1	Diffraction and transmission synchrotron imaging at the German light source ANKA – potential
2	industrial applications
3	Alexander Rack ^{1,2*} , Lukas Helfen ¹ , Timm Weitkamp ^{1,2} , Andreas N. Danilewsky ³ ,
4	Rolf Simon ¹ , Daniel Lübbert ¹ , Tilo Baumbach ¹
5	¹ Institute for Synchrotron Radiation – ANKA, Forschungszentrum Karlsruhe / K.I.T., Postfach 3640, D-76021
6	Karlsruhe, Germany, *e-mail: arack@snafu.de
7	² European Synchrotron Radiation Facility, BP 220, F-38043 Grenoble Cedex, France
8	³ Crystallographic Institute, University Freiburg, Hermann-Herder-Str. 5, D-79104 Freiburg, Germany
9	Abstract
10	Diffraction and transmission synchrotron imaging methods have proven to be highly suitable for investigations
11	in materials research and non-destructive evaluation. The high flux and spatial coherence of X-rays from modern
12	synchrotron light sources allows one to work using high resolution and different contrast modalities. This article
13	gives a short overview of different transmission and diffraction imaging methods with high potential for
14	industrial applications, now available for commercial access via the German light source ANKA
15	(Forschungszentrum Karlsruhe) and its new department ANKA Commercial Service (ANKA COS,
16	http://www.anka-cos.de).
17	Keywords: X-ray topography, laminography, micro-tomography, radiography, micro-diffraction, synchrotron
18	radiation
19	PACS : 07.85.Qe, 81.70.Tx, 61.72.Ff
20	1. Introduction
21	The synchrotron light source ANKA (Ångstromquelle Karlsruhe), located near Karlsruhe (Germany) as part of
22	$the\ For schungszentrum\ Karlsruhe\ (Karlsruhe\ Research\ Centre-FZK\ /\ K.I.T.)\ is\ operated\ by\ the\ Institute\ for\ New Centre-FZK\ /\ K.I.T.$
23	Synchrotron Radiation (ISS). First user experiments were carried out in 2003. The 2.5 GeV storage ring delivers
24	radiation for currently 13 beamlines (2 of them under commissioning, 2 additional insertion device beamlines are
25	in the construction phase) which are mostly situated at bending magnet ports. The energy spectrum available at
26	ANKA is well suited for all kinds of X-ray diffraction analysis and imaging methods. As large scale facility of
27	the Helmholtz Association of National Research Centres, ANKA is part of the national and European
28	infrastructure offered to scientific and commercial users for performing excellent science and technological
	1

26

27

- 1 development [1]. Commercial users have full access to professional services of the ANKA facility and the FZK 2 infrastructure on a contractual basis via ANKA's Commercial Service department (ANKA COS) [2]. 3 In 2005 an X-ray imaging group has been established within the ISS for the development of instrumentation and 4 methods and their application in scientific users' and commercial customers' projects. Currently a bending 5 magnet beamline named TopoTomo is available for imaging, a dedicated insertion device beamline is under 6 construction and further experimental stations are accessible via external cooperations (University of Karlsruhe, 7 European Synchrotron Radiation Facility). A cooperation with the Fraunhofer Institut für Techno- und 8 Wirtschaftsmathematik (Kaiserslautern, Germany) is the basis for development and application of quantitative 9 image analysis methods on three-dimensional (3D) volume data [3]. The imaging group also has a leading role in 10 a European research project for the development of novel X-ray detectors based on thin scintillating crystals (SCIN^{TAX} - http://www.scintax.eu). These detectors will be characterized and used at TopoTomo for topography, 11 12 micro-tomography and -radiography [21, 22]. 13 2. White beam synchrotron X-ray topography and diffraction imaging 14 White beam synchrotron X-ray topography is a non-destructive characterisation method for defects and strain in 15 bulk crystals, electronic devices and epitaxial layers. The strength of the method is the easy operation in 16 combination with high spatial resolution. The principle idea is sketched in figure 1: the white beam is diffracted 17 by a crystal. Every diffraction vector which fulfils Bragg's law results in one topograph from the same sample 18 area, by using X-ray films one can record a Laue pattern of topographs with one single exposure. Every 19 inhomogeneity (e.g. dislocations) in the crystal structure leads to a violation of Bragg's law and therefore to an 20 intensity modification in the corresponding topograph. An example of a topograph can be seen in figure 2 (left) 21 [4, 5]. Large area and section topography allow for a quantitative analysis of the type of dislocations and the 22 dislocation density. For highly absorbing materials the back reflection geometry is applied to investigate 23 dislocation networks and small angle grain boundaries. A grazing incidence method is used to characterise strain 24 and defects as a function of depth by varying the tilt of the sample [4, 5].
 - 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

14

15

16

17

18

19

20

21

22

23

24

25

26

27

- 1 imaging pixel detectors are one approach to replace X-ray films. They combine comparable spatial resolution,
- 2 high dynamic range and high frame repetition rates [7, 8]. As their field of view is limited (see as example figure
- 3 2 left), moving the detector to different positions is required to obtain a complete Laue pattern of topographs
- 4 [8]. An application of this so-called digital white beam synchrotron X-ray topography is to record a selected
- 5 topograph, e.g. of a large wafer, and via a mapping to image stress, strains or dislocations over the whole wafer
- 6 area (for up to 300 mm diameter) see figure 2 (right) [6].
- 7 X-ray topography can be extended to more general full field micro-diffraction imaging schemes, e.g. rocking
- 8 curve imaging. Here, rocking curves corresponding to different areas of the sample are measured by recording
- 9 classical topographs with a monochromatic synchrotron beam while tilting the sample around a rocking angle –
- 10 see figure 3 (top). By analysing the rocking curves one can measure stress, mosaicity and curvature in any kind
- 11 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].

3. Synchrotron micro-radiography and -tomography

Micro-radiography 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 sub-micrometre scale, to reduce noise, beam hardening and cone beam artefacts 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 degree of spatial coherence that allows to use interference effects, e.g. phase contrast and holo-tomography, in order to increase the contrast [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 micro-tomography allow to image objects in three dimensions with a spatial resolution up to the sub-micrometre range, e.g. bio-ceramics in regenerating bone tissue. Subsequent 3D image analysis, for example by methods derived from stochastic geometry, can be used for the determination of size distributions, orientations or spatial correlations within tomographic, multi-constituent volume images [3].

At ANKA we use two different optical systems for the indirect detection of X-rays by projecting the

- 1 luminescence image of a scintillator magnified via visible light microscope optics onto, for example, a CCD.
- 2 One is optimised for moderate resolutions and large fields of view used to image bigger objects with more than 1
- 3 cm diameter (macroscope). The second one is optimised for highest resolutions down to sub-micrometre scale.
- 4 These optics can be combined with different digital cameras depending on the application. A commercially
- 5 available PCO.4000 (PCO AG / Cooke Cooperation) with a large (11 megapixel, interline transfer CCD) chip
- 6 can be used with the macroscope as well as for high resolution with the microscope in order to extend the field of
- 7 view. A FReLoN 2k14bit fulfils the needs for high dynamic tomographic scans with short exposure times
- 8 (<< 1 s) and a high duty cycle, e.g. with the microscope system and 8x to 20x magnification [23].
- 9 Currently high speed CMOS cameras are applied by the ISS imaging group with the aim to being able to work
- 10 with frame rates of several thousand images per second employing a one megapixel chip and spatial micro-
- 11 resolution. By using a region of interest, frame rates of several 10 000 FPS are possible [20]. Additionally
- 12 ANKA and the ESRF developing together the FReLoN 2k14bit further in order to increase the cameras
- 13 efficiency down to 400 nm, allowing one to achieve higher resolutions and to use faster and more denser
- scintillating materials. A first new FReLoN will be commissioned in the second half of 2008.

4. Synchrotron computed laminography

15

16

17

18

19

20

21

22

23

24

25

26

27

Micro-tomography 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 artefacts. In order to obtain volume image data of laterally extended samples, digital laminography for laboratory applications was implemented in the 1990s. Here, an X-ray source is rotated around the flat sample with an inclined angle of projection [15]. Recently, synchrotron computed laminography has been developed and 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 micro system application like the detection of mm-sized voids within solder bumps [16].

1 5. Summary

- 2 This article gives a short overview of imaging methods for industrial applications accessible via our department
- 3 ANKA Commercial Service (ANKA COS) [2]. Further imaging activities at ANKA which have not been
- 4 mentioned in detail in this article are the development and production of refractive lenses fabricated by deep
- 5 synchrotron radiation lithography [17], fluorescence imaging [18] and the construction of an insertion device
- 6 beamline at ANKA, dedicated to host different imaging methods [1].
- 7 This contribution was funded by the CAARI2008 local organizing committee.
- 8 1. http://www.fzk.de/anka/ last visit July 2008, ANKA Instrumentation Book (November 2007/3).
- 9 2. http://www.anka-cos.de/ last visit July 2008.
- 10 3. http://www.itwm.fhg.de/mab/projects/MAVI/ last visit July 2008.
- 11 4. T. Tuomi, K. Naukkarinnen, P. Rabe, Phys. Stat. Sol. (a), 25 (1974), 93.
- 12 5. R. Simon, A. N. Danilewsky, Nucl. Instr. & Meth. Phys. Res. B 199 (2003), p. 550.
- 13 6. A. N. Danilewsky, J. Wittge, A. Rack, T. Weitkamp, R. Simon, P. McNally, J. Mat. Sci.: Mat. in Electr.
- *in print* (2008), DOI 10.1007/s10854-007-9480-5.
- 15 7. U. Bonse, F. Busch, Prog. Biophys. Molec. Biol. 65 (1996), p. 133.
- 16 8. A. N. Danilewsky, A. Rack, J. Wittge, T. Weitkamp, R. Simon, H. Riesemeier, T. Baumbach, Nucl.
- 17 Instr. & Meth. Phys. Res. B 266 (2008), p. 2035.
- 18 9. D. Lübbert, T. Baumbach, J. Härtwig, E. Boller, P. Pernot, Nucl. Instr. & Meth. Phys. Res. B 160
- 19 (2000), p. 521.
- 20 10. J. Goebbels, B. Illerhaus, Y. Onel, H. Riesemeier, G. Weidemann, in: Proc. 16th World Conference on
- Nondestructive Testing (WCNDT2004), Montreal, Canada, (2004),
- www.ndt.net/article/wcndt2004/pdf/radiography/559_goebbels.pdf .

- 1 11. J. Banhart (Ed.), 'Advanced Tomographic Methods in Materials Research and Engineering', Oxford
- 2 University Press, Oxford, 2008.
- 3 12. B. P. Flannery, H. W. Deckmann, W. G. Roberge, K. L. D'Amico, Science 237 (1987), p. 1439.
- 4 13. A. Koch, C. Raven, P. Spanne, A. Snigirev, J. Opt. Soc. Am. 15 (1998), p. 1940.
- 5 14. P. Cloetens, W. Ludwig, J. Baruchel, D. Van Dyck, J. Van Landuyt, J. P. Guigay, M. Schlenker, Appl.
- 6 Phys. Lett. 75 (1999), p. 2912.
- 7 15. U. Ewert, J. Robbel, C. Bellon, A. Schumm, C. Nockemann, Materialforschung 37 (1995), p. 218.
- 8 16. L. Helfen, T. Baumbach, T. Mikulik, D. Kiel, P. Pernot, P. Cloetens, J. Baruchel, Appl. Phys. Lett. 86
- 9 (2005), 071915.
- 10 17. V. Nazmov, E. Reznikova, M. Boerner, J. Mohr, V. Saile, A. Snigirev, I. Snigireva, M. DiMichiel, M.
- Drakopoulos, R. Simon, M. Grigorie, AIP Conference Proceedings (SRI2004) 705 (2004), p. 752.
- 12 18. R. Simon, G. Buth, M. Hagelstein, Nucl. Instr. & Meth. Phys. Res. B 199 (2003), p. 554.
- 13 19. X-ray diffraction imaging (X-ray topography) –
- 14 http://www.esrf.fr/UsersAndScience/Experiments/Imaging/ID19/Techniques/Diffraction/Overview (last visit
- 15 August 2008).
- 16 20. F. Garcia Moreno, A. Rack, L. Helfen, T. Baumbach, S. Zabler, N. Babcsan, J. Banhart, T. Martin, C.
- 17 Ponchut, M. Di Michel, Appl. Phys. Lett. 92 (2008), 134104.
- 18 21. K. Dupré, M. Couchaud, T. Martin, A. Rack, German Patent Application, ref. no. 10 2007 054 700.7
- 19 (2007).
- 20 22. A. Cecilia, A. Rack, T. Baumbach, P.-A. Douissard, T. Martin, M. Couchaud, K. Dupré, J.
- 21 Luminescence, *submitted* (2008).
- 22 23. J.-C. Labiche, O. Mathon, S. Pascarelli, M. A. Newton, G. Guilera Ferre, C. Curfs, G. Vaughan, A.
- 23 Homs, D. F. Carreiras, Rev. Sci. Instr. 78 (2007), 091301.

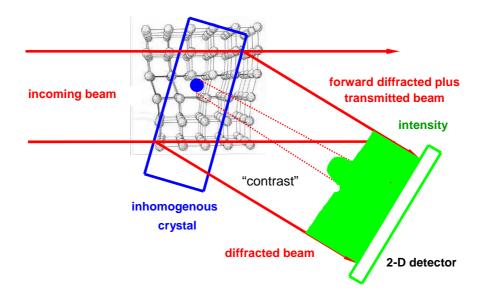
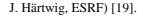


Figure 1. Contrast in X-ray topography: defects in a single-crystal do not fulfil the Bragg condition for a selected diffracted beam, which leads to a change of intensity in the corresponding topograph (Figure courtesy



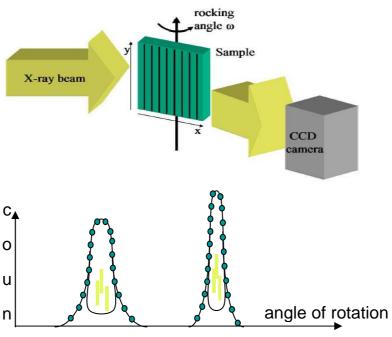


Figure 3. Top: rocking curve imaging - a monochromatized synchrotron radiation beam is diffracted by the sample; the spatial variation of the diffracted intensity over the surface of the sample is recorded by a pixel detector (classical topography). By recording a series of such images for different 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].

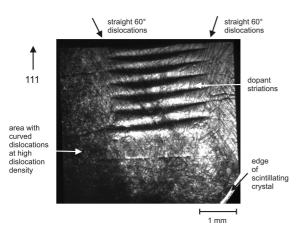




Figure 2. Left: digitally recorded 111 topograph of a highly sulphur doped InP crystals [8]. Right: experimental stage for mapping large wafers (up to 300 mm diameter) with digital white beam synchrotron X-ray topography [6].

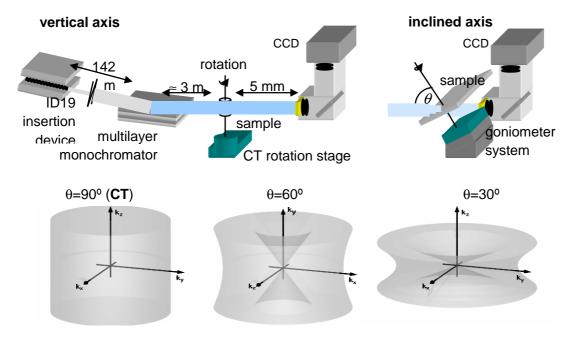


Figure 4. Top: Sketch of the synchrotron micro-tomography facility at the beamline ID19 of the ESRF [14] (left)

and modified setup for synchrotron laminography [16] (right). Bottom: comparison of sampling Fourier space

for micro-tomography (CT) and laminography (inclined axis of rotation) [16].