates on the surface to form ($\sqrt{3} \times \sqrt{3}$) structure and the structure vanishes above 320°C to give (1x1) structure while Al still segregates. The specimen was exposed to oxygen at different temperatures. The amount of oxygen uptake was not structure dependent but temperature dependent. Below 320°C, only a small amount of oxygen adsorbed. Between 325 and 600°C, oxygen adsorbed surface showed amorphous LEED pattern. The specimen was annealed at 800°C after oxygen exposure. When the specimen was exposed oxygen below 600°C, the oxygen Auger intensity decreased significantly by annealing and the annealed surface showed ($\sqrt{3} \times \sqrt{3}$) structure at room temperature. When the specimen was exposed to oxygen at 600°C, diffused spots developed newly in LEED pattern but the pattern disappeared after 800°C annealing while oxygen Auger intensity stayed almost constant. Exposing the specimen to oxygen at 725°C resulted in clear spots in LEED pattern, which were attributed to ($7/\sqrt{3} \times 7/\sqrt{3}$) structure.

SS-WeP17 The Diffusion of Single CO Molecules and Dimers on Pd(111), T. Mitsui, Lawrence Berkeley National Laboratory, M.K. Rose, E. Fomin, Lawrence Berkeley National Laboratory and University of California, Berkeley, D.F. Ogletree, M. Salmeron, Lawrence Berkeley National Laboratory

The diffusion of individual CO molecules on Pd(111) has been studied by scanning tunneling microscopy in the temperature range 40-50 K. By following the random walk motion as a function of temperature an activation energy barrier of 0.116 ± 0.005 eV and a pre-exponential factor of $10^{-3.5}$ cm²-sec⁻¹ were determined. Attractive interactions between CO molecules give rise to the formation of dimers that diffuse as a unit. Diffusion rates for dimers and energy parameters for the pair potential between CO molecules were also obtained. Coadsorbed hydrogen more than doubles the diffusion barrier of CO monomers.

SS-WeP18 Pd adatom-Adatom Interactions on the W (211) Surfaces, T.-Y. Fu, Y.-J. Hwang, National Taiwan Normal University, ROC, T.T. Tsong, Academia Sinica, Taiwan

Adatom-adatom interactions play an important role in controlling the formation of surface layers, thin film growth and catalysis. Direct observations in the field ion microscope make it possible to determine the probability P(R) of finding two atoms at a separation R at equilibrium on a surface at temperature T. The free energy of interaction F(R) can be obtained from the relation: P(R) is proportional to $\exp[-F(R)/kT]$. On W (211) planes, the interactions of two Pd atoms are studied in following cases: two Pd atoms in the same channel, in two nearest neighboring channels, in two next nearest neighboring channels, and so on. When two Pd atoms are in the same channel, they combine easily to form a dimer of bond length 2.74 Å and the dimer can diffuse along the channel. In the other cases, the oscillatory behaviors of interactions along the channel direction are observed. The more is the number of the separate channels, the smaller is the interaction energy maximum. The energy maximum in the same channel, two nearest neighboring channels, and two next nearest neighboring channels are 37.5 meV, 23.0 meV, and 9.0 meV, respectively.

SS-WeP19 Temperature Dependence of Self-Assembled Pb Domains on Cu(111), R. van Gastel, N.C. Bartelt, G.L. Kellogg, Sandia National Laboratories

Pb deposited on Cu(111) organizes into self-assembled domain patterns.¹ It has been hypothesized that stress differences between the Pb/Cu surface alloy-phase and the Pb overlayer phase stabilizes the domains.¹ The domain patterns can be ordered arrays of dots ("droplet" or "inverted droplet" phase) or alternating rows ("striped" phase). Their structure evolves as a function of Pb coverage. The feature size, area fraction and degree of long range order of the domain patterns also show a strong dependence on temperature. To understand this behavior, we have investigated the temperature dependence of the domain boundary energy by studying the thermal fluctuations of striped patterns. We observed an increase in thermal fluctuations with increasing T. Our measurements show that the boundary energy decreases by a factor three in the temperature range 570 K to 640 K. Since theory predicts that the stripe width decreases with decreasing boundary energy, this observation can explain the observed T dependence of the stripe pattern. The absolute value of the boundary energy provides an estimate of the surface stress - we discuss this number in the context of first principles calculations. We have also measured the change in area fraction with temperature of the surface alloy and overlayer phases. We observe a decrease in area fraction of the surface alloy phase with increasing T. We

attribute this change in area fraction mainly to a change in the Pb-density of the alloy phase. This work was performed at Sandia National Laboratories, a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S. Department of Energy under Contract DE-AC04-94AL85000.

¹R. Plass, J. Last, N. C. Bartelt and G. L. Kellogg, *Nature* 412, 875 (2001).

SS-WeP20 Using Ti Interlayers as an Interface Stabilizer to Promote Epitaxial Growth of Fe on Al(100) Surfaces¹, C.V. Ramana, R.J. Smith, Montana State University, B.S. Choi, Jeonju University, Korea

An approach is described to promote epitaxial growth of thin metal films on single-crystal metal substrates by stabilizing the interface with an extremely thin metallic interlayer. A single atomic layer of a metal is deposited at the interface prior to the growth of the metal film of interest to produce an abrupt, epitaxial interface in a system that is otherwise characterized by interdiffusion or chemical roughness. The stabilized interface prevents interdiffusion and serves as a template for ordered film growth. Using high-energy He⁺ backscattering and channeling techniques along with low-energy electron diffraction and low-energy He⁺ scattering, it is demonstrated that an atomically thin layer of Ti metal deposited at the Fe-Al interface, a system well known for considerable intermixing at room temperature, prevents interdiffusion and promotes the epitaxial growth of Fe films on the Al(100) surface. The resulting structure is observed to be stable for temperatures up to about 200 C.

¹Work Supported by NSF Grant DMR-0077534.

SS-WeP21 Surface Structural Studies of Epitaxial Ag/Ni/Pt(111) and Ni/Ag/Pt(111) Ultrathin Films, C.W. Su, Y.W. Chu, H.Y. Ho, C.S. Shern, National Taiwan Normal University

The introduction of Ag films on the epitaxial x ML Ni/Pt(111) metal surface, x=1-5, as the capping layer Ag/Ni/Pt(111) or the buffer sandwich Ni/Ag/Pt(111) was investigated by means of Auger electron spectroscopy (AES) and ultraviolet photoelectron spectroscopy (UPS) in UHV. The evidences from monitoring the initial annealing temperature of the Ag/Ni/Pt(111) corresponded to the signals began to decrease in AES show that 1 ML overlayer of Ag retards the bulk diffusion of the Ni atoms toward the Pt substrate. Compared with our previous study,¹ the initial temperatures of the system capped with Ag are higher than those capped without it. As for the behavior of atomic exchange, from Ni/Ag to Ag/Ni, has been observed on the annealed Ni/Ag/Pt(111) surface. Ag atoms migrate to the top at about 400 K and then reach an exchanging equilibrium at a high temperature. The thicker the Ni films, the higher the equilibrium temperature. The variations of electronic valence band by UPS though the annealing processes also attracted our attention. In addition to the variations of the Fermi edge intensities and of the Ag-Ni hybrid d-band peaks during annealing, an extra peak in UPS appears gradually with the binding energy at about 11 eV on either the spectra of the Ag/Ni/Pt(111) or the Ni/Ag/Pt(111) even the temperature up to 825 K. The heating brought about the changes was contributed to additional chemical adsorption emerged from the surface. The most interesting case was the density of state (DOS) unexpectedly affected by an extra positive or negative bias on the sample. The shift of the UP spectra and the variation of the DOS were also remarkable. Finally, the possible explanations of the phenomena are to be discussed.

¹C. W. Su, H. Y. Ho, C. S. Shern, and R. H. Chen, *Surface Science* 499, 103 (2002).

SS-WeP22 The Importance of Pb-Vacancy Attraction on Diffusion in the Pb/Cu(001) Surface Alloy, M.L. Anderson, B.S. Swartzentruber, Sandia National Laboratories

Vacancy-mediated diffusion has recently been shown to be a dominant mass transport mechanism on the Cu(001) surface.¹ Not only are vacancies responsible for self-diffusion of surface-layer Cu atoms, but also for diffusion of impurity atoms such as Pd, In, and Pb, embedded in the surface layer. We use atom-tracking STM to measure the diffusion of embedded Pb atoms directly as a function of temperature to extract the diffusion energies. We find a Pb-vacancy attraction that dramatically affects the diffusion statistics. A diffusion event consists of a "burst" of a series of rapid exchanges between an embedded Pb atom and a surface vacancy. The population density of surface vacancies and their migration energy in the Cu surface layer govern the burst-to-burst time interval statistics. Because a single diffusion event entails a number of Pb-vacancy exchanges, the net displacement of Pb is often much more than a single lattice constant. The details of this displacement distribution are measurably affected by the Pb-vacancy interaction. The long length scale of the displacement distribution due to Pb-vacancy attraction is in stark contrast to Pd, which has a repulsive interaction. The energetics extracted from the measurements in comparison with first-principles calculations lead to a better understanding of the driving forces responsible for surface alloy formation and kinetics. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed

Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

¹ J. B. Hammon, et al., *Phys. Rev. Lett.* 79, 2506 (1997); R. van Gastel, et al., *Phys. Rev. Lett.* 86, 1562 (2001); M. L. Grant, et al., *Phys. Rev. Lett.* 86, 4588 (2001).

SS-WeP23 Bias Voltage Dependence of Apparent Local Barrier Height at Constant Tip-Sample Separation, S. Yagyu, M. Yoshitake, National Institute for Materials Science, Japan

The bias voltage dependence of apparent local barrier height (LBH) corresponding to "work function" at nanometer-scale has been measured by STM on Au (111) surface with Au tip at constant tip-sample separation (CS). The obtained main result is that in the bias voltage range of I-V curve showing nearly straight (ohmic), the LBH does not depend on the bias voltage and beyond this range the LBH decrease with increasing the bias voltage. The bias voltage dependence has been reported at relatively high bias voltage above 1.5 V. However, these reports had been measured at constant current (CC) conditions generally used in STM / LBH measurements. To measure its dependence under CC, the bias voltage is varied, which causes change in separations. In this situation, the measured results contain both effects (bias voltage and separation). Therefore, to measure the effect of bias voltage on LBH truly, the separation effects have to be eliminated. We have measured bias voltage dependence of LBH at CS using the current on I-V curve, and have compared results at CS with CC. At CS measurements, in the ohmic range (below 0.1V), LBH does not depend on bias voltage within the experimental error. Beyond this range, that is off ohmic, LBH decrease with increasing the bias voltage. On the other hand, at CC measurements, the range of I-V curve showing the ohmic and a slightly off the ohmic, the LBH increase with increasing the bias voltage, because of the increasing in separation. Beyond this range, LBH decreases with increasing the bias voltage as CS case, indicating that separation does not affect any more.

SS-WeP24 Electronic Friction at the High T_c Superconductor-Adsorbate Interface, X.F. Hu, P. Guptasarma, University of Wisconsin-Milwaukee, R.W.C. Hansen, Synchrotron Radiation Center, C.J. Hirschmugl, University of Wisconsin-Milwaukee

Electronic friction at a substrate-adsorbate interface can be examined using grazing incidence Infrared Reflection Absorption Spectroscopy (IRAS). Adsorbed molecules provide extra scattering sites for substrate electrons in metallic substrates with electron mean free paths longer than the skin depth. This electron energy loss is observed as a broad absorption feature in the change in reflectivity with and without adsorbates. In superconductors, substrate electrons have varying mean free paths above and below the superconducting transition temperature. We will present IRAS results for CO adsorbed on surfaces parallel to and perpendicular to the ab-plane in near-optimal doped (T_c ~ 95K) large single crystals of Bi₂Sr₂CaCu₂O_{8-y} (BSCCO). The single crystal was grown by the float-zone technique in an infrared image furnace. Measurements were taken both above and below the superconducting transition temperature at the Synchrotron Radiation Center.

SS-WeP25 An UHV Analytical Tribometer to Evidence Tribochemistry, T. Le-Mogne, J.M. Martin, Ecole Centrale de Lyon, France

Increasing demands in the mechanisms of lubricant additives and thin films have resulted in the development of new tribometers coupled with surface analysis techniques. In this paper the so-called AES/XPS analytical tribometer is presented. In-situ surface analyses inside and outside wear scars without air exposure are possible thanks to a tribometer directly installed in the UHV analytical chamber. Two experimental approaches are developed to study the tribochemical reactions of lubricant additives. The first one consists in UHV friction experiments on tribological films previously formed. Thanks to AES and XPS mapping, the good friction reducing properties of molybdenum dithiophosphate (MoDDP) are explained by the formation of MoS₂ in the sliding contact. The second one studies the reactivity of fresh surfaces with gaseous compounds which are chemically representative of lubricant additives. Interesting results have been obtained by comparison between 1-hexene and n-hexane on steel at different partial pressures.

SS-WeP26 Coupling of XPS and XANES to Characterize Sulfides in Films Formed from Lubricant Additives, M.I. De Barros, J. Bouchet, T. Le-Mogne, J.M. Martin, Ecole Centrale de Lyon, France

The nature and properties of each species formed in both the antiwear action of zinc dithiophosphate (Zndtp) and in the friction reduction of molybdenum dithiocarbamate (Modtc) are investigated. Special attention was carried out to the advantage of coupling two surface analytical techniques on the Zndtp, Modtc and Zndtp/Modtc combination tribofilms: XPS and XANES spectroscopies. XANES is a bulk analysis of the film

compared to XPS which is much more surface sensitive. A Cameron-Plint friction machine was used to generate large tribofilm areas in mild/severe tribological conditions. First XANES spectroscopy at the P K-edge, Mo K-edge and S K-edge was carried out in order to investigate any differentiation between phosphate and sulfides. Afterwards, an XPS analysis was performed in the same location of the tribofilm. Special attention has been paid to the deconvolution of Fe2p, Zn2p, S2p, Mo3d and O1s photopeaks and Auger ZnLMM lines. Analytical results are discussed and compared with theoretical predictions from the Chemical Hardness model for Zndtp, Modtc and Zndtp/Modtc tribofilm formation.

SS-WeP27 Scanning Tunneling Microscopy Studies of a Cu₃Pt(111) Surface and Its Dependence on Preparation Conditions, K.T. Rim, T. Müller, G.W. Flynn, Columbia University, A.V. Teplyakov, University of Delaware

A single crystal Cu₃Pt (111) sample was studied using Scanning Tunneling Microscopy (STM) and Low Energy Electron Diffraction (LEED). The preparation conditions in ultrahigh vacuum have been analyzed by varying annealing temperatures following Ar⁺ sputtering on the alloy surface. The STM study reveals that, despite a sharp 1x1 LEED pattern at three different annealing temperatures (825, 845, and 855K), the alloy surface changes from very corrugated to atomically flat over a relatively narrow temperature range. When the sample is annealed at 845K following Ar⁺ sputtering, 10-20nm wide terraces are formed and an ordered 2x2 phase is observed. The atomically resolved STM image of the ordered 2x2 surface shows that bright protrusions correspond to Pt atoms surrounded by Cu atoms that appear dark. This ordered 2x2 phase covers approximately 10 - 15% of the surface, and a chemically mixed, disordered 1x1 phase covers approximately 85 - 90% of the surface. The 2x2 ordered phase also exhibits some defects and antiphase boundaries. Along the step edges of terraces, Pt atoms alternate with Cu atoms, and very few Pt-Pt pairs are observed on the terraces. The chemical reactivity of these two distinct surface phases with respect to HCl and 1-hexene will also be described.

SS-WeP29 Studies of Ballistic Electron Emission Microscopy on p-n Junction Structures, E.R. Heller, J.P. Pelz, C. Tivarus, The Ohio State University

One class of experiments using Ballistic Electron Emission Microscopy (BEEM) makes nm-scale studies of hot-electron transport through a deposited film¹ or 'stack' of metal films (e. g. magnetic multilayers)² and/or insulating layers (e. g. magnetic tunnel junctions).³ For this class of experiments, a high quality (non-leaky) metal/semiconductor Schottky has been required to block thermal electrons. However, desired film deposition techniques (e.g. sputtering) and non-ideal surface preparation often produce very leaky and non-uniform Schottky barriers, making BEEM studies complicated or impossible. We will discuss an alternate approach for such studies which uses a buried pn junction in the semiconductor as the thermal electron blocking layer. We find this permits much higher signal-to-noise for sample structures which are incompatible with good Schottky barriers. By putting the blocking layer inside the bulk of the semiconductor, a leaky or non-ideal metal/semiconductor interface becomes largely irrelevant. A well-designed pn junction still can have low hot-electron attenuation⁴ and extremely good rejection of thermal electrons. We will also discuss related on-going approaches which can permit BEEM studies at reduced temperatures with sensitivity down to the aA (10⁻¹⁸ amp) level. This work was supported by NSF Grant No. DMR-0076362.

¹Lu R. P. et al., J Appl. Phys. 87, 5164 (2000).

²W. H. Rippard and R. A. Buhrman, Appl. Phys. Lett. 75, 1001 (1999).

³W. H. Rippard, A. C. Pirella, and R. A. Buhrman, Appl. Phys. Lett. 78, 1601 (2001).

⁴L. D. Bell et al., Phys. Rev. B 48, 5712 (1993).

SS-WeP30 Monolayer Functionalization of Atomic Force Microscopy Tips for Chemical Sensitive Imaging, L.W. Zilch, A.T. Woolley, M.R. Linford, Brigham Young University

While atomic force microscopy (AFM) provides nanoscale information of surface topology, it is often incapable of detecting chemical differences between regions on surfaces. Thus, the usefulness of this technique increases when the AFM tip is chemically modified so as to perform chemical force microscopy. It is then possible to relate tip forces to both surface topology and chemical interactions between the tip and the surface. We are currently working on modifying silicon AFM tips with a variety of organic monolayers using gas and liquid phase silanes (on silicon oxide) or using 1-alkenes (on hydrogen-terminated silicon). Monolayers are advantageous because they do not substantially degrade the resolution of the technique by increasing the radius of curvature of the tip. After coating the tip with a monolayer we then intend to perform a variety of gas phase reactions in the presence of these coated tips to introduce useful functional groups onto them. Ultimately we intend to create an all gas-phase process for functionalizing AFM tips. We will use the resulting AFM tips to study

surfaces that have regions of different hydrophobicities and chemical functionalities. Two suggested substrates for testing these tips are silicon and gold surfaces microcontact printed with silanes and thiols, respectively.

SS-WeP31 Contrast Mechanisms of Secondary Electron Images from Electron, Ion and X-Ray Excitation, Y. Sakai, T. Tazawa, Y. Iijima, JEOL Ltd., Japan, C. Nielsen, JEOL Inc., T. Ichinokawa, Waseda University, Japan

The atomic number (Z₂) effect of the secondary electron yields for metals was compared by using secondary electron microscope (SEM), secondary ion microscope (SIM) imaging and X-ray photoelectron spectroscopy (XPS) imaging. The atomic number (Z₂) effect of the secondary electron yields for metals has recently experimented in secondary electron microscope (SEM) and secondary ion microscope (SIM) imaging (1). The Z₂ dependence of the secondary electron yields is opposite to each other and the brightness of the secondary electron image by electron bombardment increases with increasing Z₂, while the brightness of the secondary ion image by Ga⁺ or Ar⁺ ion bombardments decreases with increasing Z₂. In this experiment, the Z₂ dependence of the secondary electron yields in electron and X-Ray excitation are the same and opposite to that in ion excitation. The brightness of the secondary electron images by X-Ray bombardment increase with increasing Z₂. These phenomena have been found by this experiment and compared with a difference of emission mechanisms for electron, ion and X-Ray bombardment for metal. The experiment was carried out by using an X-ray photoelectron spectroscopy (JPS-9010MC). A photoelectron analysis for XPS has become a well-recognized in surface chemical analysis. The secondary electron peak at about 1 to 20eV was not used for the chemical analysis. But this peak will use for imaging observation, because the intensity of its is higher than the photoelectron peaks. A scanning secondary electron images by X-Ray excitation were measured by a stage scanning imaging method. The analyzing area was selected with the 200um aperture and scanned with a motor controlled stage. A specimen is the plate of Al, Cu, Ag and Au. The secondary electron energy from each metal as excited Mg-Kα(1253.6eV) and monochrometored Al-Kα(1486.6eV) was measured with a hemi-spherical electron analyzer at a constant retarding method (0.6%). For metal, the Z₂ dependence of the secondary electron yields in X-Ray excitation increases with increasing Z₂.

SS-WeP32 How Old is Surface Science?, E. Paparazzo, Consiglio Nazionale delle Ricerche, Italy

Some philosophical and literary testimonies from the Classical World on solid surfaces are reviewed, and their implications discussed in the light of Surface Science. While Plato (V-IV century BC) thought the surface to be a real, material entity, Aristotle (IV century BC) considered it but a merely conceptual abstraction having no existence of its own. Subsequently, the Stoic philosopher Posidonius (I century BC) regarded plane surface as existing both in thought and in reality, although the implications involved in his view were remarkably different from those of Plato's. While Aristotle's view enjoyed a virtually unanimous consensus in antiquity, some passages in book 34 of the Elder Pliny's *Naturalis Historia* (I century AD) make a notable exception, as they refer to the surface of metal objects as to a region whose nature and behavior are quite different from those of the bulk: in other words, this bears witness to what appears to be an "ante litteram" example of Surface Science. Indeed, Pliny records the peculiar and dramatic ways in which these surfaces are affected by physico-chemical agents from the environment, and he also describes the remedies which best mitigate the effects of such phenomena. Further analogies between "old" and "modern" Surface Science are considered: purely-geometrical Plato's surface is found to compare favorably to a single-crystal surface, whereas the "corporeal" surface involved in Posidonius' view is best likened to an air-oxidized, or otherwise ambient-modified surface. I shall finally argue that the long-standing dominance of Aristotle's view from antiquity onwards has greatly delayed theoretical speculation into solid surfaces.

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