

Tuesday Morning, November 11, 2014

Novel Trends in Synchrotron and FEL-Based Analysis

Focus Topic

Room: 312 - Session SA-TuM

Characterization of Nanostructured and LD Materials Using Synchrotron-Based Methods

Moderator: Maya Kiskinova, Elettra-Sincrotrone Trieste, Italy

8:00am **SA-TuM1 Analysis and Speciation of Nanoscaled Materials by Means of Grazing-Incidence and High-Resolution X-ray Spectrometry.** *Burkhard Beckhoff, M. Gerlach, I. Holfelder, P. Hönicke, J. Lubeck, M. Mueller, A. Nutsch, B. Pollakowski, C. Streeck, R. Unterumsberger, J. Weser*, Physikalisch-Technische Bundesanstalt (PTB), Germany **INVITED**

The development of efficient nanoscaled materials requires the correlation of the materials' functionality with their chemical and physical properties. To probe these properties, analytical methods that are both sensitive and selective at the nanoscale are required. The reliability of most analytical methods relies on the availability of reference materials or calibration samples, the spatial elemental composition of which is as similar as possible to the matrix of the specimens of interest. However, there is a drastic lack of reference materials at the nanoscale. We address this challenge by means of a bottom-up X-ray analytical method where all instrumental and experimental parameters are determined with a known contribution to the uncertainty of the analytical results. This approach does not require any reference materials but a complete characterization of the analytical instruments' characteristics. X-ray spectrometric methods allow for the variation of the analytical sensitivity, selectivity, and information depth needed to effectively reveal the spatial, elemental, and chemical specimen parameters of interest. Examples of interfacial speciation, elemental depth profiling as well as layer composition and thickness characterizations in various nanoscaled materials will be given.

References

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- [7] C. Streeck, S. Brunken, M. Gerlach, C. Herzog, P. Hönicke, C.A. Kaufmann, J. Lubeck, B. Pollakowski, R. Unterumsberger, A. Weber, B. Beckhoff, B. Kanngießner, H.-W. Schock and R. Mainz, *Appl. Phys. Lett.* 103 (2013) 113904
- [8] J. Lubeck, B. Beckhoff, R. Fliegau, I. Holfelder, M. Müller, B. Pollakowski, F. Reinhardt and J. Weser, *Rev. Sci. Instrum.* 84 (2013) 045106
- [9] I. Holfelder, B. Beckhoff, R. Fliegau, P. Hönicke, A. Nutsch, P. Petrik, G. Roeder and J. Weser, *J. Anal. At. Spectrom.* 28 (2013) 549

8:40am **SA-TuM3 X-Ray Photoemission Spectromicroscopy: Recent Achievements and Future Applications.** *Claus Schneider*, Forschungszentrum Juelich GmbH, Germany **INVITED**

Complex material systems play a pivotal role in many areas of modern technology, such as information storage and processing or energy conversion and storage. In particular, modern information technology exploits the full potential of very different material systems for the meticulous control of alternative state variables. These state variables are used to represent the individual information bits and may be electron charges in semiconductor nanoelectronics, electron spins in the case of spintronics, or local redox configurations in resistive switching elements. Due to this broad range, the materials encompass intermetallic compounds, oxides or chalcogenides, elementary and compound semiconductors or even molecular components. In addition, the functional elements, for example,

individual memory cells or transistor structures often involve nanometer dimensions and operate on nanosecond timescales or even below. This imposes considerable challenges on the characterization of electronic, chemical and magnetic states in the steady state or during operation.

Immersion lens microscopy with synchrotron radiation has matured into a versatile and powerful tool to investigate a broad range of issues in condensed matter physics and materials science. In its energy-filtered version photoemission microscopy combines high-resolution imaging with spectroscopic capabilities in a unique fashion, enabling access to valence and core electronic states. The excitation with photons ranging from the soft to the hard x-ray regime ensures element selectivity and variable information depth. Choosing the polarization state of the synchrotron radiation enables one to distinguish different states of magnetic order, such as ferro- and antiferromagnets. The intrinsic time structure of the synchrotron radiation permits the study of processes with picosecond time-resolution via pump-probe approaches.

This contribution will review the present status of x-ray photoemission spectromicroscopy with emphasis on applications in information technology. In particular, we will cover model systems in spintronics and in resistive switching [1,2]. The results address both static properties and dynamic processes. We will also discuss new developments, such as photoemission microscopy with hard x-rays and the implementation of imaging spin polarimetry.

Y. Aoki, et al., *Nat. Commun.* (2014) 3473.

C. Lenser et al., *Adv. Funct. Mater.* (2014) online publ.

9:20am **SA-TuM5 Growth and Characterization of Low Dimensional Materials for Applications in Energy and Sensor Devices.** *Andrea Goldoni*, Elettra-Sincrotrone Trieste, Italy **INVITED**

Nanostructured low dimensional systems, in particular carbon nanotubes and oxide nanopyllars, have a number of aspects that make them suitable for applications in the fields of environmental gas sensors and energy devices. Here we present the growth and characterization of some nanostructures of interest [1-3] made by well-ordered tin oxide nanopyllars on ITO and hybrid/functionalized carbon nanostructures, suitable both as electrodes for solar and electrochemical cells, as well as for extremely fast (and sensitive) gas sensors [4-6].

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[2] L. D'Arsiè et al., "Tubular Sn-Filled Carbon Nanostructures on Indium Tin Oxide: Nanocomposite material for multiple applications", *Carbon* 65, 13 (2013).

[3] P. Mbuyisa et al., "Controlled Synthesis of Carbon-Based Nanostructures Using Aligned ZnO Nanorods as Templates", *Carbon* 50, 5472 (2012).

[4] F.M. Toma et al., "Efficient Water Oxidation at Carbon Nanotube/Polyoxometalate Electrocatalytic Interfaces", *Nature Chemistry* 2, 826 (2010); S. Piccinin, et al., "Unifying Concepts in Water Oxidation Catalysis: Thermodynamic and kinetic pathways powered by a molecular, tetra ruthenium-oxo complex as the blue-print of metal-oxide surfaces", *PNAS* 110, 4917 - 4922 (2013).

[5] F. Rigoni et al., "Enhancing the sensitivity of pristine carbon nanotubes to detect low-ppb ammonia concentrations in the environment", *Analyst* 138, 7392 (2013); F. Rigoni et al., "Water selective, high sensitivity gas sensors based on single-wall carbon nanotubes functionalized with indium tin oxide nanoparticles: towards low ppb detection of ammonia concentrations in the environment", submitted (2014).

[6] V. Aljani et al., "Extremely Sensitive and Fast Sensors Made of Vertical Nanostructures of SnOx on Indium Tin Oxide", submitted (2014).

11:00am **SA-TuM10 Novel 2D Electron Gases at the Surface of Transition-Metal Oxides: Role of Topology and Spin-Orbit Coupling.** *Andrés F. Santander-Syro*, Université Paris-Sud, France **INVITED**

Novel 2D electron gases at the surface of transition-metal oxides:

role of topology and spin-orbit coupling

Andrés F. Santander-Syro

CSNSM – Université Paris-Sud (France)

Transition-metal oxides (TMOs) are correlated-electron systems with remarkable properties, such as high-temperature superconductivity or large magnetoresistance. The realization of two-dimensional electron gases (2DEGs) at surfaces or interfaces of TMOs, a field of current active

research, is crucial for harnessing the functionalities of these materials for future applications. Additionally, these 2DEGs offer the possibility to explore new physics emerging from the combined effects of electron correlations and low-dimensional confinement.

Recently, we discovered that a 2DEG is simply realized at the vacuum-cleaved surface of SrTiO₃, a transparent, insulating TMO with a gap of 3.5 eV. We directly imaged its multiple heavy and light subbands using angle-resolved photoemission spectroscopy [1]. In this talk, I will show that one can also create and tailor 2DEGs in other TMO surfaces, opening vast possibilities for the study of correlations in low dimensions in materials showing diverse functionalities. I will first discuss the specific case of KTaO₃, a wide-gap insulator with a spin-orbit coupling 30 times larger than in SrTiO₃. I will show that quasi-2D confinement in this system results in comparable scales for the Fermi energy, the subband splitting, and the spin-orbit coupling, leading to a complete reconstruction of the orbital symmetries and band masses [2]. Then, I will show that by choosing various surface terminations of different symmetries one can modify the electronic structure of the 2DEGs at the surface of TMOs [3]. Additionally, I will discuss the experimentally observed effects of spin-orbit coupling in the 2DEG at the surface of SrTiO₃. All these results demonstrate that, in TMOs, the strong correlations, together with the electron confinement and the surface-lattice symmetry, can lead to novel states at the surface that are not simple extensions of the bulk bands.

[1] A. F. Santander-Syro *et al.*, Nature **469**, 189 (2011).

[2] A. F. Santander-Syro *et al.*, Phys. Rev. B **86**, 121107(R) (2012).

[3] C. Bareille *et al.*, Sci. Rep. **4**, 3586 (2014).

11:40am **SA-TuM12 Effects of Interfacial Interaction: Electronic Structure of Graphene on Metallic and Insulating Surfaces, Petra Rudolf**, University of Groningen, The Netherlands **INVITED**

The interaction of graphene with substrates and its influence on the electronic properties is of paramount importance for designing novel electronic and optoelectronic devices. However, the capability of disentangling the surface contribution of the support from those of the graphene single layer is a challenging and unresolved problem. In this contribution I shall present results concerning the electronic states of a typical metallic interface, namely graphene/Cu, where the coupling is weak. These results will be compared to the electronic states of suspended graphene and graphene on an insulating substrate.

When the properties of the occupied electronic band structure were investigated by angle-resolved photoelectron spectromicroscopy, we demonstrated that a suspended CVD grown graphene membrane locally shows electronic properties comparable with those of samples prepared by micromechanical cleaving of graphite. CVD grown graphene on the Cu(111) surface was instead found to be slightly doped, while on an insulator surface it was demonstrated to fully preserve the intrinsic properties, implying that the graphene monolayer is totally decoupled as if it were freestanding and not doped.

The unoccupied surface states at the weakly coupled graphene/Cu interface were studied by non-linear angle resolved photoemission spectroscopy. In particular, by comparing the band dispersion of the unoccupied image potential states and the occupied surface states of the interfaces graphene/Cu(111) and graphene/ polycrystalline copper foil, we were able to identify and characterise the Shockley surface state and the n=1 image state of the Cu(111) surface and the symmetric n=1 image state of the single layer of graphene.

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