

Book of Abstracts

Research with High Energy X-Rays at Ultra-Low Emittance Sources



Propagation-based phase-contrast imaging: application and current limitations

Content:

Imaging the development of model organisms over the course of time and in three spatial dimensions is an essential tool in cell and developmental biology or to model human diseases. Numerous techniques are employed to study such organisms. None of these, however, allows cell behaviors to be observed with micrometer-scale resolution throughout the optically opaque, living embryo over developmental time. E.g., in vivo using fluorescence microscopy is limited to translucent objects and a few cell layer below the surface, microscopic magnetic resonance imaging lacks spatial and temporal resolution, and conventional X-ray absorption tomography suffers from a lack of contrast. Using a (quasi-)monochromatic and spatially (partially) coherent beam of X-rays, single-distance propagation-based phase contrast allows bridging this gap since early developmental stages of model organisms are essentially pure-phase objects for hard X-rays. A protocol for time-lapse X-ray phase-contrast microtomography was established for in vivo imaging and analysis of the morphogenesis of optically opaque *Xenopus* frogs [1,2].

In Fresnel theory, we study the formation of 2D intensity contrast upon free-space propagation from a given phase object and how linear approximations to the inverse problem of phase retrieval from a single-distance intensity measurement break down for large propagation distances and strong phase variations. Certain properties of linear contrast transfer, which are conserved for a wide range of phase variations and propagations distances, are exploited in order to devise a phase-retrieval method which maintains a high spatial resolution even at low photon statistics [3,4,5]. Further, we discuss constraints imposed by in vivo imaging and the partial coherence of the X-ray beam and how these can be alleviated [5]. Data from in vivo experiments demonstrates the potential of propagation-based phase contrast imaging.

References:

- [1] Moosmann et al. Nature Protoc. 9 , 294 (2014)
- [2] Moosmann et al. Nature 497, 374 (2013)
- [3] Hofmann et al. Opt. Express 19, 25881 (2011)
- [4] Moosmann et al. Opt. Express 19, 12066 (2011)
- [5] Hofmann et al. Opt. Express 24, 4331 (2016)

Primary authors:

Dr. MOOSMANN, Julian (Institute of Materials Research, Helmholtz-Zentrum Geesthacht)

A Perspective of X-ray Phase Imaging

Content:

X-ray phase imaging has been studied to investigate weakly absorbing materials, which cannot be imaged clearly by conventional X-ray radiography and microscopy relying on absorption contrast. A characteristic beyond simple acquisition of phase-contrast pictures is that quantitative image formation of amplitude, phase, and scattering is feasible through digital image acquisitions and image processing. In combination with X-ray tomography, X-ray phase imaging has been advanced to a high-sensitive three-dimensional visualization method (phase tomography). The development of synchrotron radiation has been promoting these research of X-ray phase imaging so far. It should be also noted that spin-off technologies using X-ray phase imaging concepts out of synchrotron radiation facilities have been developed for practical applications to medicine and industry. Thus, the spectrum of the research field of X-ray phase imaging is extensive now. In such a situation, current limitations of X-ray phase imaging are reviewed and a perspective with PETRA IV will be discussed.

Primary authors:

Prof. MOMOSE, Atsushi (Tohoku University)

Imaging opportunities of PETRA IV in materials science and regenerative medicine

Content:

Three-dimensional imaging is often based on X-rays as the photon energy can be tuned to make the object of interest semitransparent. In this absorption contrast mode, the bones of the human body or the pores in foam could be easily made visible. Soft tissues around strongly X-ray absorbing bone or implants, however, are not accessible. As the X-ray absorption of soft tissues resembles that of water, a differentiation between the soft tissue components is hardly recognized. Therefore, staining and embedding methods were developed to generate a significant difference in X-ray absorption between the tissues. A valid alternative is measuring the electron density distribution via the phase shifts. Highly coherent small X-ray sources enable us to take advantage of in-line methods to discriminate between the soft tissue components and to make visible soft tissues in the direct contact to bone or medical implants. The size of such X-ray beams, however, is generally limited to a few millimeters. Beamlines, where the object of interest can be positioned several hundred meters from the source, could yield centimeter-size X-ray beams, as often required in the field of biomedical engineering. Dedicated X-ray optics may further widen the beam to visualize entire organs such as the human kidney with an extension in the range of 10 cm. Research teams are interested in the hierarchical imaging of the human body down to the true nanometer scale. For example, the human brain is a fascinating organ, which should be visualized within the skull, before extraction and cutting to finally reach the level of individual cells or even subcellular structures on the true nanometer level. A dedicated imaging beamline at PETRA IV could address the anatomy of animals and humans as well as the structure of medical implants with the aim to improve health.

Primary authors:

MÜLLER, Bert (University of Basel)

High resolution reciprocal space mapping for in-situ monitoring of individual subgrains in dislocation structures during cyclic loading

Content:

Changing mechanical loads enforce substantial modifications in the deformation structure, e.g. evolving dislocation structures are partially destroyed when the applied load is reversed. During repeated load reversal, dislocations organize into ordered structures with dislocation walls of high dislocation density separating almost dislocation-free subgrains. The developing characteristic structures, as the ladder structure in persistent slip bands, play a decisive role for the materials life time; fatigue caused by repeatedly changing loading conditions is responsible for about 90% of all material failures. Insight in the structural reorganization during cyclic deformation of polycrystalline aluminium is gained by monitoring individual subgrains with high energy synchrotron radiation. By high resolution reciprocal space mapping a large number of individual subgrains can be resolved in the bulk of polycrystalline specimens and their fate, their individual orientation and elastic stresses, tracked during entirely different loading regimes as loading and unloading, strain patch changes or stress relaxation. In case of cyclic deformation, the evolution of the deformation structure can be followed during a large number of strain reversals. Additionally, the particular changes during individual load cycles can be resolved. Tracing these yet unknown elementary processes will shed light on the formation mechanisms of the particular dislocation structures and further the insight in the initiation of failure.

Primary authors:

Dr. PANTLEON, Wolfgang (DTU Mechanical Engineering)

Insights into hierarchical materials from Diffraction Scattering Computed Tomography

Content:

Hierarchical materials abound in technology and biology. For example, bone is an anisotropic hierarchical composite material formed from nanocrystals of bio-apatite and collagen fibrils as well as a wealth of additional molecules. The multi-length-scale structures of bone remain far from understood, which impedes the understanding of bone diseases and their treatment. The same holds for most other hierarchical materials.

To understand such hierarchical nanomaterials, tools are needed that allow unravelling several length scales at once. This can be achieved by Diffraction Scattering Computed Tomography (DSCT) that combines the information from atomic (diffraction) or mesoscale (small angle scattering) length scales with the 3D position resolution of tomography. This is achieved by using a pencil beam to collect diffraction or scattering data as a function of position and viewing angle and tomographically reconstructing the diffraction/scattering information. I will give examples of the applications of this technique to a range of materials science problems.

DSCT benefits tremendously from high energy X-ray synchrotron sources that provide high penetration power. Equally important, high energy X-rays allow extracting detailed information about lattice constants and microstructure because sample thickness smearing effects become negligible. With high energy X-rays it also becomes possible to perform in situ DSCT under a range of conditions. I will discuss our implementation of in situ loading studies to map how a macroscopic mechanical load is distributed across bone. This type of knowledge can only be obtained by multi-length-scale techniques like DSCT.

Primary authors:

Prof. BIRKEDAL, Henrik (Aarhus University)

In situ diffraction studies of batteries using high energy synchrotron X-ray radiation

Content:

Many present and future technological devices are depending of functional materials (inorganic and organic) where it is important to investigate the relation between structure and physical/chemical properties. It is often not possible to extrapolate from structural properties at ambient conditions; to really understand the materials it is necessary to obtain information at operative conditions. Therefore, detailed characterization at in situ and operando conditions is of significant interest for a fundamental understanding of reactions and operational mechanisms of functional materials and for development and improvement of existing materials and technology.

High energy synchrotron X-ray diffraction is a powerful tool for studying materials in their operating environment. The excellent penetrating power of high energy X-rays allows studies of materials inside e.g. real fuel cells, catalytic reactors and batteries. By using high intensity X-ray radiation, even quite fast reactions and processes may be followed in situ. I will in this lecture focus on in situ studies of batteries, from commercial batteries to micro battery cells made specifically for in situ studies.

In order to improve present day rechargeable battery technology and develop new types of batteries, a detailed understanding of the mechanisms, reactions, structural properties and interface formation is necessary. Characterization and modeling from atomic and nano-scale to entire battery cells and battery packs is necessary.

Primary authors:

NORBY, Poul (DTU, Denmark)

Mesoscale Opportunities at the HEXM beamline of the upgraded APS

Content:

My talk will describe a planned high-energy x-ray microscope (HEXM) as part of the Multi-Bend Achromat (MBA) upgrade to the Advanced Photon Source, which would provide new capabilities to investigate structure and its evolution within bulk materials. HEXM requires the source characteristics enabled by the MBA-upgrade - increased coherence and brilliance at high-energies (35-120 keV) - in order provide non-destructive measurements at the highest spatial and temporal resolutions. Current high-energy x-ray techniques practiced at the APS will be discussed in the context of enhanced MBA characteristics, with a goal to enable multi-modal imaging spanning millimeters to angstroms in single experiments. New capabilities would benefit a wide suite of materials classes. Selected applications will be presented, including thermo-mechanical loading, in situ ion-irradiation and additive manufacturing. Efforts to couple this multi-scale information with modelling approaches, which together promise to revolutionize our ability to accelerate material development, will be discussed.

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Strain analysis of plastically-deformed bulk metallic glasses: challenges and opportunities

Content:

Bulk metallic glasses (BMGs) are metastable alloys with a disordered liquid-like structure retained at room temperature. The absence of structural periodicity in their atomic arrangement leads to remarkable mechanical properties, including near-theoretical strength and an unusually large elastic limit, which makes BMGs of considerable interest for structural applications. Unfortunately, the lack of a periodic arrangement of atoms also renders the detailed understanding of their deformation mechanism a very challenging task because structural studies cannot rely on a regular crystal structure and on the dynamics of related defects, such as dislocations.

Important information regarding structural changes occurring during deformation of BMGs can be gained by measuring the atomic strains using high-energy XRD. The combination of focused hard X-ray beam, large q range and high-resolution detectors with fast acquisition time has been extensively used for the in-situ evaluation of the elastic strains arising in BMGs during mechanical testing, leading to significant progress in describing the elastic deformation of amorphous materials. In contrast, limited work has been performed so far to analyze the atomic rearrangements occurring in BMGs plastically deformed at room temperature, where the plastic strain is accommodated by highly-localized, discrete shear bands. Such a heterogeneous deformation makes the distinction between plastic and (residual) elastic strain extremely difficult because it would require the use of a local analytical probe with size comparable with the thickness of the shear bands (10-20 nm) combined with the exact knowledge about the position of any shear band present in the sample.

In this overview, the characteristic structural features of elastic and plastic deformation in metallic glasses evaluated by high-energy XRD are presented. Experimental data for the strain arising in plastically-deformed BMGs over multiple length scales from macroscopically-strained areas comprising several shear bands down to the elastic matrix around an individual shear band are discussed with emphasis on the possible mechanisms involved in the generation and propagation of shear bands. Finally, challenges and possible solutions for further improving the knowledge of the elasto-plastic deformation mechanism of these materials are examined.

Primary authors:

Dr. SCUDINO, Sergio (IFW Dresden)

Fundamental Science in Tight Place

Content:

The impact of high-pressure studies on fundamental physics and chemistry, and especially on the Earth and planetary sciences, has been enormous. While experiments in diamond anvil cells (DACs) at pressures of $\sim 250 - 400$ GPa are proven to be very difficult but possible, at higher static pressures any matter has not been investigated until very recently. We have developed a method of synthesis of balls and semi-balls (of 10 to 50 μm in diameter) made of nanodiamond (with individual nano-particles of linear dimensions below 30 nm) and used them as second-stage or indenter-type anvils in conventional DACs. In experiments on simple metals and binary compounds we prove that it is possible to generate static pressures above 1000 GPa. However, samples are extremely small (about 3 μm in diameter and 0.5 μm thick), surrounded by strong X-ray scatters (like rhenium), and it makes diffraction experiments enormously demanding. In order to study novel physics of materials in TPa pressure range breakthrough in diffraction technique is necessary.

Primary authors:

Prof. DUBROVINSKY, Leonid (BGI)

Synchrotron-Based Extreme Condition Research Using Large Volume Presses

Content:

Large Volume Presses (LVP) allow experiments on relatively large sample volumes of up to 1 cm³ under extreme conditions of pressure and temperature (2 to 100 GPa, 300 to 2500 K). In combination with synchrotron radiation, LVP are used for designing new materials and alloys, study deep Earth materials and processes, as well as the effect of pressure, temperature and deviatoric stresses on their physical properties. This contribution will present recent developments using the LVP for studies combined with synchrotron radiation and how the future evolution of synchrotron radiation sources will open up new possibilities to advance scientific research at extreme conditions.

Primary authors:

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Coherent X-ray imaging with hard x-rays

Content:

In this talk applications of coherent x-ray imaging for hard x-ray imaging applications will be discussed.

This will include a proposal for the development of an atomic resolution x-ray imaging microscope.

Discussion of coherence based experiments with the samples under high pressure conditions at the coherence beamline P10 of PETRA III will be presented

Primary authors:

VARTANIAN, Ivan (Photon Science, DESY, Germany)

Nuclear resonance scattering above 30 keV: success and opportunities

Content:

The promising improved performance of PETRA IV will provide opportunities for nuclear resonance scattering at energies above 30 keV. Currently, the source size and divergence are limiting factors for the flux and beam size at the sample position, in large part because of the unavoidably limited angular acceptance of the utilized high- or medium-resolution monochromators. Nevertheless, promising results have been obtained both with the inelastic nuclear scattering and the nuclear forward scattering methods. Phonon spectroscopy using the 35.4, 37.2 and 39.8 keV nuclear resonances of Te-125, Sb-121, and Xe-129 is possible with 1 meV resolution or better. Time domain Mössbauer spectroscopy with typical count rates of 0.3-10 Hz is possible for nuclear resonance between 60 and 90 keV, such as Ir-193 or Ru-99. Recent success and applications will be discussed as well as development opportunities. Notably efficient focusing at high-energy holds promises for qualitative improvement of measurements under high-pressure.

Primary authors:

Prof. HERMANN, Raphael (Oak Ridge National Laboratory)

Fast Compression Experiments at Ultra-Low Emittance Light Sources: Opportunities and Limitations

Content:

Compression rate dependence of solid/solid and solid/liquids phase transition, as well as the existence of meta-stable phases has attracted significant attention over the last decade in the field of high-pressure physics as well as in the field meteorite/asteroid impact studies. In both science areas there has been a divide between static and ultra-fast compression experiments, resulting in conflicting information on e.g. the position of phase transitions as consequence of different strain rates. Thus, there have been significant efforts in developing techniques that enable dynamic compression experiments with intermediate compression rates such as the piezo actuator and membrane driven (dDAC and mDAC) to try to close the divide between these two techniques and to study more systematically the structural changes that occur as function of increased compression rates. While earlier structural changes were identified by fast micro-photography and -Raman spectroscopy, today 3rd generation light sources with optimized and high efficiency fast detectors enable collection of diffraction information in the kHz regimes. Currently, we are able to reach compression rates of 10s of TPa/s entering strain rate regimes of 10¹-10² s⁻¹ depending on the material system and thus we see first indication for rate dependent changes of phase transitions. However, there are many unresolved questions in terms of what happens during the fast compression of the sample in the dDAC, e.g. where nucleation takes place during transitions (edge of the gasket or at the diamond interface).

In this presentation we will summarize the current status of the field, emerging scientific questions and technical development. We will elaborate on what scientific advances can be expected from Diffraction Limited Storage Ring (DLSR) such as PETRA IV in terms of employing decreased emittance as well as coherence to e.g. conduct combined fast diffraction and normal/coherent imaging.

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New possibilities at Petra IV for XPCS experiments using high energy X-rays

Content:

The novel Petra IV ring that is proposed as replacement for Petra III offers extended possibilities for coherence experiments due to a record low emittance. This results in a larger coherent fraction and hence a higher coherent X-ray flux, notably in the hard X-ray range which has been difficult to exploit until now for instance in XPCS experiments. In the talk examples of XPCS experiments are given that I believe will benefit from a higher photon energy and where the availability of Petra IV could be decisive. A parallel development of pixel detectors and experimental setups compatible with high energy XPCS is also required as discussed in the talk.

Primary authors:

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High energy X-ray scattering for nanostructure analysis: Materials chemistry between molecules and particles

Content:

New developments in materials chemistry have made it possible to prepare novel nanomaterials with almost atomic precision, opening for a new class of compounds with potential applications in energy storage and conversion.¹ These new materials challenge the conventional methods for structural characterization, as the atomic arrangements do not exhibit long-range order.² Using high energy X-rays, we apply total scattering measurements with Pair Distribution Function (PDF) analysis to elucidate the structure of new materials.³ By applying a high flux X-ray beam, in situ studies furthermore allow us to follow the formation of new, complex materials. In this talk, I will present some of our recent work, and introduce new applications and possibilities with a high energy beam at PETRA IV.

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2. Billinge, S. J. L.; Levin, I. *Science* 2007, 316, (5824), 561-565.
3. Jensen, K. M. O.; Juhas, P.; Tofanelli, M. A.; Heinecke, C. L.; Vaughan, G.; Ackerson, C. J.; Billinge, S. J. L. *Nat Commun* 2016, 7.

Primary authors:

JENSEN, Kirsten M. Ø. (Department of Chemistry, University of Copenhagen)

Exploring interacting electron systems at a 4th generation synchrotron: some perspectives

Content:

Complex materials with strongly interacting electrons exhibit a vast variety of outstanding macroscopic properties. Just to name a few, the latter include high-temperature superconductivity, colossal magnetoresistance or unusually strong magnetoelectric couplings. These macroscopic effects attract a lot of attention, since they do not only challenge our basic understanding of condensed matter, but also bear potential for technology. Intense research efforts are therefore devoted to clarifying the underlying microscopic physics at work. It turned out that the most fascinating electronic states are usually not perfectly ordered and homogeneous. On the contrary, interacting electron systems are found to have a strong tendency towards formation of nano- or sub-nanoscale inhomogeneities and states with a significant degree of disorder. These complex electronic phases remain largely elusive, because their exploration requires new experiments, which allow probing non-periodic modulations in a bulk sensitive way and at atomic length scales. Typically complex sample environments are necessary as well. Focusing on examples of my own research, I will provide a short introduction to the topic, describe major experimental challenges in this field and give some perspectives on how these can be tackled using a 4th generation synchrotron.

Primary authors:

GECK, Jochen (TU Dresden, Germany)

Exploring nanoscale complexity of functional materials with record resolution

Content:

The physics of complex materials and the emerging coherent macroscopic states, like superconductivity, is an important topic in condensed matter. The recent discovery of CDW in the $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ compounds reanimated the debate about the competition between the striped-orders with the high temperature superconducting state in cuprates. There is a strong technological interest in understanding how the competition, between these orders forming nano-domains, plays a role in the promotion of the material's functionality at the macroscopic scale. The first step to understand the competition between these multiple striped orders is to investigate their spatial-organization. Unfortunately up to now there are only few works in literature reporting the imaging of how SDW, CDW and defects get organized into the material. On this purpose an important solution could come by the development of scanning hard X-ray nano-diffraction (HXRnD). Due to the intrinsic dimension of the x-ray beam available at the actual 3rd generation Synchrotron sources, focusing down to few nanometers it is not possible and most of the measurement have been performed with micrometric resolution. On this purpose the development of a diffraction limited source like PETRAIV will bring enormous benefits. Indeed it would be possible to focus the x-ray beam down to the record size of 1-10 nm pushing the HXRnD technique to the nanoscale limit increasing the resolution of about two orders of magnitude. Thanks to these unique capabilities, the proposed diffraction-limited source PETRAIV, will make finally possible the investigation of the nanoscopic mechanism that governs high temperature superconductivity and more in general will shed light on the role that nanoscale complexity has in the functionality of quantum materials.

Primary authors:

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High-speed x-ray imaging and diffraction at the Advanced Photon Source

Content:

Many scientifically and/or technologically significant phenomena are transient in nature, such as interaction of water droplet with hydrophobic surfaces, atomization of fuel spray out of high-pressure engineer nozzles, reactions of highly energetic materials, fracture of materials on impact, rapid melting and solidification of metals in laser processing, material phase transformation induced by thermal or mechanical shocks, among many others. At the 32-ID beamline of the Advanced Photon Source, we've been developing high-speed hard x-ray imaging techniques to help users interrogate critical problems in various highly dynamic systems. High-energy x-ray photons possess great penetration power, which allows us to look through the samples and capture every single detail of the internal structure evolution. Through years' of efforts, 6.5 MHz frame rate, 100 ps temporal resolution, and 1 μm spatial resolution have been achieved. Recently, high-speed x-ray diffraction technique and instrument have been developed at our beamline, which well complements the imaging technique by providing sample structure information in reciprocal space. Different from conventional pump-probe x-ray scattering techniques, our approach aims at understanding the physics in irreversible and/or non-repeatable dynamic processes (like those aforementioned) by collecting multiple frames of scattering data from a single event. In the presentation, I will introduce our current beamline capabilities and user programs, and specifically highlight the application of high-speed x-ray imaging and diffraction techniques in studying the metal additive manufacturing processes. Moreover, I will discuss the potential beamline enhancements an ultra-low emittance hard x-ray source can provide.

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Depth profiling of perovskite nanoparticles in porous Si wafers by high energy X-ray microdiffraction

Content:

We have used nanoporous solid state host matrices, i.e. thin (20 – 40 μm) nanoporous Si films, as a growth template for the nucleation of perovskite nanoparticles from solution. The nanoparticles show strong quantum confinement effects, i.e. strongly blue shifted photo- and electroluminescence [1].

We employed high energy microdiffraction in order to determine the nanoparticle size with help of the Scherrer equation. Here the high energy microbeam of P07 has several benefits. First of all, we can determine the depth profiles of the thin films with a resolution of few micrometers, by measuring in transmission with the beam parallel to the surface of the sample. Second, the 80 keV beam energy allows for rather long beam paths within the sample without significant peak broadening, as scattering angles are inherently small for high energy. We plan to complement these Bragg scattering experiments by SAXS experiments, which would allow us to determine also pore size and particle shape from the same samples in one experiments. Since beam damage is generally lower at higher energy, we see great potential for SAXS and WAXS experiments at higher energies and we expect that these experiments will benefit from the reduced emittance in the projected upgrade of PETRA.

[1] Stepan Demchyshyn, Janina Roemer, Heiko Groiss, Herwig Heilbrunner, Christoph Ulbricht, Dogukan Apaydin, Uta Rütt, Florian Bertram, Günter Hesser, Markus Scharber, Bert Nickel, Niyazi Serdar Sariciftci, Siegfried Bauer, Eric Daniel Glowacki, Martin Kaltenbrunner, arXiv:1607.0466

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