A Dedicated High-Energy X-ray Beamline for the Studying the Mechanical Behavior of Materials

Dean R. Haeffner & Jonathan D. Almer

Science

High-energy x-rays are an increasingly popular tool for measuring strain and texture during thermo-mechanical deformation and material evolution during processing. Obtained information is critical to understanding the mechanical behavior of materials, and can often be used to validate (or refute) theoretical models as well as to improve material synthesis. The combination of penetration power (to insure bulk measurements) and high flux is unique to high-energy x-rays and is crucial to study many materials systems or processes.

High-energy x-ray scattering techniques are applied in a variety of ways to a large range of scientific subjects. The proposed upgrade will allow us to explore time scales and spatial resolutions that are currently not possible at the APS--or anywhere else. To illustrate this, we will limit our scientific examples to the representative, and important, area of heterogeneous materials.

One important use of high-energy x-rays scattering is to study composite materials. Composite materials are ubiquitous and their use in economically important materials is commonplace. These materials are by definition heterogeneous with the goal being to have the aggregate performance being considerably greater than sum of the parts. To achieve this goal, there are a large number of variables that can be manipulated. For example, in metal matrix composites (MMC) these include: reinforcing material size, shape, composition, amount, distribution, and thermal processing, as well as matrix composition.

The size of components in many composites is such that microfocused high-energy x-rays can be used to probe the individual constituents *in situ*. Also, as a nondestructive probe, we can follow the evolution of various properties (e.g., chemical and phase composition, strain, texture) as a function of processing. For example, the load sharing between a ceramic fiber and its surrounding matrix can be monitored during fatigue testing, and as a function of temperature. A common goal of such experiments is to use the resultant data to develop or validate models that can be used to understand a broader class of materials behavior.

One of the emerging areas for high-energy x-ray composite experiments is on biological materials, especially hard tissue such as bones and teeth. These natural nanoparticulate-reinforced composites possess complicated hierarchical structures and due to their obvious importance have been the subject of intense study by the medical community. However, the ability to measure the response of distinct constituents to mechanical load is key new information that we can now provide using high-energy x-rays. In addition to diffraction from the crystalline phases, high-energy small-angle scattering provides information on the amorphous phases (e.g., collagen). The aim is to better understand the mechanical behavior and related material degradation (e.g., osteoporosis) by examining their micro-mechanical response to load at characteristic length scales. Further, bio-implants can be studied to elucidate load sharing and cohesion with surrounding bone.

Another major area for studying heterogeneous materials with high-energy x-rays is layered systems. Examples of such systems are protective coatings (including thermal-barrier coatings), environmental-barrier coatings, metal-nitride coatings, and solid oxide fuel cells. As in composites, the ability to microfocus high-energy x-rays is key. Nondestructive measurements of texture, strain, porosity, and chemical and phase composition as a function of depth provide valuable information in these complicated materials. Many of these materials are designed to be used in non-ambient (often extreme) conditions, so *in situ* measurements are needed to obtain the most relevant information. For example, it is a goal of this program to measure the x-ray scattering from an operating fuel cell.

Added value to the medium term upgrade

The upgrades listed below will add value to the subject area in several ways. The first is obvious, but none-the-less of critical importance. Increased flux and faster detection will dramatically increase the ability to make faster measurements. Many interesting kinetic phenomena lie outside of our current capability to measure, either because the signal is too weak, or because the phenomena are too fast to catch with our current resolution. Examples include many phase transitions and strain rate determined mechanical behavior. The expected increases in measurement speed will make dramatic changes in the type of science that can be achieved.

A second obvious effect of faster measurements is that many more measurements can be made in a given time. This allows the extension of parametric studies to include more variables, which is of critical importance for studies of materials processing, where there are often many knobs that can be turned. In some studies, an example being the biomechanics of bone, sample-to-sample variation is often difficult or undesirable to control, and the added capability to measure many samples to determine the range of behaviors is crucial for appropriate interpretation.

One way to use increased brilliance is to “spend” it in ways that produce smaller beams and/or monochromatic beams with increased energy resolution. In particular, we expect to push the spatial resolution of the high-energy x-ray beam to well under a micron, which can be used in a multitude of ways to study heterogeneous samples, a key part of the proposed scientific agenda for this beamline.

The aspect of dedicating an entire beamline to mechanical behavior measurements has the obvious effect of making more beam time available for such experiments, but there are subtler effects that are also very important. The current situation on 1-ID requires instrumentation to be assembled and disassembled frequently. This has many detrimental effects: it is hard on equipment, causing considerable wear and tear; the setups are never “tweaked” for optimal performance, and the overall quality is compromised; compromises are made in the designs of equipment to allow for quick and relatively simple disassembly, which sometimes effects overall performance; and the operation of the beamline becomes considerably harder for the staff. Also, if the beamline scientific program is streamlined (as proposed), many efficiencies are gained in setup, operation, and particularly in data handling and analysis. Overall, the scientific efficiency is improved by considerably more than the simple ratio of available beam time.

Expected user communities

There exists a large community interested in the study of mechanical behavior in materials and in the processing of structurally important materials. Currently beam time at 1-ID for such experiments is heavily oversubscribed, and the staff has largely curtailed all outreach efforts to recruit new users. If the program is expanded as proposed here, it will be easy to fill the newly available beam time with high quality experiments.

Major neutron facilities (ISIS, LANCE, and the SNS) all have dedicated instruments in the proposed area of research and much of the work done on these instruments could be strongly complemented by high-energy x-ray scattering.

We believe that significant user communities in industry and the defense community (i.e., Navy, Air Force) could be developed if enough beam time was available.

Enabling technology and infrastructure

The proposed enhancements are as follows:

General Beamline

Currently 1-ID is split between three high-energy programs. Roughly half of the current beam time for this beamline is given to science covered by this proposal. We propose to dedicate an entire insertion beamline to this effort. This could be done by leaving the current program on 1-ID with a modified version of the current 1-ID Phase II Upgrade proposal and moving the other two programs, or by moving the program for this proposal to a new “green-field” beamline.

Source

Several changes are proposed to improve the source of x-rays for the beamline. First, we propose modification of the straight section to be a “long” straight section allowing for the use of more than the current five meters for insertion devices. Second, we would populate that straight section with insertion devices optimized so that the ensemble provides maximum brilliance consistent with continuous energy coverage from 45 keV to 120 keV. To achieve both optimal brilliance and the maximum tunability for a given insertion device, we request a storage ring chamber allowing the insertion device to be closed to 9.5 mm. Simultaneous use of multiple insertion devices may require that the front end for the beamline be upgraded. However, the use of several devices with differing periods will allow for the brilliance to heat load ratio to be considerably better than for the currently used insertion devices.

Detectors

Great opportunities for improving beamline performance exist in the area of upgrading detectors. A key feature of high-energy x-ray diffraction is that scattering from samples is strongly forward directed, allowing for collection of scattering data out to large Q on area detectors placed downstream of the sample. There is strong synergy between the various high-energy x-ray efforts in this respect. For the subject of this proposal, a key enabling technology is the ability to perform simultaneous SAXS measurements with the WAXS measurements. A proposed detector is shown in Figure 1. This detector configuration uses currently available technology (e.g., from Perkin-Elmer). Each of the panels is 400 X 400 mm and can be read out in 30 msec (1k X 1k mode). The smaller SAXS detector would have similar or better temporal resolution.

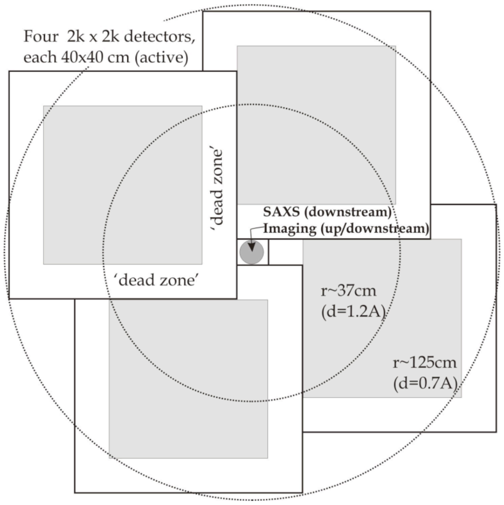

Figure 1. Proposed quad-panel configuration, using panel scaling for existing a-Si detectors. For reference, Debye ring radii and corresponding d-spacings are given for E = 80 keV and a distance = 3 m.

A third detector is requested for imaging measurements that will be used in conjunction with scattering measurements. It will need to be fast (at least 30 msec readout), have good spatial resolution (~5 micron resolution), be reasonably efficient at high energies, and be sufficiently radiation hard to survive for several years.

Optics

Currently, all focusing on the 1-ID beamline is vertical. We propose to add sagittal focusing to the proposed beamline to increase the flux density on sample by an order of magnitude. Some instrumental R&D will be necessary to determine if this is best done through crystal diffraction, a multilayer mirror or refractive optics. In order to permit 3D spatially-resolved studies, we will utilize conical and/or spiral slit systems.

Ancillary Equipment

A key science driver for this proposal is the ability to measure high-energy scattering from *in situ* samples. The recent commissioning of the MTS load frame on 1-ID has clearly demonstrated that it is necessary to have top-notch ancillary equipment as well as top-notch x-ray capability. Furnaces, cryostats, additional load frames, and other equipment will be procured and installed. To fully utilize this equipment, it is necessary to have sufficient space on the beamline to allow the setups to be installed and left in place. Currently usage, such as on 1-ID, where equipment is routinely removed from the beamline to make room for subsequent experiments, significantly compromises the performance of the ancillary equipment.

Software

Data handling and analysis are increasingly a bottleneck for producing science from synchrotron experiments. For example, it is crucial to provide sufficient analysis capability for quasi-real-time feedback to make intelligent choices during an ongoing experiment. Using the detectors described above, terabytes of data will be collected in a relatively short time. Our current computer infrastructure, both hardware and software, is ill suited to handle this massive amount of data. This is already a problem for many experiments at 1-ID, and if the upgrades being proposed are even partially implemented, it will become rapidly much worse.

Partnerships and user interest

Several potential partners exist for subject of this proposal. Both the Air Force and Navy have large programs that would benefit from this facility, and many industries could also be interested. The biomechanics research could be of interest to NIH, NSF, or the biological part of DOE. The fuel cell research could be partnered with either SECA, DOE, or another of the growing number of groups working in the sustainable energy field.

The user interest is very strong, as demonstrated by the sustained oversubscription of 1-ID. A list of current and interested users is too long to be given here.

Industry and technology transfer

The techniques enabled by this proposal have direct relevancy to industry and could lead to a variety of technological advances to be subsequently transferred. Many issues exist if this is a strong goal of the program, but most of those are political and bureaucratic, not technical.

Estimated Budget

A rough budget for the proposed upgrade is as follows: if program is on the upgraded 1-ID beamline (as in Phase II)—$2M, $1M for detectors, $200K for optics, $300K for ancillary equipment, and $100K for computer infrastructure hardware. (Total = $3.6M) If the program is moved to a greenfield beamline, the first item will significantly increase, but will make 1-ID available for other programs. We have no estimate for storage ring, insertion devices, front-end items, or computer infrastructure manpower.