

Face Page

TITLE OF PROPOSED RESEARCH: A High-Resolution Powder Diffractometer for the APS

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Date 23-Jan-03

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Yes (attach an explanation) _____ No x

16. ORGANIZATION'S NAME, ADDRESS AND CERTIFYING REPRESENTATIVE'S NAME, TITLE, AND PHONE NUMBER

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SIGNATURE OF ORGANIZATION'S CERTIFYING REPRESENTATIVE _____
Date 23-Jan-03

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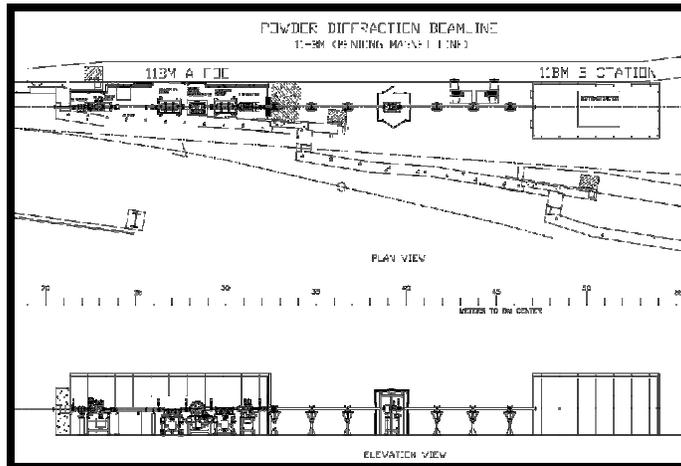
U.S. Department of Energy—Office of Science
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Enhanced Research Capabilities at DOE X-ray and Neutron Facilities

A High-Resolution Powder Diffractometer for the APS

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Table of Contents

<i>Abstract</i>	4
<i>Narrative</i>	5
I. Introduction	5
II. Scientific Justification	6
II.1 Condensed Matter Physics	6
II.2 Materials Science and Chemistry	9
II.3 Proteins and Pharmaceuticals	9
II.4 Mineralogy/Geosciences	10
III. Instrument Design	11
III.1 X-ray Optics	12
III.2 Experimental Station	14
IV. Instrument Operation	16
V. User Community and Policy	16
V.1 Instrument User Community	16
V.2 User Policy	17
<i>Literature Cited</i>	19
<i>Budget and Budget Explanation</i>	21
<i>Other Support of Investigators</i>	30
<i>Biographical Sketches</i>	31
<i>Description of Facilities and Resources</i>	41
<i>Appendices</i>	42
Letter of Support from APS Management	43
External Advisory Committee Review	44
Potential Users of a High Resolution Powder Diffractometer at the APS	49

Abstract

Definitive knowledge of the crystal structure of a material—inorganic, organic, or biological—is the gateway to understanding its physical properties, its chemical reactivity, and/or its biological functionality. It is the most fundamental aspect of any material. The increasingly complex chemistry and physics of modern materials demands that this structural information be obtained in a routine fashion and with state-of-the-art precision. This is true for materials of interest to fundamental physics, materials science, mineralogy, and biology. All of these types of materials impact DOE missions that are in the national interest including energy storage, remote sensing, environmental remediation, and metallurgical testing and validation. Because most of these materials only exist as polycrystalline solids, the definitive structural experiment requires high-resolution x-ray powder diffraction. We propose a high-resolution x-ray powder diffractometer that exploits the high flux, high-energy bandwidth, and precise energy tuning of the third-generation synchrotron source at the Advanced Photon Source (APS). As the goal of this proposal, we will design, build, and commission a world-class instrument that will bring state-of-the-art capability to the powder diffraction user community and will encourage the rapid growth of that user community at the APS. The result of this project will be a user-friendly, high-resolution, high-throughput instrument positioned to initiate leading structural science of importance to fields ranging from condensed matter physics and materials chemistry to pharmaceutical and biological sciences.

Narrative

I. Introduction

We propose a high-resolution x-ray powder diffractometer that exploits the unique capabilities of the third-generation synchrotron source at the Advanced Photon Source (APS). We will design, build, and commission a world-class instrument that will bring state-of-the-art capability to the powder diffraction user community and will encourage the rapid growth of that user community at the APS. The outcome of such a project will be a user-friendly, high-resolution, high-throughput instrument that will deliver world-leading structural science. This instrument will be used by investigators from a wide spectrum of scientific backgrounds, ranging from condensed matter physicists to materials chemists and materials scientists, from mineralogists and geologists to biologists and protein crystallographers. Our goal is to produce an instrument that will deliver critical structural information in a rapid, straightforward, and user-friendly package.

Definitive knowledge of the crystal structure of a material—inorganic, organic, or biological—is the gateway to understanding its physical properties, its chemical reactivity, and/or its biological functionality. It is the most fundamental aspect of any material. The increasingly complex chemistry and physics of modern materials demands that this structural information be obtained in a routine fashion and with state-of-the-art precision. This is true for materials of interest to fundamental physics, materials science, mineralogy, and biology. All of these impact DOE missions that are in the national interest including energy storage, remote sensing, environmental remediation, and metallurgical testing and validation. Because most of these materials only exist as polycrystalline solids, the definitive structural experiment requires high-resolution x-ray powder diffraction. This proposal addresses that need. Illustrative examples of the current and future opportunities for the fundamental scientific output of the diffractometer—which is the driver for this proposal—are highlighted in Section II.

Availability of high quality structural data to a wide user community is the guiding principle behind the design and operation of this proposed instrument, and our goal is to provide a system with the users' needs at the forefront. The instrument design outlined in Section III envisions a high energy (4.8 – 40 keV), high-resolution ($\Delta d/d = 2 \times 10^{-4}$) instrument with an automated sample changer to exploit the high throughput made possible by third generation synchrotron technology. Likewise, the stable constant beam provided by the top-up operation—unique to the APS among the world's synchrotron facilities—will further optimize data quality and throughput of the system.

With the users' needs in mind, our design goal is to produce a full, refinable, high-resolution dataset in one hour. With such capability, users will be able to study how their materials respond parametrically under a variety of environments, including variable temperature (4–1500 K) and variable processing atmospheres (vacuum – pure oxygen). Precision energy tuning ($\Delta E/E = 10^{-4}$) will permit resonant scattering for elemental contrast studies across a substantial fraction of the periodic table. When combined with automated sample exchange and environment control, these capabilities will render high-resolution parametric studies of complex materials routine, with little or no intervention necessary by the user or beamline scientist. As a key part of a user facility we envision as part of this proposal a laboratory at the APS to facilitate final sample

preparation leading to the diffraction experiments. A user access policy summarized in Section V will facilitate single experiments, long-term proposals and rapid access to the instrument, allowing both extensive, detailed study of materials as well as immediate response to "hot" topics.

As highlighted in its support letter (Appendix I), APS management has given its full commitment to this project, allowing us to plan aggressively for the design, construction, and commissioning of the instrument. Our target is user operation by October, 2005. Details of the project scope, timeline, and initial budget estimates indicate that this schedule is achievable at an estimated cost of \$3.6 M.

II. *Scientific Justification*

Many of the most compelling materials being studied today are not available in single-crystal form during the critical period following initial discovery, if ever. It is precisely during this phase that structural information is most critically needed. Powder diffraction provides the crucial tool for determining structure, for following structural changes parametrically (temperature, field, etc.), and for defining future synthetic approaches for enhancement of some desired property (conductivity, thermal expansion, biological activity, etc.). The dedicated high-resolution powder diffractometer proposed in this document will address current opportunities across structural science and will be ready to answer the future needs of users as they appear. Below are representative examples of such current and future opportunities, emphasizing the extensive utility and demand we anticipate for this instrument. The fields of study are broad, ranging from condensed matter physics to materials chemistry and from proteins and pharmaceuticals to geoscience. It is this broad ranging utility of powder diffraction that argues for user-friendly availability of state-of-the-art instrumentation. The instrument proposed here embraces this scientific breadth while fully exploiting the unifying advantages of the APS: high energy, wavelength tunability, and high flux.

II.1 Condensed Matter Physics

The atomic arrangements, bond lengths and angles, and coordination geometries in large part determine the electronic and magnetic properties of materials. Thus, an understanding of crystal structure is an extremely powerful tool for inferring the underlying physics of condensed matter systems. This section illustrates a few important current topics in condensed matter physics. These examples illustrate just some of the types of problems of interest to condensed matter physics in which powder diffraction can have a substantial impact. Specifically, we describe how researchers will be able to exploit the unique capabilities of the proposed powder diffractometer to study these and other problems.

Charge and Orbital Order The behavior of charge and orbital degrees of freedom is recognized as a powerful organizing principle for understanding the physics in transition metal oxides and chalcogenides.^{1,3} For example, the properties of colossal magnetoresistive (CMR) oxides depend critically on the relative occupation of z^2 and/or x^2-y^2 orbitals on Mn. Recent study of the spinel CuIr_2S_4 has revealed a novel charge ordering motif in which Ir^{3+} octamers interleave with Ir^{4+} octamers.² Examples of other systems that manifest charge and/or orbital order are Li-

intercalated CoO_2 and spin-crossover compounds (e.g. cobalt oxides). Importantly it is the cooperative ordering of the charge and orbitals on either short- or long-range length-scales that determines the rich physics of manganites and other related systems. The examples given above demonstrate that the full breadth of charge order and orbital order remains to be discovered and understood. Anomalous scattering at the appropriate x-ray absorption edge for the oxidation states of interest is a powerful method to study charge ordering,^{4,6} a critical design element of our proposed instrument.

Relaxor Ferroelectrics Complex perovskite oxides such as $\text{Pb}(\text{Mg}_{1/3}\text{Nd}_{2/3})\text{O}_3$ (PMN) are known as relaxor ferroelectrics because frustration (arising from compositional fluctuation and/or field inhomogeneities) prevents long-range ferroelectric phase development.⁷ Rather, polar nanoregions (PNRs) form on cooling, yielding a broad maximum in the dielectric function. The mechanism of such relaxor behavior remains an extremely active area of research in condensed matter science.⁸⁻²¹ As in the case of charge- and orbitally-ordered materials, subtle changes in coordination around the Pb atom are markers of the ferroelectric distortion. Crystallographic studies have recently revealed new symmetries²² in the solid solution of PMN with PbTiO_3 (PT) and are exploring how PNRs coarsen into bulk ferroelectric phases as the PT concentration increases.²³ Indeed, the impact of nanoscale ordering on bulk properties is of general importance to condensed matter physicists, implying an extensive milieu in which this instrument will find importance. PDF analysis of high-resolution synchrotron data, described below, is one way to model these effects.

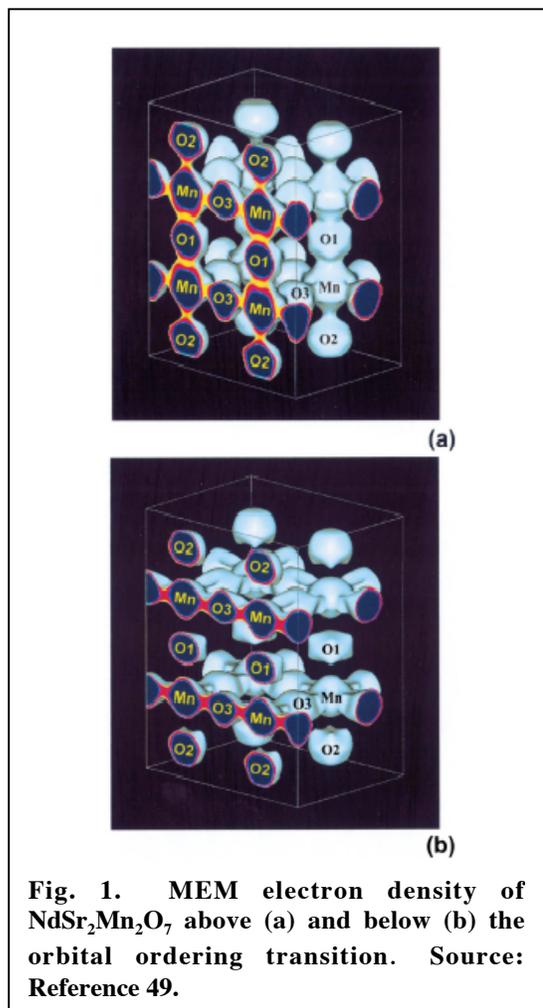
Exotic Superconductors The discovery of superconductivity in layered copper-oxide compounds in 1986 opened a new era of superconducting materials with structures far more complex than the intermetallic compounds that had previously been the main focus of this research. It was realized that the full power of both neutron and x-ray powder diffraction techniques was needed to understand these materials. Even after many years of research, single crystals are often not available for study of the properties over the wide chemical composition ranges used to control the properties. Neutron diffraction is particularly useful because of its sensitivity to the "light" atoms, such as oxygen. High-resolution x-ray diffraction is needed for structure solution, investigation of metal-site mixing, and characterization of important microstructural issues such as twin domain formation and stacking faults. Such microstructural features manifest themselves in the Bragg peak shape in high-resolution data. Recent work on compounds such as $\text{GdSr}_2\text{RuCu}_2\text{O}_8$,²⁴⁻²⁶ which exhibits both superconductivity and ferromagnetism, illustrates the how state-of-the-art neutron and x-ray powder diffraction, and electron microscopy, must be combined to understand the behavior. The recent reports of superconductivity in layered carbide and nitride compounds²⁷⁻²⁹ illustrate the kinds of new superconducting compounds that will offer similar challenges for understanding how structural properties affect superconducting behavior.

Even superconductors with simple crystal structures can pose some challenging questions to be resolved by diffraction methods. For example, in the case of MgB_2 , theoretical models indicate that anharmonicity in a particular phonon of the hexagonal B sheet is critical to superconductivity.³⁰ Subtle changes in unit cell dimensions near the superconducting transition temperature may be associated with the strong coupling of the electronic structure to the crystal lattice.³¹ Observing and studying such changes parametrically requires both high-resolution and rapid collection of high quality data. Such issues are not exclusive to MgB_2 , and recent interest

in borocarbides and related materials (interplay of superconductivity and magnetism),³² "115" intermetallics,³³⁻³⁶ etc. highlight that this is a growth area where detailed structural studies will impact the understanding of fundamental physics.

The powder diffractometer described in this proposal will become a powerful tool to study problems in condensed matter physics such as those described above. For example, high-resolution parametric studies will deepen the understanding of phase transitions in novel transition metal oxides. The ability to rapidly explore phase space will make phase diagram studies routinely accessible to the general community interested in these complex materials, opening the door to discovery of new systems. The frontier of crystallography depends on moving beyond the average structure encoded in the Bragg reflections. One such approach for studying local structure is pair distribution function (PDF) analysis. PDF study of ferroelectrics has been championed by Egami, et al.,³⁷ and the technique has also found application to semiconductors³⁸, high- T_C superconductors,³⁹ and CMR manganites.^{40, 41} Our instrument will provide the well-resolved high- Q data crucial for such researchers to accurately model the structure factor, $S(Q)$, using this technique. These models of local structure will enhance characterization of the subtle distortions developing in a broad spectrum of materials, for example the ferroelectric distortions evolving in relaxor materials. Likewise, the study of charge ordering in transition metal oxides and chalcogenides requires readily tunable energy for resonance-enhanced scattering to highlight differences in oxidation state. Energy tuning across a broad energy bandwidth is a highlight of instruments at the APS. This proposed instrument will have usable energy range, 4.8–40 keV, with sufficient energy resolution ($\Delta E/E \sim 10^{-4}$) for probing the absorption edges of interesting transition metals (Ti-La K edge and the L_3 edge of metals above Cs). Such anomalous scattering experiments are also relevant to doped compounds (intermetallics, oxides, etc.), where knowledge of site occupancies are of importance for understanding their chemistry and physics.

Orbital ordering has heretofore been inferred from M-O bond lengths and/or from complex single crystal measurements relying on the polarization dependence of anomalous scattering.⁴² The Maximum Entropy Method (MEM) is a powerful alternative that uses powder diffraction to directly generate the electron distribution in the crystal. As a model-independent technique, MEM is well-suited to study problems beyond the spherical-atom approximation and has been applied to intermetallics,⁴³ MgB_2 ,⁴⁴ semiconductors,⁴⁵ fullerenes⁴⁶ and other related systems.^{45, 47, 48} In



particular, Takata et al.⁴⁹ have applied MEM to charge and orbitally ordered manganites. Here, the orbital order-disorder transition in $\text{NdSr}_2\text{Mn}_2\text{O}_7$ was studied as a function of temperature, validating theoretical models of the low-temperature orbital occupation (Fig. 1). The high flux and high resolution of the proposed instrument will expand the range of such problems where subtle changes in electron distribution strongly impact the physics. In addition, MEM has recently been applied to polarized neutron scattering to extract spin density distributions.⁵⁰ This positions a complementary x-ray and neutron MEM study as an extremely powerful approach for generating a full picture of the magnetic and electronic structure of the crystal.

II.2 Materials Science and Chemistry

The need for increasingly high-performance materials drives the search for such systems into increasingly complex chemistries. Structural study of these complex materials (e.g. framework solids, ferroelectrics, and solid oxide fuel cell materials requiring four or more components, etc.) requires high-resolution data to map out phase stability and composition. High throughput for phase diagram exploration is required to enable parametric composition studies in complex phase fields. As in the case of condensed matter physics, precise tunability of the energy is also required to sort out multiple atom site occupancies *via* the effect of resonant scattering on atom scattering contrast. For example, the electronic properties of Type I clathrate materials (e.g., $\text{Eu}_8\text{Ga}_{16}\text{Ge}_{30}$ and $\text{Sr}_8\text{Ga}_{16}\text{Ge}_{30}$)⁵¹ are predicted to depend strongly on the distribution of cations over the Zintl ion framework.⁵² When the atomic numbers of the species in the framework are similar, traditional x-ray techniques are unable to resolve these site distributions. Recently, a resonant x-ray study on the Type I clathrate $\text{Cs}_8\text{Cd}_4\text{Sn}_{42}$ identified that the Cd ions selectively occupy a single crystallographic site, and that there are no vacancies in the framework.⁵³ The technique clearly has broad applicability in other complex materials, such as zeolites.

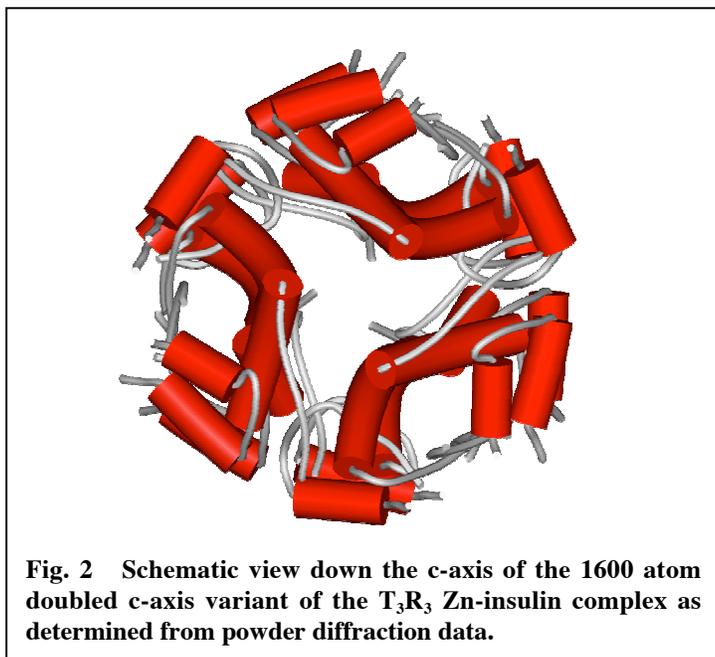
Commonly, developments in materials science arise from synthesis and subsequent characterization of materials with certain properties of interest (conductivity, thermal expansion, magnetoresistance, catalytic effect, etc.). Understanding of these materials originates from precision structure determinations coupled with parametric studies (T, $p\text{O}_2$, composition, etc.). As most of these materials rarely come as single crystals, high-resolution powder diffraction is essential for structural work. A recent example comes from the field of dielectric ceramics, in which complex phase equilibria in the $\text{LaCa}_{0.5}\text{Zr}_{0.5}\text{O}_3 - \text{SrTiO}_3$ pseudobinary have been studied.⁵⁴ Phase diagrams and structure-property relationships were determined for several samples quenched from synthesis conditions. The proposed instrument would dramatically increase the rate at which the phase space in such systems can be mapped, as well as offering *in situ* monitoring of reactivity and phase transformations at elevated temperatures.

II.3 Proteins and Pharmaceuticals

Both small molecule pharmaceuticals and proteins exhibit complex phase behavior depending on crystallization conditions (solvent composition, pH, etc.) that can affect crucial biological activity. This is not surprising considering the internal flexibility of these materials. The structures of these biological molecules are not only scientifically interesting, but also commercially important, as slight changes in processing conditions can affect polymorph formation impacting drug efficacy as well as patent position. For example, hemozoin—formed

by the action of the malaria bacterium *Plasmodium* on hemoglobin—was examined by high-resolution synchrotron powder diffraction.⁵⁵ *Ab initio* solution of the centrosymmetric 43 atom problem showed that the structure consisted of dimers formed by pairs of heme molecules instead of a previously assumed polymeric structure. This example illustrates new developments in structure solution techniques from powder diffraction data using simulated annealing approaches to explore the suite of values for a limited number of structural degrees-of-freedom, in this case torsion angles.

Although now in its infancy, protein powder diffraction will likely become a tool of considerable power for exploring protein structure variation upon synthetic mutation (amino acid substitution), protein-ligand complex formation, and changing crystallization conditions.



Recently the structure of the 1600 atom protein crystal structure of a Zn(insulin) complex has been solved from high-resolution powder diffraction data.⁵⁶ A simple, three-parameter rigid body Rietveld refinement gave the location and orientation of the two Zn(insulin) complexes; a subsequent stereochemical restraint Rietveld refinement gave a full description of this 1600 atom structure (Fig. 2). It was subsequently found⁵⁷ that a single crystal of this material surrounded by cryoprotectant gave the same doubled c-axis variant of this Zn(insulin) complex at low temperatures. The structure was found to be essentially identical to that given from powder diffraction.

In each of these cases a single point detector/analyzer system was used to collect the high-resolution powder diffraction data. A recent exploratory study at the APS using an image plate showed this could be considerably improved. Indeed, a 40 sec exposure from ~1 mg of material gave useful diffraction data for stereochemical restraint Rietveld refinement (Fig. 3); the quality of the diffraction data obtained in this way is a 10^4 - 10^6 improvement over that obtained with the single point analyzer/detector technique. The proposed instrument will readily obtain similar quality data, thus facilitating protein powder diffraction studies while following structural changes driven by complex formation, pH/solvent changes, radiation damage, etc.

II.4 Mineralogy/Geosciences

Understanding geological processes requires knowledge of crystal structures under conditions far from ambient: at high and low temperatures, at elevated pressures, and under controlled-atmosphere conditions. In addition, many reactions occurring under controlled-atmosphere conditions and at elevated temperatures take place rapidly, requiring the capability to obtain

diffraction data as quickly as possible. Indeed, some redox and hydration/dehydration reactions would benefit from the ability to collect data in a matter of seconds, providing heretofore-unavailable kinetic information (*e.g.*, Ståhl et al.⁵⁸) Atmosphere control would also provide the ability to study oxidation-reduction reactions in real time under geologically relevant temperature conditions. Many hydration reactions are sufficiently rapid that their kinetics cannot be studied with conventional diffraction systems, but the ability to obtain structural information on the scale of minutes to seconds will allow separation of crystal structural effects from macroscopic diffusion-related effects, ultimately providing more accurate information on mechanisms (*e.g.*, Fridriksson et al.⁵⁹).

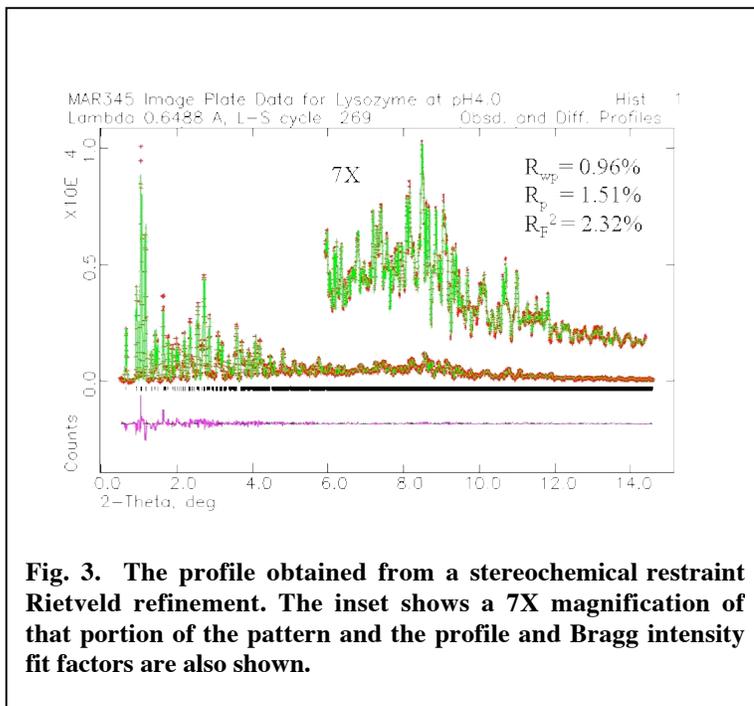


Fig. 3. The profile obtained from a stereochemical restraint Rietveld refinement. The inset shows a 7X magnification of that portion of the pattern and the profile and Bragg intensity fit factors are also shown.

The broad energy range of the proposed instrument will make it very well suited for resonant scattering studies of cation order-disorder in most geologically relevant systems. Cation order-disorder is particularly important in understanding and interpreting conditions of formation and alteration in minerals, and many systems do not lend themselves to simple site-occupancy refinement (*e.g.*, Fe-Mn systems).

The proposed diffractometer responds to the geosciences community by providing a user-friendly platform with precise energy tuning and extensive environmental control. Operation in the low-resolution mode (*vide infra*) will make kinetics studies on seconds to minutes time scales routinely available to the geosciences community.

III. Instrument Design

The purpose of this beamline is to provide a dedicated and reliable facility for a wide-ranging powder diffraction user community and to do so on an accelerated schedule. Toward this end, all of the components of the proposed beamline utilize existing and proven technologies. We have taken other successful high-resolution powder diffraction beamlines into our design consideration and will modify their plans as needed. Adopting these proven technologies leverages our ability to follow an aggressive construction and commissioning timeline, bringing the instrument to users on an accelerated schedule. Some modification and improvement of existing designs are needed in some components, such as the multi-analyzer detector system and auto sample-exchanger, to maximize the productivity and flexibility of the facility. We will also

implement new improvements such as a robotic sample exchanger, described below. However, these modifications will impact neither the design goals nor delivery schedule.

The scientific justification in Section II illustrates the types of experiments the user community will carry out. These experiments define a set of design specifications that we must incorporate to exploit fully the capability of the APS and to deliver the performance our user will demand. With this in mind, the design specifications proposed for this instrument will be focused on achieving the following experimental specifications:

- Energy range – 4.8-40keV (2.58 – 0.31Å)
- Energy resolution – $\Delta E/E \sim 10^{-4}$
- Diffraction resolution – $\Delta d/d \sim 2 \times 10^{-4}$ at high resolution; $\sim 10^{-3}$ at low resolution
- Measurement time – <1hr at high resolution; seconds at low resolution
- Parametric experiment control for T (4-1500K), pO₂ (vacuum – 10 atm)

To meet these specifications we will construct the diffractometer on the 11-BM (bending magnet) port at the Advanced Photon Source, Argonne National Laboratory. This port will give sufficient flux over the desired energy range and has adequate available space on the experimental floor of the APS for construction of the required hutches, shielded beam lines, etc. needed to house the beam line optics and diffraction instrumentation. The beamline layout for the proposed powder diffraction facility at 11-BM appears in Fig. 4. The facility will consist of a first optical enclosure (FOE) containing all white-light components which condition, focus and monochromatize the x-ray beam and a large monochromatic experimental station hutch with a high precision diffractometer and special environmental equipment and an interconnecting shielded beam line. Each of the major components is described in detail below.

III.1 X-ray Optics

The first component in the first optics enclosure (FOE) will be a beam defining slit (APS L3-30 or equivalent) which will limit the horizontal width of the beam to a maximum of ~ 2 mrad and minimize the heat load on subsequent components in the beamline.

A collimating mirror (APS 1BM design Y5-30 or equivalent) is the next beamline component. This optical element will minimize the vertical divergence and thereby increase the energy resolution of the beamline monochromator while maintaining the maximum x-ray flux. The beamline is designed to operate in the energy range 4.8–40 keV (2.58– 0.31 Å). This energy range will cover the *K* absorption edge for elements from Ti to La, and *L* absorption edges for elements above Cs for resonant scattering studies. This design goal constrains the mirror angle and mirror coating. With an incident angle of 2 mrad, a Pt coated mirror will reflect x-rays up to an energy of ~ 40 keV, a Ni coated mirror at a 2.5 mrad incident angle has an energy cutoff of ~ 22 keV, and a Si mirror at this angle has a cut-off energy of ~ 10 keV. Therefore to effectively collimate the x-ray beam, eliminate harmonics and provide a spectrum free of edge jumps from the mirror coatings the mirror system must be able to translate between Pt and Ni coated stripes and an uncoated region of the Si substrate at incident angles ranging from 2.0 to 2.5 mrad. The vertical opening angle of an APS bending magnet (73 μ rad) and the 2 mrad width of the beam require the mirror to be at least 1000 mm long and 200 mm wide. A silicon or SiC substrate is

required since the APS bending magnet heat load of ~ 70 Watts/mrad for the 2 mrad of incident radiation would require cooling to remove ~ 140 Watts of incident power and a GLIDCOP mirror would have too high a cutoff energy at 2.5 mrad.

Following the collimating mirror, the x-ray beam is monochromated by a double crystal fixed exit monochromator (Si 111) with a sagittally bent second crystal for horizontal focusing. Modified versions of either the 1-BM (PSL), 2-BM (Kohzu), or 12-BM/20-BM BESSRC-PNC monochromator designs with a 25 mm offset will provide EXAFS monochromator performance over the required energy range. Commercially available sagittal bender designs have been successfully employed at the APS and ESRF and should also be suitable for this application.

The next component in the FOE will be a un-cooled monochromatic mirror which will serve the dual purpose of deflecting the x-ray beam back into the horizontal plane and to optionally focus vertically onto the sample or detector in the experimental station. The constraints in energy and beam size require this mirror to have Pt and Ni coatings, be used at incident angles of 2-2.5 mrad and be approximately 200 mm wide and 1000 mm long. Since white light operation is not required a SiO_2 substrate such as zerodur is a suitable material.

An adaptive optic mirror such as a bimorph design (e.g. HP-CAT, ACCEL mirror) is one possible choice for the bending mechanism. This design has the advantage of allowing the mirror to be bent into either a flat or curved profile. The use of an adaptive optic mirror bender will also allow achieving the highest possible resolution and best peak shape by adjusting the mirror bend profile (close to flat) to satisfy users with experiments requiring a high degree of peak separation,

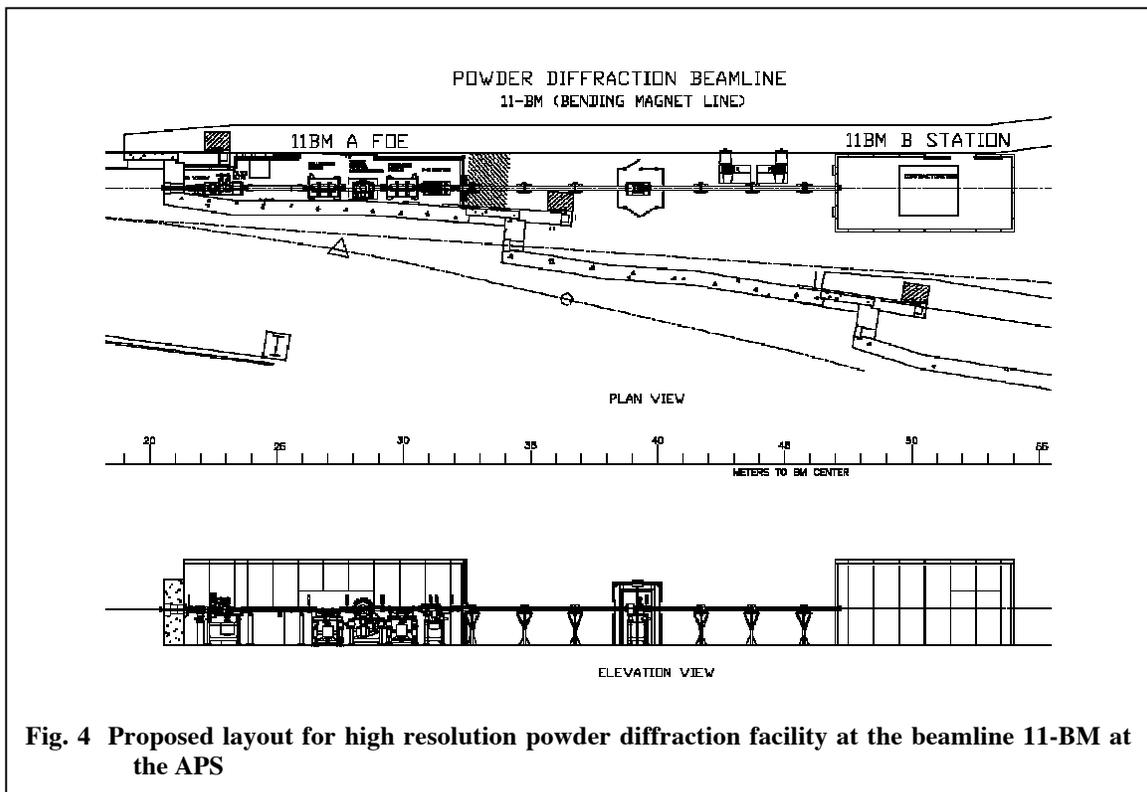
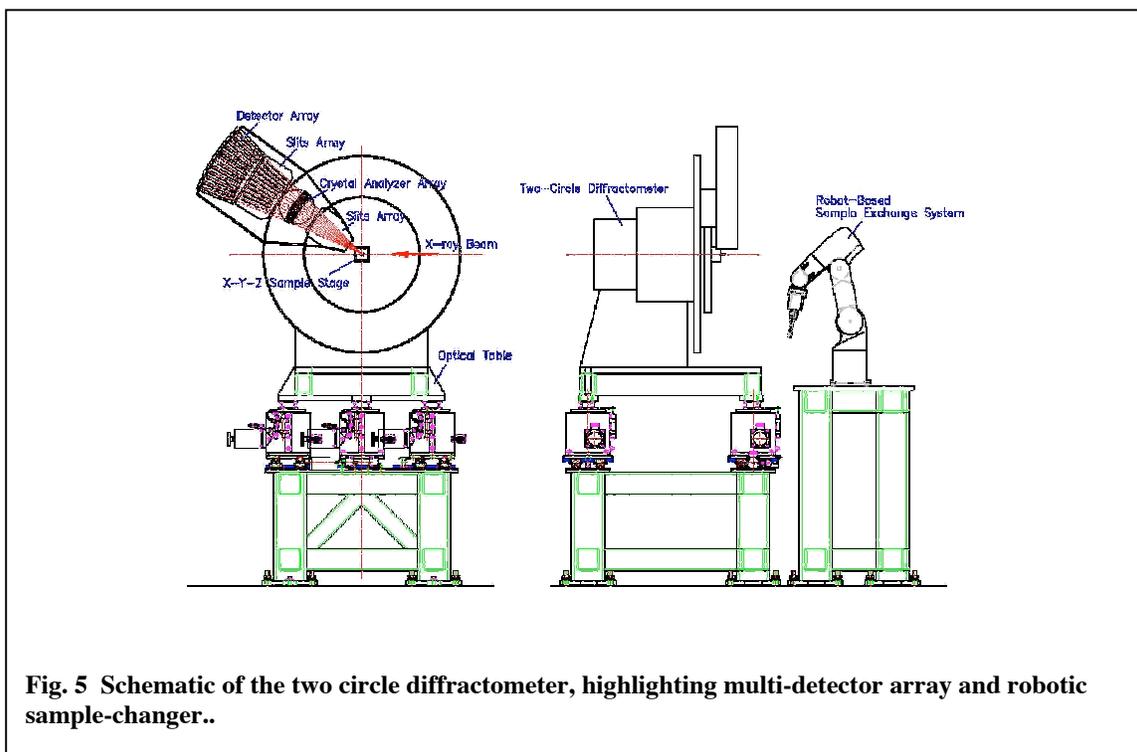


Fig. 4 Proposed layout for high resolution powder diffraction facility at the beamline 11-BM at the APS

or allowing other users to obtain a high photon flux density on a small area by bending the mirror to focus the beam when samples are small.

III.2 Experimental Station

The monochromatic experimental station will enclose a two-circle diffractometer with high accuracy ($<3 \times 10^{-4}$ deg) and high precision ($< 10^{-5}$ deg). This goniometer will also allow continuous angle scans with high precision. As schematically shown in Fig. 5, this diffractometer will be constructed from two heavy-duty high precision rotary tables mounted and aligned coaxially. The rotary tables will be fitted with encoders giving the resolution and accuracy specified above. For room temperature data collection, an x-y-z sample stage can easily position the sample at the center of rotation of the diffractometer. A sample spinner will be built inline with the translation stage to allow spinning of a capillary sample or rocking of a flat-plate sample for powder averaging during the data measurements. This rotation and translation combination stage will be mounted on and aligned coaxially with the two-circle diffractometer. A multi-analyzer detector system will be built and mounted on the 2 θ rotation table of the two-



circle diffractometer. Twelve Ge(220) or Si(111) crystal analyzer detector systems spread over $24^\circ 2\theta$ will be employed and will be able to record twelve different angle range powder patterns simultaneously. This multi-analyzer detector system will be designed to be capable of adjusting each analyzer individually under computer control to select the correct angle for the chosen x-ray energy and to optimize the performance of each analyzer as the incident x-ray energy is changed for resonant scattering studies. Sufficient shielding will be employed to prevent cross-talk between the individual analyzer detector systems. Two parallel slit sets, one before and one after each analyzer crystal, will collimate and separate the diffracted x-rays for each analyzer and its

associated scintillation detector. This multi-channel detector system will greatly reduce the data collection time and facilitate high-resolution time resolved experiments. With this combination of beamline optics and analyzer diffraction geometry, this powder diffractometer should achieve a resolution of $\Delta d/d = 2 \times 10^{-4}$.

To achieve higher data collection rates for parametric studies and experiments on samples subject to radiation damage, we propose use of an area detector positioned some distance (0.1-1.0m) downstream from the sample position. With adjustment of the primary beam focus onto the detector surface, data collection times in seconds should be achievable with a diffraction resolution of $\Delta d/d \sim 10^{-3}$. With future improvement of detector technology, a faster data collection rate may be anticipated.

For high-resolution data collection, we anticipate that <1 hr will be required for each data collection. Much faster rates will be obtained with the lower resolution area detector system. Consequently, the time spent for manual sample mounting and alignment will come to be an unacceptable portion of the beam and manpower time. Because of this problem at several neutron facilities, they have used sample-exchange robotic systems for regular room temperature measurements for several years. More recently, the protein crystallography beamlines at synchrotron facilities have developed robotic systems that can handle samples at liquid nitrogen temperatures. In order to achieve maximum efficiency, we will design and build an automatic sample-exchange system for the proposed powder diffractometer. This system will include a industrial robot to mount and retrieve the samples, a specially designed sample mounting device that permits removal and remounting with high precision, a multi-sample cartridge that will hold a large number of samples, and an auto alignment system to properly position the sample at the center of rotation of the diffractometer.

The instrument will be capable of studying samples under a variety of environmental conditions. Dedicated low temperature and high temperature environmental devices are proposed; the latter will have controlled atmosphere capability that can also maintain at different pressure conditions from vacuum to 10 atm. These devices will be mounted on a platform opposite the two-circle rotating base with suitable x-y-z translations for easy centering. The mounting platform will be designed to carry heavy environmental control devices with high precision, such as pressure cells or other ancillary equipment supplied by the user community.

Online data analysis is an important tool in the sample screening process and for time resolved experiments. It gives facility users real-time feedback to determine how the experiment should proceed, allowing the experimenter to use the beam time wisely and increase beamline productivity. Therefore, a push-button, simple-to-use data collection software system will be designed that incorporates remote access capability. This system will be constructed from components developed in the EPICS format for control of instrumentation at APS. A remote access capability can also be adapted into this system—as demonstrated recently by Sector 4 at APS—and will be part of our long-term operations plan. The data processing user interface will be developed to convert raw data to data formats for all popular data analysis software such as GSAS, Fullprof, etc.

Since careful sample preparation is an integral part of any powder diffraction experiment, we include the acquisition of certain essential laboratory equipment, such as a precision analytical balance, centrifuge and powder grinding mill, as part of this request. The basic laboratory set up as well as sufficient supplies required for final sample preparation before a diffraction measurement will also be included.

IV. Instrument Operation

Following construction and commissioning, the powder diffractometer will be operated by the APS as a general user instrument. The APS has committed to support operating staff and to provide the necessary resources (e.g., electronics, computation, etc.) to operate the instrument. The instrument scientist and team will ensure that the beamline is maintained and that it performs according to design specifications. They will lead the development of ancillary equipment for use on the diffractometer and will code the data collection and analysis software. They will communicate with users regarding scheduled experiments and will work with users to optimize productive use of the instrument and its ancillary equipment. They will assist or collaborate with users on data collection and data analysis as needed.

In addition to a staff dedicated to operating the instrument, the existing neutron and x-ray diffraction program based in the Materials Science Division (MSD) will help formulate and guide the scientific directions of the instrument. We believe that such scientific input will be critical for keeping the instrument attuned and responsive to the needs of leading edge research during its lifetime. The current emphasis of the MSD program is on neutron diffraction at the Intense Pulsed Neutron Source (IPNS) at Argonne and other facilities, including the ISIS Facility in the UK and preparations for an active program at the Spallation Neutron Source. We envision a growth of synchrotron x-ray diffraction until it plays a role comparable to that now played by neutron diffraction. The advantages of having both state-of-the-art neutron and x-ray diffraction available to the MSD research programs will be fully exploited. As at present, this research will be fully integrated into the research programs of MSD. Funding for this evolving program is not requested as part of the present proposal but will derive from existing and/or new funding initiatives in MSD.

V. User Community and Policy

The benchmarks of success for this instrument will be the accomplishments of the user community to which it appeals. The design of a general-purpose instrument with considerable flexibility lays the foundation for such a community to flourish. From the outset, we have taken an active approach to seeking advice and support from the user community and in adopting a user-oriented operations policy.

V.1 Instrument User Community

As part of the development of this proposal we have assessed the user community base in two ways: (a) by holding a proposal review workshop, and (b) by seeking out interest in the community from mailing lists, scientific meeting rosters, etc. Both of these outreach efforts have encouraged us that our instrument will have considerable scientific appeal and will be in demand

among the powder diffraction community as broadly defined in Section II. Indeed, we anticipate that the demand for the proposed instrument will be high, and that it will rapidly become oversubscribed.

We held a proposal review workshop at the APS on November 14, 2002 to discuss the scientific justification and preliminary instrument design goals for the diffractometer. A report of the workshop is attached as Appendix II. We were pleased that David Cox, Paolo Radaelli, and Andrew Fitch were present at this workshop to assist us in validating the design parameters of our instrument. We also benefited from user community representatives, including Pat Woodward, Angus Wilkinson, and Jim Kaduk. It is our intention to invite each of these participants to be charter members of the instrument scientific advisory panel. All workshop participants were of a single voice in urging us to pursue construction of the instrument, and all felt the user community base would be strong. Several recommendations from the group have helped to shape the present document, including issues surrounding focusing optics, the critical importance of peak shape reproducibility, and full utilization of the unique high-energy capabilities of the APS. Fitch in particular—whose instrument at ESRF is a model for our design and operation—admonished us to employ adequate staff support to meet the rigorous schedule of a high-throughput instrument. We anticipate such staff commitments from APS management as an operations follow-on request to this construction proposal.

Attached to this proposal as Appendix III is a list of scientists from far-ranging backgrounds and fields of expertise, all of whom have expressed interest in and support for the proposed instrument. This list of names represents the spectrum of research institutions from the National Laboratory system, to universities nationwide, to industry. This pool of supporting investigators will form the initial mailing list designed to update progress on instrument construction, to solicit initial beam time requests, and to draw upon for proposal review teams. This list will, of course, will be dynamic as the user base develops and evolves.

Importantly, the number, quality, and diversity of interested scientists validate our premise of the need for a user-friendly, high performance powder diffractometer at the APS. To address this need and to accommodate differing experimental projects (scope, timelines), we have adopted in consultation with APS management a user policy described in the next section.

V.2 User Policy

The user policy for general user instruments at the APS is found on the APS web site: http://www.aps.anl.gov/aps/frame_user_info.html. Users may apply for beam time through three types of proposals:

- An "individual proposal" for a single experiment.
- A "program proposal" for an experimental program that may require a series of visits to the APS over a two-year period (less than 10% annually of user time on a given beam line).
- A "rapid-access proposal" requesting unallocated general user time during the current run.

Individual and program proposals will be accepted three times per year (with deadlines in November, February, and August) for running cycles beginning approximately three months

later. Rapid-access proposals will be accepted at any time. If beam time is not approved for immediate access, the proposal will be reconsidered during the next proposal cycle.

This flexible user policy is intended to satisfy the diverse needs of different users. For example, the program proposal can be used to obtain beam time over an extended period as part of Ph.D. thesis research or an ongoing research program. The rapid-access proposal can be used to request time to study newly discovered materials where there is intense competition, as has been the case for new superconducting materials, or to provide researchers the opportunity to access the instrument quickly for short routine measurements that are needed in the course of exploratory materials research.

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Budget and Budget Explanation

DOE F 4620.1 (04-93) All Other Editions Are Obsolete	U.S. Department of Energy Budget Page (See reverse for Instructions)	OMB Control No. 1910-1400 OMB Burden Disclosure Statement on Reverse
ORGANIZATION Argonne National Laboratory		Budget Page No: <u>1</u>
PRINCIPAL INVESTIGATOR/PROJECT DIRECTOR John F. Mitchell		Requested Duration: <u>4</u> (Months)
A. SENIOR PERSONNEL: P/PI/D, Co-PI's, Faculty and Other Senior Associates (List each separately with title; A.6. show number in brackets)	DOE Funded Person-mos.	Funds Requested by Applicant
	CAL ACAD SUMR	Funds Granted by DOE
1. Robert B VonDreele	4.00	
2.		
3.		
4.		
5.		
6. () OTHERS (LIST INDIVIDUALLY ON BUDGET EXPLANATION PAGE)		
7. (1) TOTAL SENIOR PERSONNEL (1-6)	4.00	\$60,593
B. OTHER PERSONNEL (SHOW NUMBERS IN BRACKETS)		
1. () POST DOCTORAL ASSOCIATES		
2. () OTHER PROFESSIONAL (TECHNICIAN, PROGRAMMER, ETC.)		
3. () GRADUATE STUDENTS		
4. () UNDERGRADUATE STUDENTS		
5. () SECRETARIAL - CLERICAL		
6. () OTHER		
TOTAL SALARIES AND WAGES (A+B)		\$60,593
C. FRINGE BENEFITS (IF CHARGED AS DIRECT COSTS)		
Fringe benefits and divisional overhead included in the standard monthly rates. See Budget justification page.		
TOTAL SALARIES, WAGES AND FRINGE BENEFITS (A+B+C)		\$60,593
D. PERMANENT EQUIPMENT (LIST ITEM AND DOLLAR AMOUNT FOR EACH ITEM.)		
Beamline Stations 470K Beamline Infrastructure 165K Beamline Optics 350K		
TOTAL PERMANENT EQUIPMENT		\$985,000
E. TRAVEL		
1. DOMESTIC (INCL. CANADA AND U.S. POSSESSIONS)		
2. FOREIGN		
TOTAL TRAVEL		
F. TRAINEE/PARTICIPANT COSTS		
1. STIPENDS (Itemize levels, types + totals on budget justification page)		
2. TUITION & FEES		
3. TRAINEE TRAVEL		
4. OTHER (fully explain on justification page)		
TOTAL PARTICIPANTS () TOTAL COST		
G. OTHER DIRECT COSTS		
1. MATERIALS AND SUPPLIES		
2. PUBLICATION COSTS/DOCUMENTATION/DISSEMINATION		
3. CONSULTANT SERVICES		
4. COMPUTER (ADPE) SERVICES		
5. SUBCONTRACTS		
6. OTHER		
TOTAL OTHER DIRECT COSTS		
H. TOTAL DIRECT COSTS (A THROUGH G)		\$1,045,593
I. INDIRECT COSTS (SPECIFY RATE AND BASE)		
NOTE: ANL uses multiple rates and basis for distribution of indirect expenses. See budget justification page.		
TOTAL INDIRECT COSTS		\$98,501
J. TOTAL DIRECT AND INDIRECT COSTS (H+I)		\$1,144,094
K. AMOUNT OF ANY REQUIRED COST SHARING FROM NON-FEDERAL SOURCES		
L. TOTAL COST OF PROJECT (J+K)		\$1,144,094

DOE F 4620.1
(04-93)
All Other Editions Are Obsolete

U.S. Department of Energy
Budget Page
(See reverse for Instructions)

OMB Control No.
1910-1400
OMB Burden Disclosure
Statement on Reverse

ORGANIZATION Argonne National Laboratory				Budget Page No: <u>2</u>	
PRINCIPAL INVESTIGATOR/PROJECT DIRECTOR John F. Mitchell				Requested Duration: <u>12</u> (Months)	
A. SENIOR PERSONNEL: PI/PI/D, Co-PI's, Faculty and Other Senior Associates (List each separately with title; A.6. show number in brackets)				DOE Funded	
				Person-mos.	
				CAL	ACAD
				SUMR	
				Funds Requested	Funds Granted
				by Applicant	by DOE
1.	Robert B VonDreele		12.00		
2.					
3.					
4.					
5.					
6.	() OTHERS (LIST INDIVIDUALLY ON BUDGET EXPLANATION PAGE)				
7.	(1) TOTAL SENIOR PERSONNEL (1-6)		12.00		\$190,805
B. OTHER PERSONNEL (SHOW NUMBERS IN BRACKETS)					
1.	() POST DOCTORAL ASSOCIATES				
2.	() OTHER PROFESSIONAL (TECHNICIAN, PROGRAMMER, ETC.)				
3.	() GRADUATE STUDENTS				
4.	() UNDERGRADUATE STUDENTS				
5.	() SECRETARIAL - CLERICAL				
6.	() OTHER				
TOTAL SALARIES AND WAGES (A+B)					\$190,805
C. FRINGE BENEFITS (IF CHARGED AS DIRECT COSTS)					
Fringe benefits and divisional overhead included in the standard monthly rates. See Budget justification page.					
TOTAL SALARIES, WAGES AND FRINGE BENEFITS (A+B+C)					\$190,805
D. PERMANENT EQUIPMENT (LIST ITEM AND DOLLAR AMOUNT FOR EACH ITEM.)					
Beamline Infrastructure 5K Beamline Optics 125K					
General Instrumentation 100K Station Instrumentation 1080K					
Beamline Vacuum Hardware 134K					
TOTAL PERMANENT EQUIPMENT					\$1,444,000
E. TRAVEL					
1. DOMESTIC (INCL. CANADA AND U.S. POSSESSIONS)					
2. FOREIGN					
TOTAL TRAVEL					
F. TRAINEE/PARTICIPANT COSTS					
1. STIPENDS (Itemize levels, types + totals on budget justification page)					
2. TUITION & FEES					
3. TRAINEE TRAVEL					
4. OTHER (fully explain on justification page)					
TOTAL PARTICIPANTS () TOTAL COST					
G. OTHER DIRECT COSTS					
1. MATERIALS AND SUPPLIES					
2. PUBLICATION COSTS/DOCUMENTATION/DISSEMINATION					
3. CONSULTANT SERVICES					
4. COMPUTER (ADPE) SERVICES					
5. SUBCONTRACTS					
6. OTHER					
TOTAL OTHER DIRECT COSTS					
H. TOTAL DIRECT COSTS (A THROUGH G)					\$1,634,805
I. INDIRECT COSTS (SPECIFY RATE AND BASE)					
NOTE: ANL uses multiple rates and basis for distribution of indirect expenses. See budget justification page.					
TOTAL INDIRECT COSTS					\$144,401
J. TOTAL DIRECT AND INDIRECT COSTS (H+I)					\$1,779,206
K. AMOUNT OF ANY REQUIRED COST SHARING FROM NON-FEDERAL SOURCES					
L. TOTAL COST OF PROJECT (J+K)					\$1,779,206

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U.S. Department of Energy
Budget Page
(See reverse for Instructions)

OMB Control No.
1910-1400
OMB Burden Disclosure
Statement on Reverse

ORGANIZATION Argonne National Laboratory				Budget Page No: <u>3</u>	
PRINCIPAL INVESTIGATOR/PROJECT DIRECTOR John F. Mitchell				Requested Duration: <u>12</u> (Months)	
A. SENIOR PERSONNEL: PI/PD, Co-PI's, Faculty and Other Senior Associates (List each separately with title; A.6. show number in brackets)				DOE Funded Person-mos.	
				CAL	ACAD
				SUMR	Funds Requested by Applicant
					Funds Granted by DOE
1. Robert B VonDreele				12.00	
2.					
3.					
4.					
5.					
6. () OTHERS (LIST INDIVIDUALLY ON BUDGET EXPLANATION PAGE)					
7. (1) TOTAL SENIOR PERSONNEL (1-6)				12.00	\$200,298
B. OTHER PERSONNEL (SHOW NUMBERS IN BRACKETS)					
1. () POST DOCTORAL ASSOCIATES					
2. () OTHER PROFESSIONAL (TECHNICIAN, PROGRAMMER, ETC.)					
3. () GRADUATE STUDENTS					
4. () UNDERGRADUATE STUDENTS					
5. () SECRETARIAL - CLERICAL					
6. () OTHER					
TOTAL SALARIES AND WAGES (A+B)					\$200,298
C. FRINGE BENEFITS (IF CHARGED AS DIRECT COSTS)					
Fringe benefits and divisional overhead included in the standard monthly rates. See Budget justification page.					
TOTAL SALARIES, WAGES AND FRINGE BENEFITS (A+B+C)					\$200,298
D. PERMANENT EQUIPMENT (LIST ITEM AND DOLLAR AMOUNT FOR EACH ITEM.)					
Beamline Infrastructure 115K General Instrumention 180K					
Station Instrumentation 145K					
TOTAL PERMANENT EQUIPMENT					\$440,000
E. TRAVEL					
1. DOMESTIC (INCL. CANADA AND U.S. POSSESSIONS)					
2. FOREIGN					
TOTAL TRAVEL					
F. TRAINEE/PARTICIPANT COSTS					
1. STIPENDS (Itemize levels, types + totals on budget justification page)					
2. TUITION & FEES					
3. TRAINEE TRAVEL					
4. OTHER (fully explain on justification page)					
TOTAL PARTICIPANTS () TOTAL COST					
G. OTHER DIRECT COSTS					
1. MATERIALS AND SUPPLIES					
2. PUBLICATION COSTS/DOCUMENTATION/DISSEMINATION					
3. CONSULTANT SERVICES					
4. COMPUTER (ADPE) SERVICES					
5. SUBCONTRACTS					
6. OTHER					
TOTAL OTHER DIRECT COSTS					
H. TOTAL DIRECT COSTS (A THROUGH G)					\$640,298
I. INDIRECT COSTS (SPECIFY RATE AND BASE)					
NOTE: ANL uses multiple rates and basis for distribution of indirect expenses. See budget justification page.					
TOTAL INDIRECT COSTS					\$44,000
J. TOTAL DIRECT AND INDIRECT COSTS (H+I)					\$684,298
K. AMOUNT OF ANY REQUIRED COST SHARING FROM NON-FEDERAL SOURCES					
L. TOTAL COST OF PROJECT (J+K)					\$684,298

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Budget Page**
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OMB Burden Disclosure
Statement on Reverse

ORGANIZATION Argonne National Laboratory				Budget Page No: <u>4</u>	
PRINCIPAL INVESTIGATOR/PROJECT DIRECTOR John F. Mitchell				Requested Duration: <u>28</u> (Months) 6/03 thru 9/05	
A. SENIOR PERSONNEL: PI/PI, Co-PI's, Faculty and Other Senior Associates (List each separately with title; A.6. show number in brackets)				DOE Funded Person-mos.	
				Funds Requested	
				Funds Granted	
				by Applicant	
				by DOE	
1.	Robert B VonDreele	28.00			
2.					
3.					
4.					
5.					
6.	() OTHERS (LIST INDIVIDUALLY ON BUDGET EXPLANATION PAGE)				
7.	(1) TOTAL SENIOR PERSONNEL (1-6)			28.00	\$451,695
B. OTHER PERSONNEL (SHOW NUMBERS IN BRACKETS)					
1.	() POST DOCTORAL ASSOCIATES				
2.	() OTHER PROFESSIONAL (TECHNICIAN, PROGRAMMER, ETC.)				
3.	() GRADUATE STUDENTS				
4.	() UNDERGRADUATE STUDENTS				
5.	() SECRETARIAL - CLERICAL				
6.	() OTHER				
TOTAL SALARIES AND WAGES (A+B)					\$451,695
C. FRINGE BENEFITS (IF CHARGED AS DIRECT COSTS)					
Fringe benefits and divisional overhead included in the standard monthly rates. See Budget justification page.					
TOTAL SALARIES, WAGES AND FRINGE BENEFITS (A+B+C)					\$451,695
D. PERMANENT EQUIPMENT (LIST ITEM AND DOLLAR AMOUNT FOR EACH ITEM.) Beamline Stations 470K Beamline Infrastructure 285K Beamline Optics 475K Beamline Vacuum Hardware 134K General Instrumentation 280K Station Instrumentation 1225K					
TOTAL PERMANENT EQUIPMENT					\$2,869,000
E. TRAVEL				1. DOMESTIC (INCL. CANADA AND U.S. POSSESSIONS)	
				2. FOREIGN	
TOTAL TRAVEL					
F. TRAINEE/PARTICIPANT COSTS					
1. STIPENDS (Itemize levels, types + totals on budget justification page)					
2. TUITION & FEES					
3. TRAINEE TRAVEL					
4. OTHER (fully explain on justification page)					
TOTAL PARTICIPANTS ()				TOTAL COST	
G. OTHER DIRECT COSTS					
1. MATERIALS AND SUPPLIES					
2. PUBLICATION COSTS/DOCUMENTATION/DISSEMINATION					
3. CONSULTANT SERVICES					
4. COMPUTER (ADPE) SERVICES					
5. SUBCONTRACTS					
6. OTHER					
TOTAL OTHER DIRECT COSTS					
H. TOTAL DIRECT COSTS (A THROUGH G)					\$3,320,695
I. INDIRECT COSTS (SPECIFY RATE AND BASE)					
NOTE: ANL uses multiple rates and basis for distribution of indirect expenses. See budget justification page.					
TOTAL INDIRECT COSTS					\$286,902
J. TOTAL DIRECT AND INDIRECT COSTS (H+I)					\$3,607,597
K. AMOUNT OF ANY REQUIRED COST SHARING FROM NON-FEDERAL SOURCES					
L. TOTAL COST OF PROJECT (J+K)					\$3,607,597

WBS	Activity Name	Start Date	Finish Date	FY 03 K\$	FY 04 K\$	FY 05 K\$	BUDGET K\$	2003			2004			2005				
								J	F	M	J	A	S	O	N	D	J	F
1.6	Beamline Vacuum Hardware																	
1.6.1	Ion pumps & controllers	3/1/04	8/6/04		45.0		45.0											
1.6.2	Ion gauges & controllers	3/1/04	8/6/04		15.0		15.0											
1.6.3	Inline valves	3/1/04	8/6/04		15.0		15.0											
1.6.4	Roughing valves	3/1/04	8/6/04		4.0		4.0											
1.6.5	Bellows	3/1/04	9/24/04		10.0		10.0											
1.6.6	Beam monitors	3/1/04	8/6/04		10.0		10.0											
1.6.7	Pumping station	3/1/04	9/24/04		20.0		20.0											
1.6.8	Be Windows	3/1/04	10/29/04		5.0		5.0											
1.6.9	Miscellaneous vacuum hardware	3/1/04	6/4/04		10.0		10.0											
1.7	General Instrumentation																	
1.7.1	PSS	10/4/04	3/14/05			75.0	75.0											
1.7.2	EPS	10/4/04	4/8/05			5.0	5.0											
1.7.3	Controls	3/15/04	6/24/05		50.0	50.0	100.0											
1.7.4	Sample preparation Lab equipment	6/7/04	6/17/05		50.0	50.0	100.0											
1.8	Station Instrumentation																	
1.8.1	Optical table	1/26/04	1/28/05		90.0		90.0											
1.8.2	Diffractionmeter	1/26/04	1/28/05		200.0		200.0											
1.8.3	Diffractionmeter table	1/26/04	1/28/05		90.0		90.0											
1.8.4	Multi-analyzer Detector System	5/24/04	5/27/05		250.0		250.0											
1.8.5	CCD Detector	5/24/04	5/27/05		250.0		250.0											
1.8.6	Close Cycle cryostat	11/22/04	6/24/05			35.0	35.0											
1.8.7	Cryo Stream System	11/22/04	6/24/05			35.0	35.0											
1.8.8	High Temperature furnace	11/22/04	6/24/05			20.0	20.0											
1.8.9	Auto sample exchange system	4/5/04	5/27/05		200.0		200.0											
1.8.10	Beam transport	11/22/04	5/13/05			10.0	10.0											
1.8.11	Riso slits	11/22/04	5/13/05			20.0	20.0											
1.8.12	Turbo pump	10/18/04	2/4/05			20.0	20.0											
1.8.13	Small pumps	10/18/04	2/4/05			5.0	5.0											
1.9	Contingency (10%)	5/5/03	9/30/05	98.5	144.4	44.0	286.9											
1.10	Overhead (13%)	5/5/03	9/30/05	140.9	206.5	62.9	410.3											
				1224.4	1794.9	546.9	3566.2											

Personnel:

- J. F. Mitchell: Project coordinator and scientific liaison between MSD and instrument design and construction team at the APS.
- J.D. Jorgensen: Instrument construction consultant and user community liaison.
- R.B. Von Dreele: Leader of design, construction and commissioning efforts.
- P.L. Lee: APS coordinator for dedicated powder diffraction beamline design and construction.
- M. A. Beno: Technical advisor for X-ray optics, beamline design, and construction.

The project coordinator, J.F. Mitchell is a chemist in Materials Science Division and will be responsible for project oversight and will be heavily involved in the commissioning phase of the project. He will also act as a scientific liaison between MSD and the APS, insuring that the instrument will dovetail with existing and new programs in MSD. J.D. Jorgensen has built the Special Environment Powder Diffractionmeter at the Intense Pulsed Neutron Source (IPNS) and has led an extremely productive user program on this instrument. He will contribute his expertise in powder diffraction to the construction phase of the project and his experience in establishing and maintaining a user program during operation. Mark Beno and Peter Lee have extensive experience designing and building APS instruments and will be involved at all levels

of construction and commissioning of the instrument. No salary for any of these four participants is requested as part of this proposal.

We are requesting new support at the level of one full time equivalent (FTE) for R.B. Von Dreele, whose effort will be charged for each month of this activity. Von Dreele's experience in powder diffraction and instrument design/construction at Los Alamos National Laboratory will be critical to the success of the project. All other staff support (engineers, technicians, etc.) during the duration of this proposal will be furnished by the APS.

Argonne National Laboratory is a government-owned facility operated by the University of Chicago. As a contractor for the Department of Energy, Argonne National Laboratory must comply with DOE general policies and procedures on budgeting and accounting. The Laboratory's procedures are based on the assumption that all costs incurred will be recovered. The costing procedures use standard rates, which are used throughout the Laboratory on a consistent basis and uniformly applied to all work supported by the Department of Energy and other federal agencies.

Standard rates are established at the beginning of the fiscal year for each research division, and are monitored and revised as necessary. All labor costs are distributed using standard rates which are developed by the laboratory's budget office for each major payroll classification within a division. The standard rates are an average of the base rate and fringe benefits (32.5% for a regular staff and clerical, and 11% for postdoctoral appointees), plus a factor for divisional overhead and for paid absences.

Effort is escalated each year by a rate provided by the Argonne Budget Department.

Equipment:

As an instrument construction proposal, the majority of requested funds will be allotted to capital equipment purchases, as detailed in both the budget statements and the timeline.

The following equipment will be purchased for use in this study:

1. Beamline Stations	470K
2. Beamline Infrastructure	285K
3. Beamline Optics	475K
4. Beamline Vacuum Hardware	134K
5. General Instrumentation	280K
6. Station Instrumentation	<u>1225K</u>
TOTAL EQUIPMENT:	2869K

Beamline experimental stations (1.3) are standard x-ray radiation shielding for the APS facility. A-station (1.3.1) is a white light enclosure with shielding appropriate for the radiation levels encountered from an APS bending magnet source. B-station (1.3.2) is a standard monochromatic enclosure large enough to accommodate the instrumentation for a dedicated powder diffraction facility. A mini-enclosure (1.3.3) is included in the beamline design to provide radiations

shielding for beam-defining slits and optic components designed to provide beam diagnostics. Similarly the shielded beam transport (1.4.2) provides radiation shielding for the vacuum x-ray beam transport between these enclosures. This transport pipe is supported by shielded supports (1.4.1), which carry the mechanical load of the beam pipe and also provide radiation shielding for the vacuum joints.

Beamline optics (1.5) are necessary to define and condition the radiation from the source: white beam slits (1.5.1) defines the beam size, the collimating mirror system (1.5.2) provides a parallel beam vertically for high energy resolution and optimum peak shapes, the double crystal monochromator (1.5.3) produces a tunable, low-bandpass beam without changing the beam divergence, a sagittal crystal bender (1.5.4) focuses the beam horizontally, and an adaptive mirror system (1.5.5) focuses the beam vertically. A monochromatic shutter (1.5.6) allows beamline components to stay at a stable operating for increased beam stability. These optical components require highly stable, adjustable mounting tables (1.4.5)

Beamline vacuum components (1.6.1 - 1.6.9) are required to allow a high vacuum path from the x-ray source to the sample. Long term operations of the white light mirror, monochromator and focusing mirror systems require UHV conditions in the principal beamline components. A mobile pumping station (1.6.7) is also required to maintain these components.

Control systems (1.7) are required for the safe and reliable operation of the beamline optical components and experimental stations. Radiation safety concerns for the operation of the experimental stations requires the fabrication of a personnel safety system (PSS, 1.7.1). Safe and reliable operations of the beamline optical components require the fabrication of an equipment protection system (EPS, 1.7.2). Electronics and instrumentation required to operate and monitor the optical components and experimental instruments (1.7.3) are included. The standard laboratory equipment (1.7.4), such as dry box, hood, balance, centrifuge, etc., for sample preparation will also be purchased.

The experimental station instrumentation (1.8) is required to obtain high performance from this dedicated powder diffraction beamline. An optical table (1.8.1) is required for beam conditioning and monitoring. Slits (1.8.11) and beam transport (1.8.10) are mounted on this table. A high-resolution diffractometer with a stable adjustable table (1.8.2-1.8.3) will be used for collection of powder data. A multi-analyzer detector system (1.8.4) to be designed constructed and tested as part of this proposal is required to increase data rates while maintaining accuracy and resolution. For experiments where a large portion of the diffraction pattern must be observed simultaneously, i.e. for time-dependent diffraction studies, a CCD-detector system (1.8.5) will be purchased.

Ancillary equipment for special sample environments includes: a closed cycle cryostat (1.8.6) and cryo stream system (1.8.7) for low temperature data collection, and a high temperature furnace (1.8.8). A turbo pumping station (1.8.12) and small vacuum pumps are required for the experimental station vacuum needs and ancillary equipment operation.

Indirect Costs

The Laboratory uses multiple indirect rates and basis for distribution of indirect expenses. The indirect rates range from 12.2% to 29.2%. The total indirect cost depends on the mix of costs from the various expense categories.

Other Support of Investigators

All Argonne investigators in this proposal are supported by the Department of Energy, Office of Science under Contract No. W-31-109-ENG-38. R. B. von Dreele is currently an employee of Los Alamos National Laboratory.

Biographical Sketches

John F. Mitchell

Education

University of Chicago Ph.D., Mar. 1993; M.S., Dec., 1989
Cornell University B.A. *summa cum laude*, May, 1987

Professional History

Argonne National Laboratory
Chemist, October 2000-present
Assistant Chemist, July 1996-September 2000
Technical Staff Member, Jan.-June 1996
DOE Distinguished Postdoctoral Fellow, Jan. 1993-Jan. 1996

Honors and Awards

- Fellow of the American Physical Society November, 2001
- Presidential Early Career Award for Scientists and Engineers, April 2000
- DOE Young Investigator Award, April 2000.
- DOE Distinguished Postdoctoral Research Fellow, Jan. 1993-Jan.1996

Selected Recent Publications

- "Tricritical point and the doping dependence of the order of the ferromagnetic phase transition of $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$," D. Kim, B. Revaz, B.L. Zink, F. Hellman, J.J. Rhyne and J.F. Mitchell *Phys. Rev. Lett.* **89**, 227202 (2002)
- "Structural and magnetic ordering in $\text{Pr}_{0.65}(\text{Ca}_y\text{Sr}_{1-y})_{0.35}\text{MnO}_3$: Quantum critical point versus phase segregation scenarios," G.R. Blake, L. Chapon, P.G. Radaelli, D.N. Argyriou, M.J. Gutmann and J.F. Mitchell *Phys. Rev. B* **66**, 144412 (2002)
- "Glass transition in the polaron dynamics of colossal magnetoresistive manganites," D.N. Argyriou, J.W. Lynn, R. Osborn, B. Campbell, J.F. Mitchell, U. Ruett, H.N. Bordallo, A. Wildes and C.D. Ling *Phys. Rev. Lett.* **89**, 036401 (2002)
- "Spin correlations and magnetoresistance in the bilayer manganite $\text{La}_{1.2}\text{Sr}_{1.8}\text{Mn}_2\text{O}_7$," S. Rosenkranz, R. Osborn, L. Vasiliu-Doloc, J.W. Lynn, S.K. Sinha and J.F. Mitchell *Physica B* **312**, 763-765 (2002)
- "Indications of intrinsic chemical and structural inhomogeneity in lightly doped $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$," T. Shibata, B. Bunker, J.F. Mitchell and P. Schiffer *Phys. Rev. Lett.* **88**, 207205 (2002)
- "Structure of nanoscale polaron correlations in $\text{La}_{1.2}\text{Sr}_{1.8}\text{Mn}_2\text{O}_7$," B.J. Campbell, R. Osborn, D.N. Argyriou, L. Vasiliu-Doloc, J.F. Mitchell, S.K. Sinha, U. Ruett, C.D. Ling, Z. Islam and J.W. Lynn *Phys. Rev. B* **6501**, 014427 (2002)
- "Structural effect on colossal magnetoresistivity in manganites: Bond versus band," D. Louca, T. Egami, W. Dmowski and J.F. Mitchell *Phys. Rev. B* **6418**, 180403 (2001)
- "Spin, charge, and lattice states in layered magnetoresistive oxides," J.F. Mitchell, D.N. Argyriou, A. Berger, K.E. Gray, R. Osborn and U. Welp *J. Phys. Chem. B* **105**, 10731-10745 (2001)
- "Composition dependence of the spin wave stiffness parameter in $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ CMR materials,"

- J.J. Rhyne, H. Kaiser, L. Stumpe, J.F. Mitchell, T. McCloskey and A.R. Chourasia *J. Magn. Magn. Mat.* **226**, 775-776 (2001)
- "Impact of oxygen partial pressure on the Ruddlesden-Popper series $\text{Nd}_{1.2}\text{Sr}_{1.8}\text{Mn}_2\text{O}_7$: Oxygen vacancy formation and ordering," J.E. Millburn and J.F. Mitchell *Chem. Mat.* **13**, 1957-1966 (2001)
- "Magnetic phase diagram of layered manganites in the highly doped regime," J.F. Mitchell, C.D. Ling, J.E. Millburn, D.N. Argyriou, A. Berger and M. Medarde *J. Appl Phys.* **89**, 6618-6620 (2001)
- "Effect of layering and doping on the magnetic anisotropy of the layered manganites $\text{La}_{2-2x}\text{Sr}_{1+2x}\text{Mn}_2\text{O}_7$ ($x=0.3-0.4$)," U. Welp, A. Berger, V.K. Vlasko-Vlasov, H. You, K.E. Gray and J.F. Mitchell *J. Appl Phys* **89**, 6621-6623 (2001)
- "Phase separation and low-field bulk magnetic properties of $\text{Pr}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$," I.G. Deac, J.F. Mitchell and P. Schiffer *Phys. Rev. B* **6317**, 172408 (2001)
- "Mesoscopic and microscopic phase segregation in manganese perovskites," P.G. Radaelli, R.M. Ibberson, D.N. Argyriou, H. Casalta, K.H. Andersen, S.W. Cheong and J.F. Mitchell *Phys. Rev. B* **6317**, 172419 (2001)
- "Competition of charge, orbital, and ferromagnetic correlations in layered manganites," D.B. Romero, Y. Moritomo, J.F. Mitchell and H.D. Drew *Phys. Rev. B* **6313**, 132404 (2001)
- "Manipulating coincident charge and spin order with pressure and field in a doped manganite," A.S. Roy, A. Husmann, T.F. Rosenbaum and J.F. Mitchell *Phys. Rev. B* **6309**, 094416 (2001)
- "Polaron ordering in ferromagnetic colossal magnetoresistive oxides," L. Vasiliu-Doloc, R. Osborn, S. Rosenkranz, J. Mesot, J.F. Mitchell, S.K. Sinha, O.H. Seeck, J.W. Lynn and Z. Islam *Inter. J Modern Phys. B* **14**, 3711-3718 (2000)
- "Interplay of spin and orbital ordering in the layered colossal magnetoresistance manganite $\text{La}_{2-2x}\text{Sr}_{1+2x}\text{Mn}_2\text{O}_7$ ($0.5 \leq x \leq 1.0$)," C.D. Ling, J.E. Millburn, J.F. Mitchell, D.N. Argyriou, J. Linton and H.N. Bordallo *Phys. Rev. B* **62**, 15096-15111 (2000)
- "Oxygen stoichiometry in $\text{Sr}_3\text{Mn}_2\text{O}_{7-\square}$: A raman scattering investigation," I. Guedes, J.F. Mitchell, D. Argyriou and M. Grimsditch *Phys. Rev. B* **62**, 13809-13811 (2000)
- "Soft spin waves in the low-temperature thermodynamics of $\text{Pr}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$," M. Roy, J.F. Mitchell, A.P. Ramirez and P. Schiffer *Phys. Rev. B* **62**, 13876-13879 (2000)
- "The electronic structure of $\text{La}_{0.66}\text{Ca}_{0.33}\text{MnO}_3$ and $\text{La}_{1.2}\text{Sr}_{1.8}\text{Mn}_2\text{O}_7$ studied by angle resolved photoemission," R. Liu, W.C. Tonjes, C.G. Olson, J.J. Joyce, A.J. Arko, J.J. Neumeier, J.F. Mitchell and H. Zheng *J. Appl Phys* **88**, 786-789 (2000)
- "Magneto-optical imaging of the first order spin-flop transition in the layered manganite $\text{La}_{1.4}\text{Sr}_{1.6}\text{Mn}_2\text{O}_7$," U. Welp, A. Berger, D.J. Miller, V.K. Vlasko-Vlasov, K.E. Gray and J.F. Mitchell *J. Appl Phys* **87**, 5043-5045 (2000)
- "Low-energy spin-wave excitations in the bilayer manganite $\text{La}_{1.2}\text{Sr}_{1.8}\text{Mn}_2\text{O}_7$," S. Rosenkranz, R. Osborn, J.F. Mitchell, L. Vasiliu-Doloc, J.W. Lynn and S.K. Sinha *J. Appl Phys* **87**, 5816-5818 (2000)
- "Time dependent effects and transport evidence for phase separation in $\text{La}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$," M. Roy, J.F. Mitchell and P. Schiffer *J. Appl Phys* **87**, 5831-5833 (2000)
- "Pressure-induced phase segregation in single-crystal $\text{La}_{2-2x}\text{Sr}_{1+2x}\text{Mn}_2\text{O}_7$ ($x=0.32$)," J.S. Zhou, J.B. Goodenough and J.F. Mitchell *Phys. Rev. B* **61**, R9217-R9220 (2000)
- "Neutron scattering studies of phase segregation in $\text{Pr}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$," P.G. Radaelli, R.M. Ibberson, S.W. Cheong and J.F. Mitchell *Physica B* **276**, 551-553 (2000)

James D. Jorgensen

Education

- B.S. Physics, Brigham Young University, Provo, Utah, May 1970.
- Ph.D. Physics, Brigham Young University, Provo, Utah, April 1975.

Professional History

- Senior Physicist, Materials Science Division, Argonne National Laboratory, Argonne, IL. 1989 - present.
- Group Leader of the Neutron and X-ray Scattering Group, Materials Science Division, Argonne National Laboratory, Argonne, IL. 1981-1982, 1986 - present.
- Physicist, Solid State Science, Materials Science and Technology, and Materials Science Divisions, Argonne National Laboratory, Argonne, IL. 1980 - 1989.
- Assistant Physicist, Solid State Science Division, Argonne National Laboratory, Argonne, IL. 1977 - 1980.
- Postdoctoral Research Assistant, Solid State Science Division, Argonne National Laboratory, Argonne, IL. 1974 - 1977.

Honors and Awards

- University of Chicago Award for Distinguished Performance at Argonne National Laboratory, 1983.
- Argonne National Laboratory Pacesetter Award, 1986. (with M. B. Brodsky, D. W. Capone II, D. G. Hinks, H.-B. Schuttler, and K. Zhang)
- U. S. Department of Energy 1987 Materials Sciences Research Competition Award for Outstanding Scientific Accomplishments in Solid State Physics, 1987. (with M. A. Beno, D. W. Capone II, D. G. Hinks, I. K. Schuller, L. Soderholm)
- Argonne National Laboratory Director's Award for FY 1987, 1988. (with M. B. Brodsky, D. W. Capone II, D. G. Hinks, H.-B. Schuttler, and K. Zhang)
- Bertram E. Warren Diffraction Physics Award, American Crystallographic Association, 1991.
- U. S. Department of Energy 1991 Materials Sciences Research Competition Award for Sustained Outstanding Research in Solid State Physics, 1991.
- Charles E. Barrett Award in Powder Diffraction, Denver X-ray Conference, 1997.
- Among 100 most highly cited physicists (ISIhighlycited.com) 2001.

Selected publications, 2000-2002

- "Crystal and Magnetic Structures of Ferromagnetic Superconducting $\text{RuSr}_2\text{GdCu}_2\text{O}_8$," O. Chmaissem, J. D. Jorgensen, H. Shaked, P. Dollar, J. L. Tallon, Phys. Rev. B 61, 6401-6407 (2000)
- "Study of the Mixed Conducting $\text{SrFeCo}_{0.5}\text{O}_y$ Ceramic Membrane Material by in situ Neutron Powder Diffraction," B. J. Mitchell, J. W. Richardson, C. D. Murphy, B. Ma, U. Balachandran, J. P. Hodges, J. D. Jorgensen, Materials Res. Bull. 35, 491-501 (2000)
- "Connection Between Oxygen-Ion Conductivity of Pyrochlore Fuel-Cell Materials and Structural Change with Composition and Temperature", B. J. Wuensch, K. W. Eberman, C. Heremans, E. M. Ku, P. Onnerud, E. M. E. Yeo, S. M. Haile, J. M. Stalich, and J. D. Jorgensen, Solid State Ionics 129, 111-133 (2000)

- "Evolution of Oxygen-Vacancy Ordered Crystal Structures in the Perovskite Series $\text{Sr}_n\text{Fe}_n\text{O}_{3n-1}$ ($n=2,4,8$ and \bullet) and the Relationship to Electronic and Magnetic Properties", J. P. Hodges, J. D. Jorgensen, X. Xiong, B. Dabrowski, S. M. Mini, C. W. Kimball, *J. Solid State Chem.* 151, 190-209 (2000)
- "Neutron Diffraction Study of the Structural Distortions in $\text{Sr}_3\text{Ru}_2\text{O}_7$," H. Shaked, J. D. Jorgensen, O. Chmaissem, S. Ikeda, Y. Maeno, *J. Solid State Chem.* 154, 361-367 (2000)
- "Temperature and Pressure Effects on the Crystal Structure of $\text{Sr}_3\text{Ru}_2\text{O}_7$: Evidence for Electronically-Driven Structural Responses," H. Shaked, J. D. Jorgensen, S. Short, O. Chmaissem, S. Ikeda, Y. Maeno, *Phys. Rev. B* 62, 8725-8730 (2000)
- "Structural Phase Transition and the Electronic and Magnetic Properties of $\text{Sr}_2\text{FeMoO}_6$ ", O. Chmaissem, R. Kruk, B. Dabrowski, D. E. Brown, X. Xiong, S. Kolesnik, J. D. Jorgensen, and C. W. Kimball, *Phys. Rev. B* 62, 14197-14206 (2000)
- "Magnetic Ordering in the Superconducting Weak Ferromagnets $\text{RuSr}_2\text{GdCu}_2\text{O}_8$ and $\text{RuSr}_2\text{EuCu}_2\text{O}_8$," J. D. Jorgensen, O. Chmaissem, H. Shaked, S. Short, P. Klamut, B. Dabrowski, J. L. Tallon, *Phys. Rev. B* 63, 054440 (2001)
- "Neutron Diffraction Study of the Defect Structure of Indium-Tin-Oxide," G. B. Gonzalez, J. B. Cohen, J.-H. Hwang, T. O. Mason, J. P. Hodges, J. D. Jorgensen, *J. Appl. Phys.* 89 (5), 2550-2555 (2001)
- "Pressure-Induced Cubic-to-Orthorhombic Phase Transformation in the Negative Thermal Expansion Material HfW_2O_8 ," J. D. Jorgensen, Z. Hu, S. Short, A. W. Sleight, J. S. O. Evans, *J. Appl. Phys.* 89 (6), 3184-3188 (2001)
- "Lattice Properties of MgB_2 versus Temperature and Pressure," J. D. Jorgensen, D. G. Hinks, S. Short, *Phys. Rev. B* 63, 224522 (2001)
- "The Complex Nature of Superconductivity in MgB_2 as Revealed by the Reduced Total Isotope Effect", D. G. Hinks, H. Claus, J. D. Jorgensen, *Nature* 411, 457-460 (2001)
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Peter L. Lee

Education

State University of New York at Buffalo Ph.D., Physical Chemistry, Feb., 1991
National Cheng-Kung University, Taiwan B.S., Chemistry, May, 1980

Professional History

Argonne National Laboratory
Physicist, April 1999-present
Assistant Physicist, September 1995-March 1999
Postdoctoral Research Associate, July 1992-August 1995
Brookhaven National Laboratory
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State University of New York at Buffalo
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Selected Publications

- "Time-Resolved Devitrification Studies of Zr-Pd and Zr-Pd-Cu Metallic Glasses " M. J. Kramer, M. F. Besser, N. Yang, E. Rozhkova, D. J. Sordelet, Y. Zhang and P.L. Lee, *J. Non-crystalline Solides*, in press.
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Education

Ohio State University	Ph.D. 1979: Physical Chemistry
Marquette University	B. S. 1973: Chemistry and Mathematics

Professional History

Argonne National Laboratory	
Group Leader Materials Science Division	9/2000 - Present
Group Leader APS Experimental Facilities Division	1/2003 - Present
Deputy Director Basic Energy Sciences Synchrotron Radiation Center	7/1999 - Present
Scientist Materials Science and Chemistry Divisions	5/1990 - 7/1999
Assistant Scientist Chemistry Division	9/1982 - 12/1987

Honors and Awards

- 1990 DOE-BES Materials Sciences Award winner for Outstanding Scientific Accomplishment in Materials Chemistry.
- 1987 DOE-BES Materials Science Award for Outstanding Scientific Accomplishments in Solid State Physics.
- 1985 DOE-BES Materials Science Award for Outstanding Scientific Achievement in Materials Science.

Recent Publications

"Magnetic Compton Scattering Using the Elliptical Multipole Wiggler at Sector 11-ID-B" P. A. Montano, U. Ruett, M. A. Beno, G. Jennings, C. W. Kimball, *J. Phys. Chem. Sol.* **61**, 353-356, (2000).

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Description of Facilities and Resources

The instrument will be built and operated at station 11-BM at the APS, one of the premier synchrotron facilities in the world. With 47 beamlines instrumented, the APS has the track record and all requisite facilities and expertise required to design, build, and operate the proposed diffractometer. Extensive on-site fabrication facilities (machine, optical, electronics shops) and expertise in construction, software design, etc. position the APS to provide the facilities and resources needed for this proposal. APS management has voiced a strong commitment to the project, and has already contributed to the development of a construction timeline and to budget planning. APS management has also committed to support the requisite support staff during construction and operation. Thus, the physical plant, on-site resources, and management commitment are in place for the successful completion of this instrument construction project.

Appendices

- I. Letter of support from APS Director, J. Murray Gibson
- II. External Advisory Committee Review
- III. Potential Users of a High Resolution Powder Diffractometer at the APS

Appendix I

Letter of Support from APS Director, J. Murray Gibson

ARGONNE NATIONAL LABORATORY

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Advanced Photon Source

Phone: (630) 252-7990

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January 23, 2003

Dr. John Mitchell
Argonne National Laboratory
Materials Science Division
Building 223
9700 S. Cass Avenue
Argonne, Illinois 60439

Dear John:

It is with great pleasure that I lend my strong support to your proposal to build a high-resolution powder diffraction beamline on a bending magnet at the APS. Your Letter of Intent to build the beamline has been reviewed and approved enthusiastically by the APS Scientific Advisory Committee (SAC). To quote from the SAC report: "There is a clear need in the materials science community for a dedicated facility of this type. The importance of powder diffraction in elucidating the structure and the subtle phase relations in complex oxides such as high-temperature superconductors and manganites are recent examples. The PIs are all well recognized for the contributions in these areas and are well qualified to oversee the development of a world-class facility. The SAC is pleased to note that this facility will be operated from inception as a "general user facility."

We at APS are committed to supporting the operation of your beamline if it is funded. We are also happy to play a strong collaborative role in the design and construction of the beamline.

Best wishes for success,



J. Murray Gibson
Associate Laboratory Director

JMG/ngc

OPERATED BY THE UNIVERSITY OF CHICAGO FOR THE UNITED STATES DEPARTMENT OF ENERGY

Appendix II

External Advisory Committee Review of the Proposal to Build a General-User High-Resolution Powder Diffractometer at the APS

14 November 2002
Argonne National Laboratory

The goal of this review meeting was to present the ideas for our proposal to a small group of highly qualified scientists and to receive their input prior to submitting the final proposal. We were very pleased that the attendees included some of the most prominent scientists worldwide in the area of high-resolution synchrotron powder diffraction. These scientists have already built instruments like we are proposing and they have operated successful general user programs. The attendees also included some prominent users of such instruments. The discussion included the science case, the details of the instrument design, the instrument operation for general users, and ideas for strengthening the proposal. The review meeting was organized to encourage candid discussion and many ideas were then incorporated into the final proposal.

Meeting attendees were:

Non-ANL:

- David Cox, BNL-retired: Built and operated a similar instrument at the NSLS.
- Andrew Fitch, ESRF, Grenoble, France: Built and operates the instrument at ESRF.
- Paolo Radaelli, ISIS Facility, Rutherford Appleton Lab, UK: PI on a proposal for a similar instrument at Diamond.
- Richard Harlow, Dupont-retired: Experienced user.
- James A. Kaduk, BP-Amoco, Naperville, IL: Experience user from industry.
- Angus P. Wilkinson, Georgia Tech: Experienced user.
- Patrick Woodward, Ohio State Univ: Experienced user.

ANL:

- James D. Jorgensen, MSD.
- John F. Mitchell, MSD.
- Robert B. VonDreele, On leave at IPNS, ANL from LANL.
- Peter L. Lee, APS.
- Mark A. Beno, MSD (BESSRC).
- Dennis Mills, APS.
- Mohan Ramanathan, APS.
- Roger Klaffky, APS.
- Dean Heffner, APS.
- Deming Shu, APS

Agenda

External Advisory Committee Review of the Proposal to Build a General-User High-Resolution Powder Diffractometer at the APS

**14 November 2002
Argonne National Laboratory
Conference Room A5000, Building 401**

9:00 AM	Dennis Mills	Welcome
9:10 AM	James D. Jorgensen	Introduction to the Proposal and its PIs
9:20 AM	James D. Jorgensen and Robert Von Dreele	Brief Overview of the Science Case
9:35 AM	Peter L. Lee, Mark A. Beno, Mohan Ramanathan	Instrument Performance Goals, Detailed Instrument Design, Construction Plan, Timeline, Budget
10:15 AM	BREAK	
10:45 AM	Peter L. Lee, Mark A. Beno, Mohan Ramanathan	Continuation of discussion above
11:30 AM	Robert Von Dreele	Operation of the Instrument
11:45 AM	Roger Klaffky	General User Program at the APS
12:00	LUNCH	Argonne Guest House (no host)
1:30 PM	David E. Cox Andrew Fitch Paolo Radaelli Richard Harlow James A. Kaduk Angus P. Wilkinson Patrick Woodward	Short Presentations or Comments about Science, Instrument Design or Operation (15 minutes each)
~2:30 PM	BREAK	<i>Will probably come in the middle of the 7 presentations above.</i>
3:30 PM	John F. Mitchell	Summary discussion, including ideas for generating support for the proposal.
4:00 PM	ADJOURN	

Overview of the Meeting

The meeting began with an introduction from Dennis Mills, representing the APS. Mills expressed a strong commitment on the part of APS to help build the instrument and later to operate it on behalf of the general user community.

James Jorgensen and Robert Von Dreele gave a brief overview of the science case for the instrument. Their presentations were intended to provide the foundation for the desired instrument performance. The instrument design is driven by the science it is intended to do. There was vigorous discussion about these topics. Review committee members strongly encouraged less emphasis on routine structural studies and more emphasis on cutting edge science and science for which an instrument at the APS would have a clear advantage. Examples of this include:

- High energies for penetration of sample environments and weak signal experiments and as a means for minimizing experimental errors.
- Anomalous scattering.
- Diffraction at high pressure, but not in the extreme pressure range, which is the focus of HP-CAT or GEO-CAT.
- Electron density distributions (as being explored at Spring-8), which requires data of very high accuracy.
- Structure solution.
- Nanoscience, such as the study of microdomains, domain boundaries, microstrain.
- A stronger connection with strong ANL programs, or proposed future programs such as the catalysis center.

The committee encouraged that the science, and user community, be as wide ranging as possible without compromising the unifying concepts of the instrument design that are best exploited at the APS -- in particular, the primary design goal of high resolution with well-defined peak shapes should not be compromised.

Peter Lee and Mark Beno presented the proposed instrument design in considerable detail. This generated a lot of discussion about various aspects of the design, which ultimately led to some important changes in the final proposal. The meeting attendees were unanimous on the following issues:

- The energy should be pushed to a maximum of 40 keV. This exploits the strength of the APS and is important for many experiments.
- High resolution with a well-defined peak shape (that is handled well by refinement codes) should not be compromised to achieve higher intensity. Andy Fitch (ESRF) said that, to achieve the highest possible resolution and best peak shape on his instrument at the ESRF, he removed the focusing optics four years ago. Others expressed similar concerns about the use of focusing optics. These comments led us to modify our instrument design so that the focusing optics could be removed if desired.
- Careful attention should be given to various aspects of the instrument design that are important for anomalous scattering experiments. It is critically important to eliminate

harmonics. Anomalous scattering at low energy places special requirements on the instrument, e.g., the use of a helium-filled beam path. Some provision should be made for experimentally find the edges.

- Stability issues become very important for high-resolution instrument. For example, some users had experienced problems with peak positions shifting as a function of time. This must be avoided by careful attention to thermal expansion effects at the mirror and monochromator.

Additionally, there was considerable discussion of the analyzer/detector configuration. It was suggested that the use of channel-cut analyzers might be explored. It was also suggested that 12 detectors might be too ambitious and make it difficult to achieve the desired energy range. These issues can't be addressed without careful analysis and no consensus was formed at the meeting. Lee and Beno later explored these issues in detail to arrive at the final design. It was also suggested that provision for the installation of a curve image plate detector in addition to the analyzer/detector system should be explored, as long as this did not compromise the high-resolution operation of the instrument.

Additional discussion focused on ancillary equipment that should be provided as part of the standard capability made available to users. It was felt that furnaces should go to higher temperatures than specified in the preproposal and should be able to work in various atmospheres with the provision for safely exhausting gases. Low-temperature (e.g., 4 K) to about 800 K should be provided in one device. There should be provision for spinning capillaries at low temperature. The ability for experiments at moderate pressure (diamond-anvil cells) was discussed, but it was the opinion of the group that there should not be an attempt to compete with existing APS CATs focused on high-pressure research. The proposal to have an automatic sample changer for short, fast-access runs was enthusiastically received.

Mohan Ramanathan presented details of how the instrument construction and commissioning would be accomplished, including a detailed timeline and budget profile required to achieve the proposed timeline. His work on this was very impressive and meeting attendees felt that the proposed timeline and budget were achievable.

Robert Von Dreele discussed the plan for operation of the instrument in the user mode. There was a consensus that the staff levels discussed in the preproposal were woefully inadequate. Those who have operated such instruments for general users felt that the required staff for an aggressive user program was two staff scientists, two postdocs, and one technician.

Roger Klaffky discussed the newly developed APS policies for general users. The user policy for general user instruments at the APS is found on the APS web site: www.aps.anl.gov/aps/frame_user_info.html. Users may apply for beam time through three types of proposals:

- An "individual proposal" for a single experiment.
- A "program proposal" for an experimental program that may require a series of visits to the APS over a two-year period (less than 10% annually of user time on a given beam line).
- A "rapid-access proposal" requesting unallocated general user time during the current run.

Each of these was discussed. Some concerns were expressed that the proposal process could be unnecessarily cumbersome for both the users and the APS in the case of a large number of very short, fast-access experiments. Meeting attendees felt that it was important to have the instrument available to be used in this manner as a uniquely powerful analytical tool by scientists across the US. For such experiments, they should be able to submit samples for single data sets and have the data sent back electronically without coming to the APS. APS is open to further discussion on this topic in order to meet this need in the best way.

During the afternoon, each of the non-ANL attendees of the meeting was given an opportunity to present whatever ideas they felt were most important. Their comments have already been woven into the narrative above. All attendees expressed strong support for building a dedicated general-user powder diffractometer at the APS. Their involvement in this meeting at an early stage in the planning and design was invaluable in focusing the proposal in a way that will best meet the needs of the targeted users.

Appendix III

Potential Users of a High Resolution Powder Diffractometer at the APS

These potential users have been contacted about the instrument proposal and have agreed that their names can be used. This list has been compiled at the time of the proposal deadline and is dynamic. It is known to be incomplete at this time.

Name and Affiliation	Brief Description of Science Envisioned for the APS Powder Diffractometer
Ross Angel Virginia Tech	High-resolution powder diffraction to determine structural details of minerals and related synthetic inorganics that are not possible with laboratory instrumentation.
Davor Balzar Univ. of Denver	Studies of strains and crystalline defects in materials, utilizing the high resolution and high intensity of the instrument.
Simon Billinge Michigan State Univ.	Studies of phase transitions, phase separated samples, charge ordering, etc. in inorganic materials.
David Bish Los Alamos National Laboratory	Structural studies of hydrous materials, including time/temperature-dependent studies of hydration.
Peter C. Burns Univ. of Notre Dame	Studies of actinide minerals and compounds, including phase transitions in these materials.
Omar Chmaissem Northern Illinois Univ.	High resolution structural studies with emphasis on exact structural symmetries and phase separation in oxide materials.
Christopher L. Cahill George Washington Univ.	Resonant diffraction studies of Fe, Mn, Zn-containing ferrite nanoparticles.
Paul C. Canfield Ames Lab, Iowa State Univ.	Structural and magnetic properties of new or exotic materials, including superconductors, magnetic superconductors, and normal metals with highly anisotropic local moments and metamagnetic phase transitions.
Bryan Chakoumakos Oak Ridge National Laboratory	Studies of subtle phase transitions such as in the perovskite type oxides and ab initio structure solution of a wide range of high technology materials.
Julia Chan Louisiana State Univ.	Structure-dependent properties such as superconductivity and magnetism in rare earth intermetallics. Crystal growth of novel bulk materials. Nanostructured magnetic multilayer thin films, which are important for GMR applications.
Dhanesh Chandra Univ. of Nevada	Determination of deuterium positions in the lattice of metal hydrides as a function of loading.

<p>Abraham Clearfield Texas A&M University Bogdan Dabrowski Northern Illinois Univ.</p>	<p>Structures of organic thermal energy storage materials. Solid state chemistry. Currently interested in site selectivity in rad-waste materials. Relationship between structure and properties in complex materials, including superconductors, magnetoresistive materials, half-metallic magnets, fast ion conductors.</p>
<p>Evgeny V. Dikarev SUNY at Albany</p>	<p>Structural studies to guide the design of host supramolecular networks from the vapor phase for guest selection applications.</p>
<p>Robert Downs Univ. of Arizona</p>	<p>Real-time analysis of the structural state of oxide minerals undergoing phase transitions at simultaneous temperature and pressure.</p>
<p>Yan Gao GE Global Research Center, NY</p>	<p>Structure solution and refinement for inorganic materials and engineering ceramics.</p>
<p>John Greedan McMaster Univ., Canada</p>	<p>Transition metal oxides exhibiting strong electronic correlations where the high resolution would be used to investigate systems that exhibit complex twinning and/or psuedo symmetry and/or phase separation or to solve structures when single crystals cannot be grown.</p>
<p>Lee A. Groat Univ. of British Columbia</p>	<p>Crystal chemistry of minerals. with an emphasis on anomalous scattering and studies as a function of chemical substitution, temperature, and pressure.</p>
<p>P. Shiv Halasyamani Univ. of Houston</p>	<p>Structural characterization of new mixed-metal oxide materials for nonlinear optical applications, i.e. second-harmonic generation (SHG). As SHG can only occur in crystallographically noncentrosymmetric materials, the crystal structure of the material is paramount.</p>
<p>Richard Harlow Consultant, Harlow, Inc.</p>	<p>Structure solution and refinement on problems from a wide range of customers.</p>
<p>Peter J. Heaney Penn State Univ.</p>	<p>Ceramics with low coefficients of thermal expansion.</p>
<p>John B. Higgins Air Products and Chemicals, Inc.</p>	<p>Many projects, including: ion-transport membranes, low-k dielectrics, and hydrogen storage materials.</p>
<p>Camden Hubbard NIST</p>	<p>Studies of phase stability, temperature dependent phase transformations, crystal structure determination at various temperatures, real time studies (kinetics), and residual stresses near the surface. Materials include fuel cell and gas separation membranes (oxygen ion conductors, protonic conductors), super magnets, bulk</p>

<p>James A. Ibers Northwestern Univ.</p>	<p>amorphous alloys, steels, aluminum, magnesium, gas clatherates, intermetallics, silicides, refractories, and engine exhaust gas catalysts. Study of various solid-state materials, especially the oxides, sulfides, selenides, and tellurides of a variety of metals, including rare-earths, Th, and U with an emphasis on structure solution, followed by refinement, for new materials where single crystals cannot be grown.</p>
<p>James A. Kaduk BP-Amoco, Naperville, IL</p>	<p>Structure solution using powder data where high resolution is required. Studies of metal-oxide catalysts, including the use of resonance scattering and controlled environments.</p>
<p>Susan Kauzlarich Univ. of California at Davis</p>	<p>Structure solution and refinement for a wide range on inorganic materials.</p>
<p>Piotr W. Klamut Northern Illinois Univ.</p>	<p>Precise structural characterization of complex perovskite and perovskite-like oxides.</p>
<p>Matthew J. Kramer Ames Lab/Iowa State Univ.</p>	<p>Resolving substitutions of either transition metals or different rare earths in complex intermetallic compounds. Investigating subtle changes in either states of stress or composition during phase transformations. Particular interest in high-energy anomalous scattering.</p>
<p>Trudy Kriven Univ. of Illinois at Urbana-Champaign</p>	<p>Advanced ceramics, including in situ high-temperature studies.</p>
<p>Phil Lightfoot Univ. of St. Andrews, UK</p>	<p>Structure solution and structural changes vs. temperature nanoporous and framework solids.</p>
<p>Chun Loong Argonne National Laboratory</p>	<p>Crystal structures of ceramic materials (complex oxides, borates, carbides and oxynitrides), defect structure in crystalline nano-micro-mesostructured ceramic composites, and crystal and microstructures in ceramic films (ferroelectrics).</p>
<p>Thomas Mason Northwestern Univ.</p>	<p>Structural and point defect characterization studies (by Rietveld analysis in combination with neutron diffraction data) of technologically important and emerging electroceramic materials, including ferroelectric oxides and transparent conducting oxides, including use of in situ (high temperature/controlled atmosphere) capabilities.</p>
<p>Scott Misture NYS College of Ceramics at Alfred Univ.</p>	<p>Structure solution and refinement for electronic ceramics, including ionic conductors, mixed conductors, and ferroelectrics. Use of the brilliance to improve time resolution in high temperature studies of phase transformations in electronic ceramics as well as for structure solution of organometallic polymorphs that evolve</p>

<p>Simon C. Moss Univ. of Houston</p>	<p>with temperature. Anomalous scattering studies of the structure of various molecular species in the cages of selected zeolites. Studies of nanocrystalline biopolymers, including the various forms of natural and synthetic melanins, especially material in which there is a metal ion attached which can be probed via anomalous scattering.</p>
<p>Alexandra Novrotsky Univ. of California at Davis</p>	<p>Oxide materials, including defect materials such as perovskites and pyrochlores, zeolites and other low temperature phases, and nanomaterials.</p>
<p>John Parise SUNY at Stony Brook</p>	<p>Microporous materials which utilize selective ion exchange in applications such as rad-waste and toxic metal clean-up. Use of high resolution to solve and refine crystal structure and pdf analysis to probe local ordering phenomena.</p>
<p>Jeffrey Post Smithsonian Institution</p>	<p>Structures and behaviors of environmentally important minerals, e.g. clays, Mn and Fe oxides, many of which, occur only as fine-grained masses.</p>
<p>Surendra Saxena Florida International University Arthur Sleight Oregon State Univ.</p>	<p>High-temperature and high-pressure materials science. Structural studies of negative-thermal-expansion materials and other inorganic compounds. Emphasis on using high resolution to determine correct symmetry.</p>
<p>Hugo Steinfink Univ. of Texas at Austin</p>	<p>Studies of Bi-Pb-transition metal phosphates, vanadates and arsenates.</p>
<p>Angus P. Wilkinson Georgia Tech</p>	<p>Resonant scattering studies of materials with potential for thermoelectric energy conversion.</p>
<p>Patrick Woodward Ohio State Univ.</p>	<p>Studies of electronic and magnetic phase transitions in transition metal oxide perovskites. Ab-initio structure solution from powder diffraction data, including extended solids, such as defect fluorite materials, and molecular solids Structural characterization (using Rietveld refinement) of various electronic oxide materials, both under ambient and non-ambient conditions and kinetics of phase formation at high temperature.</p>
<p>Winnie Wong-Ng NIST</p>	<p>Solving structure from powder data of the new materials for lithium batteries, many of which cannot be obtained in single crystal form. Also, use of anomalous scattering near the absorption edge to distinguish similar elements (e.g. transition metals) when other methods fails or don't work.</p>

<p>Chong Zheng Northern Illinois Univ.</p>	<p>Structural characterizat on of superconducting and magnetic materials, including the use of anomalous scattering to enhance sensitivity for specific elements.</p>
<p>H-C. zur Loye Univ. of South Carolina</p>	<p>Rietveld refinement of structures of oxide compounds, with particular emphasis on understanding subtle distortions and incommensurate structures.</p>
<p>Josef W. Zwanziger Indiana Univ.</p>	<p>Studies of crystal growth in glass ceramics. In these materials, crystal phases grow within glass matrices, and following this process in detail requires rapid data collection of the powder diffraction pattern.</p>