Mid Term Report (Sachbericht)

Fördermaßnahme: Virtuelle Institute	
Förder-Nr.:	VH-VI-302
Titel des Vorhabens:	Femtosecond x-ray science: FLASH imaging of nanoparticles and biosamples
Sprecher der Kollaboration:	Prof. Dr. Thomas Möller
Projektadministration:	Dr. Gerhard Grübel
Federführendes Helmholtz-Zentrum:	Deutsches Elektronen-Synchrotron DESY
Beteiligte Universitäten und andere Partner:	TU Berlin, Uppsala University, Universität Hamburg
Berichtszeitraum:	*1.3.2010 bis 31.12.2010*

Mit Term Report

a) Progress on the working plan of the proposal

The Virtuelles Institut (VI) 'Femtosecond x-ray science: FLASH imaging of nano-particles and biosamples' planned and organized 2 meetings in 2010 uniting the involved groups from the universities of Uppsala, Berlin and Hamburg as well as the group from DESY. The first meeting took place on January 27, 2010 in the context of the HASYLAB Users Meeting in Hamburg. Progress and measures as well as the budget situation within the program were discussed. The second meeting was held in the context of the "Science at FLASH" Workshop, held on September 27 in Hamburg. The granted extension of the project (without additional funds) until August 31, 2011 will allow to achieve the anticipated project goals.

W**ork-package 1** the FLASH SPIDER project was postponed to a later time commensurate with the FLASH schedule (see report 2009). The funds for the SPIDER project (29 kEuro) were re-distributed to work-package 5 allowing the purchase of an urgently needed soft X-ray CCD camera.
Within **work-package 5** ("Coherence and correlations of FEL sources") the first experiments characterizing the transverse and longitudinal coherence properties in the hard x-ray regime (λ=1.37Å) of the LCLS FEL were carried out. These experiments were carried out in single shot mode by using either colloidal liquids (for the low Q regime) or a gold nano-powder (for the large Q regime). Figure 1 shows a single pulse hard X-ray speckle pattern. Figure 2 shows the speckle-contrast, the intensity histogram and an intensity autocorrelation function for the low Q regime. As can be seen from the figure the contrast is 0.94(3) supporting the fact of a fully coherent source. The large Q data (Q=2.6Å-1) show a speckle contrast of about 0.3 in agreement with the expectations. Detailed data analysis is ongoing.

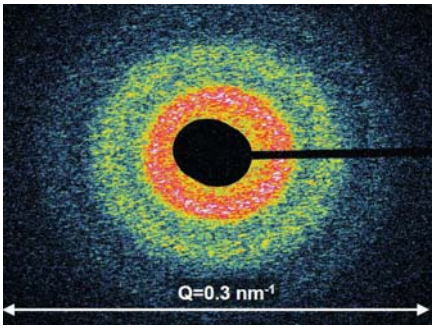

Figure 1: *Single pulse hard X-ray speckle pattern captured from nano-particles in a colloidal liquid (photon wavelength λ = 1.37 Å). The inset shows a cut of the photon intensity.*

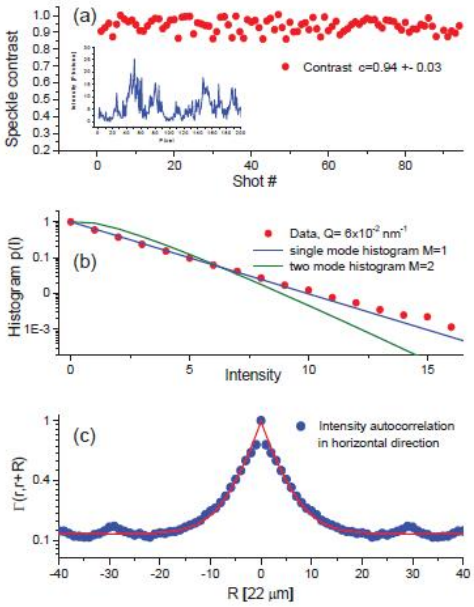

Figure 2: *(a) Single pulse speckle contrast (visibility) for different shots from the LCLS recorded in SAXS setup ($Q=6\cdot 10^{-2}$ nm^{-1}. (b) Histogram of the intensity distribution from a typical single pulse speckle pattern. Blue solid line: fit according to Eq. 1 assuming a single transverse mode (fully coherent). For reasons of comparison: Green line shows Eq. 1 assuming two transverse modes present. (c) Intensity autocorrelation function in the horizontal direction yielding the speckle width and illumination function (R is the pixel coordinate on the camera).*

Work on the **workpackages 2 and 4**, coordinated by the TU-Berlin, has continued in 2010. In **workpackage 4** the concept for time resolved measurements was further developed. In parallel analysis of results from LCLS obtained in collaboration with the groups of J. Hajdu (Uppsala) and T. Ditmire (Austin) has started. In these experiments ionisationsynamics in Argon-, Xenon- and Methane-Clusters was studied between 800 eV and 2000 eV. In a second experiment in May 2010 the TU Berlin group collaborated with the CAMP consortium to carry out IR-pump X-ray probe experiments. This experiment was very successful revealing the diffraction images from "exploding" clusters as a function of the delay time.

Work-package 3 made major progress in 2010:

Two papers published on coherent diffractive imaging in Nature:

***i) Femtosecond X-ray protein nanocrystallography* (Chapman et al. 2011).**

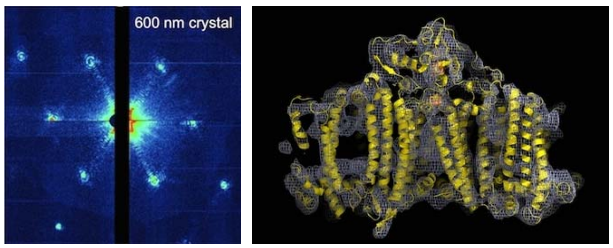

Figure 3: *Diffraction from a photosystem I nanocrystal and the 3D structure of this membrane protein from many such diffraction patterns.*

Many macromolecules yield poorly diffracting crystals, even after extensive efforts. In conventional measurements, the necessary increase in X-ray dose to record data from nanocrystals leads to extensive damage before a diffraction signal can be recorded. We mitigate the problem of radiation damage in such measurements by using pulses briefer than the time-scale of most damage processes and have used this method for the structure determination of target proteins that form nanocrystals. It is particularly challenging to obtain large well-diffracting crystals of membrane proteins, for which less than 300 unique structures have been determined. We developed a method for structure determination where single-crystal X-ray diffraction "snapshots" are collected from a fully hydrated stream of nanocrystals using femtosecond pulses from the LCLS. We tested this concept on Photosystem I, a large membrane protein complex. Over 3 million diffraction patterns were collected

in 5 days in this study, and a 3D data set was assembled from a subset of these exposures from which an electron density map could be calculated.

***ii) Single Mimivirus particles intercepted and imaged with an X-ray laser* (Seibert et al. 2011).**
Mimivirus is the largest known virus and it is visible in an optical microscope. It is too big for a full three-dimensional reconstruction by electron microscopy, and its core is surrounded by fibrils, preventing crystallisation. The size of the viral core is comparable to the size of the smallest living cells. The identification of this organism as a virus is transforming virology.

Purified mimivirus particles were transferred into a volatile buffer. The aerosol (in a wet helium atmosphere) passed through a differentially pumped aerodynamic lens. Particles focused by the lens entered the interaction zone and were intercepted randomly by the LCLS pulses. The diffraction patterns of free-flying virus particles (a, b) were exceptionally clean and background scattering from residual gas did not exceed the detector readout noise. (a,b) Diffraction patterns. (c) EM image. (d) Autocorrelation function for (a). (e) Reconstructions for (a,b). The reconstructions are 2D projections from two different orientations. The results show no measurable sample deterioration during exposure in the virus (Seibert et al. 2011).

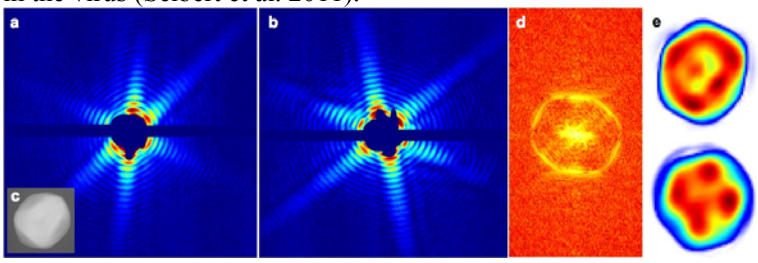

Figure 4 : *Single Mimivirus particles intercepted and imaged with an X-ray laser (Seibert et al. 2011).*

The orientation of the 260 diffraction patterns was recovered from the diffraction data alone using a new expectation maximalisation algorithm in the Hawk software package). The phases of the Fourier density were recovered from these data by iterative phase retrieval algorithms. The reconstruction did not impose any symmetry on the object, yet returned the pseudo-icosahedral shape of the particle while also revealing (for the first time) the asymmetric structure of the interior of a virus. If confirmed, this is a breakthrough result. The picture shows a compartmentalized interior structure that exhibits an unexpected distribution of dense material (presumably the viral DNA genome). The penetration depth of X-rays permits studies on the interiors of large objects. The CXDI methods applied here require no modifications to the sample such as staining, freezing, sectioning, radiolabelling or crystallisation, and can also be used to image cells that are alive at the time of the exposure.

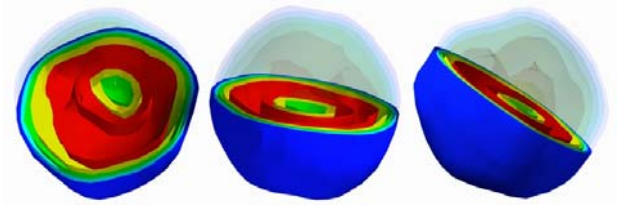

Figure 5: *First 3D reconstruction of an intact biological object (mimivirus) from diffraction data obtained with an X-ray laser (Tomas Ekeberg et al.manuscript in preparation).*

A world-wide data bank for coherent X-ray diffractive imaging.
We were the first to make publicly available coherent diffraction data collected at FLASH and the LCLS. Databases containing experimental data are crucially important for research and education. The Protein Data Bank is a remarkably successful example of such a database. Our Coherent X-ray Imaging Data Bank (CXIDB, http://www.cxidb.org) is dedicated to the archival and sharing of data

from FEL experiments. FEL data are currently available only to an extremely limited number of people. CXIDB aims to let anyone upload experimental data and browse the data deposited by others.

Publicly available software for analysing, assembling, and phasing continuous diffraction patterns. A new software package has been developed in Uppsala for processing diffraction data from FEL experiments (*Hawk,* http://xray.bmc.uu.se/hawk). This is the first publicly available suite of programs for reconstructing images from continuous diffraction patterns (Maia et al. 2010). *Hawk* handles all steps of reconstruction from raw diffraction patterns to a reconstructed image, including geometry determination, background correction, masking and phasing. It also includes 3D support and support for GPUs using the CUDA architecture. *Hawk* provides a framework for creating high-performance imaging algorithms. The package is built on cross-platform open-source libraries and works in all major operating systems, so that it is usable by as many people as possible. Our aim is to turn *Hawk* into a software package similar to the CCP4 suite of programs in protein crystallography.

The goal of **work-package 6** is to explore the possibilities to manipulate (by optical means) nanoscale materials under vacuum conditions with the ultimate goal to position samples in the FEL beam. In 2010 the laser set-up for trapping nano-scaled particles was finished and tested. The originally projected integration of the laser system into the vacuum vessel of the Uppsala group has not been possible yet, since it was not installed at the Hamburg FEL facility. The modified geometrical design of the particle injector system built by the Uppsala group has turned out incompatible with the large numerical aperture (0.9) required for producing the tight laser focus. The required modifications of the focussing system have been implemented. In view of the difficulties to synchronize the activities of the Uppsala team and us, we have decided upon setting up a small vacuum system in Hamburg, which should allow us to test laser trapping with dielectric micron sized particles without having to resort to the Uppsala vacuum system.

b) Achieved milestones

- First single shot coherence characterization of LCLS hard x-ray FEL beam.
- Analysis of LCLS data onpoing.
- Analysis of FLASH and LCLS data
  taken on cluster systems, molecules and protein nano-crystals.
- 2 publications in NATURE with TU berlin participation
- All milestones outlined in the proposal have been reached.
- The optics required for forming a tightly focused optical dipole trap has been modified in order to be compatible with the geometric limitations in the Uppsala vacuum set-up. With inhouse means we have started to set up a small vacuum test chamber. In 2011 we hope to perform first experiments in this chamber.

c) Adherence to the time and financial plan.

- Progress according to time- and budget plan.
- The timeplan and the budget planning could be maintained. The succession of C. Bostedt tool longer than expected. The position was advertised and filled in the meantime. Another delay is caused by a maternity leave of a phD student. This will however not compromise the project since a 6 month extension of the project was granted.
- WP3 operates within budget and within the expected time plan of the project.
- The work in WP6 has proceeded in accordance with the financial plan.

d) Publications, Talks

Toward a single mode Free Electron Laser for coherent hard X-ray experiments
Sooheyong Lee, Zhirong Huang, Yuantao Ding, Paul Emma, Wojciech Roseker, Gerhard Grübel and Aymeric Robert
Optics Express, submitted

Development of a hard X-ray delay line for XPCS and jitter-free pump-probe experiments at XFEL sources
Wojciech Roseker, Hermann Franz, Horst Schulte-Schrepping, Anita Ehnes, Olaf Leupold, Federico Zontone, Sooheyong Lee, Aymeric Robert and Gerhard Grübel
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