Final 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:	Prof. Dr. Gerhard Grübel
Federführendes Helmholtz-Zentrum:	Deutsches Elektronen-Synchrotron DESY
Beteiligte Universitäten und andere Partner:	TU Berlin, Uppsala University, Universität Hamburg
Berichtszeitraum:	1.3.2008 bis 31.12.2011

Aims and Goals of the VI

The prime motivation of the VI was to explore and develop experimental techniques which allow structure determination and imaging of non-crystalline objects such as macromolecules with single femtosecond X-ray pulses. The function of macromolecules and of inorganic particles is directly linked to their geometric structure, especially the organisation of molecular entities and functional groups. Despite tremendous progress in X-ray science over the past decades, structure determination of non-crystalline objects, such as several classes of macromolecules, gas phase nanoparticles and living cells is still one of the great challenges.
The research with x-ray FELs joined together in this VI created the ability to perform experiments on the nature of matter under the extreme conditions in intense x-ray laser pulses. A key aspect is that the extremely high brightness and short pulse duration of FELs allows a new regime of x-ray microscopy to be explored. Intense x-ray pulses from FELs allow overcoming the resolution limit in conventional x-ray microscopy resulting from severe radiation damage. The concept of flash imaging with FELs can ultimately be extended to atomic resolution where it seems possible that single molecules could be imaged. A main part of the VI was to push the concept of flash imaging to the limits of obtaining atomic resolution and to gain detailed understanding of the ultrafast processes, such as electron dynamics and plasma formation in the sample

The main physical questions and goals were:

- investigation of electron dynamics and chemical changes in the non-linear regime and the consequences for structure determination
- obtaining detailed understanding of physical processes limiting the resolution
- development and improving the tools (focusing optics, detectors, image reconstruction) for single shot imaging techniques
- characterization of FEL pulses and development of ultrafast diagnostic tools
- sample preparation, transport, and trapping of individual particles
- performing demonstration experiments on large molecular aggregates, gas phase nanoparticles, and living cells.

The planned work was organised into six work-packages:

Workpackage 1:	**Development of techniques for FEL pulse structure diagnostics**
Workpackage 2:	**Pump-probe optics and experimental setup**
Workpackage 3:	**High-resolution imaging of picoplankton**
Workpackage 4:	**Time-resolved imaging and spectroscopy of clusters and nanodiamonds**
Workpackage 5:	**Coherence and correlation of FEL sources**
Workpackage 6:	**Transport and trapping of individual particles**

The workpackages 1 and 5 are coordinated by DESY.

The workpackages 2 and 4 are coordinated by the TU Berlin.

Workpackage 3 is coordinated by U. Uppsala.

Workpackage 6 is coordinated by U.Hamburg.

a) *Results of the proposal*

Workpackage 1

The **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 could not be realized as originally planned 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. The evaluation of the results turned out to be far more complex as originally intended and thus not compatible with the given time frame of the VI. 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.
[1] U. Frühling et al., Nature Photonics 3, 523 (2009)

Workpackage 2

For work-package 2 the team from the TUB has developed a new optics concept for pump-probe experiments using different harmonics of the FLASH machine. The key component is a split mirror which consists of a inner part focussing the third harmonic of the FEL while the outer part focuses the first harmonic. The optics was successfully tested at FLASH in 2008. In collaboration with Lothar Strüder (Max-Planck Halbleiter Labor) a new combined detector set-up was developed which allowed recording very high quality data at FLASH in April 2009 at the fundamental harmonic of single clusters (see figure 1). Data with the third harmonic could also be achieved, but not under single cluster conditions, probably the transmission of filters was not sufficient. Very successful test and results from the combined detector poured into the development of the CAMP instrument constructed and build up by the Advanced Study Group at CFEL [5]. The set-up of the TUB was further upgraded by the integration of optics for infrared pump, XUV probe experiments on single clusters.
[5] L. Strüder et al., Nucl. Instr. Meth. A 610, 483 (2010)

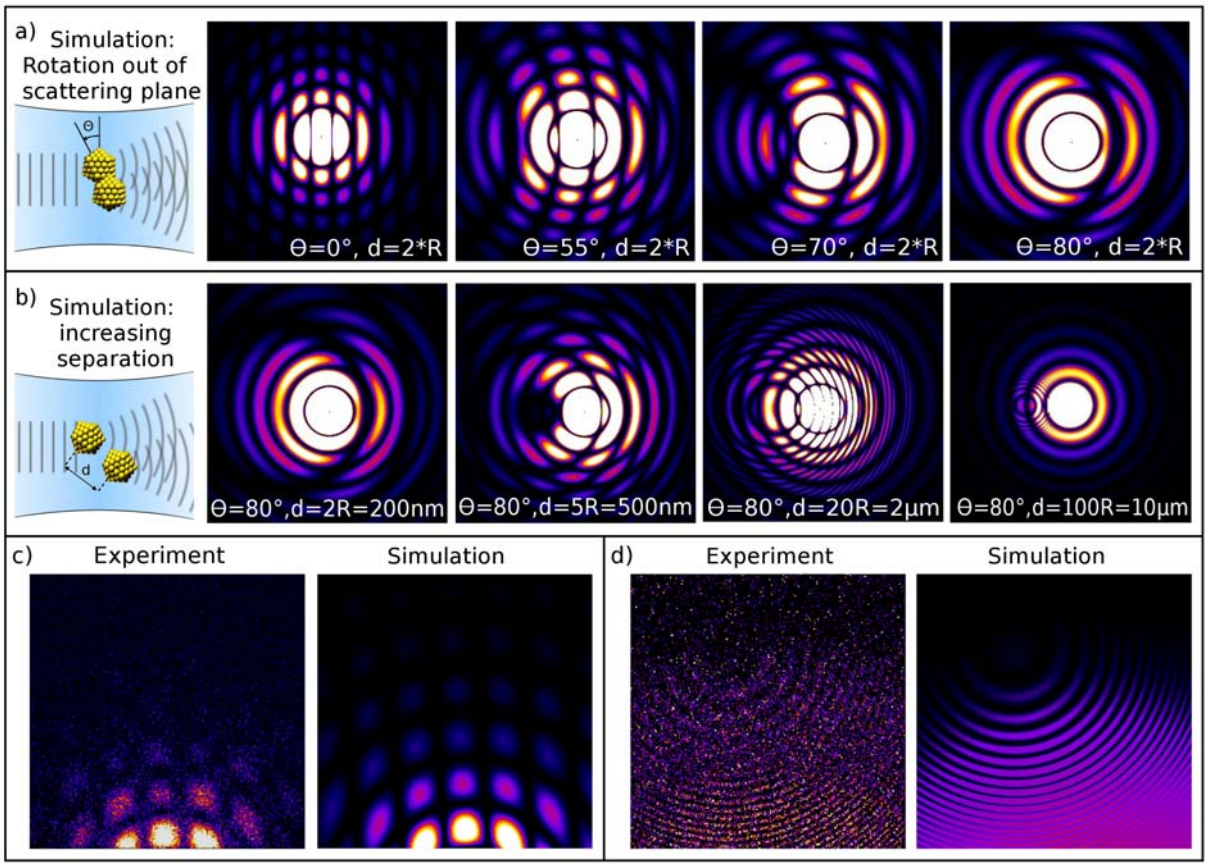

Figure 1: *Comparison between experimental and simulated scattering patterns. Simulations for a) two clusters in contact with shrinking size of the second cluster and b) two equally sized clusters with shrinking distance. D. Rupp et al.*

Workpackage 3

Development of an injection systems for rapidly injecting intact cells into the FEL focus for diffraction imaging. Development of damage models and software packages for high resolution image reconstruction. Hereby, one important aim is to extract depth information from the single diffraction images. The workpackage will benefit from the time-resolved experiments (WP2 and WP4) which will yield important input for the damage models about the ion and electron dynamics in FLASH experiments. In a long term vision, the results of this workpackage and the sample handling techniques to be developed in WP4 will allow imaging of even smaller structures down to macromolecules and clusters. The program will lead to very high-resolution single-shot images of living picoplankton, and this will seed new science in new areas of systems biology. It will also lead to potentially new horizons in cell biology.

Specific projects involved:

1) High-resolution imaging of picoplankton

2) Small beam foci. 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).

3) Investigation of sample dynamics as a source of structural heterogeneity on the resolution in a reconstructed small protein molecule (Maia et al. 2009).

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

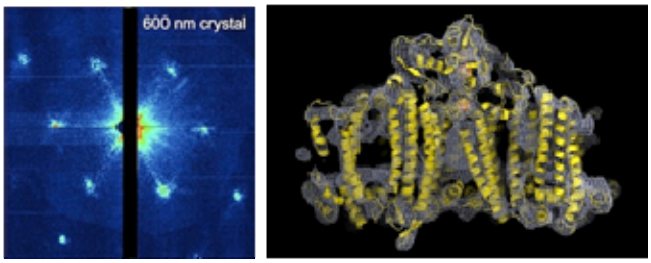

Figure 2: *Diffraction from a photosystem I nanocrystal and the 3D structure of this membrane protein from many such diffraction patterns.*

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.

5) 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.

6) 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.

7) 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).

Workpackage 4

Workpackage 4 focusses on the time resolved imagaing of clusters and nanoparticles. Initial pump-probe experiments using the autocorrelator at FLASH were recorded in Summer 2009. Using time of flight mass spectroscopy the ionisation dynamics of Xe clusters comprising up

to 10000 atoms could be studied in detailed. It turned out that the cluster density drops after 2 ps significantly [Krikunova et al]. As a result of a close cooperation between the groups in the VI a joint proposal between the group in Uppsala (Janos Hajdu) and the TUB was submitted for first experiments on clusters at the LCLS X-ray Laser in Stanford. After approval, initial experiments at 800 and 2000 eV were performed at the AMO experimental station on Ar, Xe and Methane clusters. The experiments on Methane clusters exhibit a very efficient charge transfer processes from carbon to hydrogen. These results are of considerable interest for all kind of imaging experiments with X-ray lasers. In three additional runs of experiments the TUB group collaborated with the CAMP consortium and was also involed in the first experiments on protein nanocrystals [6]. The layout of the CAMP instrument allowed first single shot coincidence experiments recording scattering patterns, ion and electron spectra from single clusters. Surprisingly, at the highest power density of 10^{16} W/cm^2 the ion spectra of large Xe clusters show atomic fragment ions in extremely high charge states peaking at 25, while the low charges states are completely absent (see figure 2). The results give strong evidence that ion-electron recombination is strongly suppressed in X-ray produced nanoplasmas [Gorkhover et al, PRL sumbitted]. In experimental runs in 2010 and 2011 the collaboration carried out IR-pump X-ray probe experiments. The experiments were very successful by revealing the diffraction images from “exploding” clusters as a

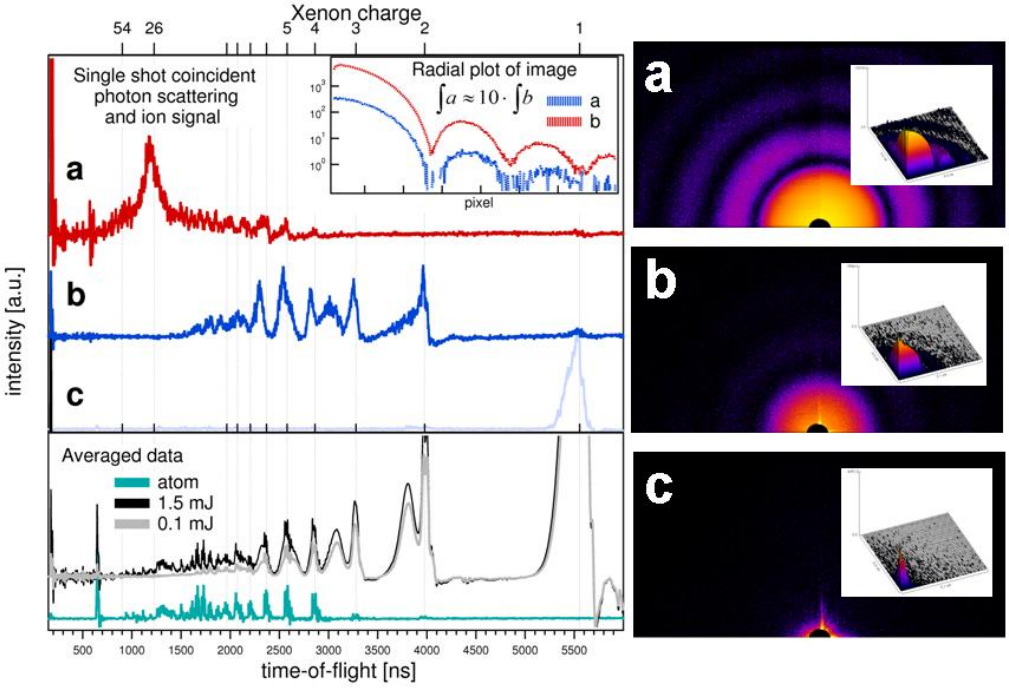

Figure 3: *Comparison between averaged (cluster and atomic) and coincident single shot ion spectra a)-c) (left part of the figure) for different intensities and simultaneously recorded scattering patterns (right part). [Gorkhover et. al, PRL submitted]*

function of the delay time. The experimental set-up of the TUB was upgraded in 2010 by incorporating a time of flight mass spectrometer into the scattering detector. This upgraded set-up allowed to perform first single shot ion-photon coincidence measurements of clusters at FLASH. Thanks to the high sensitivity scattering patterns with detailed fine structure could be recorded which reveal the shape of single clusters being far from being spherical. The new results give very detailed insight into the cluster growth process and open the door for time resolved studies on phase transitions in nanoparticles.

[6] H. N. Chapman et al., Nature 470, 73 (2001)

Workpackage 5

In **work-package 5** we presented for 2008/2009 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].

[2] C. Gutt et al., Phys. Rev. B 81, 100401(R), (2010)

We have developed a X-ray delay line capable of splitting and delaying single X-ray pulses has been developed with the aim of performing X-ray photon correlation spectroscopy (XPCS) and X-ray pump–probe experiments at hard X-ray free electron laser sources [3] . The performance of the device was tested with 8.39 keV synchrotron radiation. Time delays up to 2.95 ns have been demonstrated. The feasibility of the device for performing XPCS studies was tested by recording static speckle patterns. The achieved speckle contrast of 56% indicates the possibility of performing ultra-fast XPCS studies with the delay line.

[3] W. Rosecker at al., J. Synchr. Rad. 18, 481 (2011)

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 2010/2011. 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.

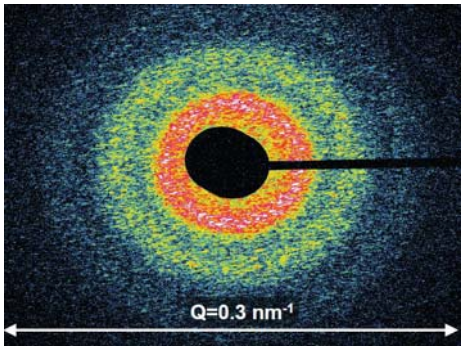

Figure 4: *Single pulse hard X-ray speckle pattern captured from nano-particles in a colloidal liquid (photon wavelength λ = 1.37 Å). The inset shows a cut of the photon intensity.*

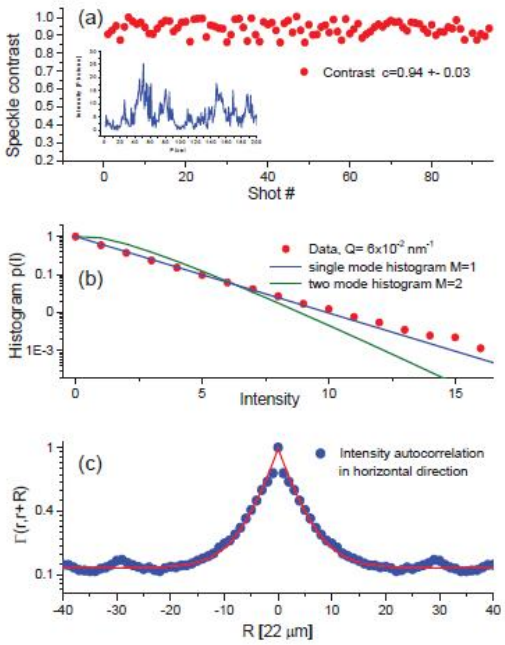

Figure 5: *(a) Single pulse speckle contrast (visibility) for different shots from the LCLS recorded in SAXS setup (Q=6• 10−2 nm−1.*

(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).

Speckle patterns from a gold nanopowder recorded with atomic resolution allowed us to measure the shot-to-shot variations of the spectral properties of the x-ray beam [4]. The focused beam is in the transverse direction fully coherent with a mode number close to 1. The average number of longitudinal modes behind the Si(111) monochromator is about 14.5 and the average coherence time τ = 2:0 +/- 1 fs. The data suggest a mean x-ray pulse duration of (29 +/-14) fs behind the monochromator for (100 +/-14) fs long electron pulses.

[4] C. Gutt et al., Phys. Rev. Lett., 108, 024801 (2012)

Workpackage 6

After the necessary decision in 2010 to set up our own vacuum system, in order to Implement an in-house test system for working out appropriate trapping protocols for nano-sized particles, we have made extensive efforts in this direction. We now have an operative system at hand. 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. The laser system for dipole trapping is operative since 2010. We have begun to explore in-vacuum nano-particle preparation and handling. Faster progress has been impeded by the fact, that no resources were available for the necessary vacuum equipment such that we had to resort to on board means, which were only available in limited portions. A further issue was the need for suitable source technology for in-vacuum nano-particle preparation and handling. We have started a collaboration with Tübingen university to import respective technology. We plan to continue our work within the BEXI framework, which should provide us with the additional funding required in this project.

With in-house means we have set up a vacuum test chamber. We have begun to explore in-vacuum nano-particle preparation and handling.
Our work has proceeded in accordance with the financial plan. The timing for his project has turned out unrealistic with the limited funding available.

b) Selection of achieved milestones

- Experimental bunch-by-bunch characterization of the longitudinal coherence length.
- Experimental bunch-length characterization of LCLS at 9 keV.
- Tests of two color multilayer mirror for time resolved experiments.
- Injection of live picoplankton and other cells into the FEL beam.
- Quantitative study of damage thresholds for (magnetic) multilayer samples.
- Successful cluster experiments at LCLS
- Set-up of a NdYAG fiber laser system and the optics required for forming a tightly focused optical dipole trap.
- FLASH and LCLS data taken on cluster systems, molecules and protein nano-crystals.
- Time resolved pump-probe experiments at FLASH on single clusters revealing the fs-ps ionization dynamics of clusters.

c) Adherence to the time and financial plan.

- The finance and time plan (including a 6 month extension) was kept.

d) Publications

The following talks and publications originated from the work within the VI:

Resonant magnetic scattering with soft x-ray pulses from a free-electron laser operating at 1.59 nm
C. Gutt et al.,
Phys. Rev. B79, 212406 (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)*

High Wave Vector Temporal Speckel Correlations at the Linac Coherent Light Source
Sooheyong Lee et al., *Optics Express,* submitted
Development of a hard X-ray delay line for XPCS and jitter-free pump-probe experiments at XFEL sources
Wojciech Roseker et al., *J. Synchr. Rad.* 18, 481 (2011)

Characterizing Coherence of Hard X-ray LCLS Pulses via a Single Shot Speckle Visibility Analysis
Sooheyong Lee et al.,
Phys Rev. B, submitted

Measurement of single shot spatial and temporal coherence properties of the LCLS in the hard X-ray regime
C. Gutt et al.,
Phys. Rev. Lett., 108, 024801 (2012)

Nanoplasma formation and neutralization of soft X-ray irradiated clusters
M. Hoener et al.,
J. Phys. B 41, 181001 (2008)

Numerical simulation of small angle scattering (SAXS) for large atomic clusters
A.R.B. de Castro et al.,
J. Ele. Spectr. Rel. Phen. 166–167, 21–27 (2008)

Multi-Step Ionisation of Argon Clusters in intense femtosecond XUV pulses
C. Bostedt et al.,
Phys. Rev. Letters 100, 133401 (2008)

Femtosecond non-equilibrium dynamics of clusters irradiated with short intense VUV pulses
B. Ziaja et al.,
New J. Phys. 10, 43003 (2008)

Atomic clusters of various sizes irradiated with short intense VUV pulses
B. Ziaja et al.,
Euro. Phys. Letters 82, 24002 (2008)

Experiments at FLASH
C. Bostedt et al., Nucl. Instr. Meth. A, 601, 18 (2009)

Femtosecond time-delay X-ray holography
H. N. Chapman et al.,
Nature 448, 676 (2007)

Energetics, Ionization and expansion dynamics of atomic clusters irradiated with short intense vacuum-ultraviolet pulses
B. Ziaja et al.,
Phys. Rev. Lett. 102, 205002 (2009)

Shell explosion and core expansion of xenon clusters irradiated with intense femtosecond soft x-ray pulses
H. Thomas et al.,
J. Phys. B. 42, 134018 (2009)

Experiments with fast spectroscopic pnCCDs at BESSY and FLASH VUV-FEL
C. Reich et al.,
IEEE Nuclear Science 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 et al.,
Nucl. Instr. Meth. A 610, 483 (2010)
Fast electrons from multi-electron dynamics in xenon clusters induced by inner-shell ionization
C. Bostedt et al
N. J. Phys. 12, 083004 (2010)

Clusters in intense FLASH pulses: Ultrafast ionsiation dynamics and electron emission studied with spectroscopic and scattering techniques
C. Bostedt et al., J.Phys. B. 43 194011 (2010)

Femtosecond X-ray protein nanocrystallography
H. N. Chapman et al.,
Nature 470, 73 (2011)

Heterogeneous clusters as a model system for the study of ionization dynamics within tampered samples
B. Ziaja et al.,
Phys. Rev. A 84, 033201 (2011)

Ultrafast x-ray scattering of xenon nanoparticles: Imaging transient states of matter
C. Bostedt et al.,
Phys. Rev. Lett., submitted

Ultra-Efficient Ionization of Heavy Atoms by Intense X-Ray Free-Electron Laser Pulses
B. Rudek et al.,
Nature Physics, submitted

Ionization dynamics in expanding clusters studied by XUV pump-probe spectroscopy
M Krikunova et al.,
J. Phys. B submitted

Identification of twinned gas phase clusters by single shot scattering with soft X-ray pulses
D. Rupp et al.,
New J. Physics, submitted

Massively parallel X-ray holography
Stefano Marchesini et al., Nature Photonics, 2, 560 (2008)

Single Particle X-ray Diffractive Imaging
Michael J. Bogan et al., NanoLetters, 8, 310, (2008).

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.
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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)

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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)

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Seibert, M.M., et al., J. Phys. B - Atomic Molecular and Optical Physics **43**, Article Number: 194015 (2010).

Time-resolved pump-probe experiments at the LCLS
James M. Glownia et al., *Opt. Express* **18**, 17620-17630 (2010).

Hawk: the image reconstruction package for coherent X-ray diffractive imaging
Maia, F.R.N.C., et al., *J. Appl. Cryst*. **43**, 1535-1539 (2010).

On the Feasibility of Nanocrystal Imaging Using Intense and Ultrashort X-ray Pulses
C. Caleman et al., , *ACS Nano* **5**, 139-146 (2011).

Saturated ablation in metal hydrides and acceleration of protons and deuterons to keV energies with a soft-x-ray laser
Andreasson, J., et al., *Physical Review E* **83***,* 016403 (2011).

Single Mimivirus particles intercepted and imaged with an X-ray laser
Seibert, M.M., et al., *Nature* **470**, 78–81 (2011).

Multipurpose Modular Experimental Station for the DiProI Beamline of Fermi@Elettra Free Electron Laser
Emanuele Pedersoli et al., Review of Scientific Instruments, Accepted.

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Anton Barty et al., Nature Photonics **6**, 35-40 (2012).

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T. Gorkhover, et al,
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