Applied Surface Science
Room: C-106 - Session AS-ThM

Practical Surface Science I
Moderator: R. Hull, University of Virginia

9:00am **AS-ThM3 Arrays of Chemomechanically Patterned Patches of Homogeneous and Mixed Monolayers of 1-Alkenes and Alcohols on Single Silicon Surfaces**, ***T.L. Niederhauser***, *Y.-Y. Lua, G. Jiang, S.D. Davis, R. Matheson,* Brigham Young University, *D.A. Hess, I.A. Mowat,* Charles Evans & Associates, *M.R. Linford*, Brigham Young University

We have demonstrated a facile, chemomechanical method of simultaneously functionalizing and patterning silicon with single organic monolayers by scribing it while it is wet with 1-alkenes,[1] 1-alkynes,[1] and 1-haloalkanes.[2] This method can be used to create different monolayer coatings in distinct and precisely controlled regions on an individual surface. Like microcontact printing, this technique allows multiple, patterned, surface features to be prepared with ease. To create these arrays a Si surface is 1) wet with a reactive compound, 2) scribed in a specific region with a computer-controlled diamond-tipped rod, 3) rinsed with a solvent, and 4) dried. Without moving the Si surface from its original position, this process is then repeated to create monolayer coatings in regions distinct from the first. With this technique we have prepared arrays of functionalized, scribed regions on single Si surfaces of a) the homologous series of 1-alkenes from 1-pentene to 1-octadecene, b) a series of alcohols and, c) a series of mixed monolayers on scribed Si from two 1-alkenes or from a 1-alkene and an alcohol. The preparations were performed in the air without any special treatment or degassing of chemicals. The ability to create surfaces with different monolayer coatings in precisely controlled regions should prove technologically valuable, for example, in creating functionalized surfaces to perform multiple bioassays.

[1] T. L. Niederhauser, G. Jiang, Y.-Y. Lua, M. J. Dorff, A. T. Woolley, M. C. Asplund, D. A. Berges, M. R. Linford, Langmuir 2001, 19, 5889-5900.

[2] T. L. Niederhauser, Y.-Y. Lua, Y. Sun, G. Jiang, G. S. Strossman, P. Pianetta, M. R. Linford, Chem.Mater. 2002, 14, 27-29. .

9:20am **AS-ThM4 Surface Analytical Characterization of SiO_2 Gradient Membrane Coatings on Gas Sensor Microarrays**, ***M. Bruns***, Forschungszentrum Karlsruhe GmbH, Germany, *H. Baumann,* Universität Frankfurt/Main, Germany, *M. Frietsch, E. Nold, V. Trouillet,* Forschungszentrum Karlsruhe GmbH, Germany, *R. White, A. Wright,* Thermo V.G. Scientific, England

The growing demand for inexpensive, space-saving and intelligent gas sensor systems led to the development of a gas sensor microarray at the Forschungszentrum Karlsruhe. The microarray currently comprises 38 sensor elements on an area of 4x8mm^2 and is based on an 150 nm thick SnO_2 layer, the electrical conductivity of which is highly sensitive to the composition of the ambient atmosphere. The basic structure of the microarray is manufactured by R.F. magnetron sputtering, applying a shadow masking technique. Parallel platinum strip electrodes for the conductivity measurement are sputtered on top of the metal oxide, thus subdividing the latter into the initially identical sensor elements. In order to modify the gas response of individual sensor segments, a gas-permeable SiO_2 membrane with a thickness variation of approximately 2 to 50 nm was deposited across microarray using ion beam assisted deposition.[1] Auger electron spectroscopy already has been proven to be a powerful tool in quality control of the fabrication of gas sensor microarrays[2] and, therefore, was used for rapid evaluation of the geometrical integrity of the electrode pattern. The chemical state determination of the layer constituents was carried out by X-ray photoelectron spectroscopy. Parallel ARXPS (angle resolved XPS) data collected with the Thermo VG Theta Probe can provide thickness information from the SiO_2 membrane in a non-destructive manner using a well-controlled analysis area (X-ray spot size 50 µm). In addition, ellipsometry, nuclear resonant reaction analysis, and non-Rutherford backscattering spectrometry were used to achieve a comprehensive characterization.

[1] M. Frietsch, L.T. Dimitrakopoulos T. Schneider, J. Goschnick, Surf. Coat. Technol., 120-121 (1999) 265

[2] R. Schlesinger, M. Bruns, Thin Solid Films, 366 (2000) 265.

9:40am **AS-ThM5 Surface Properties of Chemically Processed Niobium**, ***E.S. Gillman***, *A.M. Valente*, Jefferson Lab

Many high-performance accelerators, such as CEBAF at Jefferson Lab, rely on superconducting radio-frequency (SRF) technology. SRF technology at Jefferson Lab is based on the use of superconducting niobium accelerating cavities. The accelerating cavities are contained in cryomodules where liquid helium maintains the temperature at 2 K. To realize the highest energy and lowest heat loss from these accelerators means improving the performance of the accelerating cavities. We have studied the surface of chemically processed, heat-treated niobium used in these cavities with angle-resolved x-ray photoelectron spectroscopy (ARXPS) to ascertain the effect of process conditions on cavity performance. Our data indicates correlations between the passivation oxide layer thickness and stochiometry with processing conditions and that processing conditions can be directly correlated with cavity performance. This work was supported by U.S. DOE Contract No. DE-AC05-84-ER40150, the Commonwealth of Virginia and the Laser Processing Consortium.

10:00am **AS-ThM6 DC Field Emission Analysis of GaAs and Plasma-source Ion Implanted Stainless Steel**, ***C. Hernandez***, *T. Wang, T. Siggins, H.F. Dylla,* Jefferson Lab, *N.D. Theodore, D.M. Manos*, College of William and Mary, *C.E. Reece*, Jefferson Lab

Field emission studies have been performed on a GaAs wafer and a sample of its stainless steel (SS) support electrode that are part of the new photocathode gun for the 10 kW upgrade FEL at Jefferson Lab. The objective of the studies presented here is to characterize the effect of both, the cleanliness of the wafer and the plasma source ion implanted layer on the electrode to suppress field emission. Field emission is the limiting factor to achieve the required 6 MV/m at the surface of the wafer. Potential field emitters are first located on the surface of 1-inch diameter samples with a DC field emission scanning apparatus at 60 MV/m, then each emitter is characterized by SEM equipped with EDS. The GaAs wafer was hydrogen cleaned before the study. The results show three emitters caused by indium contamination during wafer handling. The GaAs wafer thus shows good high voltage characteristics and the need to maintain cleanliness during handling. The SS sample is hand polished with diamond paste to a 1-micron surface finish, then implanted with N_2/SiO_2 in a plasma source ion implantation chamber in preparation for the field emission studies.[1]

[1] This work is supported by the US Dept. of Energy and the Office of Naval Research.

10:20am **AS-ThM7 Study on Electron Emission from Some Metals and Carbon Materials and the Surface Characterization**, ***S. Kato***, KEK & The Graduate University for Advanced Studies, Japan, *M. Nishiwaki*, The Graduate University for Advanced Studies, Japan

We focus on secondary electron emission from metals and carbon materials in conjunction with potential and actual problems of photoelectron instability and electron multipactering in several particle accelerators in the world. Therefore it is quite important to explore promising materials which have secondary electron yields as low as possible. While a plenty of reports on investigation of secondary electron emission was made over years, the experimental conditions with poor vacuum have deteriorated the quality of the data and lack of surface characterization has made understanding the data and applying the data to surface engineering confusedly difficult. For this purpose, an UHV system was newly developed which consisted of a x-ray source, an ultraviolet photon source, an electron gun, an ion gun, residual gas analyzers, a hemispherical energy analyzer and a sample manipulator with cooling and heating capabilities. This system gives us useful basic informations about dependences of secondary electron yields and the energy distributions on primary electron energy, material, the material temperature and the surface state. In-situ surface characterization of the materials was also performed before and after conditionings such as heating, gas exposure and electron or ion bombardments. Prepared materials were oxygen free copper (single and poly-crystalline), pure titanium, stainless steel, isotropic graphite and carbon fiber reinforced carbon composite. Mechanical surface roughing on the materials was attempted to reduce electron emission in addition. The introduction of the system, the measured results and the discussion will be described in this paper.

10:40am **AS-ThM8 Investigation of the Interfacial Interactions Associated with the Application of a Polymer Coating onto Oxide-free Phosphate Films on Metals**, ***Y.Q. Wang***, *P.M.A. Sherwood*, Kansas State University

This paper will report the results of a continuing study focused on preparing novel surface chemistries with oxide-free phosphate films on metal surfaces. We have developed a new more easily applied process, based on our earlier studies and patent for oxide-free phosphate films. The new process is carried out under ambient conditions using aqueous phosphoric acid. The chemistry of the surface prepared by the new process and its effects on the coating of polymers on metalic aluminum and iron will be investigated by XPS to evaluate the potential of this film to serve as a corrosion inhibitor and other applications. It will also be seen that the valance band photoemission, interpreted by band structure and other calculations can be used to study the interfaces involved in, is a very effective tool for conclusively identifying the surface species present.

This materials was based upon work supported by the National Science Foundation under Grant No. CHE-0137502.

11:00am AS-ThM9 Valence Band X-ray Photoelectron Spectroscopy Studies of Different Forms of Sodium Phosphate

A.L. Asunskis, *P.M.A. Sherwood*, Kansas State University

Valence band X-ray photoemission can be used to identify subtle differences in surface chemistry. There are a very large number of phosphate species, and it is often important to distinguish between these species. Our group has been examining phosphate surfaces on metals, and has shown[1] that it is possible to prepare oxide-free phosphate films on the surfaces of a variety of metals (e.g. iron, aluminum, copper, and titanium) by treatment of the oxide-free metal surface with orthophosphoric acid. Studies of core X-ray photoelectron spectroscopy (XPS) are of little value in distinguishing between different phosphate species. We have shown that it is possible to distinguish between orthophosphate and metaphosphate from differences in the valence band region which can be understood by comparison with spectra predicted by band structure calculations. In this presentation we report the extension of our earlier studies to include linear phosphates of different chain length and other phosphate species. The spectra show that valence band XPS can be used to distinguish between these species and that the spectral differences can be interpreted by spectra predicted by band structure calculations. This material was based upon work supported by the National Science Foundation under grant No. CHE-0137502.

[1] J.A. Rotole and P.M.A. Sherwood, Chem. Mater., 13, 3933-3942 (2001).

11:20am AS-ThM10 The Study of Vanadium Phosphates by Valence Band X-ray Photoelectron Spectroscopy

D.J. Asunskis, *P.M.A. Sherwood*, Kansas State University

This paper presents a study of various vanadium phosphates synthesized by the reaction of vanadium with phosphoric acid in a variety of conditions, including choice of solvent and acid hydration. It is known that the differences in the nature of the vanadium phosphates can impact their role in catalysis, and we are interested in the nature of vanadium phosphate films on metals. In this study valence band X-ray photoelectron spectroscopy, in conjunction with core level X-ray photoelectron spectroscopy, is used for the identification of the synthesized compounds. The valence band spectra, which have been interpreted by band structure calculations, prove to be a conclusive way to identify the species present in each of these compounds. This material is based upon the work supported by the National Science Foundation under grant No. CHE-0137502.

11:40am AS-ThM11 Sorbed Water, as the Source of the Dissolved H_2 and D_2 In Metals at Friction

E.A. Rodina, ***E.A. Deulin***, Bauman Moscow State Technical University, Russia

Results shows, that concentration of the H_2 dissolved in the steel 304 after keeping in normal atmosphere (pressure of H_2 $P_{H2} = 5*10^{-2}$ Pa, pressure of D_2 $P_{D2} = 7*10^{-6}$ Pa) is $C_{H2} = 2*1019$ at/sm^3, instead of theoretically expected $C_{H2} = 2*10^{19}$ at/sm^3, similarly, for D_2, dissolved at $P_{D2} = 7*10^{-6}$ Pa, its concentration, instead of theoretically expected $C^{theor}_{H2} = 8*10^{15}$ at/sm^3 is $C_{D2} = 1*10^{18}$ at/sm^3. As for H_2 also as for D_2 it is possible to explain their increased concentration by the relay dissociation of sorbed water.[3] The results show that the residual atmosphere of H_2 or D_2 influences on ions exchange processes of D_2 and H_2 in layers of sorbed water. So, in the submitted results it is enough 0,002% dissociation of sorbed water to ensure the pointed mentioned concentration. Concentration of the dissolved gases (N=6000 cycles, Sigma= 1500 MPa, n = 600 cont/s) grows up to $C^{max}_{D2} = 2*10^{21}$ at/sm^3 and $C^{max}_{D2} = 3*10^{19}$ at/sm^3 as a result of mechanical action influence that corresponds to the 8,5% dissociation of H_2O, and corresponds to 0,1% dissociation of HDO (for used in a result of D_2 interaction from a surface).

[1] E.A. Deulin, A.A. Gatsenko, B.A. Loginov. Friction force of smooth surface of SiO2-Si02 as a function of residual pressure. Surface Science 433-435 (1999) 288-292.

[2] E. A. Deulin, R.A. Nevshupa. Deuterium penetration into the bulk of a steel ball of a ball bearing due to its rotation in vacuum. Applied Surface Science 144-145 (1999) 283-286.

[3] K.Akagi, M. Tsukada. Theoretical study of hydrogen relay dissociation of water molecules on Si(001) surfaces. Surface Science 438 (1999) 9-17.

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