2-ID Mid-Term Upgrade Plan

Introduction

X-ray microprobes at 2-ID have been used for novel studies in materials, environmental, earth and planetary, biomedical, life, and nano science. The world-class performance of these instruments has led to many ground-breaking results over the last decade, as is evidenced by publication record and ever increasing user demand. Driven by new scientific inquiries, however, the user community has expressed a critical need for significantly improved spatial resolution, better detection schemes, and specific sample environments than are presently possible. At the same time, demands for beamtime at 2-ID have risen steadily to a level that is difficult to sustain. Both issues have been documented by several recent workshops. The proposed 2-ID upgrade plan is designed to address both the quality and quantity of beamtime available. For that purpose, 2-ID will be dedicated entirely for scanning microscopy with nanoscale resolution, which will enable it to provide state-of-the-art μ-XRF, μ-XAFS, and μ-XRD capabilities at the APS. The upgrade plan (Fig. 1) calls for parallel operation of three *nanoprobes*: 1) a diffraction nanoprobe capable of in-situ and dynamical measurements (XRD), 2) a fluorescence nanoprobe that can handle dried biological or materials samples in air (RT-XRF), and 3) an in-vacuum fluorescence nanoprobe that can preserve frozen-hydrated specimens at cryogenic conditions (cryo-XRF) to allow imaging of specimens in a near native state, and combat radiation damage. All three instruments will be able to operate at spatial resolution better than 50 nm. Current versions of the first two instruments already have a large established user base who will substantially benefit from the increased beamtime and improved performance (higher flux, resolution, stability), while the cryogenic nanoprobe will provide a new and much needed capability at the APS. These dedicated medium-resolution nanoprobes (compared to 26-ID and Bionanoprobe) will be highly efficient for many studies.

The source and beamline will also be upgraded to enhance the performance of the proposed nanoprobes. Two canted undulators will be installed, one will be optimized for the 2-20 keV operation of the cryo-XRF nanoprobe while the other for the 5-30 keV operation of the RT-XRF and XRD nanoprobe. For proper operation of scanning microscopes at the nanometer scale, beam stability is one of the most important requirements. The beamline upgrade is designed to achieve high beam stability over many hours and during energy scans by eliminating all optics between a virtual source aperture (VSA) and the nanoprobes. To allow independent control, a separate VSA will be provided for each nanoprobe. The impact of the upgrade on the user community will be minimized by careful planning of the beamline upgrade and the installation schedule.

Science Drivers

Understanding the structures of films and nanostructures at the nanometer scale will enhance our ability to create nanostructures and also the development of new models to describe their behavior at this scale. This will lead to rapid advances in condensed matter, materials engineering, and nanoscience. The XRD nanoprobe will focus on addressing scientific needs in three main areas: magnetism and dynamics in condensed matter physics, strain engineering in integrated circuits, and the atomic and crystal structure of nanomaterials. It will enable novel in-situ and dynamics studies not currently feasible by incorporating controls of the incident beam polarization, sample temperature and pressure, external electric and magnetic fields, and timing resolution better than 100 ps. With sub-50 nm spatial resolution, it will be possible for instance to study the dynamics of polarization and magnetization switching (and their coupling) at the scale of single structural, ferroelectric, or magnetic domains. While strained silicon channels have been rapidly integrated in both PMOS and NMOS structures, the ability to measure strain directly in these 50-nm or smaller devices will led to better understanding of factors affecting device performance and reliability (Fig. 2a). For self-assembled nanomaterials such as ZnO nano-wires or ribbons, x-ray microdiffraction may be crucial for the development of techniques to better control the orientation, symmetry, and growth of these nanostructures. There are currently no structural probes with high enough resolution in both reciprocal space and real space for studying these devices or structures.

The proposed RT-XRF nanoprobe will routinely offer higher resolution (50 nm), flux, stability, and elemental sensitivity (sub-attogram) than current 2-ID microprobes. This will be essential for emerging nanoscale studies in the materials, earth and planetary, and life sciences. For instance, one of the main challenges for photovoltaic devices is in increasing the conversion efficiency, by controlling the metal impurities and precipitates in solar cell materials. The new RT-XRF nanoprobe will provide sufficient sensitivity and resolution for measuring these ever smaller nano-defects individually, whose composition and distribution are determining factors of the device performance (Fig. 2b). In planetary science, interstellar grains collected by the NASA's Stardust mission or other interplanetary dust particles from asteroids and comets are often less than 1 micron in size, thus requiring a higher resolution nanoprobe for elemental imaging. Analysis of these particles will lead to better understanding of the conditions in the dust clouds in interstellar and circumstellar space, and their formation in the Solar Nebula.

In life science, the 50-nm RT-XRF nanoprobe will be capable of producing high resolution images of most subcellular organelles, intracellular details of a bacterium, and even resolving individual virus particles, which can be used to answer many questions not currently possible. One important application will be in infectious diseases, e.g. tuberculosis and malaria, where the host cells often engulf the pathogens or parasites in small vacuoles, in which the local biochemical exchanges and metal transport will ultimately determine the fate of the pathogens (Fig. 2c). In addition, we will be able to resolve the internal structure of many organelles, thus being able to answer not only questions pertaining to the metal content of organelles but also questions requiring knowledge about the metal distribution within the organelles. This is critically important, for example, when investigating numerous diseases associated with mitochondria. The simplicity of working at room temperature will enable studies such as these in a manner complementary to the cryo-XRF station.

The cryo-XRF nanoprobe will enable a host of new scientific studies not previously possible in life science and soft matters. Cryo-fixation allows a specimen to be preserved in its natural hydrated state, avoiding artifacts from drying and chemical fixation, and thus yields the most faithful rendering of a living cell at high resolution. In biology, one-third of all known proteins contain a metal cofactor, and they regulate many essential cellular functions (e.g. differentiation, division, transcription, apoptosis). Not surprisingly, dysregulation of metals has been linked to diseases (Alzheimer's, Parkinson's) and tumor growth (angiogenesis). Thus there is an urgent need to study metalloproteins at the cellular and tissue level in homeostatic and pathogenic states. In medical science, metal-containing drugs had been used for treating cancer, leukemia, diabetic etc., even though their cellular distribution and transformation were not well understood. Similar studies will also be very important for the emerging field of nanomedicine. In particular, nanocomposites (e.g. DNA-TiO_2-Gd) have huge potential in allowing a single agent to combine functional diagnosis (MRI imaging by Gd targeted through DNA) with therapy (DNA scission through the TiO_2 component). XRF microscopy is uniquely well positioned to address many basic scientific questions that are critical for the development and acceptance of such agents.

Cryo-preservation is also known to increase the radiation tolerance of soft specimens, which is essential in particular for any meaningful micro-XANES analysis at higher resolution, as radiation-induced chemistry is already apparent even at the current spatial resolution of ~ 200 nm. The cryo-XRF nanoprobe will be a unique tool for environmental science which often involves heterogeneous samples down to the nano scale, and local chemical speciation is the determining factor for many reactions (e.g. transport and redox reaction of metals and environmental contaminants mediated by microbes). Additionally, the energy regime of this instrument, 2-20 keV, will allow spectroscopy at the important phosphorus and sulfur edge. The ability to study the chemical speciation of phosphorus in marine sediments, where polyphosphate is thought to be a key player, will lead to better understanding of geologic phosphorus sequestration. Access to the sulfur edge is also crucial for soil, geological, and planetary sciences.

Added Value

The upgrade is designed to enhance the quantity and quality of beamtime at 2-ID. By using canted undulators and reconfiguring the beamline to support parallel operation of the three nanoprobes, the upgrade will double the available beamtime. The beam quality will be improved by optimized undulators, much better stability, higher spatial and energy resolution. At the same time, the cost and user interruption during beamline upgrade will be minimized by utilizing as much as possible existing front end, 2-ID-A/D/E stations, beamline components, and phased installation schedule.

This upgrade builds upon the technical expertise and diverse user base developed at 2-ID over the years. In particular intimate collaboration with users had kept us at the leading position, but the decade-old instruments need to be upgraded to the next generation in order to stay competitive. Worldwide, many x-ray microprobes with nanoscale resolution will come online in the next few years at new and existing third generation synchrotron sources.

Enabling Technology & Infrastructure

The XRD nanoprobe user facility at 2-ID will take advantage of recent technical developments in x-ray optics, in both large-area and high-speed detectors, in high-resolution sample positioning, and in software. It will incorporate controls of the incident beam polarization by diamond phase plate, sample temperature from 10 K to 400 K, external electric and magnetic fields, high pressure, and timing resolution better than 100 ps. Considerable effort will be required to integrate all these controls with low-vibration mechanical design and positioning feedback into a user instrument.

Both XRF nanoprobes proposed here will benefit greatly from the design and operation experience of the 26-ID Nanoprobe, as well as advanced detector development. Single element energy dispersive detector typically intercept 3-5% of the XRF over 4π , which lead to an unacceptable 95% signal loss. Thus availability of multi-element detector with large solid angle (> 30% of 4π) and high count rate (>10^7 cps) is essential for both instruments. Optimized control and analysis software are another important development that will enhance these user instruments. In addition, the cryo-XRF nanoprobe will require careful design and development of a cryogenic sample holder and sample transfer system compatible with biological soft specimens.

Partnerships

The user community will be represented at several levels. The XMI Beamline Advisory Committee will provide general oversight of the upgrade plan. Workshops for targeted user communities will continue to be organized to facilitate scientific and technical discussions, to educate users about these instruments, and to expand the user base. Specific components of these instruments may be developed jointly with individual investigators, for instance an NSF proposal with UW-Madison had already been submitted for the development of the XRD nanoprobes. Technical partnerships with external companies and experts will also be established for the nanoprobes and detectors development.

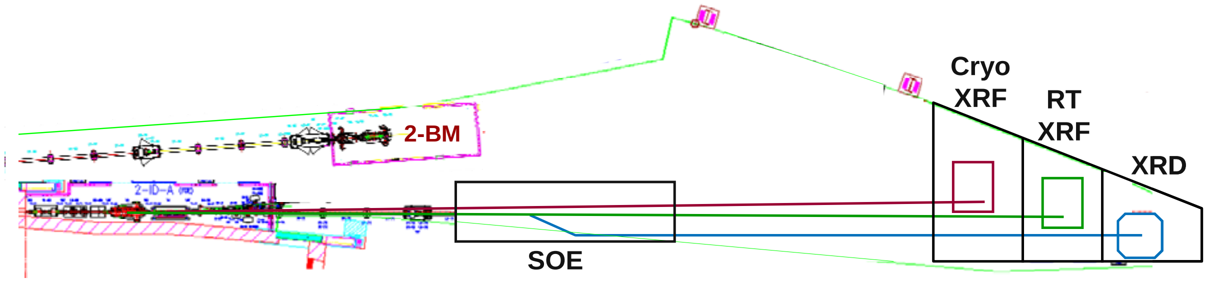

Fig. 1 Beamline layout of the 2-ID upgrade. Three nanoprobes (Cryo-XRF, RT-XRF, XRD) are located at the end of Sector 2 in a large station with three separate entrances. A second optics enclosure (SOE) will be constructed at ~ 50 m to house three virtual source apertures (VSA), various monochromators, shutters, etc. The beam offsets are ~ 0.5 m between the two XRF nanoprobes, and ~ 1 m between the RT-XRF and XRD nanoprobe.

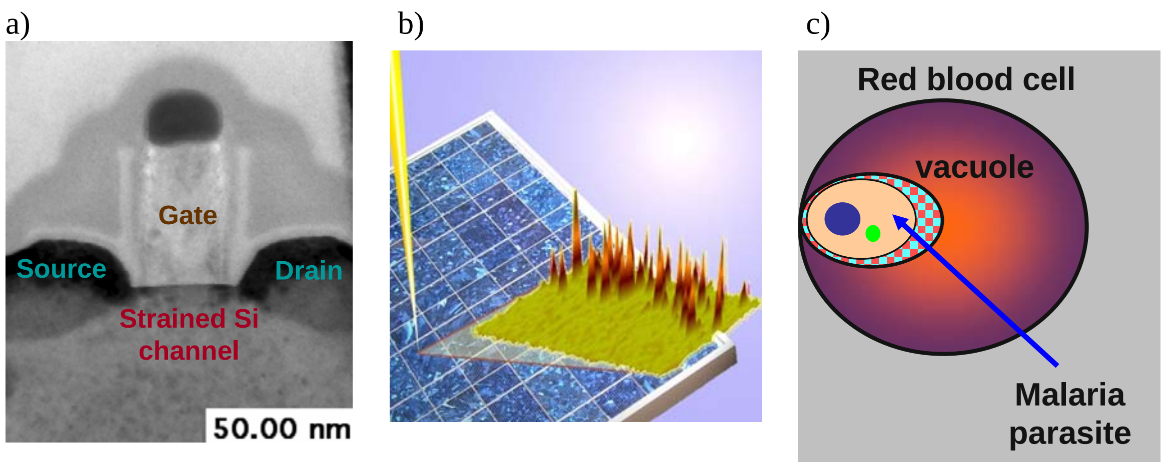

Fig. 2 Examples of scientific studies enabled by the nanoprobes. a) Strained engineering is employed in advanced microelectronic devices to increase the drive current. b) Metal nanoprecipitates act as recombination centers and limit the conversion efficiency of solar cells. c) Pathogens are engulfed by their host in small vacuoles where local chemical exchanges are crucial for the pathogen survival.

	Equipment Budget Table		Phase 1	Phase 2	Phase 3
	2-ID Beamline Upgrade ($k)	***9850***	3190	4660	1800
1.1	**Undulator Systems**	**800**			
1.1.1	2 Undulators and gap controls		700		
1.1.2	New vacuum chamber, steering magnets, etc.			100	
1.2	**Front End**	**80**		80	
1.3	**2-ID-A Station**	**1410**			
1.3.1	Sagittal focusing M1 mirror			100	
1.3.2	Dual steering mirror system			450	
1.3.3	Harmonic rejector			400	
1.3.4	Pink beam shutter			60	
1.3.5	Standard components (slits, masks, collimators…)			200	
1.3.6	Vacuum components (valves, bellows….)			120	
1.3.7	Beam transport		80		
1.4	**Second Optics Enclosure (SOE)**	**2340**			
1.4.1	SOE station with utilities		400		
1.4.2	Vertical diffracting monochromator (existing)			100	
1.4.3	Large offset horizontal monochromator		500		
1.4.4	Small offset horizontal monochromator		350		
1.4.5	Virtual source aperture (x3)		180		
1.4.6	Mono Shutter (x3)		150		
1.4.7	LN2 pump and line (x2)		350		
1.4.8	Pink beam aperture (x3)		150		
1.4.9	Vacuum components (valves, bellows…)		100		
1.4.10	Beam transport		60		
1.5	**2-ID-D Station**	**750**			
1.5.1	Hutch expansion with utilities			600	
1.5.2	temperature and humidity control			100	
1.5.3	Standard components			50	
1.6	**XRD Nanoprobe**	**1000**		1000	
1.7	**RT-XRF Nanoprobe**	**1500**			
1.7.1	RT microprobe			1200	
1.7.2	Multi-element XRF detector			300	
1.8	**Cryo-XRF Nanoprobe**	**1800**			
1.8.1	Cryo microprobe				1500
1.8.2	Multi-element XRF detector				300
1.9	**Beamline controls**	**100**	100		
1.10	**PSS/EPS**	**70**	70		