Novel Trends in Synchrotron and FEL-Based Analysis Focus Topic
Room: 312 - Session SA-MoM

Synchrotron Studies of Processes in Energy Conversion, Electronic Devices and Other Materials I
Moderator: Franz Himpsel, University of Wisconsin-Madison

8:20am **SA-MoM1 Looking Into Buried Interfaces with Soft/hard X-Ray Photoemission and Standing-Wave Excitation**, ***Charles Fadley***, University of California, Davis **INVITED**

I will present some new directions in synchrotron radiation soft x-ray photoemission (XPS, SXPS) and hard x-ray photoemission (HXPS, HAXPES, HIKE) [1-6], with illustrative examples of applications to a range of sample types. These involve combined SXPS and HXPS studies of buried layers and interfaces in magnetic and transition-metal oxide multilayers [1,2], as well as in semiconductor junctions [3]; solid-gas or solid-liquid interfaces with high ambient pressures [5]; band-offset measurements in multilayer structures [6]; and the use of standing waves from multilayer mirrors to enhance depth contrast in spectroscopy [1-5].

References

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1 “Determination of layer-resolved magnetic and electronic structure of Fe/MgO by standing-wave core- and valence- photoemission”, See-Hun Yang, Benjamin Balke, Christian Papp, Sven Döring, Ulf Berges, L. Plucinski, Carsten Westphal, Claus Schneider, Stuart S. P. Parkin, and Charles S. Fadley, Phys. Rev. B 84, 184410 (2011).

2 “Interface properties of magnetic tunnel junction La0.7Sr0.3MnO3/SrTiO3 superlattices studied by standing-wave excited photoemission spectroscopy”, A. X. Gray et al., Phys. Rev. B 82, 205116 (2010); and A.X. Gray et al., Europhysics Letters 104, 17004 (2013).

3 “Nondestructive characterization of a TiN metal gate: chemical and structural properties by means of standing-wave hard x-ray photoemission spectroscopy”, C. Papp, G. Conti, et al. J. Appl. Phys. 112, 114501 (2012).

4 “Hard X-ray Photoemission with Angular Resolution and Standing-Wave Excitation”, C. S. Fadley, invited review, J. Electron Spectrosc. 190, 165-179 (2013)

5 “Chemical-state resolved concentration profiles with sub-nm accuracy at solid/gas and solid/liquid interfaces using standing-wave ambient-pressure photoemission (SWAPPS)”, S. Nemsak et al., in preparation

6 “Band Offsets in Complex-Oxide Thin Films and Heterostructures of SrTiO3/LaNiO3 and SrTiO3/GdTiO3 by Soft and Hard X-ray Photoelectron Spectroscopy”, G. Conti, A. M. Kaiser, A. X. Gray, S. Nemšák , G. K. Pálsson, J. Son, P. Moetakef, A. Janotti, L. Bjaalie, C.S. Conlon D. Eiteneer, A.A. Greer, A. Keqi, A. Rattanachata, A.Y. Saw , A. Bostwick, W.C. Stolte, A. Gloskovskii, W. Drube, S. Ueda, M. Kobata, K. Kobayashi , C. G. Van de Walle, S. Stemmer, C. M. Schneider and C. S. Fadley, J. Appl. Phys. 113 143704 (2013).

9:00am **SA-MoM3 Hard X-ray Photoelectron Spectra (HXPES) of Bulk Non-Conducting Silicate Glasses**, ***Yongfeng Hu***, *Q.F. Xiao, X.Y. Cui, D. Wang,* Canadian Light Source, Canada, *G.M. Bancroft, H.W. Nesbitt, M. Biesinger*, University of Western Ontario, Canada

Bulk studies of non-conducting oxides and silicates, such as silicate glasses containing cations such as Na, K, Mg and Ca are important to obtain quantitative *bulk* information of bridging oxygen (Si-O-Si, so-called BO), non-bridging oxygen (Si-O-M, so-called NBO), and "free oxygen (M-O-M). These studies have been so far limited to the XPS studies using spectrometers equipped with modern charge compensation systems, such as Kratos or ESCALAB 250Xi.[1,2] Such measurements are very important for determining the chemical and physical properties of a wide variety of silicate minerals and glasses[2]. Synchrotron-based hard X-ray photoelectron spectroscopy (HXPES) has recently been applied to the characterization of surfaces and interfaces of advanced materials. In this work, we will demonstrate that the HXPES, without any charge compensation system, can avoid the large differential charging problems usually seen with bulk non-conductors using conventional XPS instruments. These problems are overcome by depositing a thin metal coating on the glass surface and by taking advantage of the large and variable probing depth offered by HXPES. We show that the optimal O 1s linewidth, matching to that of the Krotos' results, can be obtained for the non-conducting silicate glasses using HXPES. Together with the high resolution Si 1s results, these HXPES data are critical for accurate analysis of the BO, NBO and free oxide content of these silicate glasses.

[1] H. W. Nesbitt, G.M. Bancroft, G.S. Henderson, R.Ho, K.N. Dalby, Y. Huang, Z. Yan, ***J. Non-Cryst. Solids*** 357 (2011) 170.

[2] H.W.Nesbitt, G.M. Bancroft, ***Rev. Min. Geochem.*** 78 (2014) 271.

9:20am **SA-MoM4 *In Situ* Study of Plasma Assisted Atomic Layer Epitaxy of III-N Semiconductors Using Synchrotron X-ray Methods**, *N. Nepal,* Naval Research Laboratory, *M.G. Erdem,* Boston University, *S.D. Johnson, V.R. Anderson,* Naval Research Laboratory, *A. DeMasi, K.F. Ludwig,* Boston University, ***Charles Eddy, Jr.***, Naval Research Laboratory

Atomic layer epitaxy (ALE) is a relatively new method to grow crystalline materials in a layer-by-layer fashion by separating the growth reaction into two surface-mediated, self-limiting half reactions at relatively low temperatures. Recently, plasma assisted ALE (PA-ALE) has been used to grow epitaxial III-nitride films at temperatures from 180-500°C [1-2]. At these growth temperatures, the ad-atom mobility is low and the growth process is highly dependent on the quality of the growth surface. Thus, understanding the mechanism of nucleation and growth kinetics is very important to improving material quality. A promising method for *in situ* monitoring involves the use of high intensity coherent x-rays, such as from a synchrotron light source, and includes small angle reflectance/scattering, diffraction, and fluorescence.

We present initial *in situ* studies of the PA-ALE process using synchrotron x-ray radiation, and grazing incidence small angle x-ray scattering (GISAXS) , x-ray reflectivity (XRR), and in-plane x-ray diffraction (XRD) measurements. Investigations focus on the *in situ* surface preparation process and initial stages of epitaxial growth of AlN and InN on GaN template layers. Experiments were conducted in a custom PA-ALE growth facility installed at beamline X21 of the National Synchrotron Light Source. Surface evolution during the *in situ* surface preparation process was monitored by GISAXS and the nucleation and growth processes for AlN and InN were monitored using GISAXS, XRR, and in-plane XRD. Atomic force microscopy (AFM), x-ray photoelectron spectroscopy and out-of-plane XRD were employed as post growth characterizations.

In situ XRR measurements of an optimized growth process for AlN on a 450°C substrate revealed a 0.08 nm/cycle growth rate and clearly shows each half-cycle of the AlN growth process. *Ex situ* AFM measurements confirm that the surface roughness after growth was similar (RMS roughness =0.74 nm) to that of the GaN substrate. We compare the *in situ* in-plane synchrotron XRD study with previous reports[2]of AlN/GaN grown in a Cambridge Nanotech Fiji reactor to assess the material quality grown in the *in situ* chamber. The in-plane XRD measurement on pre-grown ALE AlN confirms the epitaxial nature and wurtzite structure with 60 degree symmetry in Φ-scan. These early results demonstrate that *in situ* synchrotron x-ray characterization methods are a powerful tool for exploring the epitaxial nucleation and growth mechanisms of III-nitride layers by PA-ALE.

1. N. Nepal, et al., J. Cryst. Growth and Des. **13**, 1485 (2013).

2. N. Nepal, et al., Appl. Phys. Lett. **103**, 082110 (2013).

9:40am **SA-MoM5 Application of Synchrotron Radiation Based Hard X-ray Photoelectron Spectroscopy (HAXPES) to Characterise Semiconductor Device Structures**, ***Greg Hughes***, *L. Walsh,* Dublin City University, Ireland, *J.C. Woicik,* National Institute of Standards and Technology (NIST), *P.K. Hurley*, Tyndall National Institute, Ireland **INVITED**

Hard x-ray photoelectron spectroscopy (HAXPES) is emerging as a technique which has the capability to provide chemical and electronic information on much larger depth scales than conventional XPS. This has potential applications in the study of oxide/semiconductor and metal/semiconductor buried interfaces found in device structures, particularly after annealing cycles. In this presentation results of combined hard x-ray photoelectron spectroscopy (HAXPES) and electrical characterisation measurements on identical Si and III-V based metal-oxide-semiconductor (MOS) structures will be presented. The experimental findings obtained indicate that surface potential changes at the semiconductor/dielectricinterface due to the presence of a thin metal gate layer can be detected with HAXPES. Changes in the semiconductor band bending at zero gate voltage and the flat band voltage for the case of metal gate layers derived from the semiconductor core level shifts observed in the

HAXPES spectra are in agreement with values derived from C-V measurements.

The III-V material InGaAs, shows promise as the channel material in high speed n-MOSFETs however, the issue of low resistance source/drain (S/D) contacts to InGaAs remains. A possible solution is to find a self-aligned silicide like material (salicide) to act as the S/D contacts. The search for this material has recently focussed on Ni-InGaAs, due to its promisingly low R_s and its apparent abrupt interface with InGaAs. Results of a HAXPES study of the Ni-InGaAs alloy system has been undertaken in order to determine the nature of the Ni-InGaAs interface and its evolution as a function of annealing temperature. The results show that Ni readily interacts with InGaAs upon deposition at room temperature resulting in significant inter-diffusion and the formation of NiIn, NiGa, and NiAs alloys. This information when combined with x-ray absorption spectroscopy (XAS) measurements can be used to develop a structural and chemical compositional model of the Ni-InGaAs system as it evolves over a thermal annealing range of 250-500 °C.

10:40am **SA-MoM8 Correlative Probing of the Surface Chemistry and Electron Transport of Nanodevices in Operando Mode using Scanning Photoelectron Emission Microscopy**, ***Andrei Kolmakov***, National Institute of Standards and Technology (NIST) **INVITED**

The surface as well as interfacial properties of nanoscopic devices are intimately linked to their electronic transport properties. In addition, they have a strong dependence on their dimensions, faceting and stoichiometry. As a result, the traditional measurements on the ensembles of nanostructures would suffer from significant averaging effects and need to be replaced with testing of individual well characterized nanostructure. In this report, we demonstrate few examples of correlative imaging, spectroscopy and transport measurements on individual working nanodevices using capabilities of modern synchrotron radiation based photoelectron microscopy. In particular, the surface analysis of the operating MEMS nanowire sensor model device being coupled with scanning x-ray beam induced current microscopy correlates real time changes in conductance of the nanowire with formation of the specific surface groups upon redox reaction. The effect of the electrodes and electroactive defects in the devices on their performance will be discussed. The perspectives of the in operando device characterization at real world pressures and temperatures will be outlined.

11:20am **SA-MoM10 A NEXAFS Spectromicroscope for Structural and Chemical Imaging Analysis**, ***Conan Weiland***, Synchrotron Research, Inc., *Z. Fu, C. Jaye, D.A. Fischer*, National Institute of Standards and Technology, *K. Scammon*, University of Central Florida, *P.E. Sobol, E.L. Principe*, Synchrotron Research, Inc. **INVITED**

We present the development of a Large Area Rapid Imaging Analytical Tool (LARIAT MKII) for near edge x-ray absorption fine structure (NEXAFS) surface chemical and structural analysis. This analyzer utilizes magnetostatic and grid-less electrostatic lenses to maintain the lateral distribution of electrons into a 16 mega channel detector, allowing for a 180° collection angle for high collection efficiency enabling rapid parallel imaging. The system is in development for installation at the NIST SST beamline at NSLS II. Initial images from LARIAT MKII, currently installed at NSLS, will also be presented. The images demonstrate the system's imaging capabilities, with resolution approaching 5 µm for C K-edge images.

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