DS 35: Thin Film Characterisation: Structure Analysis and Composition II

Time: Wednesday 15:00–18:15 Location: H8

DS 35.1 Wed 15:00 H8

Transrotational microcrystals: novel solid state order formed in amorphous films — •Vladimir Yu. Kolosov — Ural Federal University, Ekaterinburg, Russia

Exotic thin crystals with unexpected **transrotational** microstructures [1] have been discovered by transmission electron microscopy (TEM) for crystal growth in thin (10-100 nm) amorphous films of different chemical nature (oxides, chalcogenides, metals and alloys) prepared by various methods. Primarily we use TEM bend contour technique for crystallographic orientation analysis [2]. The unusual phenomenon can be observed ***in situ*** in TEM column: dislocation independent regular internal bending of crystal lattice planes in a growing crystal. Such transrotation (unit cell **trans**lation is complicated by small **rotation** realized round an axis lying in the film plane) results in strong regular lattice orientation gradients (up to 300 degrees per micrometer) of different geometries: cylindrical, ellipsoidal, toroidal, saddle, etc. Transrotation is strongly increasing as the film gets thinner. Transrotational crystal resembles ideal single crystal enclosed in a curved space. Transrotational micro crystals have been eventually recognized by other authors for some vital thin film materials, i.e. PCMs (phase-chage materials) for memory, silicides, SrTiO3. Atomic model and possible mechanisms of the phenomenon are discussed.

Basing on the above findings we also propose new hypothetic transrotational nanocrystalline model of amorphous state.

[1] V.Yu. Kolosov and A.R.Tholen, Acta Mater., **48** (2000) 1829.
[2] I. E. Bolotov and V. Yu. Kolosov, Phys. Stat. Sol. **69**a (1982) 85.

DS 35.2 Wed 15:15 H8

Electronic properties of $LaPO_4$ nanoparticles studied by the-hard X-Ray photoelectron spectroscopy. — •A. Gloskovskii[1], Ya. Chornodolskyy[2], V. Vistovskyy[2], O. Shevchuk[3], O. Myagkota[3], S. Syrotyuk[3], A. Zaichenko[3], A. Voloshinovskii[2], and W. Drube[1] — [1]Photon Science/DESY, Hamburg — [2]Ivan Franko National University of Lviv, Ukraine — [3]Lviv Polytechnic National University, Ukraine

Photoelectron spectroscopy using excitation by hard X-rays in the range of 2 - 15 eV (HAXPES) is rapidly developing at synchrotron light sources worldwide. Its comparatively large probing depth (10-30 nm) makes it a powerful tool for studies of complex materials, buried nanostructures and multilayered structures relevant for device applications. We are presenting a new approach for HAXPES of insulating nanoparticles by covering them with thin metal shell. It makes it feasible to probe density of occupied states (DOS) for insulating nanoparticles. $LaPO_4$-Eu nanoparticles with mean grain size of 50-60 nm were covered by 1 nm Ag resulting in core-shell particles. DOS of conductive silver shell is not overlapping with DOS of wide bandgap $LaPO_4$. We carried out HAXPES measurements with variable linear light polarizations using 5.95 keV photons. The intensity of *s*-states can be considerably suppressed by rotating the light polarization perpendicularly to the analyzer's axis. In this way the extraction of the *s*- and *p*- contributions to $LaPO_4$ valence band becomes feasible. Experimentally measured valence band DOS correlates well with calculations using projector augemented wave method.

DS 35.3 Wed 15:30 H8

Determination of thickness and refractive index of ulta-thin films via ellipsometry — Peter Nestler and •Christiane A. Helm — Institut für Physik, Greifswald University, D-17487 Greifswald, Germany

Ellipsometric measurements of layered media deliver information on film parameters like layer thickness and refractive indices with high precision. In the simplest case the substrate is covered with a single homogenous, transparent layer only. Yet, the simultaneous determination of both parameters, thickness and refractive index of the layer, via ellipsometry is immediately possible only if the layer thickness exceeds 15 nm.

Here we present a technique to cross this limitation: A series expansion of the ellipsometric ratio ρ to the second order of the layer thickness relative to the wavelength reveals the first and second ellipsometric moment. These moments are properties of the layered surface and independent of incident angle. Using both moments and one additional reference measurement of the bare substrate enables to simultaneously determine both thickness and refractive index of ultra-thin layers down to 5 nm thickness.

DS 35.4 Wed 15:45 H8

Depth-resolved image of the LaAlO3/SrTiO3 system from Resonant Soft X-ray Reflectivity — •Martin Zwiebler[1], Fabio Miletto Granozio[2], Emiliano Di Gennaro[2], Jorge Enrique Hamann-Borrero[1], Enrico Schierle[3], Eugen Weschke[3], Jochen Geck[4], Marco Salluzzo[5], and Umberto Scotti di Uccio[5] — [1]Leibniz Institute for Solid State and Materials Research, IFW-Dresden, Helmholtzstr. 20, 01069 Dresden, Germany — [2]CNR-SPIN and Dipartimento di Fisica, Complesso universitario di Monte S.Angelo Via Cinti, 80126, Naples, Italy — [3]Helmholtz-Zentrum Berlin für Materialien und Energie, Albert Einstein-Str. 15, 12489 Berlin, Germany — [4]Universität Salzburg, Hellbrunner Str. 34, 5020 Salzburg, Austria — [5]2CNR-SPIN and Dipartimento di Fisica, Complesso universitario di Monte S.Angelo Via Cinti, 80126, Naples, Italy

When LaAlO3 is grown epitaxially on a 001 Ti-terminated SrTiO3 substrate, a conductive 2D electron system (2DES) emerges, even though both parent compounds are bulk insulators. The conductivity of the 2DES is (i) tunable using the field effect, (ii) very sensitive to photon irradiation and (iii) can even show superconductivity at low temperatures. The reasons for the emergence of the 2DES at the STO/LAO interface remain elusive. Popular and intensively discussed scenarios include the layer polarity change at the interface, chemical doping via oxygen vacancies, interdiffusion, and structural changes. Here we show depth-resolved spectroscopic information, obtained from Resonant Soft X-Ray Reflectivity at the Ti L2,3 edge. Our results provide new insights about chemical interdiffusion, charge accumulation and structural distortions in these systems, which clarify the emergence of the 2DES and its response to photon irradiation.

DS 35.5 Wed 16:00 H8

Analysis of interface properties of multilayer mirrors with sub-nanometer layer thicknesses — •Anton Haase[1], Saša Bajt[2], Victor Soltwisch[1], Philipp Hönicke[1], Christian Laubis[1], and Frank Scholze[1] — [1]Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin — [2]Photon Science, DESY, Notkestr. 85, 22607 Hamburg

The spectral range from 2.2 nm to 4.4 nm, also known as the water window, has a high relevance in the investigation of biological samples in a wet environment due to the low absorbance of these particular wavelengths in water. Multilayers composed out of Cr and Sc provide enough optical contrast to serve as mirrors for this wavelength range, while maintaining low absorption. However, a respective choice of layer thicknesses down to the sub-nanometer regime directly related to the desired peak reflectivity at a certain wavelength and angle of incidence are required. Disturbances of interfaces with respect to the ideal multilayer such as interdiffusion and roughness diminish the theoretically achievable maximum reflectivity. Experimental reflectivities show values below 20%, i.e. less than half of the theoretically achievable maximum. We show how the combination of several complementary experiments, such as EUV and X-ray reflectivity, resonant EUV reflectivity at the L-edge and X-ray standing wave analysis are necessary to obtain a consistent model. Based on this results a analysis of the diffuse scattering is performed to asses the interface roughness and determine a effective power spectral density.

DS 35.6 Wed 16:15 H8

Sample thickness in Transmission Kikuchi Diffraction via Monte Carlo Simulations — •Nathanael Jöhrmann and Michael Hietschold — Institut für Physik, TU Chemnitz, Deutschland

Transmission Kikuchi Diffraction in a Scanning Electron Microscope is an interesting modification of Electron Backscatter Diffraction to get information about crystalline structures with high spatial resolution [1]. The achievable lateral resolution and the measurement time depend strongly on several parameters, particularly on sample thickness, sample tilt and acceleration voltage. To better understand measurement results, especially unindexed areas, and to optimize sample preparation and TKD setup, it would be helpful to get information about sample thickness as additional information of a measurement.

To achieve this, the background signal of measured diffraction patterns is compared with Monte Carlo simulations.

[1] R. R. Keller und R. H. Geiss, Journal of Microscopy 245 (2011), pp. 245-251

15 min. break.

DS 35.7 Wed 16:45 H8

Chemical Characterization of electrodeposited transition metal chalcogenite layers — Talha Nisar, •Torsten Balster, and Veit Wagner — Jacobs University Bremen gGmbH, Campus Ring 1, 28759 Bremen, Germany

Transition metal chalcogenides are promising materials for catalysis as well as semiconducting layers in thin film transistors, e.g. amorphous molybdenum and tungsten sulfide layers are successfully applied for water electrolysis.
In this study, we investigated the electrochemical deposition of ammonium tetrathiomolybdate (ATTM) and the subsequent annealing steps by means of x-ray photoelectron spectroscopy. The electrochemical deposition was carried out with a concentration of 0.5 mmol ATTM at room temperature in the cathodic regime with respect to Ag/Ag-Cl reference electrode. This resulted in homogenous layers with thicknesses of MoS_x (x=2..3) from 1 up to 10 nm. This layer show promising morphology to cover also complex surfaces. The deposition process results in a minor oxygen and carbon contamination, which can be reduced by proper post growth annealing conditions. Further annealing steps in an inert Ar (99.999%) atmosphere to improve crystallinity resulted in a reduction of the S content of the surface, which can be avoided in a S-containing atmosphere.

DS 35.8 Wed 17:00 H8

Surface chemistry of free-base corrole on Ag(111): Complementary insights from X-ray spectroscopy, DFT and STM. — •Mateusz Paszkiewicz[1], Stefano Tebi[2], Hazem Aldahhak[3], Wolfgang Schöfberger[4], Stefan Müllegger[2], Uwe Gerstmann[3], Eva Rauls[3], Wolf Gero Schmidt[3], Reinhold Koch[2], David Duncan[1], Francesco Allegretti[1], Johannes Barth[1], and Florian Klappenberger[1] — [1]Physic department, E20, Technische Universität München, Garching, Germany — [2]JKU, Solid state physics department, Linz, Austria — [3]University of Paderborn, Physik department, Paderborn, Germany — [4]JKU, Institute of organic chemistry, Linz, Austria

The investigated fluorophenyl-free base corrole (3H-C) is famous for its ability to act as a three-fold anionic ligand and for stabilizing metals in high oxidation states. Our multitechnic study combines X-ray photoelectron spectroscopy (XPS) and near-edge X-ray absorption-fine-structure (NEXAFS) spectroscopy with density functional theory (DFT) simulations and scanning tunneling microscopy (STM). First, we present a spectroscopic reference of all relevant regions and edges (C1s, F1, and N1s regions for XPS and C and N K-edge for NEXAFS) of the 3H-C multilayer and interpret their features on the basis of the theoretic investigations. Then, we unravel the influence of the molecule surface interaction. At room temperature we observe unordered layers of partially deprotonated species (2H-C) exhibiting a near-to-planar macrocycle. After annealing to around 400 K, ordered layers of stacked molecules are present with an intriguing spectroscopic signature.

DS 35.9 Wed 17:15 H8

Dependence of Optoelectronic Properties on Composition in CuInSe2 — •Sergiu Levcenco, Helena Stange, Roland Mainz, and Thomas Unold — Hahn-Meitner-Platz 1 D-14109 Berlin, Germany

Thin film solar cells based on chalcopyrite absorber have demonstrated more than 21% efficiency although the material used is strongly non-stoichiometric, and thus contains large quantities of defects. To investigate the effect of composition on the optoelectronic properties CuInSe2 samples were grown by coevaporation in with an intentional lateral variation of composition, leading to Cu-poor, Cu-rich and stoichiometric regions within the same sample. Photoluminescence, Raman spectroscopy and reflectometry was performed to investigate the optical and structural properties and recombination processes within on sample. The band gap is found to continuously increase when the composition approaches stoichiometry, whereas the recombination physics yields a much more complex behaviour.

DS 35.10 Wed 17:30 H8

Nanostructured surface of multilayer graphene on 3C-SiC (001) — •Victor Aristov[1,2,3], Olga Molodtsova[1,4], Sergey Babenkov[1], Dmitry Marchenko[5], Jaime Sánchez-Barriga[5], Partha Sarathi Mandal[5], Andrei Varykhalov[5], Alexei Zakharov[6], Yuran Niu[6], Alexei Preobrajenski[6], Denis Vyalikh[7], Marc Portail[8], Marcin Zielinski[9], Barry Murphy[10], Sergey Krasnikov[10], Olaf Luebben[10], Igor Shvets[10], and Alexander Chaika[2,10] — [1]DESY, Hamburg, Germany — [2]ISSP RAS, Chernogolovka, Russia — [3]TU Bergakademie, Freiberg, Germany — [4]ITMO, Saint Petersburg, Russia — [5]BESSY, Berlin, Germany — [6]Max-lab, Lund, Sweden — [7]TU Dresden, Germany — [8]CNRS-CRHEA Valbonne, France — [9]NOVASiC Le Bourget du Lac, France — [10]Trinity College, Dublin, Ireland

The results of atomically resolved scanning tunneling microscopy, low energy electron diffraction, low energy electron microscopy, micro-LEED and angle resolved photoelectron spectroscopy studies of graphene synthesized on cubic-SiC(001) will be presented. Uniform few layer graphene was fabricated on SiC(001)/Si(001) wafers using Si-atom sublimation followed by SiC surface layer graphitization during high-temperature annealing in ultrahigh vacuum. This work was supported by the RAS, RFBR grants No 140200949 and 140201234, by the BMBF-Project No. 05K12GU2, PSP-Element No. U4606BMB1211, by a Marie Curie IIF grant No 12/IA/1264, by SPP 1459 of DFG.

DS 35.11 Wed 17:45 H8

Study of amorphous chalcogenide alloys by optical and electrical investigation — •Ju-Young Cho[1] and Matthias Wuttig[1,2] — [1]I. Physikalisches Institut (IA), RWTH Aachen University, 52056 Aachen, Germany — [2]JARA-FIT, RWTH Aachen University, Germany

Amorphous phase change materials (PCMs) show unusual phenomena compared to ordinary covalent glass. Amorphous PCMs usually show significantly different local chemical order as compared with their crystalline phase, while ordinary covalent glass exhibits a similar local chemical order as compared with its crystalline counterpart. Unraveling the local structure and the relevant characteristics is highly rewarding task because this is a key to understand the unique features of the amorphous PCMs, e.g. the extremely rapid crystallization, high fragility as well as temporal drift of the electrical resistance.

When alloying PCMs, constitute elements and compositional variation significantly affect the local chemical order of amorphous PCMs. This dependency becomes more obvious when PCMs and non-PCMs are alloyed. In this study, we have performed FT-IR and Raman spectroscopy, Van der Pauw and X-ray reflectivity measurements of co-sputtered amorphous chalcogenide alloy films with various compositions and elements to exploit the change in the structural properties as well as the crystallization kinetics of PCMs. The resulting insight will reveal the correlation between the local chemical order and the properties varying with composition, possibly enabling the prediction of phase change memory characteristics.

DS 35.12 Wed 18:00 H8

Quantitative AM-FM Mode for Fast, Versatile Imaging of Nanoscale Elastic Modulus — Marta Kocun, Aleksander Labuda, Waiman Meinhold, •Florian Johann, and Roger Proksch — Asylum Research, an Oxford Instruments Company, Wiesbaden, Germany

Tapping mode AFM imaging, also known as amplitude-modulated (AM) atomic force microscopy (AFM), is fast, gentle and provides the high spatial resolution necessary for imaging nanoscale features. However, until recently, mechanical characterization with tapping mode was limited to only qualitative results. In AM-FM mode, a bimodal (dual-frequency) technique, the first resonant mode is operated in AM, whereas a higher resonant mode is frequency modulated (FM). As expected from regular tapping mode, AM-FM mode delivers topographical information. Additionally, it provides quantitative data on contact stiffness, from which elastic modulus can be calculated with appropriate models for the tip-sample contact mechanics. Experimental results on different samples such as metals, alloys and polymers will be presented to demonstrate the applicability of AM-FM mode for materials with a wide range of modulus (MPa-GPa).