An die Geschäftsstelle der
Helmholz-Gemeinschaft
Ahrstraße 45
53175 Bonn

Zwischenbericht (Sachlicher Teil)
14.3.2008

Nano-Scale Imaging Using Coherent Hard X-rays
VH-VI-203

Virtuelles Instituts im Rahmen des Impuls- und
Vernetzungsfonds des Präsidenten der Helmholtz-Gemeinschaft

Sprecher:
Georg-August-University Göttingen
Institut für Röntgenphysik
Prof. Dr. Tim Salditt
Friedrich-Hund-Platz 1
D-37077 Göttingen
e-mail: tsaldit@gwdg.de
Tel.: 0551 399427
Fax: 0551 399430

Federführendes Helmholtz-Zentrum:
Deutsches Elektronensynchrotron
Hamburger Sychrotronstrahlungslabor
Prof. Dr. Edgar Weckert
Notkestr. 85
D-22607 Hamburg
e-mail: edgar.weckert@desy.de
Tel.: 040 8998 4509
Fax: 040 8998 4475

Leitende Wissenschaftler der beteiligten Gruppen:

Deutsches Elektronensynchrotron
Hamburger Sychrotronstrahlungslabor
Prof. Dr. Edgar Weckert
Notkestr. 85
D-22607 Hamburg
e-mail: edgar.weckert@desy.de
Tel.: 040 8998 4509
Fax: 040 8998 4475

Technische Universität Dresden
Institut für Strukturphysik
Prof. Dr. Christian Schroer
Zellescher Weg 16
D-01062 Dresden
e-mail: schroer@physik.tu-dresden.de
Tel.: 0351 463 37589
Fax: 0351 463 37048

Georg-August-University Göttingen
Institut für Röntgenphysik
Prof. Dr. Tim Salditt
Friedrich-Hund-Platz 1
D-37077 Göttingen
e-mail: tsaldit@gwdg.de
Tel.: 0551 399427
Fax: 0551 309430

Deutsches Elektronensynchrotron
Hamburger Synchrotronstrahlungslabor
Dr. Ivan Vartaniants
Notkestr. 85
D-22607 Hamburg
e-mail: ivan.vartaniants@desy.de
Tel.: 040 8998 2653
Fax: 040 8998 4475

1. Joint Research Activities in 4/2007-4/2008

A coherent diffraction experiment at the newly commissioned cSAXS beamline of PSI was carried out with joint staff from all three groups. The experiments were prepared, carried out, and the data is currently analyzed in a highly synergistic manner. Special emphasis during the first year of the VI was given to the education and training of Ph.D. and Diploma students in all three groups.
The main joint beamtime was carried out with 5 Ph.D. students and 2 Diploma students, mainly in order to disseminate the corresponding knowledge.

Experimental Report: SLS Proposal ID 20070205

Abstract and Scope - We report on a Coherent Diffraction Imaging (CDI) Experiment at the cSAXS beamline of the SLS. The following goals of the experiment were reached: (A) Instrumentation for CDI methods was tested and benchmarked (pinhole fabrication for illumination, holders and manipulation of biological samples); (B) coherent diffraction data was collected in different modes (slit illumination, pinhole illumination, ptychography) from prefabricated test patterns; (C) CDI was applied to freeze-dried biological cells. To this end, samples of the slime mould Dictyostelium Discoideum (Dicty cells) in the vegetative state were measured. While it has been applied to freeze-dried cell using soft x-rays before, the present work presents is a first-time application of CDI to biological cells in the hard X-ray regime, in particular using the ptychography approach. Workpackages and goals (A) and (B) were carried out in the framework of the virtual institute (VI) for coherent x-ray diffraction. Data analysis for all project parts is ongoing.

Test samples: (i) Patterns defined in Au on thin (about 150nm Si) foils were fabricated by e-beam lithography followed by lift-off techniques. (ii) Test patterns etched in a 40-80 nm layer of Au deposited on thin Si foils were structured by a focused Ga ion beam (FIB). Both type of samples were used as test objects, with sample cross sections ranging in between 5 μm and 10 μm. The patterns comprised a siemens star, different logos and resolution objects, including a framed pattern suitable for reconstruction by direct inversion according to the recent algorithm of direct inversion (Podorov et al. 2007). For illumination, commercial electron microscopy pinholes in 200 μm thick Pt-Ir foils have been used after further treatment of edges by FIB. The test patterns were aligned optically (X-ray scintillator foil).

Biological Samples: Freeze dried Dicty cells spreading on polyimide foils were shock frozen by plunging in liquid ethane. The polyimide foils, (microspoons, Mitigen Inc.) were fixed on magnetic sample holders (Hampton research). The position of the sample in the pinhole beam was controlled in-line by phase contrast X-ray imaging (using the scintillator foil with a drilled hole at the cSAXS beamline.

Experimental settings: A photon energy of 6.8 keV (5th undulator harmonic) was set by the Si (111) double monochromator positioned upstream from the (unbent) mirror (SiO_2, 0.2° incidence angle) used for rejection of higher harmonics. Data were taken with a CCD (PI-SCX-4300) with a 1:1 tapered glass fiber system and a high resolution scintillator foil (YAG:Cr). Samples were measured in full beam and ptychography mode. Using the pinhole illumination the scintillator CCD camera allowed 0.01 sec illumination without beamstop. This allowed us to obtain intensity values for the central pixels, which are else hidden by the beamstop (BS). For long exposures, two BS sizes were used (3.5 and 1.5 mm diameters, respectively), and later combined to one image (dynamic synthesis for high dynamic range).

First Results – As illustrated in Fig. 1 and Fig.2, the farfield patterns of the FIB-cleaned pinhole series indicates that the treatment of the pinhole was very successful. In particular, the data looks much cleaner and more regular than that of untreated pinholes. More than fifty oscillations are observed, over the entire range of the CCD including the corners. The coherent superposition of the pinhole and sample is a key effect which must be included in the object reconstruction. We see that this interference signal can enhance the signal. How and if the empty pinhole will help to phase the object is subject to the ongoing work of data analysis.

From the freeze dried Dicty cells data with high signal to noise were obtained. The polyimide foils and sample holders were tested successfully, and will be used for cryogenic imaging (no freeze drying necessary) in the next beamtime.

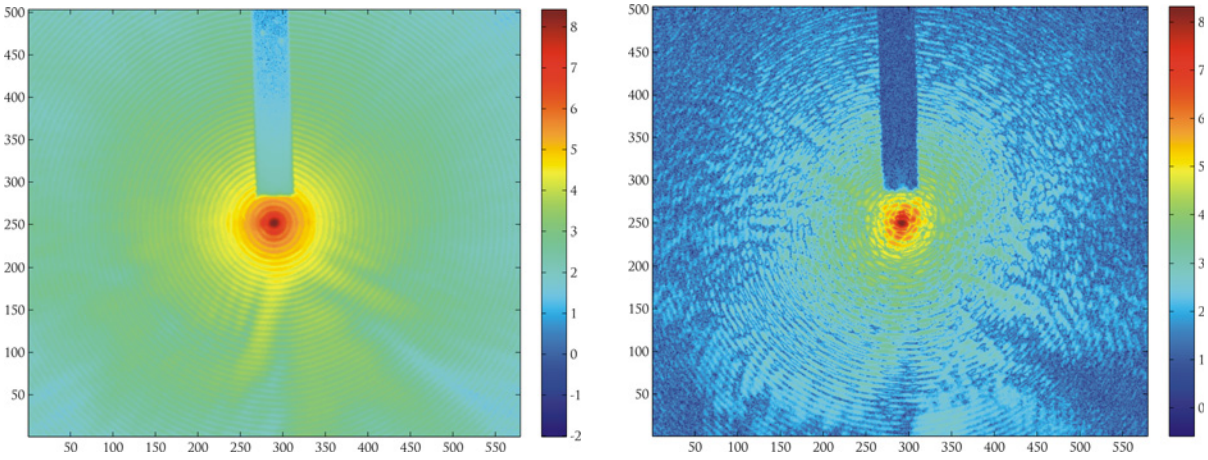

Figure 1: Farfield pattern of the (left) empty sample showing the farfield pattern of the FIB-treated pinhole, (right) the pattern of a freeze dried Dicty cell, which was scanned in the beam (Giewekemeyer et al, in preparation).

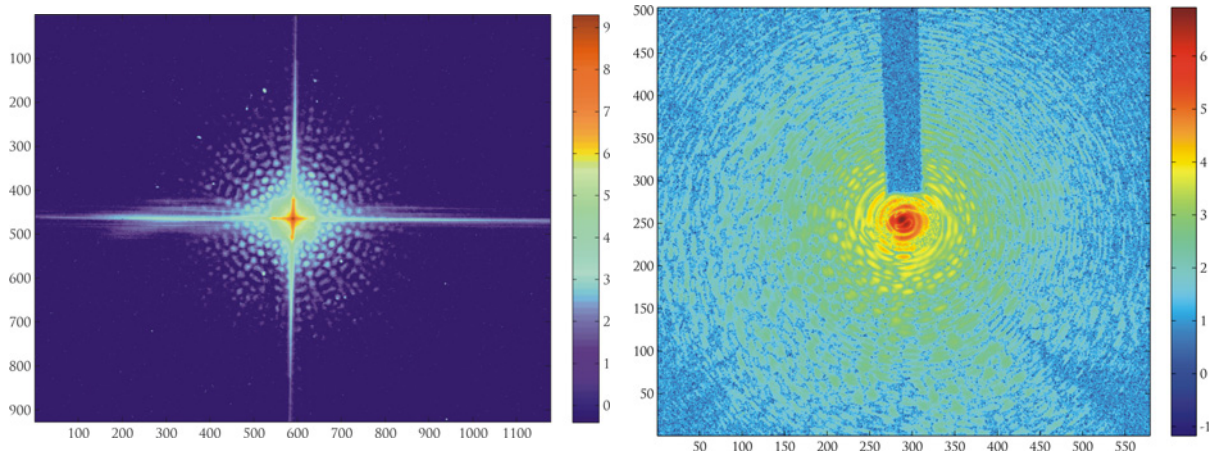

Figure 2: Farfield pattern of the same siemens star test object ("Fib2_Star"), under two entirely different illumination conditions: (left) Illumination with slit collimation. The farfield is readily identified as that of a siemens star in contrast to (right) , where interference terms between pinhole and object complicate the direct visual interpretation. Note that in (right) the object was positioned behind a 10 µm pinhole (pin#9). A 100 s exposure with beamstop has been merged with a short exposure without beamstop to illuminate the central part (dynamic synthesis). As far as can be judged at present, better quality data was obtained with pinhole illumination, since the pinhole scattering amplitude seems to add coherently to the sample amplitude, thereby enhancing the signal. Using slits, spurious (imperfect) slit scattering dominated on the orthogonal axes and in the central region, spoiling the information in the corresponding pixels (in particular at low q).

2. Joint meetings and workshops

The following VI meetings have been held:

6.7.2007 Göttingen: Opening Workshop
Talks and Discussion.
Discussion on aspects of the coherence beamline at PETRA III.

19.10.2007 Dresden
Talks and Discussion, preparation of common beamtime.

7.3.2008 Hamburg
Talls, Discussion, in particular on data analysis.

15.4.2008 TU Dresden: meeting T. Salditt, C. Schroer, discussion on VI related research activities.

3. Göttingen group

During the period of the report, the Göttingen group has addressed the following work packages and has achieved the following goals:

- Preparation of focused ion beam (FIB) test samples for CDI
- use focused ion beam (FIB) for treatment of CDI pinholes
- preparation of high transmission waveguides based on a two-component cladding
- implementation and test of plunge freezing for vitrification of biological cells
- CDI experiment on (i) freeze dried and (ii) cryogenically vitrified bacterial and eukaryotic cells
- Design and test of 3-pinhole illumination system and instrumentation for CDI
- Construction and commissioning of optical test bench for CDI (based on 632 nm He NE laser)

Publications in direct and/or indirect relation to VI project:

A. Kohlstedt, S. Kalbfleisch, T. Salditt, M. Reiche, U. Gösele, E. Lima, P. Willmott
Two-dimensional X-ray waveguides: fabrication by wafer-bonding process and characterization
Applied Physics A 91, 6-12 (2008)

C. Ollinger, C. Fuhse, S. Kalbfleisch, R. Tucoulou, and T. Salditt
Object localization with 10 nm accuracy by x-ray phase contrast projection imaging
Appl. Phys. Lett. 91, 051110 (2007)

A. Jarre, J. Seeger, C. Ollinger, C. Fuhse, C. David and T. Salditt
X-ray Waveguide Nanostructures - Design, Fabrication and Characterization
Journal of Applied Physics 101, 054306 (2007)

S. Panknin , A. K. Hartmann, T. Salditt
X-Ray Propagation in Tapered Waveguides: Simulation and Optimization
Optics Communication, in press

Talks related to VI project:

Tim Salditt

14.01.08 Physik-Kolloquium Universität Erlangen
Physik und Anwendungen von Röntgenwellenleitern

22.01.08 Physik-Kolloquium Universität Kiel
Mit Röntgenaugen im Nanokosmos

23.01.08 Nanoprobe at PETRA III Workshop, DESY, Hamburg
Hard x-ray nanoprobe of biological systems: fluorescence, diffraction and phase contrast

07.03.08 Research Course DESY, Hamburg
Imaging applications to bio-materials

15.04.08 Physik Kolloquium Dresden
Struktur und Dynamik in biologischen Modellmembranen:
Von Streuexperimenten zu Abbildungen mit Röntgenstrahlung

4. HASYLAB group

The HASYLAB group has actively pursued coherent imaging experiments both at synchrotron beamlines as well as at FLASH, and has worked on reconstruction and algorithms.

The results of the PSI/cSAXS experiment 11/2007 are now analyzed in collaboration between the different teams of the VI. Johannes Gulden from the HASYLAB team has obtained preliminary results on the reconstruction of the pinhole illumination function, see Figure 4 (work in progress).

Figure 3*: 4 best reconstructions of pinhole9 (approx. 8um) after 1950 it.*
All phases set to 0

5. TU Dresden group

The Dresden Group has performed a CDI experiment using a (coherently) focussed beam by compound refractive lenses. Placing a Au nanocrystal (size ca. 90 nm) shown in Fig. 4(a) in the focus with a beam diameter of about 150 nm, far-field images with high fringe visibility could be collected [Fig. 4(b)]. In this way it was demonstrated that indeed diffraction limited focusing yields a nanobeam with high lateral coherence (milestone 1 of coherent diffraction with nanobeam). From the diffraction pattern the gold sample was reliably reconstructed with a resolution of about 5 nm using the standard hybrid input-output algorithm [Fig. 4(c)]. The support constraint was set using the shrink-wrap algorithm (both algorithms were implemented in the *tomo* software package developed by C. Schroer and A. Schropp). The experiment demonstrates that in agreement with numerical simulations the wave front is flat in the focus and paves the way to a systematic combination of nanofocusing and coherent diffraction imaging.

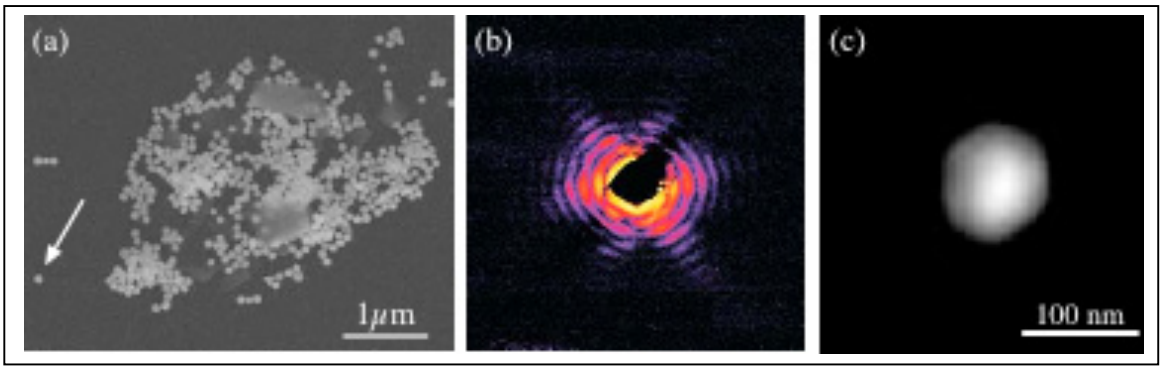

***Figure 4**: (a) Gold nanoparticles on Si_3N_4 membrane. The white arrow points to the gold particle under investigation. (b) Coherent diffraction pattern of the gold particle illuminated with a nanobeam with a diameter of about 150 nm. (c) Reconstructed gold particle.*

Publications in direct and/or indirect relation to VI project:

C. G. Schroer, P. Boye, J. Feldkamp, J. Patommel, A. Schropp, A. Schwab, S. Stephan, M. Burghammer, S. Schöder, C. Riekel
Coherent X-Ray Diffraction Imaging with Nanofocused Illumination
Physical Review Letters, submitted

Talks related to VI project:

Christian Schroer

18.04.08 404. Heraeusseminar Matter in Coherent Light, Bad Honnef
Wave-Optical Properties of Refractive X-Ray Lenses (invited)

15.11.07 Prozessnahe Röntgenanalytik ProRA2007, Berlin
Refraktive Optiken für die Röntgenmikroskopie

24.10.07 ESRF Upgrade Meeting, Grenoble (invited)
PETRA III: Hard X-ray Micro- and Nanoprobe

17.07.07 Festkörperanalytik, Wien (keynote lecture)
Mikroskopie und Tomographie mit analytischem Kontrast

04.07.07 1st Meeting of the Virtual Institute VI-203 in Göttingen
Coherent Diffraction Imaging with Small Beams

23.04.07 APS Upgrade Workshop, Argonne, IL, USA (invited)
Nanofocusing Hard X Rays with Refractive Lenses

Ivan Vartaniants

15.01.08 Physics-Colloquium Technical University, Dresden
"Coherent X-ray Diffraction of Nanometer Size Objects"

19.02.08 Workshop on Photon Science with Applications to Nano- and Life- Science, RRC "Kurchatov Intitute", Moscow, Russia
Coherent X-ray Scattering at Synchrotron and FEL Sources

23.01.08 Physics-Colloquium, Bohr Institute, University of Copenhagen, Copenhagen
Coherent Diffractive Imaging at Synchrotron and FEL Sources

07.03.08 Physics-Colloquium, Rostock University
Coherent X-ray Diffractive Imaging of Nanometer Size Crystals

Seminars and Workshops Related to VI Project

Research Course on New X-ray Sciences "New Materials in New Light" March 5-7, 2008 at DESY, Hamburg
Organizers: I. Vartaniants, R. Feidenhans'l, Th. Tschentscher

404 WE-Heraeus-Seminar "Matter in Coherent Light", March 17-20, 2008 at the Physikzentrum Bad Honnef, Germany
Organizers: E. Weckert, G. Gruebel, I. Vartaniants