# DS 27: Thin Film Characterization: Structure Analysis and Composition (XRD, TEM, XPS, SIMS, RBS,...) I

Time: Wednesday 17:15–19:45 Location: H32

DS 27.1 Wed 17:15 H32

**In-situ GISAXS study of the gold electrode growth on spiro-OMeTAD films** — •Martin A. Niedermeier[1], Ezzeldin Metwalli[1], Volker Körstgens[1], Jan Perlich[2], Ralf Röhlsberger[2], Stephan V. Roth[2], and Peter Müller-Buschbaum[1] — [1]TU München, Physik Department, LS Funktionelle Materialien, James-Franck-Str. 1, 85748 Garching, Germany — [2]HASYLAB at DESY, Notkestraße 85, 22603 Hamburg, Germany

In the fast developing field of organic electronics, thin metal films play a huge role as they are commonly applied as electrode materials. One prominent example is the field of photovoltaics which has taken on greater significance in the recent years due the energy challenges of modern society. A well-defined organic/metal interface is crucial for a good overall device performance. For a profound understanding of that interface, the morphology and the incorporation of the electrode material into the organic film is essential. However, so far only little fundamental knowledge is available on this topic.

In this work, we present an in-situ investigation of the gold electrode growth via sputter deposition on top of a spiro-OMeTAD film, which is a commonly used system in solid-state dye sensitized solar cells. We used grazing incidence small angle x-ray scattering (GISAXS) with high time resolution to follow the real time evolution of the gold electrode growth and obtain information about the growth kinetics. Additionally, enrichment layers of gold into the organic film are investigated with x-ray reflectivity. Therefore, we obtain a good fundamental understanding of the electrode/organic semiconductor interface.

DS 27.2 Wed 17:30 H32

**Gold Cluster Growth Kinetics during Sputter Deposition** — •Matthias Schwartzkopf[1], Adeline Buffet[1], Volker Körstgens[2], Ezzeldin Metwalli[2], Kai Schlage[2], André Rothkirch[1], Monika Rawolle[2], Jan Perlich[1], Gerd Herzog[1], Berit Heidmann[1], Ralf Röhlsberger[1], Peter Müller-Buschbaum[2], Rainer Gehrke[1], and Stephan V. Roth[1] — [1]DESY, Notkestr. 85, D-22607 Hamburg, Germany — [2]TU München, James-Franck-Str. 1, D-85747 Garching, Germany

The adjustment of size-dependent catalytic, electrical and optical properties of gold cluster assemblies is a very significant issue in modern applied nanotechnology. We investigate in situ the growth kinetics of gold nanostructures to a gold layer during magnetron sputter deposition with high time resolution by means of grazing incidence small-angle X-ray scattering (GISAXS). The high time resolution in the millisecond regime allows the determination of kinetics of initial nucleation and subsequent cluster growth during sputter deposition at deposition rates relevant for industrial manufacturing processes. Morphological parameters related to the growth of metallic layers such as cluster size, correlation distance and surface coverage are deduced from a general model solely based on geometrical assumptions. The temporal evolution of the model parameters reveals four stages of gold cluster growth, namely initial nucleation, diffusion-mediated coalescence, adsorption-driven cluster growth and finally grain growth above the percolation threshold. Furthermore, our study opens up the opportunity to deduce the wetting behaviour of gold nanoclusters on solid substrates.

DS 27.3 Wed 17:45 H32

**Growth kinetics of metal nanoparticles on polymer surfaces** — •Ezzeldin Metwalli[1], Volker Körstgens[1], Adeline Buffet[2], Jan Perlich[2], Stephan V. Roth[2], and Peter Müller-Buschbaum[1] — [1]TU München, LS für Funktionelle Materialien, Physik-Department, James-Franck-Str. 1, 85747 Garching, Germany — [2]HASYLAB at DESY, Notkestr. 85, 22603 Hamburg, Germany

Metal layers on polymer films [1-4] are essential for many important nanodevices such as organic solar cells and light emitting diodes. Gold in its atomic state is deposited on several homopolymer and block copolymer films by utilizing a DC magnetron sputtering deposition system. With the unprecedented time resolution of 15 milliseconds, the nucleation/growth kinetics of gold nanoparticles on the polymer surfaces is monitored using in situ real-time grazing incidence small angle x-ray scattering (GISAXS) technique. An exponential growth of metal particle size on all polymer surfaces is observed prior to the formation of a quasi uniform metal layer. Below a certain critical particle size, an initial fast particle growth is due to high particle mobility. While a slower kinetics at concentrated metal dispersion is due to the strong metal-metal interactions. This novel kinetic study of metal growth on chemically different homopolymer films clearly explains the high selectivity characteristics of metals towards a particular block of diblock copolymer nanotemplates. 1. E.Metwalli et al. Langmuir 24, 4265 (2008) 2. E.Metwalli et al. Langmuir 25, 11815 (2009) 3. G.Kaune, E.Metwalli et al. ACS Appl. Mater. Interfaces 3, 1055 (2011) 4. X.Xia, E.Metwalli, et al. J. Phys. Condens. Matter 23, 254203 (2011)

DS 27.4 Wed 18:00 H32

**In situ GISAXS characterization of Al sputtering on Alq3 thin film** — •Shun Yu, Gonzalo Santoro, Jan Perlich, Marion Kuhlmann, Johannes F.H. Risch, Matthias Schwartzkopf, and Stephan V. Roth — HASYLAB, DESY, Notkestraße 85, 22607, Hamburg

Organic light emitting diode (OLED) is an important device as the next generation light source for illumination. Its low cost, easy fabrication and relatively high efficiency have attracted many research interests. The multilayer device structure emphasizes the significance of understanding the interfacial structure and properties. In most OLEDs, Al is used as the metal electrical contact and Tris(8-hydroxyquinolinato)aluminium (Alq3) is the activating layer. Upon sputtering on Alq3, Al can diffuse into the organic layer, modifying both the morphological and electronic structures and consequently the device performance. The interaction between Al and Alq3 has been studied by different spectroscopic techniques and theoretical methods at single molecule level. Nevertheless, the growth of the Al thin film on Alq3 is scarcely studied. In this work, we have exploited in situ grazing incident small angle X-ray scattering (GISAXS) technique to monitor the growth of Al thin film on top of Alq3 layer during the high speed sputtering process in real time. As a result, we elucidate three growing stages from the out-of-plane scattering pattern. Meanwhile, both Al and Alq3 films demonstrate good correlation to substrate roughness. The results benefit the comprehension of the general industrial sputtering process.

DS 27.5 Wed 18:15 H32

**In situ study of the texture development during the growth of magnetron sputtered VC thin films** — •Sunil Kotapati[1], Bärbel Krause[1], Marthe Kaufholz[1], Stephen Doyle[2], Miguel Mantilla[3], Michael Stüber[4], Sven Ulrich[4], and Tilo Baumbach[1,2] — [1]Institut für Photonenforschung und Synchrotronstrahlung, KIT, Germany — [2]ANKA, KIT, Germany — [3]Max-Planck-Institut für Intelligente Systeme, Stuttgart, Germany — [4]Institut für Angewandte Materialien - Angewandte Werkstoffphysik, KIT, Germany

Hard coatings deposited by magnetron sputtering play a major role in enhancing the lifetime of machining tools and maintaining their productivity. In situ x-ray experiments during thin film deposition are an extremely useful tool to understand the interplay between the microstructure and macroscopic coating properties. The aim of this study is to develop a model for the structure formation of hard coatings as a function of different process parameters such as growth temperature and bias. For this, several Vanadium carbide (VC1-x) thin films were deposited at different growth conditions. In addition to in situ X-ray powder diffraction, the complementary ex situ methods AFM and TEM were used. The powder rings obtained during deposition reveal that below a transition temperature T˜250°C, a mixture of [111] and [200] textures dominates, while the preferred [111] orientation is found above T. The real time measurements also show that there are structural changes as a function of deposition time.

DS 27.6 Wed 18:30 H32

**In situ control of the structure formation of PVD hard coatings: periodic modulation of the microstructure** — •Marthe Kaufholz[1], Bärbel Krause[1], Sunil Kotapati[1], Michael Stueber[2], Sven Ulrich[2], Miguel Mantilla[3], and Tilo Baumbach[1,4] — [1]IPS, KIT, Germany — [2]IAM-AWP,KIT,Germany — [3]MPI for Intelligent Systems, Germany — [4]ANKA, KIT, Germany

Hard coatings such as Vanadium Carbide ($VC_{1-x}$) are nowadays commonly used for improving the life-time of tools. Multilayer systems are one way to enhance the hardness of such coatings. They usually consist of alternating layers of two different materials.
In situ X-ray Reflectivity measurements at the synchrotron ANKA and ex situ Transmission Electron Microscopy studies showed that the density of thin $VC_{1-x}$ films depends on the sputtering conditions. A two layer system with different densities could be grown by varying the gas flow of the sputter gas.
By repeating this process periodically, a multilayer system was successfully created. Such a multilayer system formed by only one material simplifies the synthesis process and is of great interest for optical applications.

DS 27.7 Wed 18:45 H32

**X-ray Grazing Incidence Diffraction from OTS-SAMs on Metal Oxides** — •Hans-Georg Steinrück[1], Stefan Gerth[1], Michael Klimczak[1], Moshe Deutsch[2], Ben Ocko[3], and Andreas Magerl[1] — [1]Lehrstuhl für Kristallographie und Strukturphysik, Universität Erlangen Nürnberg , Germany — [2]Bar-Ilan University, Ramat-Gan, Israel — [3]Brookhaven National Laboratory, Upton NY, USA

We investigated the in-plane structural properties of octadecyltrichlorosilane ($C_{17}H_{37}Cl_3Si$, OTS) self-assembled monolayers on amorphous $SiO_2$, quartz (001) and sapphire (001) via X-ray grazing incident diffraction.

For all three systems a powder like diffraction ring at $\mathbf{q}_r = 1.50\text{Å}^{-1}$ was found. The radial width of these peaks corresponds to a coherence length of about 4 OTS molecular diameters, which fits well with the findings of Tidwell et al. for OTS monolayers on amorphous $SiO_2$ [1].

For OTS on sapphire the azimuthal scans show increasing peak intensities towards the sapphire in-plane peak, suggesting orientational order of the OTS film which is likely related to the good lattice match with the sapphire. This epitaxial behavior depends on the headgroup properties of hydrocarbon chain monolayers, as Ocko et al. have shown perfect commensurabiltiy for $C_{18}H_{37}OH$ on sapphire [2].

Further the temperature dependence of the lateral structure was investigated yielding a decreasing intensity and an increasing lattice spacing with increasing temperature for all three systems.

[1] Tidswell et al., J. Chem. Phys. 95, 2854 (1991)
[2] Ocko et al., Phys. Rev. Lett. 106, 137801 (2011)

DS 27.8 Wed 19:00 H32

**Sol-Gel Type $Ga_{2-x}Fe_xO_3$ Thin Films: Towards Room Temperature Magnetic Oxides with an Ordered Mesoporous Morphology and Nanocrystalline Wall Structure** — •Christian Reitz, Christian Suchomski, Robert Kruk, and Torsten Brezesinski — Institute of Nanotechnology (INT), Karlsruhe Institute of Technology (KIT), 76344 Eggenstein-Leopoldshafen

Both ferri(o)magnetism and ferroelectricity are indispensable in modern technologies and have been used in devices for quite some time. To further increase the performance, the materials employed need to be reduced to the nanometer scale. The same principle also pertains to multiferroics, showing coupling of both magnetic and electric order parameters. Such materials hold great promise for many applications. Among the few single-phase multiferroics that have been discovered to date, $Ga_{2-x}Fe_xO_3$ (GFO) has recently been receiving increasing attention. In this work we focus on ordered large-pore mesoporous GFO thin films with nanocrystalline walls. These materials were produced through facile polymer templating strategies and served as model systems to obtain a better understanding of the relationships between nanoscale structure and ferroic properties. Electron microscopy, GISAXS, XPS, Raman spectroscopy and magnetization and Mössbauer studies confirmed the high-quality of the sol-gel derived thin films with ferrimagnetic ordering at room temperature and provided insight into the underlying coupling phenomena. At this point, it is envisioned that the pore cavities will facilitate the fabrication of novel magnetically exchange-coupled composites.

DS 27.9 Wed 19:15 H32

**Depth dependent influence of thin Fe layers on the magnetic properties of Pd in Pd/Fe multilayers** — •Paul Zakalek, Markus Schmitz, Ulrich Rücker, and Thomas Brückel — Jülich Centre for Neutron Science JCNS und Peter Grünberg Institut PGI, Forschungszentrum Jülich GmbH, D-52425 Jülich, Germany

Proximity effects in magnetic multilayers give rise to new physical properties which are interesting for new spintronic devices. Because of a high Stoner parameter of 0.78, Pd almost fulfills the Stoner-criterion for ferromagnetism and makes this material especially interesting. Due to hybridization with a magnetic material like Fe it is possible to induce a magnetic moment into the Pd layer.

Therefore we investigated Pd/Fe multilayers with sharp defined interfaces (layer roughnesses of less then 8 Å) with SQUID magnetometry, X-ray and polarized neutron reflectrometry measurements. The SQUID measurements could indicate an existing induced magnetic moment of Pd, which was not possible to resolve with polarized neutron reflectrometry measurements.

We will present the preparation and characterization of the Pd/Fe multilayers.

DS 27.10 Wed 19:30 H32

**Formation of supersaturated AuNi nanoparticles via solid state dewetting of Au/Ni thin bilayers** — •Andreas Herz, Dong Wang, and Peter Schaaf — Institut für Werkstofftechnik, Fachgebiet Werkstoffe der Elektrotechnik, Ilmenau, Germany

Thin films undergo agglomeration upon annealing due to their high surface-to-volume ratio which produces a large driving force for reduction of surface area. This process is known as dewetting and can occur well below the melting temperature of the layer material. In recent years, dewetting of thin metal films has therefore become a promising method for fabricating various nanostructures with potential applications in catalysis, plasmonics, or magnetic devices. However, nanoalloys may reveal new or even unique properties due to the combination of size and composition. Thus, thin bilayer systems consisting of a pair of metals, e.g. Au and Ni, could be a convenient basis for the self-organized synthesis of novel alloy nanostructures via dewetting.