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:	*1.3.2009 bis 31.12.2009*

Mit Term Report

a) Progress on the working plan of the proposal

The members of the Virtuelles Institut (VI) 'Femtosecond x-ray science: FLASH imaging of nanoparticles and biosamples' met on January 27, 2009 in Hamburg in order to review the details of the collaboration, to decide on further actions according to the working plan and to discuss details of the financial planning.

Progress was made in all domains although, due to the complexity and novelty of the project, there are small delays. **We thus ask for a prolongation of the project for 6 months.**

The prime motivation of the project is to explore and develop experimental techniques which allow structure determination and imaging of non-crystalline objects with single femtosecond x-ray pulses.

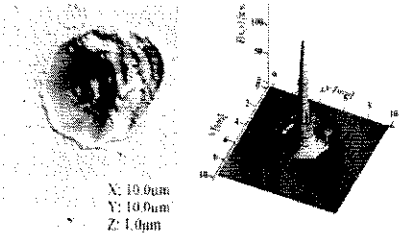
Two **work-packages (1+5)** deal with FEL pulse diagnostics and the characterization of FEL pulses. The project FLASH SPIDER of **work-package 1** aimed at the "development of a single shot full characterization of FLASH pulses by using a transposition of the SPIDER technique". It turned out that this work-package cannot be realized since the FLASH SPIDER project based on an extension of a single-shot terahertz-field-driven X-ray streak camera [1] proved to be **far more extensive** in its conduction and particularly in the evaluation of the results as originally intended and thus not compatible with the given time frame. At the same time, a set of necessary improvements of the THz-beamline at FLASH was identified. Therefore, the decision was made to delay the FLASH SPIDER project to a later time commensurate with the FLASH schedule. It is proposed to re-distribute the funds for the SPIDER project (29 kEuro) to work-package 5 allowing the purchase of an urgently needed soft X-ray CCD camera.

In **work-package 5** we presented for 2008 a first bunch-by-bunch characterization of the temporal coherence properties of the FLASH Free Electron Laser (FEL) in Hamburg. However, the unprecedented peak power of FEL sources also implies that a considerable amount of energy is deposited in the sample. The radiation damage threshold defines the borderline between non-destructive and therefore repeatable pump-probe type scattering experiments and high fluence destructive single-pulse experiments. In order to explore beam-sample interaction we performed single-pulse scattering experiments using 20.8 nm soft x-ray pulses from the free-electron laser FLASH at DESY. We could record a magnetic diffraction pattern from a Co/Pt multilayer sample with a single 30 femto-second long FEL pulse. Pulse energies of 4 mJ/cm^2 are sufficient to record a

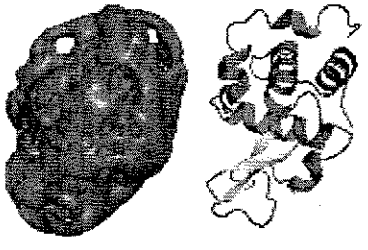
magnetic diffraction pattern within 30 fs without destroying the sample. We observed that higher pulse intensities can lead to permanent changes of the magnetic properties of the magnetic multilayer but without macroscopically observable destruction. Below that threshold it is possible to record magnetic diffraction patterns without changing the magnetic domain size distribution [2].

The team from the TU Berlin has work on the **work-packages 2 and 4**. For **work-package 2** a new optics concept for pump-probe experiments was developped allowing these type of experiments at different harmonics of the FLASH machine. In a collaboration with Lothar Strüder (Max-Planck Halbleiterlabor) high quality data at the fundamental (harmonic) were taken at FLASH in April 2009. Data taken at the third harmonic were less excellent probably due to non-adapted filters. In addition novel concepts were developped within **work-package 4** for time-resolved experiments. A proposal for LCLS was written in collaboration with J. Hajdu (Uppsala) and T. Ditmire (Austin, Texas) which was granted beamtime. In Fall 2009 the ionisation dynamics of Ar-, Xe- and Methane- Clusters were studied between 800 ev and 2000 eV.

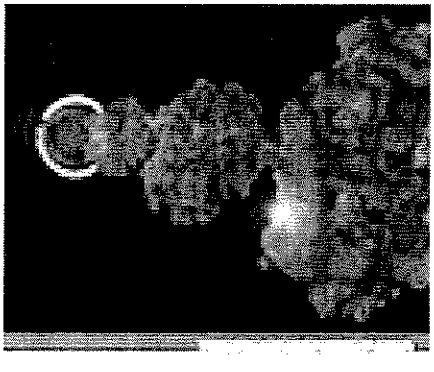
Work-package 3 made major progress in 2009:



(a) The team achieved achieved better than 1µm focus at beam line 3 of FLASH (Bajt et al. 2009; Nelson et al. 2009) at λ = 13.5 nm, pulse length 15 fs, pulse energy 10-40 µJ, 5Hz with a new off-axis parabola coated by a Mo/Si multilayer (reflectivity: 67% at 13.5 nm). This optics opens up new possibilities to study extreme states of matter at power densities exceeding 10^{17} Wcm^{-2} on the sample, and was used to create transparent aluminium. Left: AFM scan of a crater produced in a PMMA target. Right: Reconstructed beam profile shows 1 µm focus.



(b) X-ray lasers may allow structural studies on single particles and biomolecules without crystalline periodicity in the samples. We examined the effect of sample dynamics as a source of structural heterogeneity on the resolution in a reconstructed small protein molecule (Maia et al. 2009). The patterns were incoherently summed and the resulting data set was phased by oversampling. The noise introduced by the incoherent addition of dissimilar structures restricts the maximum resolution. A way of potentially reducing this effect is by grouping dynamic structures into distinct structural substates and solving them separately. This work is in progress.



(c) Simulations of protein structures in vacuo (Marklund et al. 2009) show that a small amount of water prevents unfolding over a broad temperature range. The structural conservation stems mainly from interactions with the surrounding water. The water molecules aggregate in clusters and form patterns on the protein surface, maintaining a reproducible hydrogen bonding network. The wide temperature range in which we find proteins to be stable is reassuring for the success of single particle imaging experiments.

(d) With the help of the Virtual Institute, we built a *mobile container laboratory* in Uppsala for experiments on sensitive biological samples at free-electron lasers. The first use of this lab at FLASH was made in August 2009, and at LCLS in November-December 2009. We will make the container lab available to other interested parties for experiments.

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 accordance with the project plan we have designed and implemented a NdYAG fiber laser system as the key tool for trapping nano-scaled particles. For coupling the beam into the vacuum chamber a photonic bandgap fiber has been set up. A vacuum compatible microscope objective with 0.9 numerical aperture has been designed to be used for the formation of a tight beam focus inside the vacuum. It has been agreed with the collaborators in Uppsala, that starting in august 2010 their vacuum vessel with integrated particle injector will be installed at the Hamburg FEL facility. At this point the laser system prepared by our group can be implemented.

b) Achieved milestones

- Feasibility study for FLASH Spider project.
- Quantitative study of damage thresholds for (magnetic) multilayer samples.
- Time resolved experiments. A "two-color" multilayer mirror was developped, commissioned and used.
- Successful cluster experiments at LCLS
- Sample injection for picoplankton is operational. Flash imaging experiments on single picoplankton and virus particles have been performed.
- A NdYAG fiber laser system and the optics required for forming a tightly focused optical dipole trap has been set up.

c) Adherence to the time and financial plan.

- Allocation of funds for the WP 1: SPIDER project (29 kEuro) to work-package 5 allowing the purchase of an urgently needed soft X-ray CCD camera.
- The time and spending plan for WP 2 + 4 is commensurate with the planning. Dr. Bostedt has accepted a position in Stanford. This was beneficial for the experiments at LCLS. His position at TU Berlin was advertised and recruitment is in progress.
- WP3 Funds (Euro 23479) were spent via DESY for 2 microscopes.
- Sachmittel WP3 for Uppsala University will be requested soon.
- Remaining WP3 funds (Euro 35945) are already committed and will be requested soon.
- Because of the late start of WP 6 due to a delay in finding appropriate personel a part of the financial means for 2008 were transfered to 2009. The acqusition of the NdYAG fiber laser required to combine investment funds for 2008 and 2009.

d) Publications, Talks

Single-shot terahertz-field-driven X-ray streak camera

U. Frühling et al., Nature Photonics 3, 523 (2009)

Single-pulse resonant magnetic scattering using a soft x-ray free-electron laser
C. Gutt et al., Phys. Rev. B 81, 100401(R), (2010)

G. Grübel, Invited Talks, Magnetism Workshop, Trieste, Dec. 16-18, 2009;
PNSXM 2009, Bonn, August 4, 2009; Summer School, Nanomagnetism, Bochum-Duisburg, October 12-16, 2009

C. Gutt, Invited talk, SNI Meeting Berlin 24-26 Februar, 2010

Energetics, Ionization and expansion dynamics of atomic clusters irradiated with short intense vacuum-ultraviolet pulses
B. Ziaja, H. Wabnitz, F. Wang, E. Weckert, T. Möller Phys. Rev. Lett. 102, 205002 (2009)

Shell explosion and core expansion of xenon clusters irradiated with intense femtosecond soft x-ray pulses
H. Thomas, C. Bostedt,. M. Hoener, E. Eremina, T. Fennel, K.H. Meiwes-Broer, M. Kuhlmann, E. Plönjes, R. Treusch, A.R.B. de Castro, and T. Möller, J. Phys. B. 42, 134018 (2009)

Experiments with fast spectroscopic pnCCDs at BESSY and FLASH VUV-FEL
C. Reich, R. Hartmann, H. Soltau, L. Strüder, N. Meidinger, U.Pietsch, W.Leitenberger, C. Bostedt, T. Möller, IEEE Nuclear Scieince Symposium Vol 1-9, 1860 (2009)

Large-Format, High-Speed, X-ray pnCCDs Combined with Electron and Ion Imaging Spectrometers in a Multipurpose Chamber for Experiments at 4th Generation Light Sources
L. Strüder, S. Epp, D. Rolles, R. Hartmann , P. Holl , G. Lutz , H. Soltau, R. Eckart , C. Reich , K. Heinzinger , C. Thamm , A. Rudenko, F. Krasniqi, K.U Kühnel, C.Bauer , C.D. Schröter, R. Moshammer, S. Techert , D. Miessner , M. Porro, O. Hälker, N. Meidinger, N. Kimmel, R. Andritschke, F. Schopper, G. Weidenspointner, A. Ziegler D. Pietschner , S.Herrmann, U. Pietsch, A. Walenta, W. Leitenberger, C.Bostedt, T.Möller, D. Rupp , M.Adolph, H. Graafsma , H. Hirsemann, K. Gärtner, R. Richter , L. Foucar, R. L. Shoeman, I. Schlichting J. Ullrich,
Nucl. Instr. Meth. A 610, 483 (2010)

Fast electrons from multi-electron dynamics in xenon clusters induced by inner-shell ionization with soft Xray pulses,
C. Bostedt, H. Thomas, S. Schorb, M. Hoener, T. Möller, U. Saalmann, I. Georgescu, C. Gnodtke, J.M. Rost, Phys. Rev. Lett. sumitted

Clusters in intense FLASH pulses:
Ultrafast ionsiation dynamics and electron emission studied with spectroscopic and scattering techniques
C. Bostedt, M. Adolph, E. Eremina, M. Hoener, D. Rupp, S. Schorb, H. Thomas A. R. B. de Castro and T. Möller , J.Phys. B., submitted

Sub-micron focusing of a soft X-ray Free Electron Laser beam
Bajt, S., et al.. Proc. SPIE **7361**, 73610J1-10 (2009)

Radiation damage in biological material: Electronic properties and electron impact ionization in urea
Caleman, C., et al.. Europhysics Letters (EPL) 85, 18005 (2009).

Non-thermal desorption/ablation of molecular solids induced by ultra-short soft x-ray pulses
Chalupský, L., et al.. Optics Express **17**, 208-217 (2009)

Molecular Dynamics Simulations of a Membrane Protein-Micelle Complex in Vacuo.
Friemann, R., et al. J. Am. Chem. Soc. **131**, 16606

Wavelength dependence of the damage threshold of inorganic materials under extreme-ultraviolet free-electron-laser irradiation
Hau-Riege, S. P., et al. Appl. Phys. Letts. **95**, 111104 (2009)

Structural stability of electrosprayed proteins: temperature and hydration effects
Marklund, E.G., et al.. Phys. Chem. Chem. Phys. **11**, 8069-8078 (2009).

Soft x-ray free electron laser microfocus for exploring matter under extreme conditions
Nelson, A. J., et al.. Optics Express **17**, 18271-18278 (2009)

Single-shot diffractive imaging with a table-top femtosecond soft X-ray laser-harmonics source. Physical Review Letter
Ravasio, A., et al. s, 103, 028104 (2009)

Combined X-ray and NMR analysis of the stability of the cyclotide cystine knot fold that underpins its insecticidal activity and potential use as drug scaffold
Wang, C.K., et al.. J. Biol. Chem. **284**, 10672-10683 (2009)

Encapsulation of Myoglobin in a Cetyl Trimethylammonium Bromide Micelle in Vacuo: A Simulation Study
Wang, Y.F., et al.. Biochem. **48**, 1006-1015 (2009)

Janos Hajdu: "Flash diffraction with X-ray lasers"
EBSA2009, European Biophysics Congress Genoa, Italy, 11-15 July 2009,
"Ultra - fast diffraction imaging in biology"
Int. Conf. on Bio & Medical Sciences with New Light Sources, British Embassy, Rome, Italy, 12-13 March 2009
" Flash diffraction imaging of biological objects: Experimental possibilities in the 0.5-1.0 nm wavelength range" Next Generation Light Sources: Nanoscale Coherent Imaging and Microscopy with a Soft X-Ray Laser, UC Berkeley ,17 October 2009