5.1. Scattering & Imaging

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Current activities at the Scattering and Imaging facilities are concentrated on the upgrade of existing beamlines to state-of-the-art technology and on the design and procurement for new beamlines (NANO and IMAGE).

Funding to upgrade the existing PDIFF beamline has now been secured: the upgrade is geared towards rapid in-situ diffraction experiments on bulk nanocrystalline and microcrystalline materials. Additional funding has also been secured to enable a variety of complex sample-environmental stages to be designed/purchased for in-situ scattering experiments both on bulk and on thin-film materials.

With commissioning of the 6-circle diffractometer on SCD complete this instrument is now available for user experiments which require the enhanced degrees of freedom of this instrument.

The beamline instrumentation is described in detail in the latest version of the ANKA instrumentation book 2007. In the following we report on the status of the 3 existing scattering beamlines - SCD, PDIFF and the MPI-MF beamline, together with planning/status of the imaging facility TOPO/TOMO. Additionally we report on the planning and design of the NANO beamline, which is now at an advanced stage.

ANKA-SCD

In 2007 a number of upgrade measures have been conducted in parallel on both SCD and PDIFF beamlines: these measures were necessary to improve beamline optics stability and reliability, beam diagnostics, experimental station performance, sample environment, control and data processing software and beamline infrastructure. In particular and as announced in the 2006 annual report [1], the water chillers for the 1st DCM crystal(s) have been exchanged by devices known to be more reliable and with an order of magnitude less temperature tolerance. Since their replacement no more beam intensity fluctuations due to water temperature oscillations have been observed.

Control of beam shutters and the vacuum system

The facility controlling the beam shutters and the vacuum system of the SCD beamline has been replaced by a system based on PVSS© (manufactured by etm). This has not only enhanced the reliability of the SCD beamline control system but also facilitates its extension with the components described in the next section. The system will be implemented on all forthcoming beamlines and beamlines to be upgraded. The server-client structure allows for centralized system maintenance.

Additional beamline components

To reduce the background and make X-ray reflectivity and GISAXS measurements feasible, two additional remotely controlled slit systems (collimating and anti-scatter slits) have been introduced in the SCD beamline optics. The detector entrance slits on the 6-circle instrument have been motorized to allow remote control as well.

The primary beam intensity can be reduced now in 15 steps by a remote controlled attenuator. An ionization chamber close to the sample position of the 6-circle instrument makes a precise normalization of the scattered signal with respect to the primary beam intensity possible.

Upgrade of crystallographic software

The existing SMART software for control of the Bruker AXS diffractometer and CCD detector will no longer be upgraded by the manufacturer. Thus we decided to purchase the more recent APEX2 software, which will allow us to develop a user interface unifying beamline and instrument control based on the client-server structured BIS ("Bruker Instrument Services". The new software also is much more user-friendly and intuitive to use than the old one. By swapping out data processing the frame buffer is relieved, now handling detector communication and instrument control only. This has lead to a more reliable operation.

The X-AREA control and data processing software of the STOE IPDS station has been upgraded to the most recent version 1.39.

Plans for the future

An analyzer stage for the 6-circle diffractometer is under construction and should become available in October this year. It is planned to replace the point detector on the 6-circle instrument including the electronic chain by a faster system allowing for higher count rates. Making available a 2d detector to be used in conjunction with the 6-circle diffractometer is in preparation. Translational stages for sample positioning in the Eulerian cradle are to be motorized. A sample microscope system comprising a video camera will facilitate sample alignment. Specialized sample environment equipment is under construction which will allow to perform in situ investigations of open nanostructure evolution during their treatment.

References

[1] G. Buth, S. Doyle, A. Rack, T. Weitkamp, S. Trabelsi-Bauer, ANKA Annual Report 2006, p. 200

[2] K. Eichhorn and G. Buth, ANKA Annual Report 2006, p. 202

ANKA-PDIFF

As well as the common improvements which have taken place in parallel on both PDIFF and SCD beamlines (see above) we have implemented a number of new components available either for beam-monitoring/conditioning or for sample conditioning. These include:

1. a motorised sample holder allowing X- and Y-translations and tilts of a sample has been installed: this instrument is particularly useful for the alignment and position-resolved measurements on flat samples such as wafers etc. An additional Z-translation (offering approx. ± 7mm height adjust) will be installed soon.
2. a motorised slit-system together with improved shielding for the analyser stage has been installed on the detector-arm
3. an incident beam-attenuator allowing up to 16 discrete attenuation levels
4. a liquid-nitrogen cold-finger attachment for the MRI-sample chamber to permit low-temperature powder measurements (down to approx. 80 K)

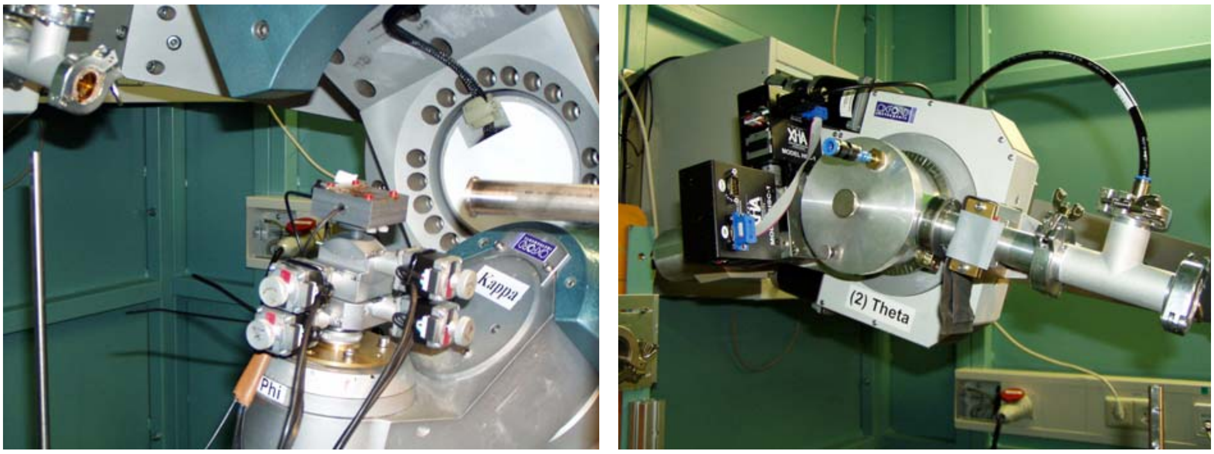


Fig. 1: *Motorised sample-adjustment stage (left) mounted on the diffractometer, and (right) detector housing and XIA motorised slits.*

The control of all new components has been, or will shortly be, implemented in the beamline spec application.

Beamline Software

Tests on the control of the diffractometer via spec were carried out successfully in May and it is now planned that during the October shutdown the control of the diffractometer via spec will be permanently implemented. Replacement of the beamline control system (RST) by the process control environment PVSS© is currently underway and will also be completed in October 2007. This will enhance both the beamline safety and reliability and enable faster and easier implementation of beamline components.

PDIFF Major Upgrade

Specifications for the required hardware for a major refurbishment of the existing PDIFF beamline are presently being defined. The new instrumentation will provide a facility for in-situ investigations on nano- and microcrystalline samples and consists of four major aspects:

- installation of both area and linear detectors for polycrystalline diffraction: it is foreseen that an area detector will be installed at the beginning of 2008 and a linear detector towards the middle of 2008.

- installation of a heavy-duty 2-circle powder diffractometer with a capability to carry moderately heavy loads (max. approx. 50 kg).
- various in-situ sample-conditioning capabilities, such as furnaces, extensometer, etc.
- installation of additional focusing optics to permit both vertical as well as horizontal increase in flux at the sample

Further details of the proposed upgrade will be presented at the user meeting in October. It is planned that all aspects of the upgrade project will be complete by the end of 2008.

NANO

The NANO beamline will be a facility for high resolution X-ray diffraction and surface/ interface scattering with the focus on in-situ and ex-situ structural characterisation of thin films, multilayers and nanostructured materials.

The beamline will consist of three parts: the optics and two experimental stations. The beamline optics including four mirrors, a double crystal monochromator and a double multilayer monochromator (see below) has been specified in full detail in such a way we will be able to select monochromatic, 'pink' as well as white beam. Furthermore, by choosing the appropriate radius of curvature for the different mirrors, the beam can be either collimated or focused on the sample position. The beamline will be operational in the energy range between 3 and 25 keV with a flux density of 10^{12} photon/s at the sample. The heart of the experimental station will be a six-circle diffractometer specified and designed to permit in-situ scattering structure investigations during the growth process of nanomaterials such as quantum dots. The diffractometer will be equipped with a crystal analyzer for high resolution measurement with the possibility to detect simultaneously two types of crystalline reflections using two different detectors arms (see figure 2 below). Moreover, it is designed to handle heavy environmental chambers up to 500 kg.

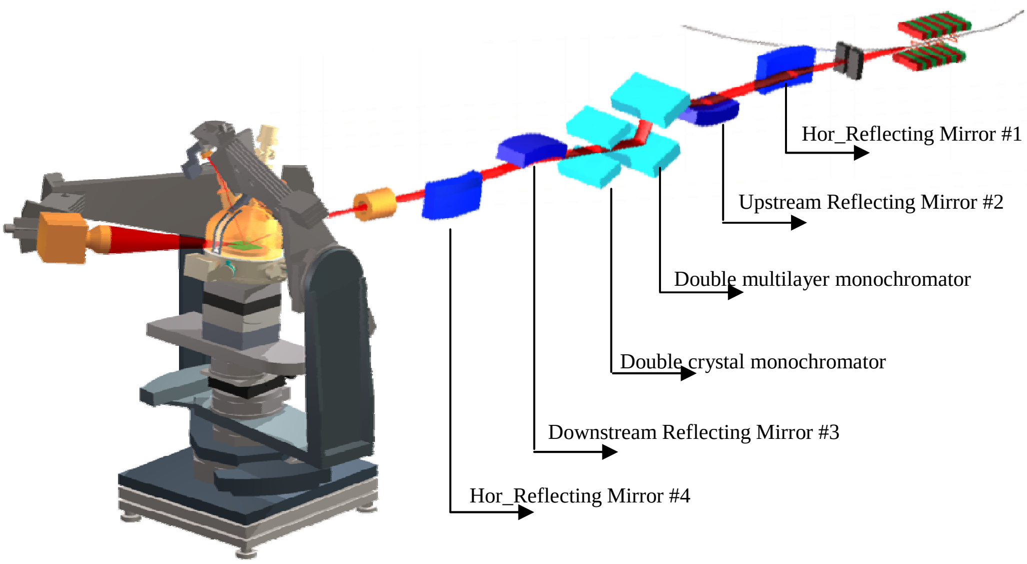
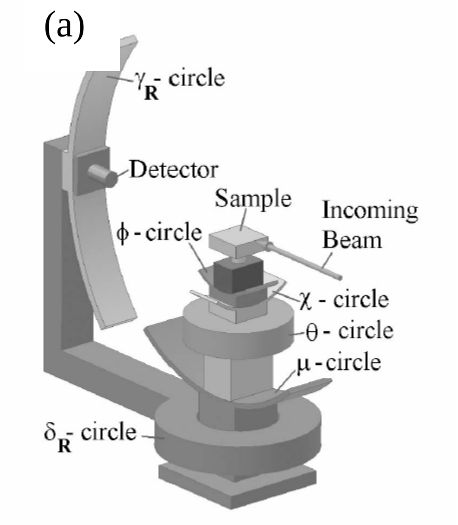


Fig. 2: *Design of NANO beamline optics and the multipurpose diffractometer of the experimental station NANO 1.*

MPI-MF

Three siginificant technical developments have been conducted on the beamline during the reporting period: these have been or will be documented in publications (see [1-3]):

1. Adoption of the diffractometer to a standard six circle geometry (Figure 3):
2.



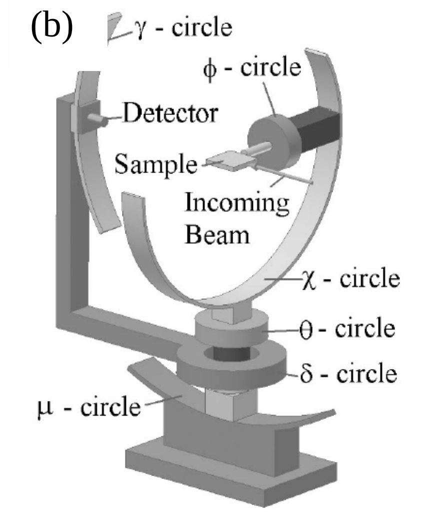


Fig. 3a: *Goniometer circle arrangement of the diffractometer at the MPI-MF beamline.*

Fig. 3b: *angular arrangement of a six circle grazing incidence diffractometer*

By appropriate choice of transformation matrices the angular configuration of a (2+2) diffractometer (a) can be traced back to a six circle diffractometer (b), which exhibits the intrinsic grazing incidence diffraction geometry: the incident angle μ tilts the whole diffractometer and γ sets the exit angle relative to the surface. This transformation was implemented into the diffractometer control software SPEC, allowing work in reciprocal space on a virtual six circle diffractometer [1].

2. Development of a new technique to study stresses in ultra-thin films in-situ using synchrotron radiation [2].

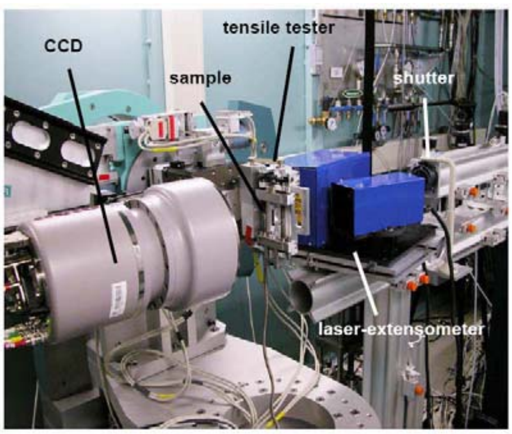


Fig. 4: *Experimental set-up for in-situ strain measurements*

Figure 4 shows the set-up for in-situ strain measurements at the MPI-MF beamline. A constant force is applied via a tensile tester to a polycrystalline metal film deposited on a Kapton substrate. At the same time, the macroscopic dilatation of the foil is traced using a laser extensometer and the microscopic change in the lattice spacing is followed in-situ by recording sets of Debye-Scherrer rings on the CCD camera. In such a way stress – strain curves of ultrathin films can be obtained.

3. Test and development of a quarter wave based digital lock-in system for x-ray magnetic circular dichroism (XMCD) measurements [3].

Diamond single crystals can be used in transmission geometry as quarter wave plates for hard X-ray synchrotron radiation. By scanning the Bragg peak rocking curve, linear polarized synchrotron radiation can be either transformed into left- or right-circular polarized synchrotron radiation, which is needed for XMCD experiments. Figure 5 below shows a schematic view of the setup and the result of a test experiment.

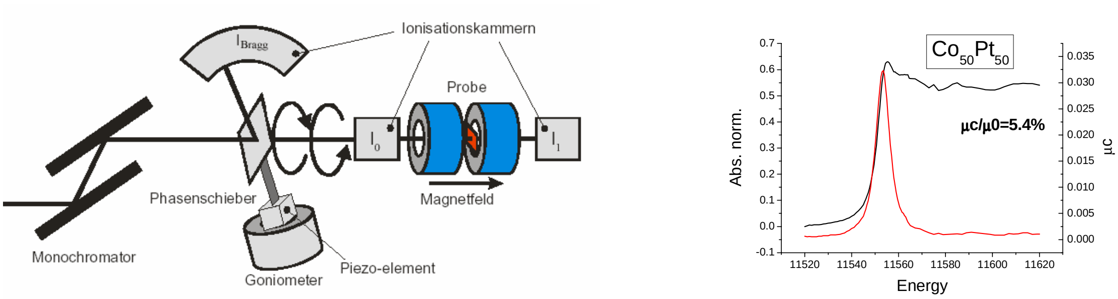


Fig. 5: *Schematic view (left) of the XMCD setup, and (right) normalized absorption at the Pt(L_{III}) edge at 11.55 keV and the corresponding dichroic signal (red curve).*

The monochromator and the beamline fulfill the stability requirements for such a sensitive measurement and the magnetic signal µc from a thinned CoPt foil can be detected with high sensitivity and very good signal-to-noise ratio.

References

[1] D. G. de Oteyza, E. Barrena, S. Sellner, J. O. Osso, and Helmut Dosch
Role of the substrate thickness for the structural properties of organic-organic heterostructures
Proceedings of the European Conference On Surface Science, accepted

[2] Böhm J, Gruber P, Spolenak R, Stierle A, Wanner A, Arzt E. Tensile testing of ultrathin polycrystalline films: a synchrotron-based technique. Review of Scientific Instruments, vol.75, pp.1110-19 (2004).

[3] E. Göring, G. Schütz, Grüner, Weigel, Low noise XMCD Spectroscopy: A quarter wave based digital Lock-In system (Rev. Sci. Instr., in prep.)

Imaging activities

Detectors

For high resolution X-ray imaging the current focus on the detectors lies on indirect systems where a luminescence screen is projected magnified via microscope optics onto the chip of a digital camera. Concerning the luminescence screens different scintillating single crystals are now available. For moderate resolutions and large field of view 1" sized CWO crystals (300 µm thick) have been ordered and commissioned, for higher resolutions 10 mm sized CWO crystals (40 µm thick) on YAG substrates are used. BGO crystals from different suppliers were tested but none of them reach the required quality level.
For highest resolutions down to sub-micrometer range we founded a small consortium consisting of the Advanced Photon Source (APS, USA), Bundesanstalt für Materialforschung und -prüfung (BAM, Germany) Swiss Light Soure (SLS) and ANKA to order LuAG:Eu crystals grown via liquid phase epitaxy on undoped YAG substrates by CEA LETI (Grenoble).

Two optical systems have been commissioned for moderate (macroscope) and high resolutions (microscope). The macroscope consists of commercial Rodenstock and Nikon objectives with large numerical apertures and high light throughput. It was already successfully applied for white beam phase contrast radiography (up to 40 FPS) and tomography. For highest resolutions we chose a microscope design from the French company OptiquePeter (Lyon). During commissioning we could reach with this detector a resolution below 1 µm (using a modified white beam, a 4 µm thick LuAG:Eu single crystal on tp of an undoped 170 µm thick YAG substrate, 0.2 µm effective pixel size, the resolving power was tested with an Xradia test pattern). For dedicated white beam optical systems currently a system is designed with the American company Iomega.

For the camera systems we ordered and received a PCO4000 (4008 x 2672 pixel, 9 µm pixel size, Kodak interline transfer chip with up to 5.000 greylevels at 1.4 FPS read-out speed) and as the first external users the famous FReLoN 2k14bit (2048 x 2048 pixel, 14 µm pixel size, ATMEL four readout port CCD with 12.000 greylevels at 10 FPS readout speed) from the European Synchrotron Radiation Facility ESRF (France). Currently ANKA and ESRF are developing the FReLoN camera further by extending the cameras quantum efficiency down to 400 nm by putting a phosphor coating on the CCD chip. The modified FReLoN allows to use faster scintillating single crystals like LSO:Ce and to achieve higher resolutions.

After feasibility studies an ultra-fast CMOS camera is in the tender process. It was proven at the ESRF's beamline ID15a that with white beam phase contrast imaging radioscopy with frame rates up to 10.000 FPS can be achieved.

Experimental station

A modular sample manipulator consisting of a z-stage and a cradle for raw adjustments (Huber GmbH) and high precision stages for sample translation and rotation (MICOS GmbH) have been delivered and commissioned. Additionally, our detector systems are now mounted on a dedicated tower for fast x- and z-translation in order to adjust the detector to the different needs of X-ray imaging or X-ray topography. For holotomography applications we have now a high load, high precision air-bed linear table available (1100 mm travel range, up to 200 kg load, Johann Fischer Aschaffenburg GmbH). Commissioning and installation will be done after the upgrade of the optical table in the experimental hutch.

Further upgrades also included a complete exchange of the control system, an extension of the experimental hutch, installation of X-ray shutters and the installation of new air condition systems for higher thermal stability. Most of these will be done during a shutdown of the beamline from middle of November 2007 to February 2008.

TOPO/TOMO beamline layout

In preparation for the installation of a double multilayer monochromator (DMM) a filter system (two holders, each consisting of three filters and a white beam option, Oxford-Danfysik) was installed. The system houses six different metal foils as absorption filters to modify the spectrum of the TOPO/TOMO bending magnet source, to suppress lower energies which otherwise would pass the DMM by total reflection and ruin the energy bandwidth, and to lower the heat load, either on the DMM first mirror or the sample in white beam phase contrast mode.

The new double multilayer monochromator is currently in the tender process. The tender will end October 2007, order will be placed by the end of 2007. As installation date the end of 2008 is foreseen. The DMM will consist of two layer stripes optimized for high flux and low beam profile influence. A bending option for the 2nd multilayer is included for high resolution or high flux pink beam applications.

Fig. 6: *FReLon camera purchased from the ESRF for high-resolution imaging applications*

Other Activities

A practical course was held in March 2007 for students attending the lecture series „Moderne Röntgenphysik" at University of Karlsruhe. During the 3-day practical two groups of students were assigned measurement and data-evaluation tasks on the SCD and PDIFF beamlines: each group was required to investigate structural aspects of different epitaxially grown semi-conductor materials and to evaluate parameters such as film thickness and composition, lattice mismatch and degree of relaxation based on measured data. The course served both as training for the students and for the implementation of thin-film scattering techniques at both beamlines. It is planned that the course will be repeated in future semesters.

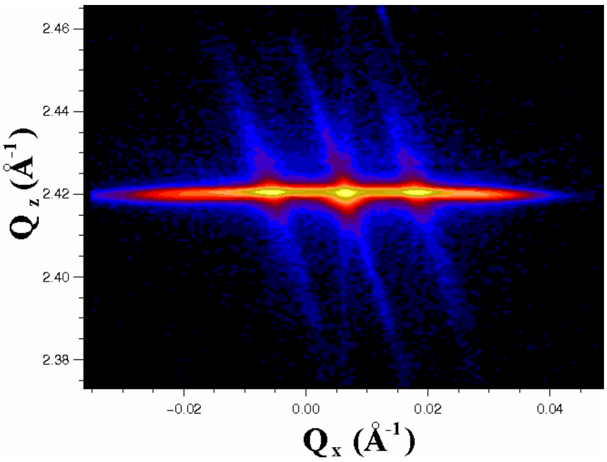


Fig. 7: Reciprocal space map of a GaN device grown by epitaxial lateral overgrowth (ELO) and measured by course participants.