

Applied Surface Science

Room: Hall 3 - Session AS-TuP

Applied Surface Science Poster Session

AS-TuP1 XPS Characterisation of Thiol-SAMs on Au Surfaces for Biological Applications, *V. Trouillet*, Forschungszentrum Karlsruhe GmbH, Germany, *S. Engin, D. Wedlich*, University of Karlsruhe, Germany, *P. Mack, R.G. White*, Thermo Fisher Scientific, UK, *M. Bruns*, Forschungszentrum Karlsruhe GmbH, Germany

Structured and oriented immobilisation of biomolecules has become subject of great interest in recent years due to the expected diversity of applications, e.g. biosensors in diagnosis, lab-on-chip technology, and modern cell culture focused on cell adhesion, migration, and differentiation. Therefore, a lot of effort has been spent to develop strategies for covalent and non-covalent immobilization of proteins, respectively. A very promising approach is surface patterning by micro-contact printing (μ CP) to produce self-assembly-monolayers (SAMs) on gold surfaces based on mixtures of benzylguanine thiol (BGT) and matrix thiol. In this case BGT is the substrate for the SNAP-tag system, allowing for covalent attachment of any protein of interest fused to this tag, whereas the inert matrix thiol acts as spacer for BGT and moreover prevents from non-specific protein adsorption.

The present contribution focuses on surface analytical characterization of pure benzylguanine and matrix thiols as well as mixtures of both by means of X-ray photoelectron spectroscopy (XPS) to achieve information on chemical binding states in a non-destructive manner. For this purpose all SAMs were prepared on gold films deposited onto glass substrates by r.f. magnetron sputtering directly prior to the thiol exposure. In particular, XPS proves the covalent binding of the thiols and concentration depth profiles constructed from angle resolved data applying the maximum entropy method reveal the orientation of the SAMs together with thickness information. The μ CP micro-structures were verified by small area XPS together with time-of-flight secondary mass spectrometry. In addition, scanning electron microscopy and ellipsometry were used to achieve a comprehensive characterization.

AS-TuP2 Dielectric Response of AISb Determined by In-Situ Ellipsometry, *Y.W. Jung, T.H. Ghong, J.S. Byun, S. Kim, Y.H. Cha, Y.D. Kim*, Kyung Hee University, Korea, *H.J. Kim, Y.C. Chang*, Academia Sinica, Taiwan and University of Illinois at Urbana-Champaign, *S.H. Shin, J.D. Song*, Korea Institute of Science and Technology, Korea

We present pseudo dielectric function data $\langle \epsilon \rangle = \langle \epsilon_1 \rangle + i \langle \epsilon_2 \rangle$ from 0.7 to 5.0 eV of oxide-free AISb at 300 K that is currently the most accurate representation of the bulk dielectric response ϵ of the material. The data were obtained by in-situ spectroscopic ellipsometry (SE). SE is a well-known method of obtaining $\langle \epsilon \rangle$ directly, without requiring Kramers-Kronig analysis. AISb is an important material for optoelectronic applications such as infrared optical devices and high-electron-mobility transistors. However, because overlayers strongly affect $\langle \epsilon \rangle$ and Al reacts readily with oxygen, the approximate determination of ϵ of any Al-containing semiconductor ordinarily requires complex processing to minimize overlayer artifacts. We avoid this by using in situ SE to obtain $\langle \epsilon \rangle$ data before oxides have the opportunity to form.

Our measurements were done on an AISb film $d = 1.5$ nm thick that was grown on a GaAs (001) substrate using molecular beam epitaxy (MBE). Since d is significantly larger than the critical thickness of AISb, the film is fully relaxed and its measured dielectric properties closely approximate those of bulk material. The growth station features an integrated spectroscopic ellipsometer. Measurements were made through strain-free windows while maintaining the AISb layer in ultrahigh vacuum. In the interference-oscillation region, ϵ was extracted with a multilayer parametric model. Compared to previously reported results, our $\langle \epsilon \rangle$ data show lower and higher values in the E_1 and E_2 spectral regions, respectively, confirming that our data are less affected by overlayers. We also observe the indirect band gap of AISb, and obtain the $E_0, E_0 + \Delta_0, E_1, E_1 + \Delta_1, E_0', E_0' + \Delta_0', E_2,$ and $E_2 + \Delta_2$ critical-point (CP) energies from numerically calculated second derivatives. Band-structure calculations done using the linear augmented Slater-type orbital (LASTO) method were performed to identify overlapping CPs in the E_2 energy region. The calculated CP energies agree well with those obtained from data, confirming the validity of the calculations. Our results will be useful in various contexts, including the design of optoelectronic devices.

AS-TuP3 Crossing of the $E_2, E_2 + \Delta_2, E_2'$ and $E_2' + \Delta_2'$ CPs in InAs_xSb_{1-x} Alloys as Determined by Spectroscopic Ellipsometry, *J.J. Yoon, T.J. Kim, S.Y. Hwang, Y.J. Kang*, Kyung Hee University, Korea, *D.E. Aspnes*, Kyung Hee University, Korea and North Carolina State University, *Y.D. Kim*, Kyung Hee University, Korea, *H.J. Kim, Y.C. Chang*, Academia Sinica, Taiwan and University of Illinois at Urbana-Champaign, *J.D. Song*, Korea Institute of Science and Technology, Korea

Owing to its small band gap, Hg_xCd_{1-x}Te is the dominant material for infrared (IR) detectors. However, due to the high Hg vapor pressures needed during growth, accurate compositions x are difficult to obtain, particularly over large areas. The low-bandgap III-V ternary alloy InAs_xSb_{1-x} exhibits important advantages, including better control of composition during growth, higher electron and hole mobilities, and the availability of high-quality, low-cost substrates. As a result, InAs_xSb_{1-x} alloys have recently been used to fabricate various IR optoelectronic devices. Although the optical properties and main critical-point (CP) energies of the binary endpoints InAs and InSb are well known, much less information is available for alloys of arbitrary x . Here, we report pseudodielectric function $\langle \epsilon \rangle = \langle \epsilon_1 \rangle + i \langle \epsilon_2 \rangle$ data from 1.5 to 6.0 eV of InAs_xSb_{1-x} alloys for selected values of x over the entire composition range, assessing the removal of overlayers in real time to ensure that overlayer artifacts are minimized and therefore, that the data most closely represent the intrinsic dielectric responses ϵ of these materials. Energies of the $E_1, E_1 + \Delta_1, E_0', E_0' + \Delta_0', E_2, E_2 + \Delta_2, E_2', E_2' + \Delta_2'$, and E_1' critical points (CPs) were obtained from numerically calculated second derivatives, and identified where necessary by band-structure calculations done using the linear augmented Slater-type orbital method (LASTO). The calculated CP energies agree well with those obtained from data, confirming the validity of the calculations. These calculations show a crossing of the $E_2, E_2 + \Delta_2, E_2'$, and $E_2' + \Delta_2'$ CP structures with increasing As-composition. Also, the band-structure calculations allow us to identify the InSb structures at 3.31 and 3.75 eV with the $\Delta_5^{cu} - \Delta_5^{vu}$ and $\Delta_5^{cl} - \Delta_5^{su}$ saddle points at $k = (0.35, 0, 0)$. These saddle points were reported previously only at low temperature. Our results will be useful in a number of contexts, including the design of optoelectronic devices based on InAsSb and obtaining a more detailed understanding of the band structure of these and related alloys.

AS-TuP4 Gold Nanodot Array on Silicon Fabricated by Scanning Probe Lithography, *M. Kanda, T. Ichii, K. Murase, H. Sugimura*, Kyoto University, Japan

Gold nanoobjects are recognized as important materials to construct the advanced nanoscale technology for electronic, optical, and sensor devices because they can transfer a near-field light between them in a well-aligned arrangement. The aligned arrangement of gold nanoobjects requires a preliminarily patterned substrate with the nanometer-scale precision. Scanning probe lithography (SPL) utilizing atomic force microscopy (AFM) is a very powerful technique to fabricate nanoscale patterns even under atmospheric condition.

In this study, a well-aligned array of gold nanodots was fabricated using SPL. A Si(111) surfaces covered with self-assembled monolayers (SAM) of 1-hexadecene (HD) were used as substrates. HD-SAMs have high chemical durability to hydrofluoric acid (HF) and were used as resist film of SPL. Nanoscale patterns of silicon oxides were fabricated by applying DC bias voltages between an AFM probe and the substrate. Since the HD-SAMs had a highly ordered structure and only 2.3 nm thicknesses, the size and the position of the oxides were precisely controlled. Then, the oxides were etched by immersing the samples into HF solution, and the underlying Si surfaces on the nanopatterns were exposed. After the HF etching, the samples were immersed into an Au electroless plating solution and gold nanodots were deposited only on the nanopatterns. The structures and the optical properties of the gold nanodots arrays were investigated by field emission scanning electron microscopy (FE-SEM) and spectrophotometer, respectively.

AS-TuP5 Ultrashort Pulse Laser Ablation as a Tool for the Depth Profiling of *Staphylococcus Epidermidis* Microbial Biofilms, *S. Milasinovic, M. Blaze, Y. Liu, Y. Zhao, J. Johnston, R.J. Gordon, L. Hanley*, University of Illinois at Chicago

Ultrashort pulse laser irradiation is known to cause minimal laser induced damage when interacting with soft biological materials and might therefore be used for depth profiling in mass spectrometric imaging. This work examines the possibility of using this technique for chemically non-destructive ablation of microbial biofilms. Biofilms of *Staphylococcus epidermidis* were grown on indium tin oxide coated glass slides. ~100 μ m thick biofilms were obtained after 3 day incubation by the drip flow method. These biofilms were then irradiated with 45 fsec pulses of 800 nm

radiation from a Ti-Sapphire laser. Effects of varying the laser fluence and numbers of pulses were investigated by focusing the laser beam to a ~200 μm spot diameter. Laser ablated craters were imaged by scanning electron microscopy and two-photon laser scanning microscopy. The ablation threshold was determined to be 0.08 J/cm². Biofilms were then spiked with Br-tyrosine, which was detected by laser desorption/ionization mass spectrometry within the intact biofilms before and after laser ablation. Mass spectra obtained at different locations were compared to determine the extent of chemical damage done by laser irradiation.

AS-TuP6 Vacuum Ultraviolet Light Induced Chemical Modification of Cyclo-Olefin Polymer Surface Dependent of the Irradiation Atmosphere, Y.J. Kim, H. Sugimura, K. Murase, Kyoto University, Japan, Y. Taniguchi, Y. Taguchi, Alps Electric, Japan

In recent years, cyclo-olefin polymer (COP) resins have been used in a variety of applications owing to their excellent properties and low cost, and the market for cop resins is growing every year[1]. For improvement in adhesivity, dyeability, and wettability, extensive research to develop practical and economical methods for the surface modification of COP has been carried out by many groups. In particular, wettability and adhesion are critical design features for many commercial products. For this purpose, corona discharge treatment, plasma etching, ultraviolet irradiation, and chemical solution etching have been employed. Although it is desirable to provide addition functions to polymer surfaces, it is also important to do so without affecting their bulk characteristics, such as mechanical, thermal, and other intrinsic properties. Ultraviolet irradiation should be the best method in this respect, since ultraviolet irradiation interacts only with the polymer surface and does not penetrate into the centre of the material. This study sought to photochemically convert the surface of COP into a hydrophilic surface consisting of oxygen functional groups such as C–O, C=O, and COO components by simple irradiation with a vacuum ultraviolet (VUV) light of 172 nm wavelength in the presence of atmospheric oxygen molecules. We found that this hydrophilic surface served as a base for the formation of adhesive chemical bonds at the interface to improve the wettability and adhesion properties. We have optimized the VUV photochemical conversion conditions for the COP surface[2].

In this study, we report how different the chemical nature of COP surfaces irradiated with the VUV light is, dependent on the VUV-irradiation atmosphere. The extent of oxygenation was evaluated by XPS and FTIR-ATR spectra, and it was shown that the surface properties, hydrophilicity, and functionalization were crucially dependent on the VUV irradiation atmosphere. Roles of oxygen, nitrogen and water molecules in the VUV-irradiation environment on the surface modification have been elucidated.

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AS-TuP7 Dynamic Solid-liquid Interaction on Hydrophilic Surfaces, Y. Yokota, N. Yoshida, The University of Tokyo, Japan, M. Sakai, Kanagawa Academy of Science and Technology, Japan, A. Nakajima, Tokyo Institute of Technology, Japan, T. Watanabe, The University of Tokyo, Japan

Dynamic solid-liquid interaction as a liquid droplet-movement on inclined hydrophilic-hydrophobic surfaces was investigated. The liquid droplet movement was affected by a surface energy, roughness, and homogeneity of the solid surface, and furthermore it is suggested that the internal fluidity detected by Particle Image Velocimetry method determined the rate. The movement also depended on the shape of a droplet, that possibly changed the internal flow drastically. In this presentation, we will discuss about the detail of relations among them.

AS-TuP8 Relationship between In-Depth Profiles and Local Structures in HfSiO Film Studied by Photoemission Spectroscopy and Extended X-Ray Absorption Fine Structure, S. Toyoda, The University of Tokyo, Japan, H. Ohfuchi, JASRI, H. Kamada, M. Oshima, The University of Tokyo, Japan, G. Liu, Z. Liu, T. Sukegawa, K. Ikeda, STARC

High dielectric (high-k) transition metal oxide materials such as ZrO₂, HfO₂, and their silicates are widely investigated to reduce the leakage current from channel to gate, because SiO₂ gate dielectrics have reached a physical thickness scaling limit of complementary metal-oxide-semiconductor (CMOS) devices. Among them, HfSiO and nitrided HfSiO are promising candidates due to appropriate band gap, soft reactivity to the Si substrate, and suppression of crystallization. In this study, we have demonstrated annealing effects on in-depth profiles and local structures in the HfSiO films by angle-resolved photoemission spectroscopy and extended x-ray absorption spectroscopy. HfSiO films with thicknesses of 2.0 nm were deposited on 0.7 nm SiO₂ interfacial layers using an atomic layer deposition (ALD) technique. Annealing was performed at 850 °C and 1050 °C for 1 min in a nitrogen gas of 1 atm by the direct current flowing

method through the Si substrate. Photoemission measurements were performed at BL-2C of the Photon Factory in High Energy Accelerator Research Organization (KEK). The total energy resolutions were estimated to be 0.20 eV for photon energy of $h\nu = 630$ eV. Photoelectron emission angles were changed from the surface normal to 60° for enhancement of surface sensitivity. To analyze in-depth profiles from angle-resolved core-level photoemission spectra measured by tilting the samples, the maximum entropy method, which was coded according to previous reports, was utilized. The validity of the in-depth profiles was confirmed by comparison with Rutherford backscattering spectroscopy (RBS). In-depth profile of the HfSiO film presents a complicated layer structure even at the as-grown stage; Si concentrations are high in both surface and interface regions and nitrogen atoms are slightly distributed at the interfacial layer. Accumulation of Si atoms at the surface is known by the ALD growth and the in-depth profile in the as-grown sample is quantitatively confirmed by RBS measurements. It should be noted that changes in the in-depth profiles suggest that Si oxide components diffuse from the interfacial SiO_xN_y layer into the HfSiO layer and concentrations of Hf relatively decrease. Coordination number obtained by Fourier transform of the EXAFS spectra decreases with increasing annealing temperature, suggesting that chemical bonding states on nearest neighbor of the Hf atoms in the HfSiO layer change with Si diffusion due to oxidation of the Si substrate.

AS-TuP9 In Situ XPS Depth-Profiling of Hydrogen Storage Material VCrTi during Thermal Annealing, J.R. Harries, Y. Teraoka, A. Yoshigoe, M. Tode, Japan Atomic Energy Agency

VCrTi is a candidate material for hydrogen-storage. The presence of a surface oxide layer, its thickness, and its composition can be expected to effect the properties of charging and release of stored hydrogen, and as such it is of interest to characterise the native oxide, and to investigate whether more desirable properties can be obtained by artificially modifying the surface composition. In this study we have used synchrotron radiation photoelectron spectroscopy to study the chemical composition of the native oxide layer of a commercial sample of V₂₅Cr₄₀Ti₃₅ alloy. The total XPS energy resolution is sufficient to provide chemical state-specific information, and spectra recorded at a range of different take-off angles provide information on the depth-profiles of the composition. The maximum entropy method is used to generate element-specific (and chemical state-specific) depth profiles from the spectra. The following conclusions are drawn:-

The thickness of the native oxide layer as determined from the XPS data is consistent with a TEM analysis, at approximately 5 nm, and consists of oxides of vanadium, chromium, and titanium, in amounts roughly corresponding to the composition of the alloy. It also contains carbon. Depth-profiling suggests that the carbon forms a sub-layer.

Thermal annealing gradually reduces the oxide coverage with increasing temperature. After heating to 800 degrees C, the dominant surface component appears to be titanium oxides, with vanadium and chromium oxides only remaining below the surface.

It is also planned to study the chemical composition of a sample irradiated with hydrogen (deuterium) ions, and study the uptake of hydrogen for samples with oxide layers of differing composition.

AS-TuP10 Automated XPS Analysis of Passivated Stainless Steel to the SEMI Standard, T.S. Nunnery, O. Mustonen, J. Wostenholme, Thermo Fisher Scientific, UK, B.R. Strohmaier, J.D. Piasecki, R.J. Lee, RJ Lee Group, Inc.

The Semiconductor Equipment Manufacturers Institute (SEMI) defines methods for analysing the elemental composition and metallurgical characteristics of metal used in components for high purity gas distribution systems. Typically stainless steel components are treated to improve surface quality and prolong their lifetime and performance. In the passivation process a chromium-rich 'passive' layer is formed on top of the steel surface. The SEMI procedures define methods to determine the surface and oxide composition and chemistry of the interior surface of chromium enhanced stainless steel tubing. The key metrics that are used to identify passivated steel are the total Cr/Fe surface atomic ratio, the Cr oxide/Fe oxide surface atomic ratio, and the total oxide thickness.

The current SEMI test methods have been used since 1992 (former SEMASPEC methods) and use both X-ray Photoelectron Spectroscopy (XPS) and Auger Electron Spectroscopy (AES) to evaluate the passivation of the stainless steel surface. Since then XPS instrumentation has developed significantly which enables the determination of the parameters quickly and easily with XPS only. This work describes how the test procedure can be automated from start to finish with combination of modern XPS instrumentation and software. In particular, procedures can be used to maintain consistency of approach in the important peak-fitting steps to eliminate inconsistencies which can be introduced by different users. The automated approach can be extended to include sputter depth profiling of

the passivation layer, resulting in a method for batch control or failure analysis of a series of samples.

AS-TuP11 Vacuum Annealing Phenomena in Ultrathin TiDy/Pd Bi-Layer Films Evaporated on Si(100) as Studied by TEM and XPS, E.G. Keim, University of Twente, The Netherlands, W. Lisowski, Polish Academy of Sciences, Poland

Using a combination of TEM and XPS, we made an analysis of the complex high-temperature annealing effect on ultra-thin titanium deuteride (TiD_y) films evaporated on a Si(100) substrate and covered by an ultra-thin Pd layer. Annealing induced deuterium evolution from such material¹ can be applied as a useful source of deuterium used in chemical and energetic reactions. In this report we show to what extent structural changes occur within the ultrathin Si/TiDy/Pd film area as a result of annealing. The TEM/XPS data are compared for two films, one taken before, the second after annealing. 10 - 20 nm thick TiD_y films on a Si substrate each covered by 10 - 20 nm thick Pd were prepared in a UHV glass system². Mass spectrometry was used to monitor in situ deuterium evolution from the film during annealing, all other analyses were performed ex situ. It was found that the Si/TiD_y/Pd assembly undergoes a microstructural and chemical conversion as a result of annealing. Energy Filtered TEM (EFTEM) mapping of cross-section images and XPS depth profiling analysis revealed both a broad intermixing between the Ti and Pd layers and an extensive inter-diffusion of Si from the substrate into the film bulk area. Selected Area Diffraction (SAD) reveals very fine crystallites of PdTi₂ and the initial stages of TiSi phase formation. Segregation of Ti towards the Pd top layer surface has been evidenced using Angle Resolved XPS (ARXPS) and the EFTEM image analysis.

References

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AS-TuP13 Investigation of Metal-Assisted SIMS and Cluster Ion Bombardment for Analysis of Polystyrene Surfaces, H.F. Arlinghaus, F. Lipinsky, Westfälische Wilhelms-Universität Münster, Germany, N. Wehbe, A. Delcorte, P. Bertrand, Université Catholique de Louvain, Belgium, L. Hoissiau, University of Namur, Belgium, A. Heile, Westfälische Wilhelms-Universität Münster, Germany

In addition to structural information, a good knowledge of the local chemistry proves to be of ever greater importance for the development of new types of materials as well as for specific modifications of interfaces and surfaces. A detailed understanding of the systems is required in multiple fields of the metal and semiconductor industries and for various biomedical and chemical applications. In particular, the ongoing miniaturization and the associated reduction of the volumes of material available for analysis constitute a challenge for the detection limits of quite a number of characterization techniques.

In the case of TOF-SIMS, several methods of secondary ion yield enhancement have been proposed. Our contribution focuses on two of these methods, i.e. metal-assisted SIMS and polyatomic primary ion bombardment (e.g. Bi_3^+ , C_{60}^+). In previous studies on thicker layers of organic molecules it was found that polyatomic primary ions generally increase the secondary ion yields and that a yield enhancement due to gold deposition can only be detected for monoatomic ion bombardment [1,2]. To investigate advantages and drawbacks of the use of Meta-SIMS and polyatomic primary ion bombardment in more detail, not only thicker layers of several ten nanometers but also layers of only some nanometers thickness of polystyrene were prepared. The samples, pristine and metallized with different amounts of gold, were analyzed using monoatomic and polyatomic primary ions.

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AS-TuP14 Surface Analysis of Electro-Polished Niobium Cavity-Samples for RF Applications, P.V. Tyagi, M. Nishiwaki, T. Saeki, M. Sawabe, H. Hayano, KEK, Japan, T. Noguchi, KAKEN Inc., Japan, S. Kato, KEK, Japan

The performance of superconducting RF cavities is restricted by the contaminations present on the surface and the surface roughness. In the recent years extensive research has been done to enhance the cavity performance by applying improved surface treatments such as mechanical grinding (MG), electro-polishing (EP), chemical polishing (CP), tumbling, etc followed by various post-treatment methods like ultrasonic pure water rinse, alcoholic rinse, high pressure water rinse, hydrogen peroxide rinse

etc. Although EP post-treated by various methods has revealed good results to obtain smooth and clean surface, still more research and efforts are required in this area. Prior XPS (x-ray photoelectron spectroscopy) analysis of EPed Nb samples demonstrate that the surface was covered mainly with the niobium oxide (Nb_2O_5) along with carbon in addition, a small quantity of sulfur and fluorine was also found in SIMS (secondary ion mass spectroscopy) analysis even they were post-treated by various methods. In this article we present the analysis of surface contaminations and roughness for a series of EPed Nb samples located at various positions of a single cell Nb cavity followed by post-treatment methods and our endeavor to understand the performance of EP in a cavity with the help of surface analytical tools like XPS, SIMS and SEM (scanning electron microscope) at KEK.

AS-TuP15 Kelvin Probe Force Microscopy on Phase-Separated Self-Assembled Monolayers of Alkanethiol and Amide-Containing Alkanethiol, S. Ikebukuro, Kyoto University, Japan

Frequency modulation atomic force microscopy (FM-AFM) is a very powerful tool for investigating various materials including organic samples. Since the chemical interaction and the van der Waals interaction between the tip and the sample are the main origin of the FM-AFM contrast, it can be applied for chemical identification of surface functional groups as well as structural analysis of organic ultrathin films[1, 2]. However, the decay-length of those interaction is less than 1 nm, the FM-AFM contrast only provides the chemical information of the sample surface and the subsurface information is difficult to be obtained. On the other hand, Kelvin probe force microscopy (KFM) is a method to investigate surface potential (SP) distribution on the nanometer scale. Since the electrostatic force, which is the origin of KFM contrast, is a long-range interaction, it is expected that we can obtain the subsurface information and discriminate two separated areas that have same surface profile while subsurface structures differ from each other using KFM.

In this study, to evaluate transitions of SP caused by the difference of subsurface structures, we investigated a phase-separated self-assembled monolayer (SAM) formed from a binary mixture of nearly isometric adsorbates, n-alkanethiol (n-dodecanethiol : DDT) and amide-containing alkanethiol (3-mercapto-N-octylpropionamide : IATC8), on a Au(111) substrate using FM-AFM/KFM. The SAMs were prepared by the coadsorption from an equimolar solution of DDT and IATC8 (1 μM in total thiol) and two separated regions were discriminated in the FM-AFM/KFM images. The same experiments were also performed on a DT/IATC8 phase separated SAMs. We compared these results and concluded that the SP of the IATC8 areas was lower by 240 mV compared to the DDT areas. Considering the differences of molecular properties between DDT and IATC8, these SP images present reasonable contrast and suggest that the difference of the subsurface structures was detected using FM-AFM/KFM.

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AS-TuP16 Surface Potential Measurement of Organo-Chalcogenide Self-Assembled Monolayers using Kelvin Probe Force Microscopy, T. Ichii, S. Nanjo, S. Ikebukuro, K. Miki, K. Murase, K. Ohe, H. Sugimura, Kyoto University, Japan

Organo-chalcogenides (sulfide, selenide, and telluride) have been expected to play an important role in molecular electronics because they can be bound onto noble metals. Functional groups with the chalcogene atoms in these molecules act as anchoring groups, and the molecule-metal bindings can be modified by changing the atom species. Some of the organo-chalcogenides are formed into self-assembled monolayers (SAMs) and they have been intensively studied as model structures of molecule-metal binding.

Kelvin probe force microscopy (KFM), which is one of scanning probe techniques, is an outstanding method for the investigation of local surface potential on the nanometer scale. Since surface potential of organic monolayers on substrates reflects dipole moment of the organic molecules and interfacial dipole moment between the molecules and the substrate, KFM allows us to clarify how the anchoring groups effect on electric properties of the molecule-metal bindings.

In this study, we investigated surface potential distribution of phase-separated SAMs composed of organosulfide and organoselenide using KFM. Dibenzyl-disulfide (BS), dibenzyl-diselenide (BSe) and 3-mercapto-N-octylpropionamide (IATC8) were used and phase-separated SAMs of BS/IATC8 and those of BSe/IATC8 were prepared. The IATC8 areas in the SAMs were used as a reference in the surface potential measurement. The surface potential of the BSe area were higher by approximately 300 mV compared to the BS regions and we concluded that it was mainly due to the difference of the interfacial dipole moment.

AS-TuP17 Lattices of Freestanding Carbon Nanofibres Characterized by Spectroscopic Ellipsometry, R. Magnusson, Linköping University, Sweden, *R. Rehammar*, Göteborg University, Sweden, *H. Arwin*, Linköping University, Sweden

Photonic crystals with lattice constant in the visible range of light can be fabricated using freestanding carbon nanofibres. These types of structures are optically complicated, with geometric effects both from the lattice and from individual scatterers. In this report, results from pilot studies of the optical properties of such samples are presented.

Ellipsometric measurements on samples with vertically aligned arrays of free-standing carbon nanofibres with lengths up to 1600 nm are presented. The carbon nanofibres were grown on silicon wafers with a 70 nm layer of titanium and a 15 nm layer of titanium nitride using plasma-CVD with nickel as catalyst. Electron beam lithography was used to create the nanofibre lattice. Samples with varying periodicity and length of the carbon fibers, i.e. film thickness, were investigated.

In this pilot study measurements were performed with a dual rotating compensator ellipsometer in the spectral range 245-1700 nm. The instrument provides the full Mueller matrix of the sample and measurements were performed at multiple angles of incidence and different sample orientations.

The samples show no difference in the optical response when rotated 90°, but at other orientations changes in the spectrum are observed below a wavelength of approximately 1000 nm. The analysis of the ellipsometric data is presented and different modeling approaches are discussed.

AS-TuP19 Spectroscopic Ellipsometry of Bulk MAX-phases, A. Mendoza-Galvan, Cinvestav-Queretaro, Mexico, *M. Rybka*, *K. Järrendahl*, *H. Arwin*, *M. Magnuson*, *L. Hultman*, Linköping University, Sweden, *M. Barsoum*, Drexel University

MAX-phases are compounds with the chemical formula $M_{n-1}AX_n$ where M is a transition metal, A is an element from column 13 to 16 in the periodic system and X represents C or N and $n = 1, 2$ or 3 . These materials are potentially technologically important as they show unique refractory and other physical properties due to the combination of metals and ceramics. From a fundamental point of view the band structure of these materials are of interest and optical reference data are important to determine. Herein we report, for the first time, on the optical properties of NbTiAlC, Nb₂AlC, TiSc₂, Cr₂GeC, TiGeC₂, Ti₂AlC, and Ti₂AlN by spectroscopic ellipsometry in the 0.03 to 6.0 eV spectral range. The ellipsometric data in the infrared range show features corresponding to Fano modes indicating the presence of a thin oxide layer a few nm thick. The optical response of these MAX-phases is represented by a Drude-Lorentz model. Thus, in the low energy range the electrical conductivity through the Drude term is evaluated and two or three interband electronic transitions which are compositional dependent can be identified at photon energies in the visible-ultraviolet range 1.0-6.0 eV.

AS-TuP20 In-Situ Synperonic Film Growth on Self Assembled Monolayers and Organic Polymers Investigation Using Quartz Crystal Microbalance in Conjunction with Spectroscopic Ellipsometry, A. Kjerstad, T. Hofman, E. Schubert, M. Schubert, University of Nebraska - Lincoln

Thin film growth in an aqueous environment of a high water content film is difficult to characterize due to the ambient conditions and unknown porosity of the film. We report on the investigation of aqueous in-situ synperonic thin films. These films are grown on self-assembled monolayers and organic polymers of varying hydrophobicities. The deposition is measured using quartz crystal microbalance (QCM) in conjunction with spectroscopic ellipsometry (SE). The strength of QCM lies in its ability to determine the mass of deposition, whereas SE is highly sensitive to changes of index of refraction and film thickness. These findings are used to model film deposition and behavior. The models are created using two instrumentation techniques, thus ensuring validity.

AS-TuP21 Ellipsometric Study of Ga-Doped ZnO Films Deposited on Large Area Substrates by Pulsed Laser Deposition, D. Agresta, K. Leedy, B. Bayraktaroglu, U.S. Air Force Research Laboratory

Transparent conductive Ga-doped ZnO (GZO) thin films are prepared on large-area substrates by pulsed laser deposition (PLD) for optoelectronic contact applications. Limited reports exist of large area PLD of oxide thin films. A previous study of Al-doped ZnO (AZO) films reported high quality material being obtained over large areas by using a combination of off-axis PLD and post deposition annealing at 400°C in forming gas [1]. This paper utilizes these same techniques for the fabrication of GZO (3% Ga₂O₃) on Si or quartz substrates up to 100 mm in diameter. We report on the effect of different PLD temperatures (400, 500, and 600°C) and pressures (5, 10, 50 mTorr) on the uniformity and stability of pre- and post-annealed GZO film properties.

The optical properties (refractive indices, absorption coefficients, and energy gaps) of the GZO thin films are extracted primarily by spectroscopic ellipsometry (SE). SE has been used to determine the optical functions of ZnO films. Known for its precision and non-destructiveness, SE is an indirect measurement technique in the sense that the film properties of interest are obtained by a nonlinear regression analysis of measured data to an optical model. This enables the extraction of both the real and imaginary parts of the dielectric function, without directly involving Kramers-Kronig analysis, while simultaneously determining the film thickness with great precision. SE spectra are obtained with a Horiba Jobin Yvon UVISEL spectroscopic ellipsometer from 0.6 to 4.7 eV and analyzed with the self-contained DeltaPsi2 (DP2) software package. Additionally, we present comparative studies using normal incidence reflectance and transmission, atomic force microscopy (AFM), optical interferometry, x-ray diffraction (XRD), and scanning electron microscopy (SEM) which will either confirm the ellipsometric optical model or may reveal parameters for their incorporation. Lastly, the electrical properties are considered by resistivity measurements.

Reference:

1. K. D. Leedy, C. V. Varanasi, D. H. Tomich and B. Bayraktaroglu, "Al-doped ZnO Thin Films Deposited on Large-area (100 mm Diameter) Substrates using Pulsed Laser Deposition for Optoelectronic Contact Applications," 5th International Workshop on ZnO and Related Material (2008).

AS-TuP22 The Optimized Wet Cleaning for Extreme Ultraviolet (EUV) Masks: Cleaning Efficiency for Residual Photoresist and Ru Capping Layer Surface, H. Seo, J.Y. Park, University of California, Berkeley and Lawrence Berkeley National Laboratory, *T. Liang*, Intel Corporation, *G.A. Somorjai*, University of California, Berkeley and Lawrence Berkeley National Laboratory

Extreme Ultraviolet (EUV) lithography is a leading technology for future top-down semiconductor device manufacturing. Since this technology relies on the beam projection by reflection, Mo-Si multilayer (ML) stacks are used for EUV masks, to maximize the reflection from the mask. It is challenging and necessary to effectively remove contaminants from the masks and mirrors without adverse effects on their surfaces. Furthermore, process-induced contaminations on mask surfaces such as residual photoresist, metalorganic compounds, and sub-micron particles during patterning, handling, and use of EUV masks also affect the ML surfaces and cause problems for resist print on the wafer. Thus, the development of an effective cleaning process is one of critical technical issues, which must be resolved in order to achieve damage-free, efficient, and reliable cleaning methods for use in EUV lithography.

In this study, the efficiency of various wet cleaning methods for EUV mask blanks were investigated using surface sensitive characterization techniques. Two types of samples were prepared for cleaning: (i) EUV mirrors capped with 3 and 6 nm Ru layer on Mo-Si multilayers and (ii) e-beam photoresist (PR) coated Ru layer on Mo-Si multilayers. These two types of samples were cleaned using various wet chemicals to evaluate both the cleaning efficiency for resist and organic contaminants and the chemical effects on the Ru surfaces. The wet chemicals used in the study include tetramethyl ammonium hydroxide (TMAH), non-toxic organic solvents and sulfuric acid. The chemical compositions of the EUV mask blanks were characterized with x-ray photoelectron spectroscopy (XPS) before and after each cleaning process. Atomic force microscopy (AFM) and Scanning Electron Microscope (SEM) were used to investigate the influence of cleaning methods on the surface morphology and roughness. The chemical analysis of the EUV masks after these treatments revealed different chemical effects on the Ru oxidation state and surface carbon concentration depending on the pH of the wet solution. A surface reaction model for the behavior of Ru and RuO₂ in wet solutions is proposed. More importantly, the systematic study to seek optimized chemical solutions for efficient residual PR removal and improved EUV mask surface reflectivity was carried out and the results are presented and discussed.

AS-TuP23 XPS and RBS Quantification of Pulsed Laser Deposition of LAO/STO Heterostructures: Unexpected Variations in Stoichiometry, M.H. Engelhard, T. Droubay, V. Shutthanandan, D.R. Baer, S.A. Chambers, Pacific Northwest National Laboratory

We report quantitative analysis of pulsed laser deposition (PLD) of LaAlO₃/SrTiO₃ heterostructures performed using X-ray photoelectron spectroscopy (XPS) and Rutherford backscattering spectrometry (RBS). Pulsed laser deposition (PLD) has been the method of choice for the growth of LAO/STO heterostructures in many laboratories. PLD practitioners generally assume that the stoichiometry of the target is preserved in the process of vaporizing and condensing the target material onto the substrate. However, researchers do not usually conduct detailed materials characterization measurements that would ensure that this assumption is

valid. We recently initiated a study of the spatial distribution of condensed La and Al on 2" diameter Si substrates held at ambient temperature during PLD of a LAO target. The substrates were fixed in position relative to the laser plume and did not rotate during growth. We found that there is considerable spatial variation in composition of La and Al deposited on the Si from a single crystal LAO target, even at laser fluences well above the ablation threshold. The on-axis geometry, in which the axis of the laser plume intersects a portion of the substrate, clearly leads to a significant enrichment of La and an Al deficiency. Although the variations are apparent in both XPS and RBS, we have further quantified the composition in XPS by comparison with standards and sensitivity factors specifically determined for our instrument. A La to Al ratio of 1:1 is achieved only over a narrow range of off-axis plume angles. XPS and RBS determination of the composition as a function of position on the substrate will be reported. We present a comparison of XPS LAO quantifications with and without overlayer correction from surface adventitious hydrocarbon contamination using the La 4d line with a relatively high kinetic (KE) energy of 1,383 eV with a lower KE La 3d_{5/2} line at 651 eV.

AS-TuP24 Complete Analysis of Materials using Complementary Techniques. V.S. Smentkowski, D. Wark, L. Le Tarte, H. Piao, J.C. Chera, S.G. Ostrowski, A. Suzuki, General Electric Global Research

A combination of complimentary analysis techniques has been used to characterize a diffusion couple sample. We will demonstrate that the results, when taken together, provide for a better understanding of the sample than the data from any one technique alone. The poster will summarize how diffusion couples are fabricated and highlight the benefits of each characterization technique. Electron Probe Micro Analysis (EPMA) provides for quantitative chemical analysis (spectrometry and imaging) of most elements (Z>4) at a concentration greater than about 0.1 % within a volume of 1-2 microns. EPMA also allows for imaging of both backscattered and secondary electrons. Auger Electron Spectroscopy (AES) has the smallest analytical spot, provides for quantitative analysis of all elements (except H and Li) present at concentrations of greater than about 0.5 atomic percent, and can perform high lateral resolution imaging. X-Ray Photoelectron Spectroscopy (XPS) provides for quantitative analysis of all elements (except H and Li) present at concentrations greater than about 0.5 atomic percent, is able to analyze insulating samples, and can provide information regarding the chemical state of the material. Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS) is able to detect all elements and high mass molecular fragment ions with high sensitivity, is able to analyze non-conductive samples, and the analyst does not need to select which species to analyze for prior to the measurement since a full mass spectrum is saved at every volume element. AES, XPS, and ToF-SIMS surface analysis instruments can operate in spectrometry modes (compositional analysis), imaging modes, as well as depth profiling modes (where one monitors the sample composition as a function of depth).

AS-TuP25 Field Emission from Two-Dimensional Electron Gas in Hetero-Junctions of InAlAs/InGaAs Multiple Quantum Wells. Y. Itakura, M. Matsumoto, K. Fukutani, T. Okano, The University of Tokyo, Japan

The two-dimensional electron gas (2DEG) in multiple quantum wells (MQWs) shows interesting electronic phenomena which are important for semiconductor device applications such as High Electron Mobility Transistor (HEMT). Due to a highly coherent feature and quantized energy levels, electron emission from 2DEG is expected to reveal spatially anisotropic patterns and a characteristic bias dependence. In this study, we applied a high electrostatic field to the surface of a semiconductor hetero-structure in aimed at exploring the physics of field emission from a 2DEG.

For the experiments, we developed a novel cleavage mechanism usable in the UHV environment to realize the high electrostatic field necessary for the tunneling through the potential barrier between the bulk and the vacuum allows for double cleavage along the <110> and <1-10> directions and thus creates a very sharp edge at the corner of a square-shaped wafer with a semiconductor hetero-structure. The cleavage under UHV is also indispensable to obtain clean surfaces free from oxidation. Observation of the apex of the cleaved edge by scanning electron microscopy confirmed that the curvature radius is sufficiently small to allow for field emission. The hetero-structure adopted for this study was an InAlAs/InGaAs modulation doped structure grown on oriented InP(001) substrate by molecular beam epitaxy (MBE). The composition of the hetero-structure was In_{0.52}Al_{0.48}As/In_{0.53}Ga_{0.47}As MQW with 2 periods of InGaAs wells, InAlAs spacers, and Si-doped InAlAs donor layers. The thickness of the spacer layer was 20 nm, and the sheet carrier density was approximately 1.0 x 10¹² cm⁻².

In this presentation, we will report the spatial distribution of the field emitted electrons (field emission pattern) and the I-V characteristics of the field emission.

AS-TuP29 Characterization of Silver Nanoparticles Synthesized on Nylon Membranes Used as Nanoreactors. L. Huerta, Universidad Nacional Autónoma de México, V. Sánchez-Mendieta, R.A. Morales-Luckie, S. Reyes-Vega, Universidad Autónoma del Estado de México, M. Flores, CUCEL, Universidad de Guadalajara, México, J. Arenas Alatorre, Universidad Nacional Autónoma de México

We used X-ray photoelectron spectroscopy (XPS) method for the characterization of surface composition of core and shell silver nanoparticles. The samples were prepared in thin nylon membranes, by means of an easy method of impregnation and reduction of metal ions, using a NaBH₄ aqueous solution, at ambient condition. Particles of less than 10 nm were obtained using the nylon fibres as nanoreactors.

The mesoporous nylon fibres, as observed by scanning electron microscopy (SEM), along to the oxygen and nitrogen density from amide moieties in nylon, provide effective sites for *in situ* reduction of silver ions, and for the formation and stabilization of Ag nanoparticles, consistent with the XPS results, showing of the N 1s core level a chemical shift with increasing concentration of metal particles of Ag. Transmission electron microscopy (TEM) analysis showed that silver nanoparticles were homogeneously dispersed in the nylon templates.

Photoelectron spectroscopy analyses confirmed the composition of the clusters to be metallic status of Ag nanoparticles.

AS-TuP32 Transport Properties of Doped SiGeSn Alloys. V. D'Costa, Y. Fang, J. Menendez, J. Kouvetakis, Arizona State University

A tunable direct absorption edge has already been demonstrated in lattice-matched SiGeSn alloys grown on Ge-buffered Si [1]. These alloys represent the first practical group-IV ternary making it possible to decouple electronic structure and lattice parameter. This opens up new possibilities in IR optoelectronics and photovoltaics [2,3]. The doping of ternary alloys is a basic requirement for device applications. N-type doping is achieved using (SiH₃)₃P whereas diborane is used to obtain p-type SiGeSn. In this paper, we focus on the transport properties of SiGeSn alloys. The dielectric function of heavily-doped alloys has been measured using infrared spectroscopic ellipsometry. The infrared response of the ternary alloys is Ge-like and is dominated by the free carrier contribution. In addition, the dielectric function of p-type alloys shows features due to optical transitions between split-off (SO), light-hole (LH), and heavy-hole (HH) bands. Our studies confirm that doping can be achieved in the ternary alloys. The resistivities and mobilities of the alloys are comparable to those found in Ge samples with similar doping concentrations. We are currently studying the transport properties of lattice-matched alloys as a function of Si and Sn concentration.

1. V.R. D'Costa et al, Phys. Rev. Lett 102, 107403 (2009)

2. R. A. Soref et al, Journal of Materials Research 22, 3281 (2007)

3. F. Dimroth and S. Kurtz, MRS Bull. 32, 230 (2007).

AS-TuP33 Topographical Study of TiN "Ion-CCD" Detector Surface: How damaging are sub-fA Ion Beams of 1-keV Energy? O. Hadjar, G. Kibelka, O.I. Analytical

The development of ion-detector arrays with high spatial resolution enabled the miniaturization of double-focusing sector-field mass spectrometers (MS). A modified imager based on a charge-coupled device (CCD) provides the spatial resolution, ruggedness, and analytical sensitivity necessary to build a transportable mass spectrometer. The transportable (~40 lb) mass spectrometer, which was introduced by OI Analytical at the 2009 Pittsburg Conference¹, is based on a double-focusing sector-field MS of Mattauch-Herzog geometry and an ion-CCD. The MS separates ions of different m/z spatially and focuses all ion beams onto a confocal plane of 2" width. The ion-CCD detects simultaneously all separated ion beams. The focal point of an ion beam is about 1500 x 300 mm², covering roughly 10 pixels. The ion-CCD consists of 2126 active pixels with a pitch of 24 μm. Fig.1 is a contact mode Atomic-Force-Microscope image showing the width (21 μm) of the pixel and the insulation gap between two adjacent pixels (3 μm). The ion-CCD as described earlier² has an upper layer of TiN, 100- nm thick. It is this layer that takes the full impact of the up to 1- keV ions at an incidence angle of 45°. In this work we will investigate the effect of the energetic ions on the ion-CCD and probe the extent of the surface damage, if any, as function of the ion flux and the overall ion-CCD exposure time. The ion-CCD is generally exposed to ion currents in the sub-fA regime and ion beam densities of up to about 10⁹-10¹¹ ions/cm² per second. We will characterize an ion-CCD chip after operating for roughly one year and compare pixels with no ion impact history to those exposed to ion beams consisting of N₂⁺, O₂⁺, Ar⁺ and Xe⁺. In our system, the impact energy of the analyzed compounds falls right on the efficient sputtering regimes, especially for ions with masses close to N and Ti where momentum transfer is optimum³.

(1) "Compact GC/MS based on the Ion-Camera mass spectrometer",
Gottfried Kibelka, Omar Hadjar, Scott Kassan, Scott Shill, Chad Cameron.
Pittsburgh Conference, Chicago. 2009.

(2) "CCD for Ion Detection" Omar Hadjar, Gottfried Kibelka, AVS
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(3) *J. Vac. Sci. Technol. A*, Volume 19, Issue 3, pp. 1004-1007 (May 2001).

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