

Abstracts of Posters

Imaging Large Superfluid Helium Droplets

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Superfluid helium droplets do not rotate as a solid body; instead, the droplet's rotation is manifested through the presence of quantized vortices. The vortices have a diameter of $\sim 2 \text{ \AA}$ and their detection remain an outstanding experimental problem. Here, we report on X-ray coherent diffraction imaging of free, single, rotating superfluid 4He droplets (diameter $D = 200 - 2000 \text{ nm}$, temperature $T = 0.4 \text{ K}$) using the Linac Coherent Light Source at SLAC. The droplets were doped with xenon atoms, which trace the vortex cores and serve as a contrast agent for imaging. The instantaneous positions and shapes of the vortices from the diffraction images were obtained using a phase retrieval algorithm developed in our laboratory at USC. In the first part of this contribution, we present an overview of the observed vortices in droplets of different sizes and shapes. Additionally, we found that doping by a large number of xenon atoms may influence the kinematics of vortices.

In the second part, we report on diffraction patterns collected from very large droplets, $D > 2000 \text{ nm}$, that are produced from the breakup of a liquid helium jet in vacuum. In contrast to the usual concentric rings in smaller droplets, the diffraction patterns in very large droplets show characteristic lobed structures that resemble surface harmonics. While further analysis is needed, we temporarily assign these unusual intensity distributions to large amplitude shape oscillations in the droplets. Quantum vortices and shape oscillations are global excitations in superfluid helium droplet, which currently can only be accessed through coherent diffractive imaging at x-ray free electron lasers (XFEL). Accordingly, XFELs emerge as powerful tools for investigating the manifestations of superfluidity in finite sized systems.

Direct observation of electron density reconstruction at the metal-insulator transition in NaOsO_3

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The metal-insulator transition in $5d$ transition metal oxide NaOsO_3 which occurs concomitantly with antiferromagnetic ($T_{\text{MIT}}=T_{\text{N}}$) has been proposed to be of Slater mechanism^[1, 2, 3, 4]. However, there is no consensus and other mechanisms such as a Lifshitz transition have also been proposed. We employed non-resonant and resonant x-ray single crystal diffraction at the Os $L_{2,3}$ edges in order to obtain details of the Os electron density deformation across T_{MIT} and to determine the microscopic mechanism of the phase transition. In case of a Slater metal-insulator transition, there should be no change in the crystallographic symmetry and, indeed, our single crystal x-ray diffraction experiments find no evidence of crystallographic symmetry breaking across the metal-insulator transition. In

addition, using an incident x-ray energy corresponding to the Os L₃ edge, we observe the emergence of a diffracted intensity at the (300) forbidden reflection at $T_{MIT}=T_N$ for a specific energy $E_A=10.878$ keV. The intensity of this space-group forbidden peak increases continuously with decreasing temperature and it is not of magnetic origin. Rather, we show that it is due to a change in the Os electron density which is associated with the onset of long range antiferromagnetic ordering. Thus, the main conclusions of our experimental results, namely the absence of crystallographic symmetry breaking and the presence of antiferromagnetic driven osmium electron density reconstruction, support the first realization of a Slater insulator, NaOsO₃.

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[2] Y. G Shi, et al, Physical Review B, 80(16), 161104 (2009)

[3] S. Calder, et al, Physical Review Letters 108(25), 257209 (2012)

[4] B. Kim, et al, Physical Review B, 94(24), 241113 (2016)

Femtosecond time-resolved and element-specific x-ray absorption spectroscopy of Fe/MgO

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A localized optical excitation of a metal/insulator heterostructure induces ultrafast dynamics through the interface in its individual compounds, which can involve charge and spin transfer processes as well as coupling to low energy excitations mediated by e.g. electron-electron and electron-phonon scattering. Femtosecond soft x-ray spectroscopy allows to separate and identify these electronic and lattice excitations directly in the time domain and, furthermore, is sensitive to the dynamics of the individual constituents itself due to its element-specific character.

We have measured time- and element-resolved x-ray absorption spectroscopy of a [2 nm Fe/2 nm MgO]₈ multilayer at the Fe L₃- and O K-edges with a time resolution of 150 fs. After a local optical excitation of Fe with a UV laser pulse of 266 nm wavelength we see a clear pump-induced effect at both edges in fs time resolution. The Fe L₃-edge shows an ultrafast 0.5 % dropdown of the signal in 240 fs, followed by an almost full recovery until 1.3 ps, while the O K-edge signal reaches its maximum only after 1 ps. At later delays the change at the Fe L₃-edge reappears, but with a slower time constant. Furthermore, we measured the transient changes at the O K pre-edge region.

Comparison of the experimental XAS spectra with layer-resolved first principles calculations in the framework of density functional theory reveal that this pre-edge feature results from interface states near the Fermi level in MgO. Here, we observe an even slower pump-induced change than the ones at the both edges. The complex response at the Fe L₃-edge represents local electronic excitations driven by the 266 nm pump, coupling to phonons in Fe, and their relaxation through coupling to the insulator constituent, as supported by complementary time-resolved electron diffraction studies. The dynamics at the O K-edge is clearly faster than the lattice relaxation in Fe, mentioned above. We currently consider e-ph coupling at the interface to be responsible for such this ultrafast process.

The slower response at the O pre-edge occurs on a similar time scale as lattice energy transfer of Fe to MgO and could be related to the specific interface nature of the probed state.

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Femtosecond X-ray Fourier holography imaging of free-flying nanoparticles

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The advance of XUV/X-ray Free Electron Lasers (FEL) enable unprecedented insights into processes in individual non-crystalline nanoparticles with high spatial and temporal resolution. While in X-ray imaging of solid state targets Fourier transform holography was used for a long time as an elegant solution to the phase problem [Eisebitt et al., Nature 432, 885-888], it was only recently realized for unsupported nanospecimen, using a X-ray FEL facility [Gorkhover et al., Nature Photonics 12, 150-153]. Here atomic gas phase clusters were used as a holographic reference scatterer to image nanometer scale bio specimen – both injected by two different sources. Using a new approach, it becomes possible to overcome problems with alignment and overlap of two particle beams by using only one injector. This simplifies the experimental setup vastly, while increasing the holographic hit rate at the same time. First results of the proof of concept experiment as well as a framework to evaluate gas phase holograms will be presented.

Revealing the three-dimensional shapes of spinning superfluid quantum droplets using extreme ultraviolet FEL pulses

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With the advent of free-electron lasers (FEL) delivering femtosecond short-wavelength pulses, coherent diffractive imaging methods have been developed to gain insight into the structure of unsupported nanoparticles such as viruses or clusters. While experiments using light pulses in the X-ray regime ultimately aim at atomic resolution [1], full three-dimensional information on the particle shape and orientation from a single diffraction pattern requires access to wide-angle scattering signal, especially available at longer (e.g., extreme ultraviolet) wavelengths [2].

Superfluid helium nanodroplets are interesting targets in this context since they can gain angular momentum when produced by a free-jet expansion from the liquid phase. As superfluid droplets cannot rotate in the classical hydrodynamic sense, quantized vortices accommodating the angular momentum are formed inside the droplets. Recently, pioneering experiments on superfluid helium nanodroplets at the LCLS FEL using intense X-ray pulses revealed such vortex arrays and showed strong deformations of the droplets [3,4] exceeding the classical stability limit for equilibrium shapes of rotating drops [5]. However, the shape analysis was restricted to the two-dimensional electron density projection reconstructed from the recorded small-angle scattering images.

In our experiment, XUV diffraction patterns of single helium nanodroplets were recorded at the FERMI FEL's LDM endstation [6]. While the majority of the bright scattering images exhibit concentric ring structures that reflect spherical droplet shapes, about 10% of the images show diffraction patterns of non-spherical particles. In particular, a tilt of a deformed droplet out of the scattering plane produces non-centrosymmetric features in the wide-angle diffraction pattern. In order to simulate these features, a multi slice Fourier transform (MSFT) algorithm was employed, similar to the one described in Ref. [2]. By assuming simple model droplet shapes and matching the MSFT simulations to our data, the droplets' axes and volume could be retrieved. When compared to a numerical model of classically rotating (i.e., non-superfluid) drops [7] our data show unexpectedly good agreement. Further, the previously reported metastable shapes were not observed.

This finding demonstrates the usefulness of XUV light pulses to uncover the three-dimensional geometry of nanoparticles. Thus, developing suitable reconstruction algorithms and combining X-ray and XUV sources at FEL facilities to simultaneously record the particle's atomic structure and its geometry would be an interesting prospect for future research.

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Covalent modification of graphene by Neutral red dye

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Functionalization of graphene is a promising approach for expanding the field of application of graphene, which has attracted the attention of scientists due to its unique properties. Covalent attachment to graphene of photoactive dyes leads to the transformation of electronic structure in resulting nanocomposite. For example, in contrast to pristine graphene, which is transparent in the visible range of spectra, the graphene-dye composite has pronounced absorption peak, furthermore, disruption of the ideal sp² bound graphene structure by incorporation of sp³ hybridized carbon atoms leads to the bandgap opening in graphene. In the present study investigation of the electronic structure of graphene covalently modified by phenazine dye Neutral red by means of soft X-ray synchrotron radiation is presented. In addition, UV-PEEM measurements with work function contrast are given.

Highly efficient end-station for space-, time- and spin-resolved photoemission spectroscopy at free electron lasers and high harmonic generation sources

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High repetition rate XUV and soft X-ray free electron lasers (FELs) such as FLASH at DESY (Hamburg) offer unique possibilities for time-resolved photoelectron spectroscopy (PES). To fully exploit these possibilities it is necessary to use very efficient photoelectrons detection schemes. Combining a time-of-flight momentum microscope with the FEL as a source is ideal for time- and angle-resolved PES and time-resolved X-ray photoelectron diffraction to study ultrafast electron, lattice and spin dynamics. The momentum microscope allows simultaneous detection of the entire band structure with unprecedented efficiency in the full surface Brillouin zone with up to 8 \AA^{-1} diameter and 4 eV binding energy range, or the angular pattern of core level photoelectrons, respectively, for each time step in a pump-probe experiment. Adding the imaging spin detector extends the capability to detect the spin polarized band structure of the material. The set up was commissioned at FLASH and first results will be presented.

Low Energy Electron Holography as a tool for imaging single proteins at high resolution

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Protein functionality is intimately linked to the protein's native 3D folding structure. Determining these structures is of tremendous importance for understanding such functionality as well as related biological processes.

Recently, Low Energy Electron Holography (LEEH) has been shown to be able to image proteins at the single molecule level (without averaging), while avoiding radiation damage [3]. Low Energy Electron Holography [1] is a lens-free imaging method in which the sample is radiated by coherent low energy electrons (50-200eV) [2] to form holograms that in principle contain full 3D information of the object.

Our setup is an in-line holography setup, consisting of an atomically sharp tungsten tip acting as an electron point source, the molecule sample on a free-standing graphene substrate and an MCP detector to record the holograms. Since graphene is transparent to electrons, part of the electrons, the reference wave, pass through the sample unscattered, hence the hologram is formed on the detector screen as the interference pattern of the electrons scattered on the molecule (object wave) with the unscattered reference wave.

The experimentally acquired holograms then need to be numerically reconstructed. Basic Kirchhoff-Fresnel propagation-based reconstruction already yields the sizes and shapes of the investigated proteins, which match known structures from the protein structure data base[3].

To reach the aim of ultimately being able to extract detailed 3D information from the data, the reconstruction process and the setup are in the process of being optimized: a model for electron-soft matter interaction at the relevant energies has to be constructed, including effects like inelastic and multiple scattering, to help obtain a stable algorithm for phase and amplitude retrieval and the setup has to be modified such that tomographic data can be acquired.

The poster gives an overview of the experimental technique, compares it to other structure determination techniques and presents the theoretical background, preliminary results and future plans.

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3D high resolution diffractive imaging of laser excited individual metal clusters

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The advent of free-electron lasers (FEL) capable of delivering intense, femtosecond x-ray pulses, has allowed for high-resolution imaging of single nanometer-sized objects in free flight. Experiments with clusters can be used to both explore fundamental light-matter interactions and, because of their relative simplicity, refine imaging techniques.

In a previous work, a large variety of structural motifs of unsupported metal clusters from a magnetron sputter source were identified using wide angle diffraction with 13.5 nm FEL pulses [Barke et al. *NatCommun* 6:6187 (2015)]. As smaller metal clusters have shown temperature dependent changes in their structure when heated [Koga et al. *PhysRevLett.* 92.115507 (2004)], we tried to image the evolution of these changes using the wide angle method. In our experiment, we heated single, unsupported, silver and gold clusters using picosecond-long, visible (400 nm) laser pulses. We then took scattering images at varying delays in a diffraction before destruction approach using soft x-ray pulses from the FEL in Hamburg (FLASH). On this poster, first indications of delay-dependent changes in the scattering images as well as in the ion time of flight spectra will be presented.

MooNpics – Metrology On One-Nanometer-Precise Optics

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With new light sources under construction or planned — free electron lasers and ultimate storage rings — diffraction limited X-Ray radiation will become available for new classes of scientific and technological applications. Therefore, highly precise diffraction limited optics are needed. Of major interest are the long X-ray mirrors for beam transport and focusing. These mirrors exhibit shapes from very flat ($r < 3000$ km) to strongly curved ($r < 10$ m) and mirror lengths up to 1 m. To ensure appropriate beam focusing and alignment it becomes mandatory to control the shape of these mirrors on the 1 nm precision level. They can be manufactured with deterministic methods like ion beam polishing. Though, the requested accuracy generates high demands on the manufacturers as well as on the metrology for the characterization of the mirrors.

For this reason, a European project called “MooNpics – Metrology On One-Nanometer-Precise Optics” has been established, as a work package of the CALIPSOplus project. In this project 12 partners, including 2 European mirror manufacturers, are collaborating to improve the mirror

metrology as well as the capabilities in mirror manufacturing techniques. Within the scope of this project it is planned to bring all X-ray optics metrology labs in Europe to the same level, to make the metrology methods and procedures available and to disseminate the respective know-how. The collaboration will consolidate the existing experience in various kinds of profilometry, as interferometry stitching and Long Trace Profiler based instrument, as well as wavefront sensing technologies for hard and soft X-rays.

Among other activities, a round-robin with three mirrors of different shapes will be executed within a period of about two years. The mirror manufacturers are actively contributing to the round-robin performing also cross-calibration measurements. For the round-robin measurements a holder concept was developed to provide a reproducible mounting system. The objective is to understand mechanical and stress influences and hence to improve mounting principles. Preliminary measurements are performed in order to investigate the present surface quality of the mirrors. With the results it will be possible to establish standards for metrology measurement methods and mirror mountings. Furthermore, calibrated test mirrors can be created. Methods for spatial characterization with wavefront metrology will be optimized, in order to provide fast and accurate beam profile characterization and optics alignment. Not only the research labs, but also the mirror manufacturers will benefit from the data achieved in the goal of having better and better mirrors available and to become competitive in an international context.