Medium-Term Upgrade Proposal for Dedicated Coherent X-ray Diffraction Facility at 34-ID

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Scientific Motivation

Most atomic-scale structural knowledge that exists today has been acquired through diffraction on homogeneous crystalline materials. As scientists expand studies to wider range of matter and focus more attention on structure-function relationships, structural information on nonperiodic and inhomogeneous materials has become increasingly more important. In biology, for example, about 40% of all proteins are not amenable to forming suitable crystals, an example of which is membrane proteins – an important component for biological function. In addition, structure information at the subcellular level is of critical importance in the post-genomic era in order to determine the functions of genes and gene products identified by modern molecular biology. In materials science, structural and strain information at the nanometer-scale on real-world heterogeneous specimens often provides the critical missing link between atomic-scale structures and macroscopic materials properties. Furthermore, many nanoparticles and nanoclusters that possess unique physical properties such as chemical reactivity, electron transport, and magnetism, are noncrystalline or highly strained, and thus require new structural determination tools to understand and tailor their structure-function relations.

Coherent x-ray diffraction imaging (CXDI) is a rapidly advancing field of lensless x-ray microscopy that provides real-space images of structural details by numerically inverting coherent diffraction patterns. This method has attracted considerable interests in the scientific community because it has many advantages compared to existing microscopy techniques. Because sample periodicity is not required, this method has the potential to perform ‘crystallography without crystals’. Compared to lens-based x-ray microscopy, the spatial resolution in coherent x-ray diffraction depends only on the diffraction signals measured at high scattering angles and thus offers the possibility to go beyond the resolution limit set by x-ray optics. Compared to widely used electron microscopy techniques, coherent x-ray diffraction has much larger penetration depths and thus can be used for studies of larger single particles and buried structures as well as in-situ investigations in complex sample environments.

While an ideal third generation source to pursue lensless imaging in the hard x-ray regime, at present APS does not have a 100% dedicated beamline to satisfy the user community needs for coherent x-ray diffraction imaging experiments. This has severely limited the potential growth of the CXDI scientific user community and has impeded further advances in CXDI methodology development. In this medium term upgrade plan, we propose to establish such a dedicated CXDI facility to enable a new range of structural investigations of biological and materials science specimens at the APS.

Proposed CXDI Facility

Specifically, we propose to design and implement: (a) a canted-undulator beamline at 34-ID with independent source and steering control that will be 100% dedicated to the coherent x-ray diffraction program, (b) a sample environmental chamber that is vacuum/helium compatible and is equipped with high-precision position and orientation manipulations for both Bragg and forward CXDI, (c) ptychography capability for studying not only isolated specimens but also extended object, and (d) an advanced area detector suite with single-photon sensitivity and high dynamic range capable of measuring accurate strong signals at low scattering angles and weak intensities at high momentum transfers.

It is estimated that the total cost of capital equipment for implementation of this upgrade plan is $5.4M over a period of approximately three years. In steady state operations, it is expected that the facility will be operated by three full-time staff scientists in X-ray Microscopy and Imaging plus a technical support person.

Partnerships/User Community

There already exists a very active user community for the proposed CXDI facility at the APS. For example, the recent Workshop on Coherent X-ray Microscopy held in May 2007 at the APS attracted more than 100 registered participants. The Workshop identified key areas of scientific applications of the CXDI technique in materials science and biology and concluded that a dedicated CXDI beamline at the APS would be needed in order to make substantial progress in this field. The following is a partial list of partnerships and collaborators that already exist in the APS user community.

Ian Robinson (University College London) is a professor of physics and a pioneer in coherent x-ray diffraction in Bragg reflection geometries. His research interests include imaging strain fields in nanocrystallites and nanowires and studying in-situ how shape and strain evolve under external influences at nanometer scale.

Oleg Shpyrko (University of California, San Diego) is a professor in physics. His research focuses on coherent x-ray diffraction studies of strongly correlated electronic systems and imaging of magnetic domains and their fluctuations.

Eric Isaacs (Center for Nanoscale Materials, ANL) is the Director for CNM at Argonne. His research interest in coherent x-ray diffraction is to image individual lattice dislocations and their interactions.

Larry Sorensen (University of Washington) is a professor in physics. His group is interested in using coherent x-ray diffraction to image domain fluctuations in condensed matter.

Veit Elser (Cornell University) is a professor in condensed matter theory. His research interest has been in phase retrieval algorithms and their applications in condensed matter physics.

Jianwei Miao (University of California, Los Angeles) is a professor in physics. He is a pioneer in the development of coherent x-ray diffraction imaging and the phasing algorithm. His current research interest focuses on imaging nanostructures in quantum materials and in biomaterials.

Thomas Earnest (Lawrence Berkeley National Laboratory) is a senior scientist in physical biosciences. His research interest is in cryo x-ray crystallography and macromolecular and cellular structures in biology.

Paul Evans (University of Wisconsin, Madison) is a professor of materials science and engineering. His research interests include structures and response of ferroelectric complex oxides under extreme fields.

Keith Nugent (Melbourne University) is a professor of physics and a pioneer in Fresnel coherent x-ray diffraction imaging using a curved wavefront. His research focuses on advanced methodology development in coherent diffraction imaging and applications in materials science and biology.

Andrew Peele (Le Trobe University) is a professor of physics. His research interests include development of Fresnel coherent diffraction on extended objects and singular x-ray optics and their applications.

Abbas Ourmazd (University of Wisconsin, Milwaukee) is a professor of physics. His research is in the area of signal recovery from noisy and limited data sets, and optimized alignment methods for coherent diffraction imaging of single particles.

Dilano Saldin (University of Wisconsin, Milwaukee) is a professor of physics. His research interests include direct imaging of surface nanostructures from x-ray diffraction data.

John Spence (Arizona State University) is a professor of physics and has a distinguished scientific career in electron microscopy. His recent research interests include coherent x-ray diffraction imaging of laser-oriented single molecules and phasing of diffraction data.

Paul Fuoss (Materials Science Division, ANL) is a physicist at Argonne and is interested in using coherent x-ray diffraction to image nanoparticles at buried interfaces.

Lee Makowski (Bioscience Division, ANL) is a senior scientist at Argonne. His research interests include application of coherent diffraction imaging to systems biology and bioinformatics.

David Dunand (Northwestern University) is a professor of materials science and engineering. His research focuses of metal nanofoam formation and their mechanical and physical properties.

Proposed Scientific Programs

The applications of coherent x-ray diffraction imaging are just being realized in recent years and continue to expand. Examples of nondestructive, high resolution lensless imaging of materials in many scientific disciplines now exist in the literature. Here we outline a few exciting examples of scientific research areas that will be pursued at the proposed CXDI facility at the APS.

Understanding physical properties in nanoparticles by in-situ imaging of structure and strain. Perhaps the greatest advantage of lensless x-ray imaging techniques is their versatility with regard to sample environment and preparation. In-situ imaging of catalytic behavior, magnetic and ferroelectric properties, surface melting, and materials under high pressure will be possible at the individual nanoparticle level. In the Bragg geometry, where coherent scattering is measured around the Bragg peaks of crystal lattices, the technique offers strong sensitivity to the distortion of the crystalline lattice due to strain (Fig.1). With the proposed CXDI facility it will be possible to image the fully three dimensional lattice distortion of a nanocrystallite. Such information will be crucial to the understanding and eventually the control of interesting physical properties of nanoparticles and nanoclusters.

Direct imaging of extended 3D lattice distortions around individual crystal defects. How lattice dislocations and other defects form in thin-films and how they interact with each other and with substrate are of directly related to the technologically important semiconductor research. Even though there is a long history in studies of lattice dislocations and defects, no method exists so far to image directly the strain field around individual dislocations because of its long-range nature. The improved CXDI facility will enable such studies for the first time. When coupled with in-situ sample environment, it will open up the exciting possibilities of controlling individual dislocation growth during thin-film deposition.

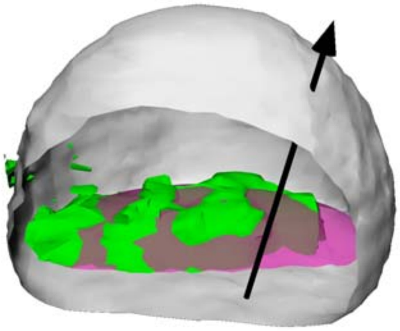

Fig.1. CXD imaging of a lead crystal of 750nm in diameter. The green isosurface represents the lattice distortion projected onto the Q vector of the Bragg reflection (arrow). To recover the entire vector field of the lattice distortion more Bragg spots from the crystal would need to be measured.

Imaging nanophase domain interactions and fluctuations in correlated electronic materials. Correlateed electronic materials such as complex oxides comprise a broad class of materials with domains of nanometer dimensions intimately associated with their electronic and magnetic properties. An example is the remarkable properties of colossal magneto-resistance and charge ordering in manganites. This behavior, occurring in materials with complex phase diagrams and competing ground states, is thought to be due to nanoscale phase separation and coupling between their many degrees of freedom in charge, spin, orbitals, and lattice. The ability to image nanoscale phases with magnetic and orbital as well as charge contrast by CXDI will substantially increase our understanding of how these degrees of freedom interact and affect the physical properties of the material.

Direct imaging of the formation and internal 3D structures of metallic nanofoams. Metal nanofoams represent a new class of nano-materials with unique and interesting physical properties that may not only lead to traditional applications in nanocatalysis and microsensors, but also be potentially useful as voltage-tunable microelectronic devices such as actuators, magnets, and resistors due to their electronically tunable physical properties. Structural imaging of these materials with CXDI in both forward and Bragg geometries at nanometer-scale resolution will provide the essential information on why these nanoporous materials behave differently from their homogeneous bulk counterparts. In addition, in situ observations on how these nanofoams are formed at nanometer scale during the initial stage of the dealloying process will be essential in the attempt to control the nanoporous network and the corresponding physical properties.

Structural imaging of biological cells, tissues, and bones at different developmental stages. Although the main focus of the proposed CXDI facility is in materials science and condensed matter physics, a significant portion of the CXDI usage will be devoted to biological studies. The main interest in this area is to image the interior architectures of whole cells and groups of cells, as well as extended structures such as tissue and bone sections, in their natural fully-hydrated or freeze-dried states. Such studies would make a tremendous impact on our understanding of subcellular organelles and parasites and multiprotein complexes at different developmental stages, and of the life cycle and internal structures of larger biological complexes such as spores. Coherent x-ray diffraction imaging of bones with different degrees of mineralization will enhance our understanding and control of biomineralization processes in bones, in particular, the apatite mineral phase, role of collagen, and effects of mineralization on the orientation of collagen fibrils. Structural imaging of other biomineralized structures such as shells may lead to better insights into the environmental processes that exist in nature.

Added Value

Currently the APS 34-ID-C is the only dedicated coherent hard x-ray diffraction imaging station in the world. It shares the 34-ID beamline with a microdiffraction program and therefore receives only up to 50% of beamtime. Addition of a second or third undulator at 34-ID would not only enable a fully dedicated CXDI user facility, but also make measurements involving changing or scanning x-ray beam energies completely independent of the other experimental station.

The proposed upgrades to the experimental instrument will enable many measurements that can only be dreamed of with the current configuration. Among them would be the ability to measure multiple Bragg peaks from single isolated nanocrystal objects. This would enable the recovery of a three dimensional image of the density of the object including the fully three dimensional lattice distortion vector field within the crystal. The proposed instrument would also be able to image, in three dimensions, the domain structures of magnetic and ferroelectric materials using the emerging techniques of Ptychography and curved-beam coherent diffraction. One particular subject of interest is the structure of the resulting charge density waves established within the magnetic domains of materials like Chromium at cyrogenic temperature. Coherent diffraction should be able to image the “strain” within the charge density wave, a property no other method could directly image. The proposed upgrades would also enable the measurement of coherent diffraction data in the small angle geometry. The promise of nanometer resolution imaging of noncrystalline objects, particularly done in-situ to environmental cells and high pressure cells, can only be practically accomplished with the proposed CXDI facility.

Enabling technology

The proposed upgrade to CXDI facility will be coordinated with the Materials Characterizatoin Group which operates the microdiffraction branchline 34-ID-E. Depending on what is possible in a single straight section, we hope for three independently configured undulators. In the three-undulator scheme, the CXDI branch would extend past the existing 34-ID hutches from a large displacement side bounce monocromator and deliver coherent monochromatic x-rays to a new experiment hutch at the end of the 34-ID territory, where the undeveloped empty space would provide >12m space for both the small angle and the Bragg CXDI experiments. At present this plan is not sufficiently developed, and thus this proposal covers only the standard double canted undulator scenario.

The basic sample manipulation is identical for both Bragg and forward-direction CXDI. A high quality optical table, perhaps composed of granite and mounted on footers extending through the experimental hall floor to maximize isolation, will accommodate high precision stages. The Bragg geometry measurement requires the ability to manipulate the sample on three rotational degrees of freedom. Modern parallel kinematic devices such as Hexapods and very low runout air bearing stages will provide the needed alignment.

One of the most important components of the CXDI measurement is to oversample the resulting diffraction pattern with a high-sensitivity and high dynamic-range area detector. We plan to acquire the newly developed pixel array detectors, which require a longer distance from the specimen.

We estimate that the total cost of capital equipment for implementation of this proposed CXDI facility will be $5.4M over roughly three years. The following table shows a detailed budget breakdown.

Estimated Capital Equipment Cost for 34-ID-C Midterm Upgrade

	Total cost for CXDI Upgrade (k$)	**Total 5370**	**Phase I 1645**	**Phase II 1635**	**Phase III 2090**
1.1	**Undulator and Front-end**	**955**		**955**	
1.1.1	New U33 undulator or equivalent			350	
1.1.2	New vacuum chamber, steering magnets, etc.			85	
1.1.3	Standard canted-undulator front-end			520	
1.3	**Station Instrumentation**	**460**	**420**	**0**	**40**
1.3.1	Control system upgrade to XOR standard		80		40
1.3.2	Coherent beam conditioner		70		
1.3.3	In-vacuum fast x-ray beam shutter		10		
1.3.4	Station workstations		20		
1.3.5	New optical table		50		
1.3.6	High resolution optical microscope		55		
1.3.7	High resolution x-ray imaging system		60		
1.3.8	X-ray fluorescence detector and electronics		25		
1.3.9	PSS/EPS upgrade		50		
1.4	**Specimen Environment & Detector System**	**395**	**245**	**100**	**50**
1.4.1	Sample manipulation stages and motors		90		50
1.4.2	Newport goniometer detector arm upgrade		20		
1.4.3	New optical bench for sample chamber		25		
1.4.4.	High vacuum sample chamber and components		45		
1.4.5	Direct detector x-ray CCD		65		
1.4.6	Low-temperature sample stage			100	
1.5	**Beamline Component Upgrades**	**580**		**580**	
1.5.1	34-ID-C white-beam slit			60	
1.5.2	New white-beam mask after existing mirror M1			50	
1.5.3	New 30cm-offset horizontal double-crystal mono			400	
1.5.4	Modified white-beam mask in existing SOE			30	
1.5.5	Miscellaneous upgrades at beamlne			40	
1.6	**High-Dynamic Range Area Detectors**	**2980**	**980**	**0**	**2000**
1.6.1	Pilatus pixel array detector 2M		980		
1.6.2	Pilatus pixel array detector 6M for forward CXDI				2000