3.1.2. X-ray IMAGING at ANKA

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Synchrotron imaging methods with spatial resolution in the micrometer and nanometer range are increasingly relevant to material research and diagnostics in engineering sciences, micro system and nanotechnologies, to structure imaging in nano-biology and life sciences, to non-destructive testing and geometrical measuring of components and devices, to paleontology, cultural heritage and other fields.
Synchrotron imaging methods allow closing the gap between conventional 2D and 3D imaging performed with X-ray table-top sources techniques and electron microscopy methods, i.e. the gap between about 10 µm and several ten nanometers. X-ray imaging techniques are largely non-destructive and allow using and combining various contrast mechanisms (absorption, X-ray fluorescence, Fresnel and Bragg diffraction, dichroism, etc.) to image geometrical, crystallographic and chemical structures of samples.
In 2006 the ANKA-IMAGING-group has been established and assigned to push instrumentation and development of components, methods and techniques of X-ray imaging. The group is developing full-field methods of projection imaging, microscopy and 3D tomographic imaging based on absorption, phase and Bragg/Laue diffraction contrast.
The main imaging projects for the period from 2008 to 2010 will be the construction and the full operation of TOPO-TOMO and IMAGE beamlines. Both will allow ANKA to cover the resolution range from 40 nm to 40 µm with X-ray energies ranging from approximately 6 to 60 keV.

The main instrumental goals of the imaging group in the reporting period were

- First stage of TOPO-TOMO beamline upgrade;
- Design and starting ordering of the insertion device for the IMAGE beamline;
- Equipment, commissioning and development of X-ray imaging detectors;
- Methodical developments.

TOPO-TOMO beamline

The TOPO-TOMO beamline is devoted to conventional X-ray topography and micro-imaging.
After microfluorescence methods have been transferred from the former FLUO-TOPO beamline to the new FLUO beamline, a major upgrade of the remaining station is launched within two stages.

– The first stage of the new **TOPO-TOMO** beamline is focused on white beam imaging methods:
- Digital white beam topography (projection and section topography),
- Digital white beam radiography and 3D microtomography, both in absorption and in phase contrast.

– In the second upgrade stage in 2009, a double multilayer monochromator will be installed and the spectrum of methods will be extended to monochromatic imaging allowing, for instance, elemental contrast.

The beamline will open to users for white-beam topography and radiography by the end of 2008 and for monochromatic imaging including computed tomography in autumn 2009.

The Double Multilayer Monochromator (DMM) of TOPO-TOMO is under construction. The use of monochromatic radiation provides quantitative absorption and phase contrast being sensitive to density and atomic numbers and to the local electron density respectively. Hence it minimizes the so-called beam hardening artefacts in tomographic reconstructions.
So far, three commercially produced DMMs are applied in the world for imaging beamlines (ID-34 @ APS – OxfordFMB design, BAMline @ BESSY – Accel design, TomCat @ SLS – Sinel design). In order to optimise the performance of our DMM we did several preliminary investigations. In cooperation with the AXO Dresden GmbH, high quality silicon substrates were acquired from General Optics and characterised

concerning their micro- and long range roughness by BESSY‘s metrology group. Afterwards, different types of multilayers with varying design and coating materials were grown on the substrates by the AXO Dresden GmbH. Intense tests of the different structures were performed at the ESRF‘s BM5 and ANKA’s SCD beamlines, allowing us to choose the optimal structure design in terms of flux, beam profile behind the DMM and useable monochromatic beam height.
Additionally, our tests at BESSY‘s BAMline proved that beam compression by using a meridional multilayer bending can be used for micro-imaging in order to improve the flux, which reduces the required exposure times.
The final design of our DMM being constructed by ACCEL, includes a two-stripe solution in order to optimise flux and avoiding to work at energies near the absorption edges of the multilayer materials. The second multilayer has an option for meridional bending.
The DMM design contains a beam position monitor suitable for white beam and pink beam as well. The DMM based on this design is foreseen to be installed in March 2009.

Upgrade of the TOPO-TOMO control system. To prepare the beamline for the DMM installation a complete upgrade of the control system has been realized. After that the beamline components can be fully operated from the control station and all the necessary cabling for the DMM are put in place.

A white beam tomographic imaging station has been set up, allowing reliable tomographic scans based on radiographic projection images with resolutions up to 2.5 µm (“microscope”) or large field of view (5 mm x 15 mm, limited by the beam size) and moderate micro-resolutions in the order of 10 µm (“macroscope”).
We can combine white beam imaging with absorption and phase contrast. White beam phase contrast radiography was successfully applied at ANKA for fast imaging of living insects, see figure 1. Frame rates of up to 250 images per second were achieved: a unique tool for micro-biologist to study the inside of living organisms.
In order to perform tomography with high quality volume renderings, ANKA developed a phase retrieval PlugIn software, allowing the users to perform phase maps reconstruction from radiographic phase contrast images as input for subsequent tomographic reconstruction. It is based on the algorithm developed by D. Paganin and coworkers [1]. The algorithm delivers volume images displaying the local phase shift rather than only edge enhanced ones. One example is reported in figure 2.

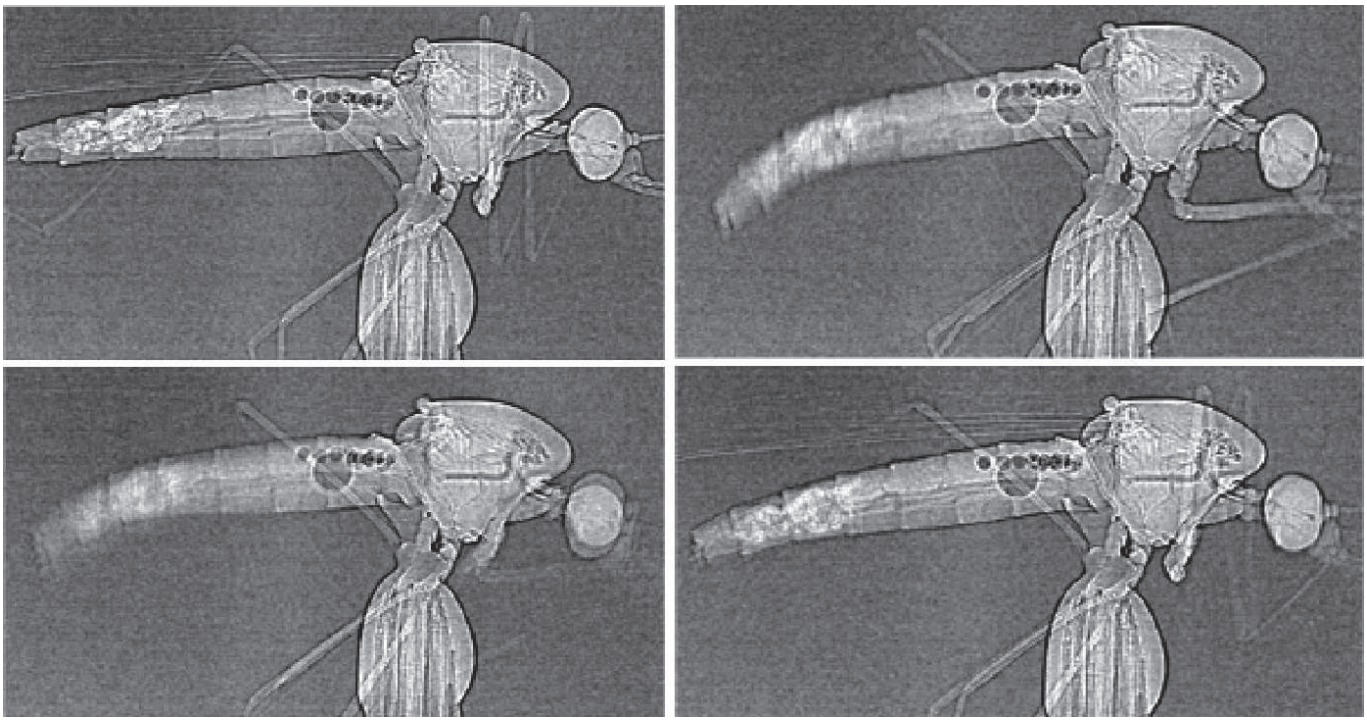

Fig. 1: *Radiographic images acquired with 4 FPS, showing the internal morphology of a living female mosquito.*

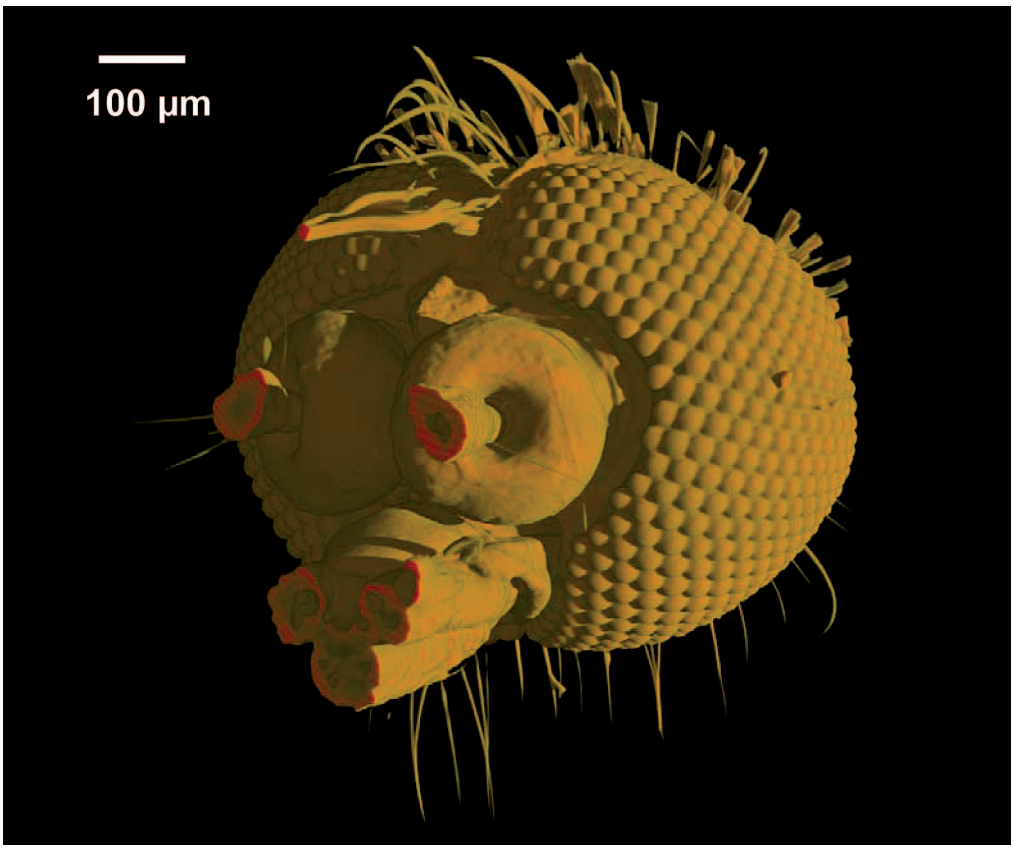

Fig. 2: *Rendered tomographic image of a mosquito head (IAEA Vienna), phase maps derived from in-line phase contrast images using phase retrieval. The spatial resolution is approx 2 µm.*

IMAGE beamline

The IMAGE beamline has received funding in autumn 2007 based on major investment of the HGF, the state Baden-Württemberg and internal resources.
It has been specified and at present it is in the phase of technical design and ordering of the first elements. The IMAGE beamline is being set up in the west straight section. The photon source will be a combined superconducting undulator/wiggler. Hence, IMAGE will cover two energy ranges (in the undulator mode from 4 keV to 20 keV and in the wiggler mode up to 100 keV) and X-ray imaging methods tailored to the performance parameters of the ANKA radiation source.
Figures 3 and 4 illustrate the overview of the methods and stations foreseen at IMAGE in the present design stage.
Three measurement stations will be established for two-dimensional and three-dimensional imaging methods in the hard X-ray range using absorption, phase, and Bragg/Laue diffraction contrast:

1. Station 1 for fast white beam imaging;
2. Station 2 for Full-field Transmission X-ray Microscope for hard X-rays;
3. Station 3 for computer tomography / laminography, including coherent imaging by in-line holography, grating interferometry and 3D micro diffraction imaging methods.

The conceptual design of the beamline has been finished and the front-end, optics and experimental stations are in the specification stage.
The optical scheme of the beamline will include both a Double Multilayer Monochromator for projection imaging and microscopy applications and a Double Crystal Monochromator allowing Full field Microdiffraction Imaging and Diffraction Enhanced Imaging. Partially coherent X-rays will be available by means of a white beam slit in the front end.
A possible bottleneck in the time schedule will be the construction and implementation of the insertion device SCUW as source of the IMAGE beamline. To bridge that time the microscopy and tomography end stations will be preliminary built and commissioned at the TOPO-TOMO beamline. A dedicated laminography set-up is already operational at the ESRF ID19 beamline, a grating interferometer for ANKA will be constructed and commissioned together with the ESRF imaging group at the ESRF ID19 beamline. Further scientific

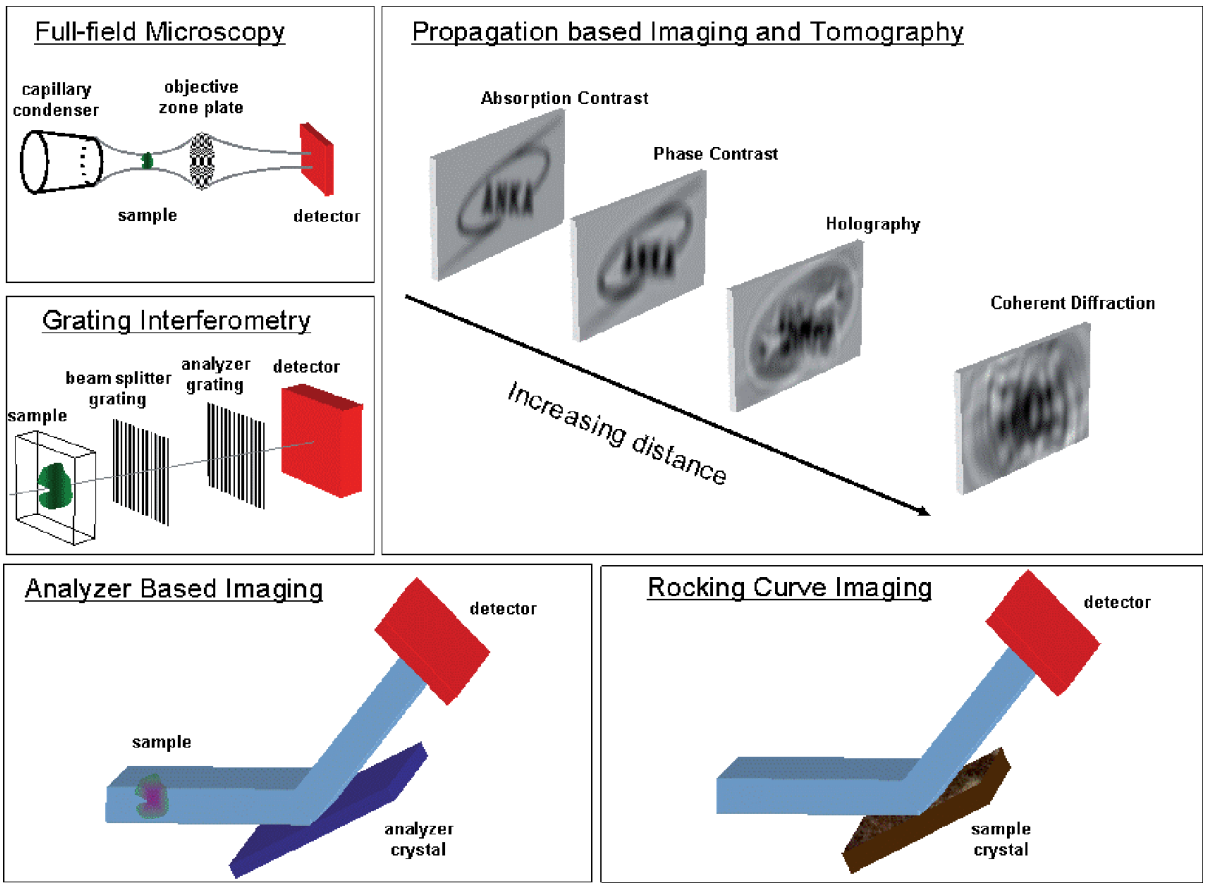

Fig. 3: *Overview of the planned methods for the Image beamline. A full-field X-ray microscope allowing high lateral resolution (down to 40 nm) via to zone plate objective lenses. A number of different phase contrast techniques will be implemented: grating interferometry, propagation-based and analyzer-based imaging will allow high phase sensitivity in the micrometer resolution range for 2D and 3D imaging. Finally the Rocking Curve Imaging technique combines Bragg diffraction information about crystal strain/stress and misorientation with a local high spatial resolution.*

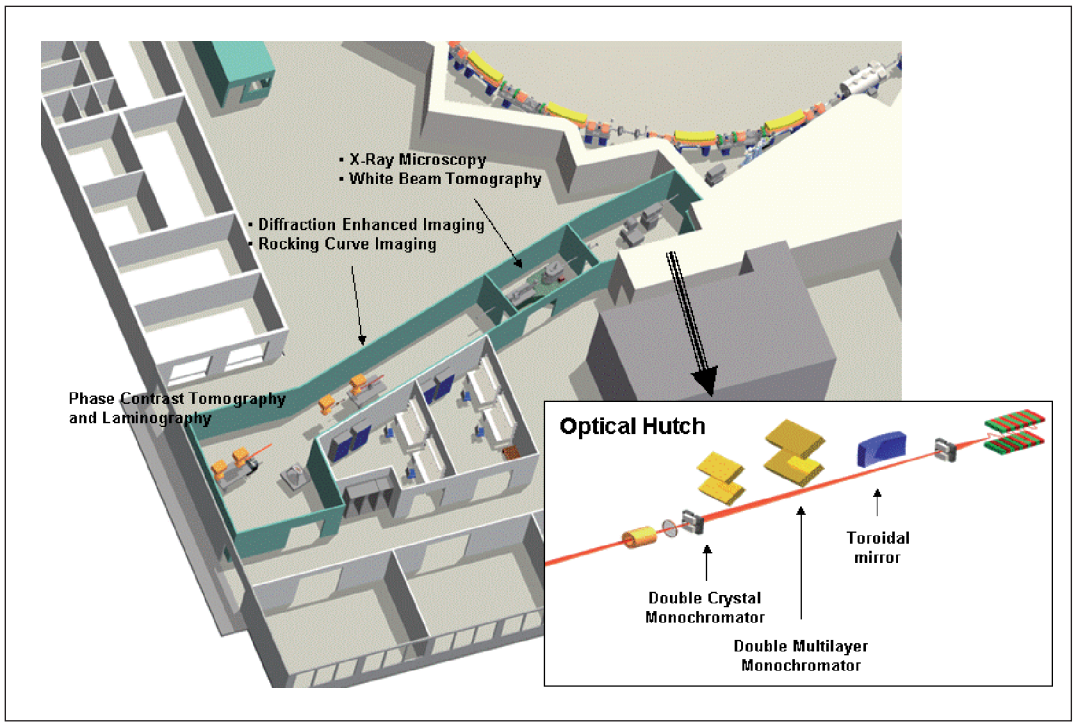

Fig. 4: *Scheme of the Image beamline and the northwest hall extension. The optical hutch is designed deliver either high flux-intensity pink beam (with the Double Multilayer Monochromator) or highly monochromatic beam (with a Double Crystal Monochromator) for the different applications. The first experimental hutch will host the X-ray microscopy station with an option to add a white beam tomography station. The other phase contrast techniques, requiring higher coherence length, will be placed in the second experimental hutch that will extend up to 42 m from the source.*

collaboration with the ESRF detector group enabled ANKA to get two FReLoN (Fast Readout Low Noise) cameras for its tomography stations, all equipped with high performance scintillators developed in the EU project ScinTax, coordinated by ANKA.
In particular the X-ray microscope is currently specified to be built for the hard X-ray energy range. It will be equipped for tomographic 3D imaging with reduced dose (if compared to soft X-ray microscopy) and will contain the capability to use elemental contrast. In contrast to station 3 it will allow for spatial resolutions below 1 µm, down to about 50 nm, special environment will be implemented for applications in nano- and microtechnologies, nanobiology and life sciences.
From 2009, the X-ray tomography and microscopy methods available at ANKA will be complemented by two of the most modern laboratory tomography instruments run by the University of Karlsruhe. From then on, services for scientific and commercial use will be bundled in the Joint Computer Tomography Lab of the Karlsruhe Institute of Technology.

Development of X-ray Pixel Array Detectors

For its imaging activities ANKA needs a pool of powerful pixel array detectors. To reach that goal ANKA has established a small group, mainly by third party funding, working within international collaborations on the development and optimization of directly converting and indirectly converting detector systems.

Indirect imaging detectors may be based on the conversion of X-rays to light by a scintillator; the luminescence image is projected via visible light optics onto a digital camera chip. The performance of such detector systems is mainly driven by the scintillating screen, the visible light optics and the read out quality of the camera. ANKA is coordinating the ScinTax collaboration in the development of scintillating materials and benefits from fruitful collaboration with ESRF in the CCD based camera development.

Direct hybrid pixel array detectors may consist of a semi-insulating pixilated sensor chip interconnected with a CMOS (Complementary Metal-Oxide Semiconductor) read out chip. This allows a high sensitivity for hard x rays, extremely useful when weak signals have to be detected. ANKA and the University of Karlsruhe are currently involved in the EDAS project *(Efficient Pixelarray Detectors for Application in the Synchrotron)* together with the University of Freiburg. The project target is the development of a fast 2D-detector (selection times <10 µs) for the detection of X-rays in highly brilliant synchrotron sources of high energy photon spanning from 20 keV to approximately 150 keV. Additionally, a pixel size of 55 µm will guarantee a sufficiently good spatial resolution. A directly converting X-ray camera will be developed based on a Hybrid Pixel Array Detector with a GaAs solid-state detector, which guarantees a high efficiency of the sensor to highly energetic X-ray radiation. In addition, sensors from CdZnTe and corroded 3D structures on silicon will be examined to investigate other possibilities of raising the sensor efficiency.

The ScinTax project

ANKA is participating and coordinating the development of an European consortium (BAM, ESRF, FZK-ANKA, CEA LETI, Optique Peter, InnospeXion, Photonic Science, FEE GmbH) funded by the European Commission for the project „ScinTax“ (STRP 033 427, FP6). The consortium aims to develop high resolution X-ray imaging detector systems based on novel scintillating thin single crystals. The scintillating crystals are based on LSO (Lu_2SiO_5) films grown by Liquid Phase Epitaxy (LPE).
During the first year of the ScinTax project, suitable substrates for the LPE process were developed and the LPE process has been optimised in order to grow LSO:Tb crystals. The grown crystals show excellent light yield and stopping power. The substrates are free of undesired parasitic luminescence that would degrade the spatial resolution.
In order to characterize the performance of the developed scintillators under excitation by synchrotron radiation, a radio-luminescence set-up has been set up at Topo-Tomo beamline. The system allows the investigation of the radio-luminescence spectrum of a wide diversity of materials under study as well as analyzing in situ changes of its scintillation behaviour (e.g. under heat load and/or intensive ionising radiation).

CCD based cameras

Besides the high quality scintillating screen, an indirect detection system requires also a sophisticated digital image acquisition system. Typically, these are CCD cameras with chips of several megapixels, trimmed for a high dynamic range. However, most of the commercial camera systems lack of high read out speed in combination with a dynamic range. Based on collaboration with the ESRF ANKA installed a FReLoN CCD camera (Fast Readout Low Noise) suitable for synchrotron-based micro-imaging. This unique in-house development of the ESRF combines outstanding dynamic range of 13.000 grey levels with high readout speed of 10 fps. Currently, the ESRF and ANKA run a common project where the standard FReLoN is upgraded with a UV coated chip. The coating extends the CCDs efficiency down to the 400 nm range, allowing a broader choice of scintillating materials to be uses which enhances the efficiency and/or the speed of the complete detector system. The coated and characterised CCD chips are now available: in 2008 a second, upgraded FReLoN camera will be implemented at an ANKA imaging set-up.
In the field of high speed imaging ANKA owns currently the fastest systems based on data acquisition by a CMOS camera. The ANKA detector group could successfully combine for the first time specially designed white beam indirect detector optics (developed at the ESRF) with the CMOS read-out. Using the white beam of ESRF's ID19 wiggler source we could reach X-ray imaging speeds of 5.000 FPS, at ID15 recently even 40.000 FPS. At ANKA we could perform in-vivo phase contrast radiographic investigation of internal morphology of insects (see image sequence in figure 3).

New synchrotron imaging techniques

In cooperation with the PSI, ESRF, APS, and the universities of Karlsruhe, Berlin, and UCSB Santa Barbara, the IMAGING group is developing new imaging methods and devices:

- New phase contrast methods, coherent imaging by the use of waveguides or gratings, diffraction enhanced imaging by using Bragg magnifiers;
- Synchrotron laminography: development of a laminography instrument for large samples, including positioning and scanning robotics;
- Full-field microdiffraction imaging;
- Theoretical modeling of coherent imaging with synchrotron sources and X-ray optics.

Grating interferometry, for instance, allows realizing phase imaging also with sources providing less coherent wave properties at the samples position such as the ANKA light source. Participation in the development of grating interferometry continues in a collaboration program with PSI and ESRF, IMT and its industrial partners.
ANKA and University of Karlsruhe are driving the development of X-ray laminography for applied research motivated by the Fraunhofer Society. Extending enables the 3D-tomographic techniques to laterally extended specimen, laminography for the first time including high-resolution 3D imaging of the micro- and nanostructure in laterally extended specimen, components, micro- and nanosystems, organic tissues and organisms, paintings, paleontological specimen etc. Its main advantage in comparison to computed tomography is the avoidance of destruction of the specimen due to sample removal and preparation. ANKA installed the method and instrumentation at ESRF (ID 19) for phase contrast laminography and at ID15 for white beam laminography (figure 5) and is transferring its know-how presently to the APS (2BM beamline). In collaboration with the imaging groups of ESRF and APS, ANKA is going to combine laminography with projection microscopy and lens based microscopy, pushing the resolution limits into the nanometer range.

Further methodical and technical developments concern

- Time resolved 2D and 3D imaging;
- Theoretical modeling of coherent imaging with synchrotron sources and X-ray optics. Theory and application of coherent diffraction imaging to nanostructures;
- Design for an integrated system for experiment control and data analysis;
- Development of fast algorithms for 3D tomographic volumes reconstruction and algorithms for 2D, 3D and 4D data processing and analysis including optical flow.

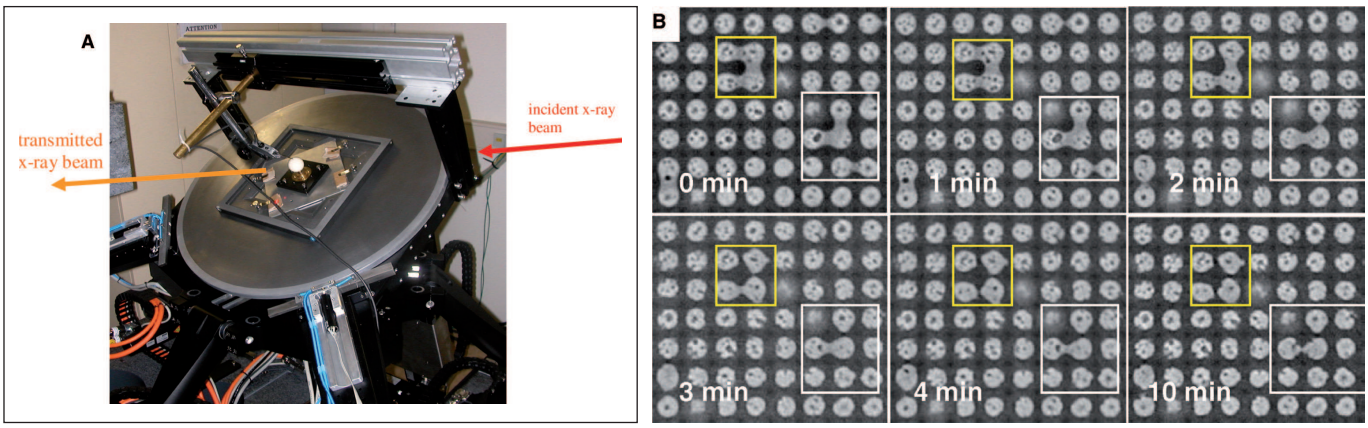

Fig. 5: *A. Manipulator stage of the Synchrotron Laminography station. The red and orange arrows indicate the propagation direction of the X-ray beam before and after transmission by the specimen; B. In situ imaging under heat treatment: AuSn solder flow on Si-GaAs assemblies; Repair of AuSn solder joint array (55 µm pitch) under heat treatment at T=300°C. Reconstructed slices after 0,1,2,3,4,10 min of heat treatment.*

Main publications

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