Science Opportunities for 3D x-ray diffraction microscopy- Sector 34

Materials' properties depend largely on mesoscopic structure and evolution on length scales from fractions of a micron to fractions of a millimeter. Nondestructive, in-situ structural, x-ray diffraction measurements provide a direct link to theory, simulations, and multi-scale modeling of structural evolution during deformation, or other strained states.

In a 2007 report, "*CONDENSED-MATTER AND MATERIALS PHYSICS: The Science of the World Around Us*"**,** the National Research Council of the National Academies Committee on CMMP 2010, Solid State Sciences Committee, identified the prospects for CMMP in the early part of the 21st century. Clearly articulated in this comprehensive report is the value of measurement techniques designed to probe the properties of matter at smaller length scales to advance the forefront of condensed-matter and materials physics research.

The APS is the recognized world leader in developing and using the 3D diffraction technique to examine the structure of materials with sub-micron spatial resolution *in all three dimensions*. To preserve this advantage and remain at the forefront in 3D structural microscopy will require both strong research programs that exploit this tool for the benefit of important materials science problems, and a vigorous development effort to improve this capability and extend it into the nano-scale regime.

Dislocation Cell Elastic Strain Distribution

The deformation behavior of metals is of central importance to the forming and eventual failure of components. Despite decades of intensive research, fundamental aspects of the evolution of dislocation structures are still poorly understood. Micro-diffraction experiments at the APS have now provided crucial, but previously inaccessible information on the deformation process: namely, (i) the evolution of local orientations and sub-grain strains during in the initial stages of deformation, and (ii) the distribution on sub-micron length scales of elastic strains within deformed materials. Understanding the mechanisms of crystal plasticity requires a comprehensive study of how and why dislocations are distributed heterogeneously in the form of 'dislocation cells' in the crystal. These recent studies using submicron sized x-ray beams have presented the first quantitative, spatially resolved measurements of elastic strains within dislocation cells [1]. As the dislocation cell elastic strains originate as backstresses from the dislocation walls, the observed variation in the cell-interior strains strongly suggest that a similarly broad distribution may exist for the dislocation walls themselves. The measurements on dislocation walls will require 50 nanometer x-ray probe or better. The new information will provide critical data for the development of detailed dislocation-based simulations and models.

Sn Whisker Growth Mechanism

Sn whisker spontaneous growth in films is a long-standing and technologically critical question, which has plagued the electronics industry since the first attempts to replace Pb-based solders [2]. It is a surface relief phenomenon of creep, driven by a compressive stress gradient. The whiskers are electrically conductive, crystalline structures and have been observed to grow to lengths of several millimeters (mm) and are typically ~1 micron in diameter. Many electronic system failures have been attributed to short circuits caused by tin whiskers that bridge closely-spaced circuit elements maintained at different electrical potentials. Tin is only one of several metals that is known to form whiskers: others include Zinc, Cadmium, Indium and Antimony, so this represents a general issue in many metal systems. Earlier measurements of Sn whiskers with the unique 3D microdiffraction capabilities at APS 34-ID-E support new evidence for preferential grain orientations and a grain-boundary network near whiskers. To detect strain near the root of whiskers, the current spatial resolution of submicron x-ray beam is not sufficient. With a new 50 nanometer x-ray diffraction microscope probe, measurements will be greatly extended by the ability to map the strain field in the whisker root, which is the key to fully understanding the whisker growth mechanism.

Diffraction from materials in extreme environments: Tera-pascal Ultrahigh Pressures

Advances in high-pressure diamond-anvil cell (DAC) technology have opened many new areas in fundamental research for materials and earth sciences [3]. The maximum attainable and sustainable pressures can be reached by using nano-fabricated diamond anvil tips to reduce the pressurized volume. Ultrahigh-pressure investigations in the terapascal regime rely upon the ability to probe samples in a sub-100 nanometer scale through the anvils, and to distinguish sample signals from the background signals from the surrounding materials. The first study using submicron x-ray probe at 34-ID-E has been very encouraging and obtained the first diffraction pattern at 270 GPa. The unique interchangeable white and monochromatic micro/nano-diffraction method provides a powerful way to probe submicron single crystals at ultrahigh-pressure. We can expect the next breakthroughs of reaching terapascal pressures and opening the vast new areas for discovery of novel materials and phenomena.

Phase Separation and Strained Domains in Manganite Systems

Current research on strongly-correlated electron systems is driven by both scientific and technological interest in areas such as high-temperature superconductivity, colossal magnetoresistance (CMR), and spintronics [1]. In particular, transition-metal oxide systems exhibit a variety of interesting and useful electronic and magnetic properties. Often, these physical properties arise from a subtle competition between competing phases with similar lattice structures. For example, lattice or charge distortions to an underlying cubic-perovskite structure in manganite systems can lead to phase transitions between charge-ordered insulators and ferromagnetic metals. Near phase boundaries, spatially inhomogeneous microstructures and self-organized domains spontaneously appear. Understanding the complex electronic, structural and spin interactions between such coexisting nanoscale domains will likely provide the key to understanding the interesting physical properties in these systems.

The development of 3D spatially-resolved x-ray nanodiffraction capabilities will enable the first quantitative measurements of the microstructure associated with phase domains in strongly-correlated electron systems. Structural modulations such charge and orbital ordering are known to exist in manganites, but no technique has been available thus far to map the orientations or spatial distributions of phase domains. Similarly, theoretical models suggest that inhomogeneous strain fields couple to the electronic order and consequently play an essential role in phase coexistence [2]. For example, strain fluctuations may create pinning or nucleation centers for domain formation. The ability to nondestructively, quantitatively map structural modulations and strain distributions with nanoscale spatial resolution would represent a breakthrough for characterizing nanoscale phase separation. This tool will enable unique new studies, leading to significant advances in our understanding of strongly-correlated electron systems.

[1] E. Dagotto, *Science* **309**, 257 (2005).
[2] K.H. Ahn, T. Lookman and A.R. Bishop, *Nature* **428**, 401 (2004).

Added value of the medium term upgrade

The nano-probe diffraction microscope will operate at the same time as the existing micro-beam platform and will serve users whose science requires a diffraction probe with spatial resolution that is approximately an order of magnitude better than currently available. The extension of such capabilities to the nano-scale has been shown feasible, and ideal optics can provide beams as small as 18 nm for polychromatic and monochromatic investigations.

Since the new nano-probe system would operate in parallel with the existing micro-focusing program, the investment will double the access of white beam Laue and monochromatic beam diffraction capabilities for 3D diffraction investigations to a diverse and growing user community.

New and greatly improved data processing and analysis software will be developed for user friendliness and high throughput.

Expected user communities

This science area brings together a very large and scientifically diverse community with interests in materials deformation, electromigration, recrystallization, fatigue, solid-solution precipitation, high pressure environments, and condensed matter physics. The user community interested in this area is large and growing. Key individuals are: Gene Ice, Bennett Larson, John Budai, Jon Tischler (Oak Ridge National Laboratory), Ho-kwang (Dave) Mao (Carnegie Institution of Washington), and Lyle Levin (National Institute of Standards and Technology).

Enabling technology and infrastructure

A canted undulator beam line will be optimized for separate micro-beam and nano-beam diffraction experiments. One branch of the beam line will be for improved microbeam capabilities, and the other branch on a separately tunable undulator will feed a new hutch that includes the nano-scale diffraction platform, and includes environmental stability control (temperature, noise, vibration).

The beam line focusing optics for each branch line will provide excellent, diffraction limited spot size at each experimental location and will be used for either white beam or mono beam studies.

New fast detectors (CCD, Pilatus, vortex) will be deployed to increase the throughput and effectiveness of the facility. Specialized sample cells will be designed to allow users to perform experiments at elevated or reduced temperatures, and for in-situ deformation studies of materials.

(If these exciting 3D diffraction microscopy activities remain co-located on a beam line that also performs coherent diffraction imaging, a new front end design may be required to enable three independently operated insertion devices).

Industry and technology transfer

While there are no specific industrial partnerships or technology transfer agreements, there are many opportunities for industrial participation in many research activities at the APS, and many opportunities for vendor collaboration – particularly through partnerships enabling technology and infrastructure.

Estimated Budget

	Capital	M&S
Year 1: ……	2000	
Year 2: ……	2000	300
Year 3: ……	720	400
Year 4: ……	0	300
Year 5: ……		
Total:	$5700 K	