MI 5: Quantitative Materialanalyse (mit KR)

Chair: Leonore Wiehl

Time: Tuesday 9:30–12:15 Location: H5

Invited Talk MI 5.1 Tue 9:30 H5

Quantitative Röntgenspektrometrie für die Analyse nanostrukturierter Materialien — •Matthias Müller, Burkhard Beckhoff, Philipp Hönicke, Beatrix Pollakowski, Cornelia Streeck und Rainer Unterumsberger — Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin

Die physikalischen und chemischen Eigenschaften nanostrukturierter Materialen bestimmen maßgeblich deren Funktionalität. Für eine zielführende und effektive Materialentwicklung ist es deshalb entscheidend, dass eine zuverlässige Analytik verfügbar ist. Die quantitative Röntgenspektrometrie hat sich für die Materialcharakterisierung als leistungsfähige und zuverlässige Methode etabliert.

Die Nutzung von Röntgenstrahlung geringer Divergenz an Synchrotronstrahlungslaboren hat die Entwicklung der RFA unter streifendem Einfall ermöglicht, wodurch die tiefensensitive Charakterisierung von Nanoschichtsystemen erreicht wurde. Der Einsatz von durchstimmbarer Synchrotronstrahlung hat die Weiterentwicklung von Quantifizierungsmodellen unterstützt, welche auf der Fundamentalparametermethode basieren. Eine weitere deutliche Verbesserung lässt sich durch den Einsatz radiometrisch kalibrierter Instrumentierung erreichen, wodurch eine referenzprobenfreie Quantifizierung erreicht werden kann.

Der Vortrag gibt einen Überblick über die verschiedenen Quantifizierungsmodelle, deren Vor- und Nachteile sowie die Entwicklungsperspektiven. Anhand aktueller Arbeiten der PTB bei BESSY II werden ausgewählte Anwendungsfelder der quantitativen Röntgenspektrometrie für die Entwicklung nanostrukturierter Materialien vorgestellt.

MI 5.2 Tue 10:15 H5

Advances in Low Energy X-ray Analysis with state of the art Silicon Drift Detectors using EPMA, SEM and STEM — •T. Salge[1], R. Terborg[1], M. Falke[1], O. Tunckan[2], A. Kearsley[3], D. Pereira da Silva Dalto[4], M.J.O.C. Guimarães[4], R. Ferhati[5], I. Bjurhager[6], S. Turan[2], M.E.F. Garcia[4], and W. Bolse[5] — [1]Bruker Nano GmbH, Berlin, Germany — [2]Anadolu University, Eskisehir, Turkey — [3]Natural History Museum, London, UK — [4]UFRJ, Rio de Janeiro, Brazil — [5]University of Stuttgart, Germany — [6]Ångström Laboratory, Uppsala, Sweden

Element analysis of ever smaller structures in bulk samples requires low electron beam energy to enhance spatial resolution. To separate overlapping peaks at low energy X-ray lines (e.g. N-K/Ti-L), the line deconvolution algorithms in EDX software is important. We describe features at the submicron scale (e.g. ceramic-metal joints) using SDDs in conventional geometries. The annular four channel SDD placed between the pole piece and sample covers a large solid angle of 1.1sr. Features with high topography from experiments with wafer irradiation and hypervelocity impact craters can be analyzed as well as beam sensitive polymer composites. For cultural heritage and biological samples, carbon coating can be avoided during low vacuum acquisition. Nano- and atomic scale analysis of electron transparent samples ideally requires not only high solid angle detector design but also adjustments in pole piece and sample holder geometry as well as a high quality electron probe. This approach will be demonstrated to allow 1 sr solid angle and single atom spectroscopy even at 0.1 sr and 60 kV.

MI 5.3 Tue 10:30 H5

Quantitative Analysis of Pyramid Textured Silicon Wafers and Size Dependence of Optical and Electronic Properties — •Jan Kegel[1,2], Heike Angermann[2], Uta Stürzebecher[3], Erhard Conrad[2], and Bert Stegemann[1] — [1]Hochschule für Technik und Wirtschaft, Berlin, Germany — [2]Helmholtz Zentrum Berlin, Berlin, Germany — [3]CiS Forschungsinstitut für Mikrosensorik und Photovoltaik GmbH, Erfurt, Germany

Wet-chemical etching in alkaline solution is used to texture monocrystalline silicon wafers for high-efficiency solar cells. This texturing result in micron-sized random pyramids on the wafer surface which reduce reflection losses and increase the absorption probability. Successful texturing is evaluated by reflection and charge carrier lifetime measurements. Both parameters are found to be influenced by the geometric surface properties as well. Thus, elaborated image processing is applied for precise and reproducible evaluation of pyramid number and size distribution. The results show a distinct dependence of the total reflection and the minority charge carrier lifetime on the pyramid size distribution. Based on these results etching parameters can be adjusted to produce optimal surface properties with respect to highest solar cell efficiencies.

MI 5.4 Tue 10:45 H5

phase diagram of nano-hydride formation: consequences for hydrogen embrittlement — •Gerard Paul Leyson, Blazej Grabowski, Johann von Pezold, and Jörg Neugebauer — Max-Planck-Straße 1, 40237 Düsseldorf, Germany

Local hydride formation around dislocations induces stress-shielding effects and is the underlying mechanism for hydrogen-enhanced local plasticity (HELP). In this work, we present an analytic model for hydride formation around fcc Ni edge dislocations that takes input from atomistic calculations. The hydrogen-hydrogen interaction is modeled using information obtained from a semi-empirical embedded atom method (EAM) potential. Within this approach, the equilibrium concentration and the binding energies of hydrogen around the dislocation are self-consistently calculated. At 300K, local hydride formation is observed with bulk hydrogen concentrations on the order of ~500ppm, consistent previous studies [1]. The onset of nano-hydride formation and with it the activation of the HELP mechanism is predicted through a parametric study of the hydride size as a function of temperature and bulk hydrogen concentration.

[1] von Pezold J, Lymperakis L and Neugebeauer J. Acta Materialia 59 (2011), 2969-2980.

MI 5.5 Tue 11:00 H5

Diffuse scattering and stacking faults in (Bi,Na)TiO3 single crystals — •Wolfgang Donner[1], Marton Major[1], and John Daniels[2] — [1]Fachbereich Materialwissenschaft, Technische Universität Darmstadt — [2]School of Materials Science and Engineering, University of New South Wales, Sydney

In our previous work we found diffuse streaks in the x-ray diffraction from the single crystal relaxor BNT-4BT [1]. These streaks connect half-order reflections associated with octahedral tilts in the sample. The diffuse streaks and diffuse half-order peaks react upon the application of an external electric field. Similar diffuse scattering patterns had been found in electron diffraction [2] from pure BNT samples and were interpreted as arising from stacking faults in the octahedral tilt sequence. The stacking fault structure could also be viewed as a twin structure of two rhombohedral domains. Here we present results from simulations of the diffuse scattering pattern based on certain stacking faults in the R3c structure and show that the model can be applied to estimate the amount of stacking faults. The stacking fault probability in turn can be used to estimate the size of the nanopolar regions in BNT-BT giving rise to the relaxor behavior.

[1] J. Daniels, W. Jo, J. Rödel, D. Rytz and W. Donner, Appl. Phys. Lett. 98, 252904 (2011) [2] V. Dorcet, G. Trolliard, Acta Mat. 56, 1753 (2008)

15 min. break

MI 5.6 Tue 11:30 H5

Comparative Study of Ion Sputtering in XPS Depth Profiling for Thin Film Analysis. — •Andrey Lyapin[1], Stefan Reichlmaier[1], Saad Alnabulsi[2], Sankar Raman[2], John Moulder[2], Scott Bryan[2], and John Hammond[2] — [1]Physical Electronics GmbH, Fraunhoferstr. 4, D-85737, Ismaning, Germany — [2]Physical Electronics, 18725 Lake Drive East, Chanhassen, MN, 55317, USA

The objective of successful XPS sputter depth profiling is to accurately identify the layer thicknesses and chemical composition of materials within thin film structures. Cluster ion beam sputtering has been widely used in recent years with the intent to address this essential analytical goal for a broader range of materials, including organic materials. C_{60} cluster ion beam sputtering provided the first access to quantitative chemical state information below the surface for many polymers, organic and inorganic oxide materials.

The recent introduction of argon gas cluster ion beam sputtering to the XPS community has further expanded the capability of successful depth profiling with an emphasis on preserving the chemical structure of challenging polymer and organic materials that exhibit rapid radiation induced damage due to the mobility and reactivity of free radicals that are formed during the sputtering process when other ion sources are used.

The purpose of this study is to present a comparative evaluation to quantify the benefits of using either C_{60} or argon gas cluster ion beam sputtering for XPS compositional depth profiling.

MI 5.7 Tue 11:45 H5

Analysis of impurity diffusion and recrystallisation processes of Fe and FeNi polycrystals with low energy electron microscopy

— •Benjamin Borkenhagen, Gerhard Lilienkamp, and Winfried Daum — Institute of Energy Research and Physical Technologies, TU Clausthal, Leibnizstraße 4, 38678 Clausthal-Zellerfeld

We use low energy electron microscopy (LEEM) and laterally resolved low energy electron diffraction (μLEED) to characterize surface properties of polycrystalline materials as well as structural and dynamic properties of grain boundaries. In this contribution, we report on our analyses of segregation and diffusion processes taking place at the surface of polycrystalline Fe and FeNi. Previously we have shown that bulk impurities, mostly sulphur, segregate from the bulk of a heated polycrystal to the surface and form two-dimensional impurity islands. At a suitable temperature we observe Ostwald ripening of these islands and, at elevated temperature, dissolution of the islands. Here we present a quantitative study of impurity diffusion processes, which yields both linear and $t^{1/2}$ time dependencies for the impurity concentrations on different grains. These different time dependencies point to different bulk impurity concentration profiles in different grains. In addition to impurity diffusion, we studied recrystallisation processes and their effects on surface topography in real time. By measuring tripel-point speeds and geometries of the grain boundaries, the rate-limiting step of the recrystallisation process – grain boundary mobility or tripel point mobility – was identified.

MI 5.8 Tue 12:00 H5

Microscopic Understanding of Ionic Thermophoresis

— Mario Herzog, •Maren Reichl, Alexandra Götz, and Dieter Braun — Systems Biophysics, LMU, München, Germany

A number of microscopic models for thermophoresis has been proposed recently. Here we measured short DNA and RNA molecules over a wide parameter range (0.4-14 nm Debye length, 5-75°C base temperature, 5-50 bases, 11 different electrolytes) [1]. The measurements confirm the capacitor model of thermophoresis with the following details [2]:

1. Thermophoresis is proportional to the Debye length when the latter is smaller than the molecule radius, but saturates for Debye lengths exceeding the molecule radius. This confirms the predicted size transition between the plate and spherical limit of the capacitor model. The fitted effective charges depend on DNA length predicted by molecular dynamics simulations of Manning condensation.
2. Depending on the electrolyte, a constant additive contribution for the Seebeck effect of the electrolyte is confirmed. It can be understood from literature data without fitting parameters.

The model allows non-trivial predictions of thermophoresis. Our work confirms in detail a local equilibrium approach to thermophoresis. The finding is likely to improve biomolecule binding studies using microscale thermophoresis (Nanotemper Technologies).

[1] Herzog M and Braun D, under review

[2] Dohnt J, Wiegand S, Duhr S and Braun D, Langmuir 23, 1674-1683 (2007)