HZB Helmholtz Zentrum Berlin

Workshop on Industrial Research

Using Synchrotron and Neutron Methods

February 19, 2015
Berlin Adlershof

09:30	**Welcome** (Christoph Genzel)
09:40	Markus Bender, AMTC Dresden *EUV Mask Manufacturing & Characterization*
10:05	Matthias Burkhard, Carl Zeiss Jena GmbH *Manufacturing of Blazed XUV Gratings with a Very Low Level of Stray Light*
10:30	Rainer Lebert, Bruker ASC *Actinic XUV Metrology Tools with Laboratory-Sources: Complementary to Beamlines like at PTB Radiometry*
10:55	**Coffee break**
11:25	Torsten Feigl, optiXfab GmbH *EUV Optics with Integrated IR Suppression Gratings*
11:50	Martin Radtke, BAM *Imaging with the Color X-ray Camera – Basics and Examples*
12:15	Ruslan Muydinov, PVcomB *Approaches towards Highly Conductive TCOs (Transparent Conductive Oxides)*
12:40	**Lunch break**
13:20	Jeremy Robinson, University of Limerick *Using Neutron Diffraction to Characterise Residual Stresses in Aerospace Aluminium Alloys*
13:45	Roberto Coppola, ENEA *SANS and Neutron Diffraction Studies of Materials for Energy Applications*
14:10	Majid Farajian, Fraunhofer Institute for Mechanics of Materials IWM *Numerical and Experimental Description of the Surface and Subsurface Residual Stresses in Metallic Components*
14:35	**Coffee break**
15:05	Edward Mitchell, ESRF *CALIPSO and NMI3: European Research Infrastructures Working for Industry*
15:30	Martina Schäfer, Bayer HealthCare *Drug Discovery in Industry Using Synchrotron Radiation*
15:55	Manfred Weiss, HZB *Facilities for Macromolecular Crystallography at the Helmholtz-Zentrum Berlin*
16:20	**End**

9:40

EUV Mask Manufacturing and Characterization

Markus Bender[1]

[1] Advanced Mask Technology Center Dresden

Among all the choices for the next generation lithography, EUV lithography remains to be the most promising technique. Decision for introduction of EUV for 7nm HVM has to be made end 2015 beginning 2016 [1].

Presently main attention is to bring source power of the EUV scanner to High-Volume-Manufacturing (HVM) productivity requirements. The next major roadblock to progress in the ongoing push to develop EUV lithography for HVM is the availability of high quality EUV Masks fulfilling the needed requirements acc defectivity and feature uniformity.

The talk will give an introduction into EUV Mask manufacturing and characterization. Major challenges for a successful introduction of EUV Masks into HVM from a view of a mask maker will be discussed.

References:
[1] EUVL Symphosium 2014, Qualcomm

10:05

Manufacturing of Blazed XUV Gratings with a Very Low Level of Stray Light

M. Burkhardt[1], C. Laubis[2], F. Scholze[2], V. Soltwisch[2], A. Gatto[1]

1 Carl Zeiss Jena GmbH, Germany

2 PTB, Germany

Due to their comparatively high diffraction efficiencies, blazed grazing incidence gratings are frequently preferred in monochromators for the EUV and XUV wavelength ranges (~1 nm to 40 nm). One of the few suitable technologies for their manufacturing is based on an interference lithographic surface patterning. Thus, blazed grating lines with a typically rather large blaze angle are initially generated in photo sensitive resist material on top of the substrate. An adapted subsequent reactive ion etching step allows for a height-scaling during the pattern transfer into the glass substrate. The scaling factor, typically much less than one, simultaneously reduces the blaze angle to the low values required for grazing incidence operation and also reduces the roughness of the photo resist image proportionally. Our continuously optimized process chain led to a new quality in terms of efficiency and homogeneity for the diffractive grating structures as well as to very low stray light. The manufacturing process will be introduced in detail to support a sound evaluation of the potential of this technology. We present and discuss recently achieved results.

References:

DGaO Proceedings 2014 ISSN: 1614-8436 - urn:nbn:de:0287-2014-P019-1, (2014),
JEOS, rapid publications 6, 11006 (2011), ISSN 1990-2573

10:30

Actinic XUV Metrology Tools with Laboratory-Sources: Complementary to Beamlines like at PTB Radiometry

Rainer Lebert[1], Christoph Phiesel[1], Thomas Mißalla[1], Azadeh Farahzadi[1], Christian Piel [1], Stefan Herbert[2], Lukas Bahrenberg[2], Serhiy Danylyuk[2]

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2 Chair for the Technology of Optical Systems, RWTH, Aachen, Germany

The Photon Instrumentation group at RI (Originating from former ACCEL, Bruker ASC and AIXUV activities) is developing and producing tools for metrology in the spectral range of 1 - 60 nm (XUV) for both synchrotron beamline and laboratory use in close cooperation with our research partners from e.g. Aachen.

When developing and building our fully autarkic stand-alone laboratory tools we are relying on our broad scope of laboratory XUV-sources of which we know how to tailor towards best working wavelength, spectral distribution and filtering, brightness or power for most efficiently meeting user demands in EUV, XUV and soft x-ray metrology. Customers often want lab tools which are much cheaper, flexible and compact than beamline installations but on the other hand expect comparable precision and accuracy as the champion national labs like PTB. Such, the two activities are actually complementary! Only with both PTB calibration and stand alone tool supply the full scope of demands can be met.

Starting from experienced design concepts and sub-unit options, we integrate the components XUV-source, sample stages, optics (lenses, mirrors, gratings, filters) and detectors as to realize the most suitable and simultaneously most economic solution for fulfilling the top-level specifications best. Hence, dedicated variants for specific tasks are easily tailored and may be extremely compact and economic.

While repeatability and reproducibility is largely an issue of source monitoring and stability, accuracy is highly demanding. With the plasma based sources, which we are using, we have demonstrated to achieve brilliant spectral precision and accuracy of up to $\lambda/\Delta\lambda > 10{,}000$, which is sub-pm for EUV at 13.5 nm exploiting tool internal calibration with plasma emission lines.

When discussing accuracy of measured reflectivities or transmission in comparison with slow spectral scanning PTB measurements at beam lines there are multiple aspects to be considered in order to achieve similar quality of fast and spectral parallel “polychromatic broad-band measurements.

Such aspects are discussed for our EUV-Mask blank-Reflectometer (EUV-MBR) and our XUV Spectrophotometer (XUV-SPM) as set up for EUV (10-20 nm: 60 – 125 eV) using either discharge produced Xenon UV-Lamp, or laser produced gold plasma source.

The results demonstrate that our polychromatic concept is such an efficient solution with lab sources e.g. for high quality characterization of EUV-mask blanks or EUV components and explore its specific advantageous for many analytical techniques. Such solutions feature the potential to be integrated as in-line/ in-situ metrology with production systems, even.

11:25

EUV optics with integrated IR suppression gratings

Torsten Feigl[1], Hagen Pauer[1], Marco Perske[1], Tobias Fiedler[1],
Christian Laubis[2], Frank Scholze[2]

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2 PTB Berlin, Abbestr. 2-12, 10587 Berlin, Germany

Today's EUV source concepts for EUV Lithography focus on laser-induced plasma generation using CO_2 lasers in combination with Sn droplets. Different approaches of CO_2 laser suppression have been discussed and realized in the past such as binary phase gratings and CO_2 AR coatings. While CO_2 AR coatings suffer from a significant EUV reflectance loss at 13.5 nm wavelength, binary phase gratings for 10.6 µm show great advantages in terms of EUV reflectance, IR suppression factors and mechanical stability. Binary grating structures for 10.6 µm are implemented in today's LPP collector mirrors. They significantly suppress the CO_2 laser wavelength of 10.6 µm and contribute to clean EUV photons in the intermediate focus.

Since pre-pulse technology significantly enhances the conversion efficiency of EUV generation, source manufacturers use this technique to condition the Sn droplets. Different types of pre-pulse lasers are in operation today: CO2 pre-pulse lasers operating at 10.6 µm and YAG pre-pulse lasers operating at 1064 nm. As a consequence the combination of a 10.6 µm main pulse CO2 laser and a 1064 nm pre-pulse YAG laser would require a spectral purity filter that suppresses both wavelengths at the same time.

The talk discusses a new approach of a dual-wavelength spectral purity filter to suppress 10.6 µm and 1064 nm IR radiation at the same time. The dual-wavelength spectral purity filter combines two binary phase gratings that are optimized for 10.6 µm and 1064 nm, respectively. The dual phase grating structure has been realized on test samples and on elliptical sub-aperture EUV collector mirrors having a diameter of 150 mm. IR suppression factors up to 1000 at 10.6 µm and 1064 nm and EUV reflectance levels of more than 60 % at 13.5 nm have been measured on the sub-aperture EUV collectors. The optical performance at 13.5 nm and the IR suppressions at 10.6 µm and 1064 µm as well as the manufacturing process of the grating structures will be discussed in the paper. The dual-wavelength spectral purity filter can be used in future EUV collector mirror generations to suppress the pre- and main-pulse IR radiation.

11:50

Experience with the Color X-Ray Camera at the BAMline

Martin Radtke[1], Uwe Reinholz[1], Heinrich Riesemeier[1] and Oliver Scharf[2]

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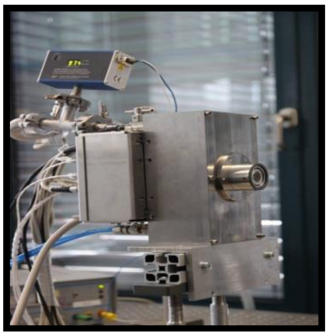
2 IFG Institute for Scientific Instruments GmbH, Berlin, Germany

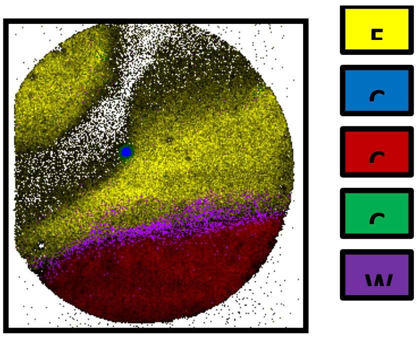
The Color X-ray Camera CXC or SLcam® is an energy-resolving X-ray camera capable of energy- and space-resolved measurements. It consists of a high-speed CCD detector coupled to a polycapillary optic that conducts the X-ray photons from the probe to distinct pixels onto the detector.

The camera is capable of fast acquisition of spatially and energy resolved fluorescence images. A dedicated software enables the acquisition and the online processing of the spectral data for all 69696 pixels, leading to a real-time visualization of the elements distribution in a sample. It was developed in a joint project with BAM, IFG Berlin and PN Sensors.

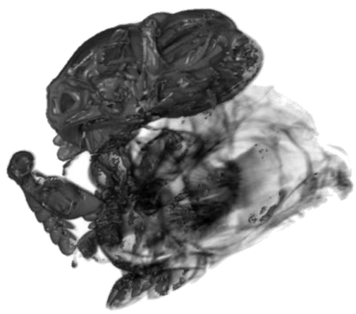
In this contribution we will discuss the use of the CXC at our beamline, the BAMline at BESSY II and imaging applications of the CXC from different areas, like biology and archaeometry.



The Color X-ray Camera



Trace element distribution in bone



3-D reconstruction of a hornet

References:
X-Ray Spectrometry 34 (2), 160 (2005)
Analytical Chemistry 83 (7), 2532 (2011)
Journal of Analytical Atomic Spectrometry 29 (8), 1339 (2014)

12:15

Towards TCOs with high mobility: approaches and restrictions

Bernd Szyszka[1], Ruslan Muydinov[1,2], Harald Scherg-Kurmes[1,2], Stefan Körner[2], Florian Ruske[3], Sebastian Neubert[2], Thomas Jung[4], Kai Ortner[4]

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2 PVcomB, Helmholtz-Zentrum Berlin, Germany
3 Helmholtz-Zentrum Berlin, Germany
4 Fraunhofer Institute for Surface Engineering and Thin Films, Germany

For most applications of transparent conducting thin films, a certain maximum sheet resistance for the TCO (transparent conductive oxide) layer is required. Due to increasing absorption losses and production costs with increasing TCO thickness, it is desirable to achieve this with a minimum film thickness. To realise low resistant very thin films, carrier concentration N_e and charge carrier mobility μ_e have to be enhanced as compared to state-of-the-art films. We consider here such approaches as an improvement of standard ZnO:Al (AZO) material and development of new materials like IOH (In_2O_3:H). The former approach includes tricks like seed layer approach and a post-deposition treatment. Seed layer approach based on nitrogen mediated crystallization, which leads to a better film nucleation for subsequently deposited doped zinc oxide films [1,2], and an annealing procedure under a protective a-Si:H capping layer, providing tuning N_e and increasing μ_e [3] are presented.
Hydrogen doped IOH has recently emerged as a TCO with an outstanding charge carrier mobility of over 100 cm^2 / Vs [4, 5]. Because of its low optical absorption as compared to conventional TCOs like AZO or ITO (In2O3:Sn) and the excellent performance reached with low temperature fabrication, IOH has a huge potential as a contact material for all types of solar cells which require contacts with both good lateral conductance and high optical transparency. In all cases we consider an improvement of magnetron sputtering as the main TCO-films' fabrication tool. For instance, evidence of O^{2-} - plasma damage of growing films [6] and approaches towards its hindering will be reported. A Gas-Flow-Sputtering technique providing soft sputtering process (without plasma damage) and being wealthy in composition tuning [7] will be presented.

[1] N. Itagaki, K. Kuwaharaa, K. Matsushimaa, K. Oshikawaa, Proc. of SPIE Vol. 8263, 2012.
[2] D.C. Look et al., Proc. SPIE 8626, Oxide-based Materials and Devices IV, (March 18, 2013)
[3] F. Ruske et al. Journal of Applied Physics, 107: 013708, 2010, pp. 1-8.
[4] T. Koida, H. Fujiwara, M. Kondo, J. Appl. Phys. 46 (2007) L685-L687.
[5] T. Koida, H. Fujiwara, M. Kondo, Sol. Energy Mater. Sol. Cells. 93 (2009) pp. 851-854.
[6] W. Dewald, V. Sittinger, W. Werner, C. Jacobs, B. Szyszka, Thin Solid Films 518 (2009).
[7] B. Szyszka et al., Hollow cathode gas flow sputtering with plasma excitation by unipolar pulsing with reverse voltage, ICCG-10 conference in Dresden (2014)

13:20

Using neutron diffraction to characterise residual stresses in aerospace aluminium alloys

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The critical step in realising the high strengths achievable in precipitation hardened aluminium alloys used in aerospace is the rapidity of quenching from the solution heat treatment temperature. When cooling is fast enough, the resulting low strength homogeneous supersaturated solid solution can be subsequently aged to form sub-micron precipitates with a concomitant very large increase in yield strength. However, another less desirable consequence of rapid cooling is the introduction of residual stresses. Large thermal gradients can develop during quenching, and if simultaneous plastic deformation occurs to relax the associated thermal strains, high magnitude residual stresses can remain in the material. Neutron diffraction has the great advantage of being able to characterise the through thickness residual stresses in large aluminium alloy components. This presentation will demonstrate how successful the technique can be in identifying the magnitude and distribution of residual stresses induced by quenching and how subsequent processing affects the residual stresses.

13:45

SANS and neutron diffraction studies of materials for energy applications

R. Coppola[1],

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This contribution will present recent results, obtained by small-angle neutron scattering (SANS) and neutron diffraction, in the characterization of materials for energy applications. SANS provides a powerful experimental tool to quantitatively investigate the microstructural effects of radiation damage in technical steels developed for nuclear applications; SANS measurements carried out on the irradiated steel Eurofer-97, european reference for fusion applications, are providing fundamental microstructural information on the simultaneous effects of high helium concentrations and damage levels. SANS results on spatially oriented hydrides in hydrogen loaded Zircalloy have also been recently obtained, in the frame of a collaborative IAEA project on nuclear safety. Neutron diffraction has been utilized, in collaboration with the HZB, for non-destructive stress evaluation in multi-layered W-Cu prototype structures, developed for plasma facing components in the ITER reactor. Neutron diffraction studies are also carried out for crystallographic characterization of engineering metallic materials as well as for innovative fuel cell components. Recent results and current projects in this area will be presented.

14:10

Numerical and Experimental Description of the Surface and Subsurface Residual Stresses in Metallic Components

Majid Farajian[1], Wulf Pfeiffer[1], Dieter Siegele[1]

1 Fraunhofer Institute for Mechanics of Materials IWM, Freiburg, Germany

It is a frequent practice to perform mechanical surface treatment e.g. shot peening, deep rolling and hammer peening in different industrial disciplines in order to improve the surface integrity of components and structures. These treatments invariably change the material properties and the residual stress state in the surface and subsurface and induce beneficial surface conditions; compressive residual stresses and cold working which in turn increase the performance of materials against fatigue, wear and corrosion. Determination of the surface and sub-surface material states is a critical point for quantitative consideration of the beneficial induced surface conditions in structural integrity assessments. Most research work has focused on experimentally determining the residual stress and its depth profile by means of x-ray diffraction and corresponding electro-polishing the surface layers or hole drilling method. This valuable body of knowledge has led to development of phenomenological-models which could describe the material behaviour during treatment qualitatively. Since there are quite a number of parameters which could influence the residual stress field after mechanical surface treatment, covering the whole possible process parameters combinations with the purpose of experimentally determining the residual stress profiles would be an impossible task. Taking into account the development of new alloys, complex geometries, mechanical surface treatment in higher temperatures and under pre-strained conditions, relaxation of residual stresses under thermal and mechanical loadings, one would conclude that the application of numerical analysis as an effective tool to treat different issues of residual stress field and its influence on the structural integrity with lower experimental costs and time is indispensable. A literature study showed that, in the numerical works, a shortcoming in general is that a quantitative description of the surface and subsurface conditions by material modelling and process simulation has not kept pace with the rapid developments in the field of characterization of polycrystalline materials by means of combination of the available diffraction techniques.

In this paper some issues in simulation and modelling of shot peening, deep rolling and hammer peening will be discussed first. The corresponding calculations are then compared with the complementary experimental analysis by means of x-ray, synchrotron and neutron diffraction methods. Further the necessity of the development of the concept residual stress engineering for metallic components in which wanted residual stress states are tailored for specific cases by appropriate means will be discussed. The possibilities of the quantitative consideration of the benefits into the structural integrity assessments will be presented in some practical examples.

15:05

CALIPSO and NMI3: European Research Infrastructures working for industry

Ed Mitchell[1]

1 European Synchrotron Radiation Facility, Grenoble, France

The creation and tailoring of new materials are at the heart of current industry challenges. New materials must meet ever more stringent requirements of performance, whilst fitting into the modern cradle-to-grave cycle of material production, use, and recycling. The properties and function expected of materials depend heavily upon their composition and their micro- or even nano-structure. Their "ultimate" characterisation is possible down to the atomic scale using the tools and techniques of large-scale facilities such as synchrotron X-rays.

Synchrotron X-rays are a non-destructive probe of material structure at length scales ranging from centimetres to the size of an atom. Special properties such as element selectivity offer a variety of analytical tools for characterising materials under in situ conditions (heat, cold, pressure, chemical, stress/tension, electrical/magnetic fields....) and in real time that are not accessible with traditional techniques. Synchrotrons therefore provide the ability to visualise the atomic, nano, and macro-structure of a huge range of complex real-world materials, often under processing or end-use conditions and in real time. This capability lends itself to an equally wide range of industrial R&D problems which, in particular, have been adopted by the healthcare industry. Beyond drug discovery and development, synchrotron facilities are also very active in providing analysis for micro- and nano-electronics, energy and smart materials, transport, chemistry and catalysis, engineering materials, and home and body care amongst others.
In Europe and worldwide, funding agencies are requesting and demanding a stronger economic return from the significant public investments made in central facilities and this is resulting a gradual but firm pressure for stronger interactions with industry. In this context, the FP7 funded CALIPSO (www.calipso.wayforlight.eu) and NMI3 (www.nmi3.eu) light and neutron/muon source networks have been working to draw together research infrastructures and industry with workshops, industry office best practice exchange and an Industry Advisory Board.

This presentation will present and discuss the increasingly critical role of such large-scale facilities in delivering ultimate materials characterisation for innovative industrial and applied R&D, looking to both the current developments and future possibilities of business, as well as review several examples of partnerships between research and industry and the impact these partnerships have on academic research.

15:30

Drug Discovery in Industry using Synchrotron Radiation

Martina Schäfer[1]

1 Bayer Pharma AG, Germany

Three dimensional co-crystal structures of pharmaceutical relevant targets in complex with small molecule inhibitors are crucial in the discovery and development of drugs. In a normal drug discovery campaign first hits originating from a High Throughput Campaign (HTS) are co-crystallized to determine their binding mode. Often more than 10 co-crystal structures of several compound classes or even similar compounds are needed to guide structure-based-drug-design efforts.

In addition to that, complex structures of a target of interest with 'fragments' (very small molecules with Mw less the 250g/ml) can also provide good starting points for a new drug discovery project.

Both approaches, either HTS or Fragment Screening (FS), need frequently high brilliant x-ray sources as it is the case with Synchrotron radiation. In my talk I will show examples where Synchrotron radiation was used to obtain excellent data of important targets with new inhibitors.

15:55

Facilities for Macromolecular Crystallography at the Helmholtz-Zentrum Berlin

Manfred S. Weiss[1], Ronald Förster[2], Michael Hellmig[1], Franziska Huschmann[3], Alexandra Kastner[1], Piotr Malecki[4], Martin Röwer[1], Karine Sparta[1], Michael Steffien[1], Monika Ühlein[1], Piotr Wilk[5], Uwe Mueller[1]

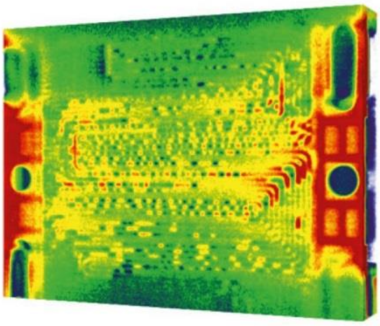
1 Helmholtz-Zentrum Berlin für Materialien und Energie, Berlin, Germany
2 Freie Universität Berlin, Germany
3 Phillips-Universität Marburg, Germany
4 Max-Delbrück-Centrum for Molecular Medicine, Berlin-Buch, Germany
5 Humbold-Universität zu Berlin, Germany

The Macromolecular Crystallography (MX) group at the Helmholtz-Zentrum Berlin (HZB) has been in operation since 2003. Since then, three state-of-the-art synchrotron beam lines (BL14.1-3) for MX have been built up on a 7T-wavelength shifter source [1,2]. Currently, the three beam lines represent Germany's most productive MX-stations, with more than 1300 PDB depositions (Status 01/2015). Beam lines BL14.1 and BL14.2 are energy tuneable in the range 5.5-15.5 keV, while beam line BL14.3 is a fixed-energy side station operated at 13.8 keV. All three beam lines are equipped with state-of-the-art detectors: BL14.1 with a PILATUS 6M detector and BL14.2 and BL14.3 with large CCD-detectors. BL14.1 and BL14.2 are in regular user operation providing about 200 beam days per year and about 600 user shifts to approximately 100 research groups across Europe. BL14.3 has been equipped with a HC1 crystal dehydration device in 2011. In addition to serving the user community mainly as a screening and test beam line, it is currently the only MX beamline in Europe with a HC1 device permanently installed. Additional user facilities include office space adjacent to the beam lines, a sample preparation laboratory, a biology laboratory (safety level 1) and high-end computing resources. In the presentation, a summary on the experimental possibilities of the beam lines and the ancillary equipment provided to the user community as well as the services provided to industrial customers will be given.

References:

[1] Heinemann U., Büssow K., Mueller, U. & Umbach, P. (2003). Acc. Chem. Res. **36**, 157-163.
[2] U. Mueller, N. Darowski, M. R. Fuchs, R. Förster, M. Hellmig, K. S. Paithankar, S. Pühringer, M. Steffien, G. Zocher & M. S. Weiss (2012). J. Synchr. Rad. **19**, 442-449.

HZB Service for Industry

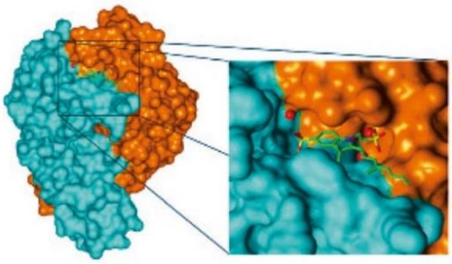


Neutron tomography provides 3D insight into a fuel cell

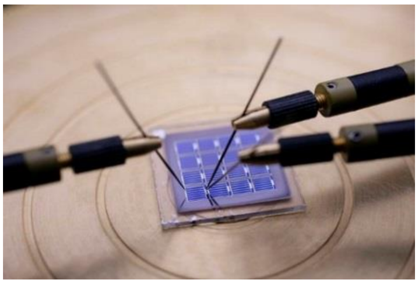
The Helmholtz-Zentrum Berlin für Materialien und Energie **HZB** operates the third generation synchrotron source **BESSY II** on Wilhelm-Conrad-Röntgen-Campus in Berlin-Adlershof and the medium flux research reactor **BER II** on Lise-Meitner-Campus in Berlin-Wannsee. HZB is home to a number of state-of-the-art **on-site laboratories** and user facilities. All these research facilities are designed to serve scientific as well as industrial research.

Dedicated areas of expertise for industry are

- Stress and strain analysis by synchrotron and neutron scattering probing volume as well as surface areas.
- 3D-Information of objects down to atomic resolution, e.g. protein structures, battery functions, nanomaterial interaction
- Structure characterization of materials, mechanical or electronic devices or biological samples
- Characterization and production of X-ray optics with sub nanometer resolution
- Novel materials and thin layer systems in photovoltaics and solar energy devices
- Solar fuels production technologies



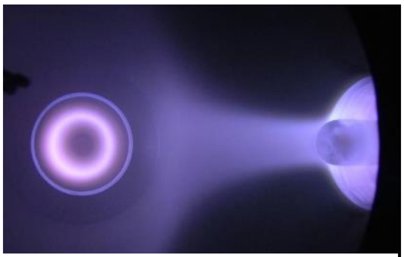
Determination of molecular structures at our macromolecular crystallography beamlines



Investigation of a thin-film solar cell

Offered methods include

- Strain and texture analysis
- Micro-tomography with synchrotrons and neutrons
- Small angle X-ray and neutron scattering
- X-ray and neutron reflectometry
- Protein crystallography
- Nanometeroptics and metrology
- Co-60 radiation
- PVcomB thin-film technology



Thin-film deposition methods such as sputtering

Contact:

Helmholtz-Zentrum Berlin für Materialien und Energie GmbH
http://www.helmholtz-berlin.de/angebote/tt-industrie/index_de.html