

## Synchrotron Radiation Based Imaging Methods for Industrial Applications at the German Synchrotron ANKA

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### Abstract

Synchrotron radiation imaging methods have been proven to be highly suitable for investigations in materials research and non-destructive evaluation. The intense flux and partial spatial coherence available at modern synchrotron light sources allows one to work with high resolutions and different contrast modes. This article gives an overview about different direct and indirect imaging methods for industrial applications, recently available for commercial access via the German light source ANKA and its department ANKA commercial services (ANKA COS).

**Keywords:** synchrotron imaging, topography, laminography, materials research, non-destructive evaluation, microtomography, radiography, micro-diffraction, phase contrast, coherent imaging

### 1. Introduction

The Synchrotron light source ANKA, located near Karlsruhe (Germany) as part of the Forschungszentrum Karlsruhe (Karlsruhe Research Center - FZK), started operation by the end of the year 2000. It is operated now by the Institute for Synchrotron Radiation (ISS) with user operation since 2003. The 2.5 GeV storage ring delivers radiation for currently 13 beamlines (2 of them under commissioning, additionally 2 insertion device beamlines are in planning) which are mostly situated at bending magnet ports. ANKA's characteristic wavelength of 2 Å is well suited for all kinds of X-ray diffraction analysis and imaging methods. As large scale facility of the Helmholtz Association of National Research Centers, ANKA is part of the national and European infrastructure offered to scientific and commercial users for performing excellent science and technological development [1].

Commercial users have full access to professional services of the ANKA facility and the FZK infrastructure on a contractual basis via ANKA's commercial services (ANKA COS) [2].

In 2005 an imaging group has been established within the ISS for the development of instrumentation and methods and their application in scientific users' and commercial customers' projects. Currently a bending magnet beamline called TopoTomo is available for imaging, a dedicated insertion device beamline is under construction and via external cooperations (University of Karlsruhe, European Synchrotron Radiation Facility) further experimental stations are accessible. A cooperation with the Fraunhofer Institut für Techno- und Wirtschaftsmathematik (Kaiserslautern, Germany) is the basis for development and application of quantitative image analysis methods on three-dimensional (3D) volume data [3]. The imaging group also has a leading role in a European research project for the development of novel X-ray detectors based on thin scintillating crystals (SCIN<sup>TAX</sup>). These detectors will be characterized and used at TopoTomo for topography, microtomography and -radiography.

## 2. White beam synchrotron X-ray topography and diffraction imaging

White beam synchrotron X-ray topography is a non-destructive characterisation method for defects and strain in bulk crystals, electronic devices and epitaxial layers. The strength of the method is the easy operation in combination with a high resolution. The principle idea is sketched in figure 1: the white beam is diffracted by a crystal. Every diffraction vector which fulfils Bragg's law results in one topograph from the same sample area, by using X-ray films one can record a Laue pattern of topographs with one single exposure. Every inhomogeneity (e.g. dislocations) in the crystal structures leads to a violation of Bragg's law and therefore to an intensity modification in the corresponding topograph, a sample topograph can be seen in figure 2 (left) [4, 5]. Large area and section topography allow for a quantitative analysis of the type of dislocations and the dislocation density. For highly absorbing materials the back reflection geometry is applied to investigate dislocation networks and small angle grain boundaries. A grazing incidence method is used to characterise strain and defects as a function of depth by varying the tilt of the sample [4,5].

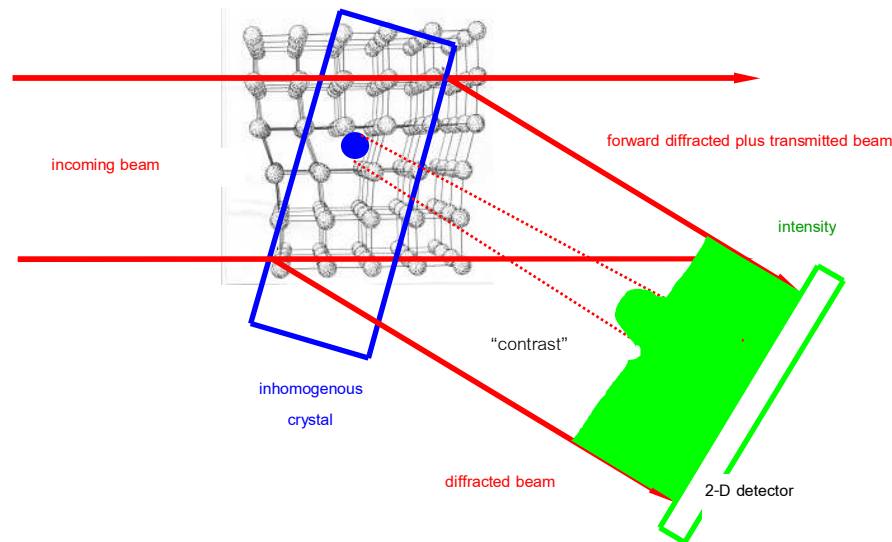


Figure 1. Contrast in X-ray topography: defects in a single-crystal do not fulfil the Bragg condition for a selected diffracted beam which leads to a change of intensity in the corresponding topograph [4].

X-ray films are frequently used to record a Laue pattern of topographs due to their large field of view, high sensitivity and high resolution. The rather long post-processing time of X-ray films usually do not allow for an automatisation and also high resolution films tend to disappear more and more from the market. High resolution imaging pixel detectors are one approach to replace X-ray films due to their high dynamic, high speed and the high resolutions available [7]. Because of their limited field of view they are currently mostly suited to record single topographs (see as example figure 2 (left)), in order to obtain a complete Laue pattern of topographs moving the detector to different positions is required. An application of this so-called digital white beam synchrotron X-ray topography is to record a selected topograph, e.g. of a larger wafer, and via a mapping to image stress, strains or dislocations over the whole wafer area (for up to 300 mm diameter) – see figure 2 (right).

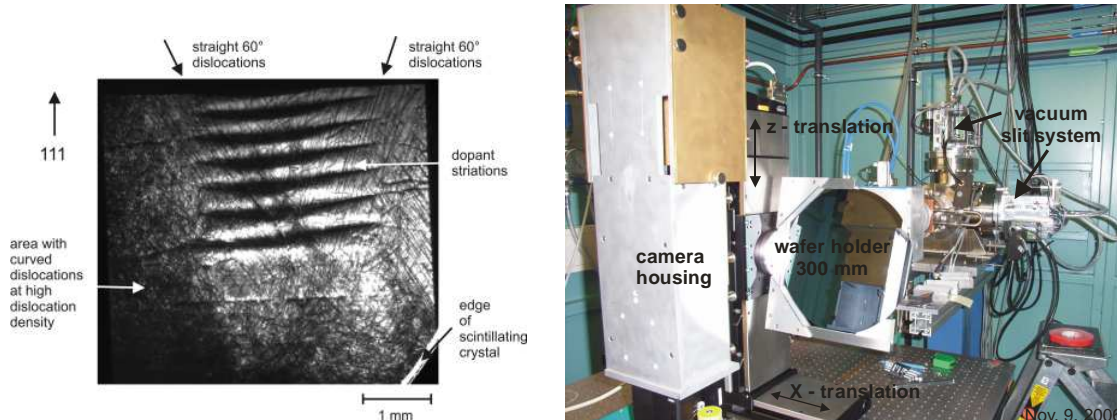


Figure 2. Left: digitally recorded 111 topograph of a highly sulphur doped InP crystals [6]. Right: experimental stage for mapping large wafers (up to 300 mm diameter) with digital white beam synchrotron X-ray topography [8].

X-ray topography extends to more general fullfield microdiffraction imaging schemes, e.g. rocking curve imaging. Here, rocking curves corresponding to different areas of the sample are measured by recording classical topographs with a monochromatic synchrotron beam while tilting the sample around a rocking angle – see figure 3 (top). By analysing the rocking curves one can measure stress, mosaicity and curvature in any kind of crystalline structure, e.g. again to be applied to wafers in order to optimize the fabrication technology. This technique is very promising for quality imaging of microelectronic devices [9].

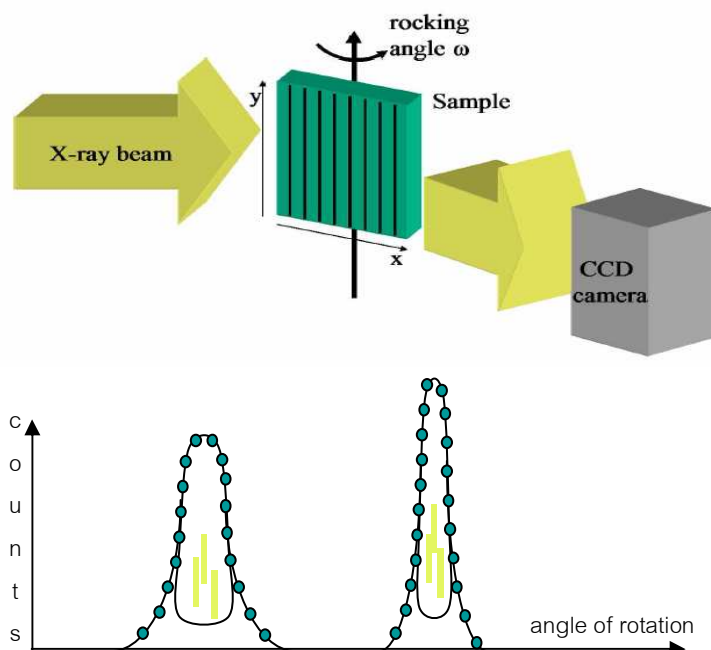


Figure 3. Top: rocking curve imaging - a monochromatized synchrotron radiation beam is diffracted by the sample; the spatial variation of the diffracted intensity over the surface of the sample is recorded by a pixel detector (classical topography). By recording a series of such images for different sample rocking angles, a whole set of rocking curves can be measured simultaneously. Bottom: rocking curve analysis – Bragg position (misorientation, stress), FWHM (dislocations, macrodefects, stress, inclusions) [9].

### 3. Synchrotron microradiography and -tomography

Microradiography and –tomography are well-established methods for the non-destructive evaluation and materials research [10, 11]. The use of synchrotron radiation instead of laboratory sources for tomography and radiography allows to extend the resolution to the submicrometer scale, to reduce noise, beamhardening and conebeam artifacts as well as increasing the contrast by the use of monochromatic radiation [7, 10, 11, 12, 13] – see also figure 4. This is due to the nearly parallel beam propagation and intense flux of synchrotron light sources. Additionally synchrotron light has a partial spatial coherence which allows one to use interference effects in order to increase the contrast, e.g. phase contrast and holo-tomography [11, 14]. High-resolution and phase contrast radiography are used to investigate micro-structured, multi-component material systems, e.g. to detect delaminations between substrates and glob tops encapsulating wire-bonded devices. Radiographs taken from different projection angles for computed microtomography allow to image objects in three dimensions with a spatial resolution down to the sub-micrometer range, e.g. bio-ceramics in regenerating bone tissue. The subsequent application of 3D image analysis methods derived from stochastic geometry can be used for the determination of size distributions, orientations or spatial correlations within the tomographic, multi-constituent volume images [3].

At ANKA we use two different optical systems for the indirect detection of X-rays by projecting the luminescence image of a scintillator magnified via visible light microscope optics onto a CCD. One is optimised for moderate resolutions and large fields of view used to image bigger objects with more than 1 cm diameter (macroscope). The second one is optimised for highest resolutions down to submicrometer scale. Further details can be found in table 1.

**Table 1. Specifications of optical systems used at ANKA for microimaging**

	<b>Macroscope "BAMline"</b>	<b>Microscope "OptiquePeter"</b>
<b>magnifications</b>	<b>3.6x, 1.6x</b>	<b>4x, 8x, 10x, 20x, 40x</b>
<b>highest resolution R / max. field of view</b>	<b>R &gt; 5 <math>\mu\text{m}</math> (2.5 <math>\mu\text{m}</math> pixel size) 22 mm x 15 mm</b>	<b>R &gt; .7 <math>\mu\text{m}</math> (0.35 <math>\mu\text{m}</math> pixel size) 7 mm x 7 mm</b>

These optics can be combined with different cameras depending on the application. Our PCO4000 uses a large (11 megapixel) chip which makes it well-suited for the macroscope as well as highest resolution with the microscope in order to extend the field of view. A FReLoN *2k14bit* fulfils the needs for high dynamic tomographic scans with short exposure times ( $\ll 1$  s) and a high duty cycle, e.g. with the microscope system and 8x to 20x magnification. Further details on the camera systems currently available at ANKA can be found in table 2.

**Table 2. Specifications of camera systems used at ANKA for microimaging**

	<b>FReLoN <i>2k14bit</i></b>	<b>PCO4000</b>
<b>chip</b>	<b>ATMEL TH7899M, 2048 x 2048, 14 <math>\mu\text{m}</math></b>	<b>KODAK KAI-11000, 4008 x 2672, 9 <math>\mu\text{m}</math></b>
<b>max. dynamic / max. framerate (full frame)</b>	<b>13.000:1 20 FPS</b>	<b>5.000:1 5 FPS</b>

Currently CMOS cameras are commissioned by the ISS imaging group with the aim of being able to work with framerates of several thousand images per second with a one megapixel chip and a micro-resolution. By using a region of interest framerates up to 10.000 FPS should be possible. A corresponding CMOS camera will be available by the end of this year. Additionally ANKA and the ESRF developing together the FReLoN *2k14bit* further in order to increase the cameras efficiency down to 400 nm, allowing one to achieve higher resolutions and to use faster and more denser scintillating materials. A first new FReLoN should be running in 2008.

#### 4. Synchrotron computed laminography

Microtomography is limited to scan objects which fit into the field of view of the imaging system. Objects which are slightly bigger than the field of view can be used as well (local tomography), frequently introducing artifacts. In order to be able to image laterally extended samples, digital laminography for laboratory applications was implemented in the nineties where a X-ray source is rotated around the flat sample with an inclined angle of projection [15]. Recently, synchrotron computed laminography has been implemented at the ESRF beamline ID19 by members of the ISS Imaging group in order to image flat and laterally extended objects with high spatial resolution in three dimensions. Due to the fact that the synchrotron radiation source is fixed, the sample is rotated with an inclined axis of rotation (cf. figure 4 (top, right – laminography and CT - left)). The inclined axis of rotation results in a different scanning of the Fourier space (see figure 4 – bottom), omitting some spatial frequencies but allowing one to image flat extended samples in three dimensions. Flip-chip bonded and wire-bonded devices are examples which show the potential of this method for typical industrial microsystem application like the detection of mm-sized voids within flip-chip solder bumps [16].

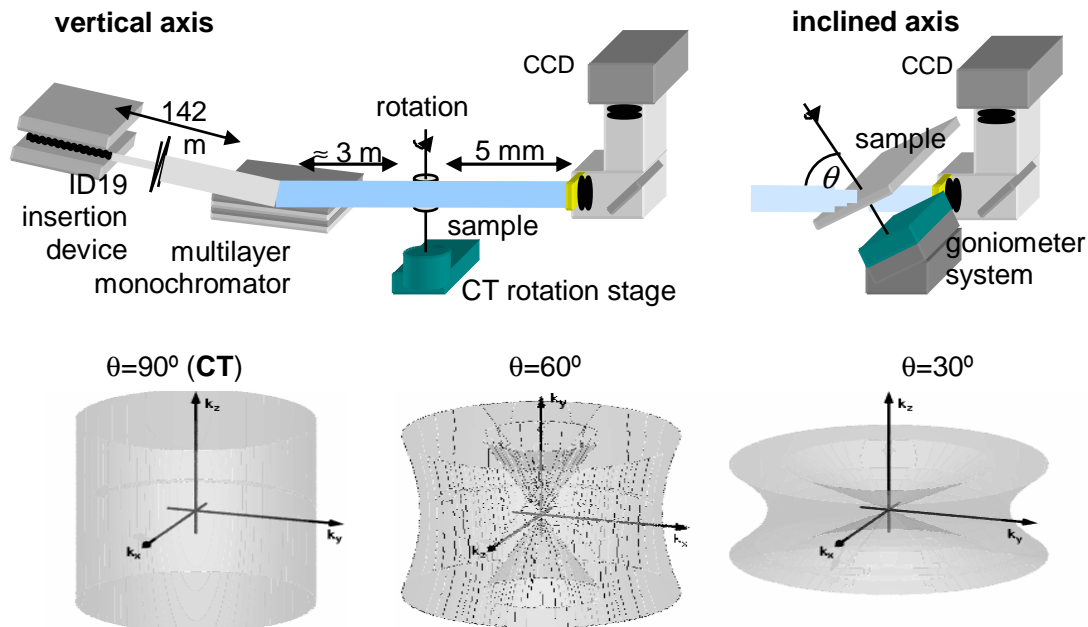


Figure 4. Top: Sketch of the synchrotron microtomography facility at the beamline ID19 of the ESRF [14] (left) and modified setup for synchrotron laminography [16] (right). Bottom: comparison of sampling Fourier space for microtomography (CT) and laminography (inclined axis of rotation) [16].

## 5. Summary

This article gives a short overview about imaging methods for industrial applications accessible via our department ANKA commercial services (ANKA COS) [2]. For different examples for applications the reader is asked to follow the citations, visit our website or directly get in touch with us.

Further imaging activities at ANKA which have not been mentioned in this article are the development and production of refractive lenses fabricated by deep synchrotron radiation lithography [17], fluorescence imaging [18] and the construction of an insertion device beamline at ANKA, dedicated to host different imaging methods [1].

## References

1. <http://www.fzk.de/anka/> - last visit July 2007, ANKA Instrumentation Book (November 2005/2)
2. <http://www.anka-online.de/> - last visit July 2007.
3. <http://www.itwm.fhg.de/mab/projects/MAVI/> - last visit July 2007.
4. T Tuomi and K Naukkarinen and P Rabe, Phys. Stat. Sol. (a), 25 (93), 1974.
5. R Simon and A N Danilewsky, 'The experimental station for white beam X-ray topography at the synchrotron light source ANKA, Karlsruhe', Nucl. Instr. & Meth. Phys. Res. B, Vol 199, pp 550-553, 2003.
6. A N Danilewsky and A Rack, 'Digital White Beam X-ray Topography', ANKA Highlights 2006.
7. U Bonse and F Busch, 'X-ray computed microtomography ( $\mu$ CT) using synchrotron radiation (SR)', Prog. Biophys. Molec. Biol., Vol 65, pp 133-169, 1996.
8. A N Danilewsky and A Rack and T Weitkamp and J Wittge and H Riesemeier and R Simon and T Baumbach, 'White beam synchrotron topography using a digital X-ray imaging detector at ANKA's TopoTomo beamline', Nucl. Instr. & Meth. Phys. Res. B, in preparation, 2007.
9. D Lübbert and T Baumbach and J Härtwig and E Boller and P Pernot, ' $\mu$ m-resolved high resolution X-ray diffraction imaging for semiconductor quality control', Nucl. Instr. & Meth. Phys. Res. B, Vol. 160, pp 521-527, 2000.
10. J Goebbels and B Illerhaus and Y Onel and H Riesemeier and G Weidemann, '3D-Computed Tomography over four Order of Magnitude of X-Ray Energies', Proc. 16th World Conference on Nondestructive Testing (WCNDT2004), Montreal, Canada, [www.ndt.net/article/wcndt2004/pdf/radiography/559\\_goebbels.pdf](http://www.ndt.net/article/wcndt2004/pdf/radiography/559_goebbels.pdf), 2004.
11. J Banhart (Ed), 'Advanced Tomographic Methods in Materials Research and Engineering', Oxford University Press, Oxford, 2007.
12. B P Flannery and H W Deckmann and W G Roberge and K L D'Amico, 'Three-dimensional x-ray microtomography', Science, Vol 237, No 4821, pp 1439-1444, 1987.
13. A Koch and C Raven and P Spanne and A Snigirev, 'X-ray imaging with submicrometer resolution employing transparent luminescent screens', J. Opt. Soc. Am., Vol 15, No 7, pp 1940-1951, 1998.
14. P Cloetens and W Ludwig and J Baruchel and D Van Dyck and J Van Landuyt and J P Guigay and M Schlenker, 'Holotomography: Quantitative phase

- tomography with micrometer resolution using hard synchrotron radiation x rays', *Appl Phys Lett* Vol 75, No 19, pp 2912-2914, 1999.
15. U Ewert and J Robbel and C Bellon and A Schumm and C Nockemann, 'Digital Laminography', *Materialforschung*, Vol 37, No 6, pp 218-222, 1995.
  16. L Helfen and T Baumbach and T Mikulik and D Kiel and P Pernot and P Cloetens and J Baruchel, 'High-resolution three-dimensional imaging of flat objects by synchrotron-radiation computed laminography', *Appl Phys Lett* Vol 86, Article 071915, 2005.
  17. V Nazmov and E Reznikova and M Boerner and J Mohr and V Saile and A Snigirev and I Snigireva and M DiMichiel and M Drakopoulos and R Simon and M Grigorie, 'Refractive lenses fabricated by deep SR lithography and LIGA technology for X-ray energies from 1 keV to 1 MeV', *AIP Conference Proceedings (SRI2004)*, Vol 705, pp 752-755, 2004.
  18. R Simon and G Buth and M Hagelstein, 'The X-ray fluorescence facility at ANKA, Karlsruhe: Minimum detection limits and micro probe capabilities', *Nucl. Instr. & Meth. Phys. Res. B*, Vol 199, pp 554-558, 2003.