NSLS-II Beamline Development Proposal

Type I [x] Type II [ ]

Powder Diffraction Beamline for *in situ* studies of structural and chemical transformations for developing advanced energy materials and industrial applications. **(IXD)**

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A. Science Case

Timely access to accurate structural information on new materials plays a central role in all aspects of materials science. Synchrotron x-ray diffraction (SXRD), especially x-ray powder diffraction (XRPD), is an indispensible tool in obtaining this information because many technologically critical materials exist only in polycrystalline form. However, knowing the static structure of a material often is insufficient to predict its physical properties and functionalities in practical applications. Therefore, as well as offering static structural information, SXRD's ability to generate detailed knowledge of the dynamic behavior of materials in *operando* is increasingly important in resolving complex problems in advanced energy-related materials studies, and their industrial applications. In many cases, this additional information is essential to the optimization of processing parameters in the synthesis of complex systems. Based on the most important scientific needs of our broad user-community, we propose to build a three-pole-wiggler (TPW) beamline at the NSLS-II to investigate the dynamic behaviors of materials using *in situ* x-ray diffraction techniques. The proposed beamline offers users the ability to perform *in situ* XRD experiments under a wide variety of conditions, including a wide temperature- (10 to 2000 K), chemical, charging/discharging, and pressure range. The high brightness of this TPW beamline compared to the NSLS's bending-magnet beamlines and its continuously tunable x-ray energy up to 25 keV make it ideal for element-specific and surface-sensitive XRD methods of materials characterization, in addition to regular XPD measurements. The proposed beamline will critically complement the existing high-energy powder diffraction beamline (XPD), benefitting users in the following areas:

1, Powder crystallography, including solving and refining crystal structures, quantitative analysis of phase fraction and size/strain analysis.
Beamline requirements: High beam-intensity, high angular- and energy-resolution, fast detectors (2D or linear detector), software (e.g., Jade/Topas/GSAS/ICDD database). Typical x-ray energy: 25 keV, beam size: 0.5x1.0 mm. Software will be available at the beamline to provide real time analysis of the data and to archive the data into a database for the user's remote access. A critical part of the program at this beamline will be the training of users in data analysis methods. The goal will be to have users (conducting routine experiments) leave the beamline with publication level analysis already completed. The beamline will be staffed to accomplish this goal.
Applications: Characterization of novel materials developed for batteries, fuel cells, catalysts, high-temperature superconductors, and thermoelectric- and ferroelectric-devices.

2, *In situ* powder-diffraction measurements to study, with sub-minute time resolution, the structural, morphological and chemical transformations in materials following changes in temperature (10 K to 2000K), pressure, reaction gas flow and charging states
Beamline requirements: The items listed in (1), plus furnaces for capillary and plate samples, cryostats, gas flow reaction cells with heater, gas handling system, residual gas analyzers, and battery charging devices. Controls for temperature and gas flow should be incorporated into the data acquisition software. The combination of the Si strip detectors and the medium energy range provided by the TPW can readily deliver full-pattern XPD measurements with a time resolution of sub-minute scale (or sub-second scale using the 125° Si trip detector being developed), and with good angular resolution for structural refinement.
Applications: Acquiring material properties, such as the phase diagram and chemical reactivity, via dynamical reactions in thermal, chemical, electrochemical, and physicochemical processes.

3, Anomalous (or resonant) x-ray diffraction studies by tuning the x-ray wavelength near the absorption edges in order to distinguish neighboring elements and oxidation states
Beamline requirements: Tunable x-ray energy, from 5 to 25 keV, to cover a wide range of elements at the K or L edges, but with still enough Q range for structural refinement, and an energy resolution up to the intrinsic resolution of the Si (111) monochromator.

Applications: Resolving cation ordering in complex lithium-ion battery cathode materials, and the structures of solid solutions and alloys.

4, *In situ* high-pressure x-ray diffraction
Beamline requirements: Small beam size (100 μm x 100 μm), realized by inserting collimating slits or in hutch focusing devices, and diamond anvil cells.
Applications: Pressure-induced phase transitions, and synthesis of new materials with novel properties

5, Residual, thermal and growth-stress measurements
Beamline requirements: The x-ray energy in many cases has to be tuned to below the absorption edges of transition metals widely used in industrial alloys. Small beam size is often used for industrial components with curved surfaces. The commonly used $sin^2\psi$ method requires a six-circle diffractometer and crystal analyzers.
Application: Residual stresses affect important design properties in materials such as fatigue life, fracture strength, onset of yield, and microcracking. The residual-stress measurement using synchrotron x-ray has wide applications in industry (see details in section A2).

6, Diffraction at grazing incidence and reflectivity
Beamline requirements: Angular (or Q-space) resolution can be increased by defocusing the mirror and/or narrowing the incidence slits to reduce horizontal divergence. An x-ray energy of about 8 keV (depending on the samples' absorption edge) is desirable for easy depth of penetration control.
Applications: Surface reconstruction (single crystal), surface coating-phase components and phase transition, and the interfacial roughness of thin films.

The categories outlined above are not mutually exclusive, e.g., the *in situ* XRD measurement of the thermal- and growth- stresses associated with the oxidation of the metallic interconnects of solid oxide fuel cells (SOFCs). We list below the science drivers of the research projects for the proposed IXD beamline:

A1, Lithium-Ion Battery Studies

One of the major science drivers for the proposed IXD beam line is to continue and enhance the current cutting-edge Li-ion battery studies at the NSLS. Electricity storage is one of the Scitech Priority Research Directions identified at the 2010 DOE /BESAC meeting. The development of durable, affordable batteries to use in advanced vehicles also is a primary objective of the DOE's Vehicle Technologies (VT) program and the office of Energy Efficiency and Renewable Energy (EERE).[1] Compared to other types of rechargeable batteries, lithium-ion batteries possess higher power, higher energy, and lighter weight. These advantages make them the most promising candidate for using in hybrid electric vehicles (HEVs) and plug-in hybrid electric vehicles (PHEVs). Research on the crystal structural and morphological changes in the electrodes during the charge/discharge cycles of these batteries greatly impacts the search for advanced cathode materials for the future batteries that must meet the four major industrial requirements: Safety, cost, extended lifetime, and performance (power and energy) over a wide range of temperature (-30 to 52°C).

The XRPD, one of the most important tools for *ex situ* and *in situ* structural characterization of advanced materials has been widely used in battery studies. The powder-diffraction beam lines at NSLS (X7B, X14A, X16C and X18A) currently host several groups studying batteries. Prof. Yet-Ming Chiang's group (MIT), exploring with *in situ* XRPD the effects of overpotential on phase stability and transformation mechanisms, revealed a remarkable dependence of the phase transition pathway on overpotential in the model olivine Li_1-

[1] FY 2009 Annual Progress Report for Energy Storage R&D, DOE Office of Energy Efficiency and Renewable Energy.

$_xFePO_4$. They found that at intermediate overpotentials a crystalline-to-amorphous phase transition dominates and the amorphization is emphasized further at nanoscale particle size.[2] The battery research team at BNL Chemistry, led by Dr. Xiao-Qing Yang, developed techniques using the combination of a high-intensity synchrotron X-ray beam and fast detectors (image plate or position-sensitive detectors) to undertake time-resolved X-ray diffraction (TR-XRD) during the thermal decomposition of charged cathode materials. They published their studies on the structural changes of layered, nickel-based cathode materials during thermal decompositions in several papers. The appendix A1 details the researches by Dr. Yang's team with a recent publication list. Other groups studying batteries at the NSLS include groups led by Dr. Jason Garetz (BNL), Prof. Clare P. Grey (SBU), Prof. Gerbrand Ceder (MIT), and Prof. Ann Marie Sastry (UMich). The battery materials researchers are forming a fast growing synchrotron x-ray user community. In the 2009 Annual Progress Report for Energy Storage R&D by the DOE's office of EERE, 22 projects conducted by 16 research groups listed were using XRPD as their structure characterization tool. Battery study groups often are funded by the EERE, and have close relationships with the electric energy storage and automobile industries. For example, Yet-Ming Chiang is the cofounder of A123 Systems, and General Motors sponsors the UMich group's battery program. Therefore, the research that these groups conduct directly impacts American industry and the economy.

One technical challenge in current battery studies is structure refinement in the quantitative analysis using the *in situ* XRPD data. The XRPD data from battery cells contain large amorphous backgrounds and some minor diffraction peaks from the electrolyte and other components in the cell, and usually possess multiple phases in the active cathode material. Proper Q-space resolution is required to resolve the overlapping Bragg peaks in the XRPD patterns. This also applies to other practical sample systems of industrial importance. On the other hand, *in situ* measurements, especially in TR-XRD, must acquire multiple data sets in sub-minutes or a shorter time scale; the best way to do so is to use position-sensitive linear or area detectors. However, the pixel size of the detector limits the detector's Q-space resolution, especially at higher x-ray energies. Experiments at the NSLS's XRPD beam lines demonstrated that the proper Q-spacing resolution for the *in situ* battery studies can be achieved using medium-energy x-rays (16 to 20 keV) with position-sensitive detectors. The quantitative analysis of the *in situ* XRPD data will help to solve the puzzles in the Li-ion battery chemistry, such as the delay of the structure changes to the charging state and the mismatched ratio of the two cathode components at different stage of charging.

A2. *In Situ* Surface Stress Analyses of High Temperature Alloys

High-temperature alloys have many important industrial applications in assuring a better performance of fossil-energy systems, and in significantly improving the equipments' thermal efficiency, productivity, and lowering their operating costs. Such alloy systems, as applied in the industrial processes, such as gas-turbine operations, usually have multicomponent structures that undergo complicated physical and chemical processes in their surfaces. *In situ* measurements of the thermal and growth stresses generated during oxidation at high temperature were conducted at X14A; a new scheme called the "multiple *hkl*" method to monitor phase growth and the evolution of stress simultaneously can be developed using the proposed IXD beamline. This method requires an x-ray energy ranging from 6 to7 keV to avoid iron-edge absorption and to maximize the inclination angle, along with small beam to improve angular resolution. The medium x-ray energy range and the high brightness of the 3PW beamline are very well suited for this element-selective and surface-sensitive method. (Detailed in Appendix A2)

A3. A Comprehensive Solution for Solid-Oxide Fuel-Cell Research

[2] "Phase Changes during Electrochemical Cycling of Olivines" by Y. Chiang, Y. Kao, M. Tang, N. Meethong, J. Bai and W. Carter, abstract #323, IMLB 2010 - The 15th International Meeting on Lithium Batteries (IMLB 2010)

Solid Oxide Fuel Cells (SOFCs) are the most efficient, cleanest energy-converters operated at high temperatures. They have the potential to replace the internal-combustion engine in vehicles, and to provide power in stationary- and portable-power applications. The Office of Energy Efficiency and Renewable Energy (EERE) invests heavily in SOFCs for their benefits in strengthening the economy, protecting the environment, and reducing dependence on foreign oil. To overcome the technological and economic obstacles to commercializing fuel cells and related technologies, research efforts mainly are focused in two directions: [3]

1. Decrease operating temperature to 500 - 800 °C to lower costs and enhance durability.
2. Increase their fuel flexibility so hydrocarbons or gasified coal are suitable as fuels.

To reach these goals, intensive materials development is crucial to finding novel materials with desirable properties for low-to-medium temperature operations. This proposed powder X-ray diffraction beamline will dedicate a major effort to advancing the technology of solid oxide fuel cells using multiple techniques. Based on the needs of our fuel-cell users, the capability for, and the availability of *in-situ* in operando characterization are highly desirable. These include *in-situ* XRPD structure analysis using powder refinement, *in-situ* surface/interface analysis using grazing incidence XRD at reaction conditions, and *in-situ* simultaneous stress and structure analysis to monitor phase growth and stress evolution at the same time. These users also are interested in the chemical environment that is directly important to their needs, for example, using H_2S in the studies of sulfur poisoning. This proposed beamline, with its variable energy, high intensity, high energy and angular resolution, fast detector, and dedicated instrumentation, will satisfy the SOFC researchers' needs.

Currently, the XRPD community at NSLS is engaged in cutting-edge research on the anode, electrolyte, and interconnect materials of SOFCs. Recently, Professor Meilin Liu's group from the Georgia Institute of Technology discovered a novel anode material, $BaZr_{0.1}Ce_{0.7}Y_{0.2-x}Yb_xO_{3-d}$[4] , that has superb anti-coking, anti-sulfur poisoning capabilities at medium temperatures. With the active participation of X14A beamline staff, a suite of SXRD techniques were employed to produce comprehensive information on minority-phase identification and quantification, multi-layering of thin films at the anode/electrolyte interface, and the chemical stability of anode materials in the presence of water, carbon dioxide, and methane under operational conditions. The results are critical for elucidating the catalytically active sites for fuel oxidation. Professor Scott Misture from Alfred University examined the oxygen-vacancy ordering structure of anode material $La_{1-x}Sr_xCoO_{3-\delta}$.[5] Professor Leopoldo Suescun from Universidad de la República Uruguay was interested in using in-situ XRD structural characterization the lattice behavior of nanometer-sized scandia-doped ZrO_2 electrolyte. Researchers at Oak Ridge National Laboratory employed the *in-situ* XRD technique to investigate surface oxidation and stress evolution in the chromia phase of alloy 441, a stainless steel (UNS S 44100) that is being developed as an SOFC interconnect material. There are continuing demands for beam time for SOFC researches.

A4. Real-Time Catalysis: *In-Situ* Structural Studies for Producing and Storing Hydrogen Fuel

Catalysis is the most critical field for sustainable energy production to improve our energy security and reduce the environmental impacts.[6] A fundamental understanding of the nature of catalysts under operating conditions, especially of active species or sites, is pivotal to designing and controlling their activity. Since real catalysts and catalytic processes are extremely complex, the availability of techniques for the real time characterization of catalytic systems as they evolve in time with a changing chemical environment is a high priority. The *in-situ*

[3] N. H. Menzler, F. Tietz, S. Uhlenbruck, H. P. Buchkremer, and D. Stover, J Mater Sci **45,** 3109 (2010).
[4] L. Yang, S. Z. Wang, K. Blinn, M. F. Liu, Z. Liu, Z. Cheng, and M. L. Liu, Science **326,** 126 (2009).
[5] J. Ovenstone, J. S. White, and S. T. Misture, J. Power Sources **181,** 56 (2008).
[6] Ctalysis for Sustainable Energy Production; edited by P. Barbaro and C. Bianchini (Wiley-VCH, 2009)

XRD capability of our proposed IXD beamline affords the effective solution to such research needs, and will ensure the continuity of service to existing user groups. At the NSLS, *in-situ* XRD techniques were used effectively in many projects directly related to EERE goals. For example, investigations into the production and storage of the ultimate cleanest fuel, hydrogen, proved fruitful and currently are progressing strongly

Two major efforts in hydrogen production are spearheaded by Brookhaven National Laboratory chemists Dr. Jose A. Rodriguez and Dr. Etsuko Fujita, one in hydrogen generation from the water-gas shift (WGS: $H_2O + CO \rightarrow H_2 + CO_2$) reaction, and another in solar photocatalytic water-splitting (H_2O + visible light $\rightarrow H_2 + O_2$). Both teams have extensive international collaborations with scientists from Spain, Japan, Venezuela, Brazil, China, and are funded by the DOE's Chemical Sciences Division. In WGS studies, they investigated, via in-situ XRD, mixed metal oxides catalysts, including $CuMoO_4$, $NiMnO_4$, $Ce_{1-x}Cu_xO_{2-x}$, and $CuFe_2O_4$ under reaction conditions, identified the active species, and correlated the oxygen vacancies with reactivity.[7] In researches on the solar water-splitting, the BNL team probed the solid-state synthesis mechanism of band-gap-narrowed photocatalysts, including nitrogen-doped TiO_2 and GaN/ZnO solid solutions, both among the most promising catalysts. The team's discovery of Ga_2ZnO_4 as an intermediate species in the synthesis of GaN/ZnO led to the rational design of a high-pressure synthesis path to produce the very useful high-Zn-content solid solution with its much improved absorption of visible light.[8] The IXD beamline will continue to support these researchers in pursuing technological breakthroughs.

A major focus of the National Hydrogen Storage Project is the chemistry of hydrogen storage materials. The most sought-after targets for materials performance are high gravimetric and volumetric density, reversibility of hydrogen uptake/release under near- ambient conditions, and fast kinetics. *In-situ* XRD studies of hydrogen storage materials at the NSLS have attracted both industrial and academic users. A GE Global Research team has been employing synchrotron techniques in their hydrogen storage R&D work. To follow the reaction pathways and phase transitions during hydrogen absorption and dehydrogenation, this team developed a reaction cell that tolerates charging of hydrogen at ~ 2000 psi while heating to 500 °C for their investigation of $2LiNH_2 + MgH_2$.[9] This cell design will be very useful for other hydrogen storage users of the future IXD beamline. Professor Turben R Jensen's team from University of Aarhus, Denmark, worked on $LiBH_4$, a material with a very high hydrogen content of 18.5 wt.%, but non-reversible in hydrogen release. This team's focus was to boost the performance of $LiBH_4$ using different additives, such as SiO_2, $TiCl_3$, LiCl, and Au, as potential catalysts to lower the hydrogen-releasing temperature, and to improve the reversibility of the reactions[10].

B. Beamline Concept & Feasibility

B1. Scientific Scope

The IXD is a powder diffraction beam line with tunable x-ray energies up to 25 keV geared towards *in situ* studies of structural and chemical transformations for developing advanced energy materials and their

[7] J. A. Rodriguez, J. C. Hanson, W. Wen, X. Q. Wang, J. L. Brito, A. Martinez-Arias, and M. Fernandez-Garcia, Catal. Today **145,** 188 (2009).
[8] H. Y. Chen, L. P. Wang, J. M. Bai, J. C. Hanson, J. B. Warren, J. T. Muckerman, E. Fujita, and J. A. Rodriguez, J. Phys. Chem. C **114,** 1809 (2010).
[9] J. Rijssenbeek, Y. Gao, J. Hanson, Q. Huang, C. Jones, and B. Toby, J. Alloys Compd. **454,** 233 (2008).
[10] Mosegaard, L., B. Moller, J.-E. Jorgensen, Y. Filinchuk, Y. Cerenius, J. C. Hanson, E. Dimasi, F. Besenbacher and T. R. Jensen, Reactivity of $LiBH_4$: In Situ Synchrotron Radiation Powder X-ray Diffraction Study, Journal of Physical Chemistry C,**112**: p.1299-1303 (2008)

industrial applications, It also will provide users with element selective and surface sensitive x-ray methods, such as anomalous diffraction, surface-stress measurements, and grazing-incident diffraction.

B2. Source

Three-Pole Wiggler

B3. X-Ray Optics

To deliver a continuously tunable high- intensity x-ray beam with high-energy resolution (up to the intrinsic resolution of the Si (111) monochromator) and small beam size, the IXD beamline must have highly efficient and flexible x-ray monochromating and focusing optics.

B3.1 Collimating mirror.

The collimating mirror is flat and bent along its length to focus the beam at infinity. It minimizes the energy spread of the monochromator, and reduces contamination from the harmonics. For a TPW source, E_c=6.8keV, and for the 3 GeV storage ring of the NSLS-II, γ=5871. Thus, the vertical divergence of the beam is 102 μrad at 6 keV, and 55 μrad at 25 keV. The Darwin width of Si(111) is 45- and 10-μrad at these two energies. Therefore, with the collimating mirror, we can achieve an energy resolution equivalent to the intrinsic limit of the Si(111) crystal, while using most of the flux from the TPW source in the vertical direction. The Pt-coated Si mirror should be water-cooled, and with an uncoated half of the Si surface left for lower x-ray energies.* The mirror should be placed as close as possible to the source to reduce the incident beam's footprint on the mirror.

* Another solution to change the cut-off energy of the Pt-coated mirror is to vary its tilt angle from 4.6 to 3.2 mrad, and change the mirror bending accordingly to keep the correct vertical focusing.

B3.2 Sagittal Focusing Fixed-exit Double Crystal Si(111) Monochromator

The monochromator should be placed after the collimating mirror with a water- cooled flat Si(111) crystal, and a sagittally bent Si(111) crystal to focus the beam horizontally. The bender assembly should have highly repeatable motors and with μm precision to allow dynamical focusing. The monochromator should be able to accept at least a 2-mrad horizontal fan of the incident beam. Position sensors, such as LVDTs, are desirable for each bender motor to monitor the real position value of each bending point. The bent crystal should have ribs with a period less than 1 mm to reduce the anticlastic effect.

The major advantage of using a sagittal focusing monochromator instead of a toroidal mirror lies in its flexibility to focus the beam at different positions. For example, we can choose to focus the beam at the detector position to increase the angular resolution, while keeping a large beam on the powder sample for better crystallite average.

The designed beam size at focusing point should be 1.0(h) x 0.5(v) mm^2.

B3.3 Vertical Focusing Mirror

The 2nd Pt-coated Si mirror is placed after the monochromator to vertically focus the beam to the positions required by the experiments.

Based on the spectral flux chart provided by NSLS-II, assuming a 2-mrad horizontal radiation acceptance, and an overall 50% efficiency of the optical components; the beam's flux at 8, 17, and 25 keV should be about $2x10^{13}$, $7x10^{12}$, and $2x10^{12}$ respectively. The current X14A measured beam flux is $3x10^{12}$ at 8 keV, and $7x10^{11}$ at 17 keV.

B4. End Station Equipment

B4.1 Motorized slits, Ion Chambers, helium or vacuum path, Compound Refractive Lenses (CRL)

The first optical component in the hutch is a motorized two-way slit, followed by a helium or vacuum path to eliminate air absorption for low energy x-rays. The 2nd motorized slit after the pass defines the beam size on the sample. For XRPD measurements using capillary samples and the position sensitive detectors, a 1.0x0.5 mm^2 beam size is desirable. For experiments needing small beams, such as high-pressure experiments with diamond anvil cells, or stress measurements using position-sensitive detectors, the He pass can be replaced by CRLs, two of which are desirable, one for higher energy (20-25 keV) and another for low energy (6 to 7 keV). Each slit will have an ion chamber attached on its downstream side to monitor the beam's intensity.

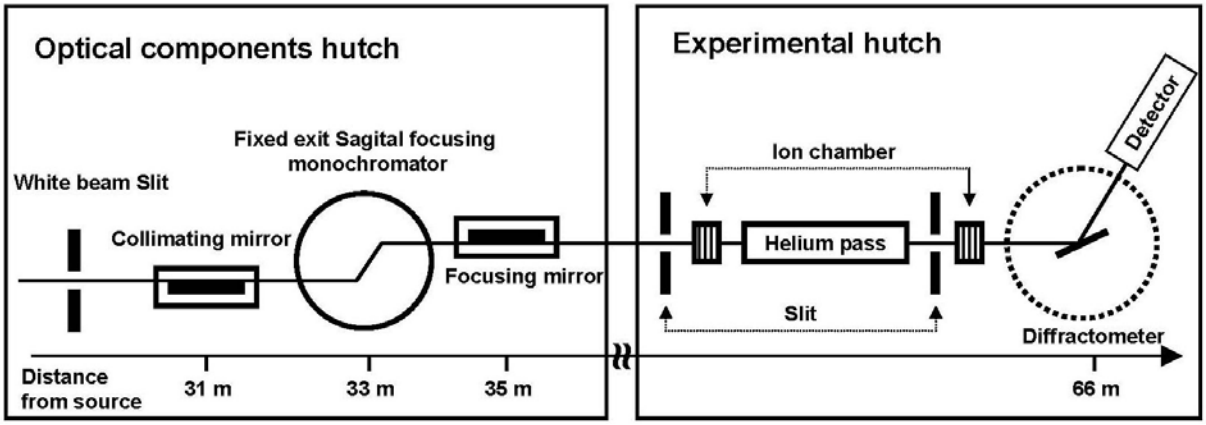
B4.2 Six Circle Diffractometer

The six-circle diffractometer can be used for regular XRPD measurements. The chi angle movement and the analyzer circle are needed for conventional stress measurements and surface diffractions. The straight Si strip detectors can be mounted on the 2 arm with a distance up to 2 meters for high angular resolution.

B4.3 Detectors

- Scintillation counters
- Avalanche photodiodes
- Ion chambers for beam monitors
- Straight Si strip detectors
- Curved Si strip detectors (125° 2 range with 0.014° resolution)
- 2D CCD or Image plate detectors

B5. IXD Optical Layout (not to scale)



B6. *In Situ* Capability

- High-temperature furnace for capillary powder samples up to 2000°C with gas-flow capabilities
- High-temperature furnace for plate powder samples up to 1100 °C in vacuum or gaslow chamber
- Gas-handling devices, hazard-gas exhaust device
- Residual gas detectors
- High-pressure diamond cells
- Battery-charging devices
- He closed-circle cryostat (Displex) for plate sample
- Cryostream for capillary samples

C. Required Technical Advances (if any)

D. User Community and Demands

XPD, the new NSLS-II high energy (> 40 keV) powder diffraction beam line that already is funded represents the most advanced XRPD instrumentation. It will create new frontiers in XRPD techniques and benefit users facing the most challenging problems in structural characterization. On the other hand, a large portion of the research projects, including many cutting-edge science programs, can very well be conducted using a beamline with medium energy x-rays. XRPD with medium x-rays is a well-developed, powerful tool for materials characterization. Indeed, all the research examples discussed in the science case section were conducted with x-rays ranging from 7 to 19 keV.

IXD aims to satisfy the practical needs for *ex situ* and *in situ* XRPD measurements by a wide user community, such as the user groups conducting studies on batteries, fuel cells, catalysis, high-temperature materials, light-metal production, and other energy industry related and EERE sponsored researches Commissioning the IXD in the early stages of NSLS-II operation will ensure a smooth transition of the well-developed user community and expertise in XRPD studies from NSLS to NSLS-II. The IXD will contribute to advancing XRPD techniques in several areas, such as creating extreme high-temperature sample environments with gas flow and fast data acquisitions of high Q-resolution XRPD patterns. IXD also will address user's needs for element selective and surface sensitive X-ray diffraction methods that are unavailable at the high- energy XPD beamline.

The High Temperature Materials Laboratory (HTML) at Oak Ridge National Laboratory is a DOE User Facility dedicated to solving materials problems that limit the efficiency and reliability of systems for power generation and energy conversion, distribution, and use. The HTML has operated X14A as a major part of its user center for research in diffraction and residual stress. HTML staff developed sophisticated, often one-of-a-kind instruments for characterizing the microstructure, microchemistry, and physical and mechanical properties of materials over a wide range of temperatures. The X14A user groups were benefited by combining their synchrotron XRPD measurements with other property characterizations provided by HTML in their materials studies. HTML supports the proposed IXD beamline and will join with the battery study group of BNL's Chemistry Department, and other EERE sponsored programs to provide support in terms of scientific personnel and instruments in operating the IXD.

Appendix D is a list of current and potential NSLS user groups that will be benefited by the proposed IXD beamline. Also listed as the appendixes to this proposal are the letters of support from the Joint Photon Sciences Institute (JPSI) and three user groups.

E. Proposal Team Expertise and Experience

Jianming Bai is a beamline scientist who operates beamline X14A for the High Temperature Materials Laboratory of ORNL since 1997. His research interests are in the areas of in situ XRPD as applied in materials sciences, x-ray optics, synchrotron beamline instrumentation. He led the teamwork to upgrade the X14A sagittal focusing monochromator to the 11 mrad horizontal radiation acceptance, which was the largest in the world. He wrote the control macros and the ray-tracing error analysis program for precise dynamical focusing of the monochromator. Together with the new mirror built at the same time, the upgrade boosted the beam intensity by 6 folds. He worked with the NSLS detector group on detector testing and developments at X14A and is a Co-PI of the NSF/MRI grant award for the development of a Silicon Detector for Synchrotron Based X-Ray Spectroscopy, X-Ray Holography and Materials Education.

Jonathan Hanson is a chemist whose main research interests are in the areas of catalysis and diffraction sciences. His research is supported by US-DOE. He is spokesperson for beamline X7B at the NSLS and has contributed to the development of combined XRD/QEXAFS at beam line X18A He has more than 18 years of experience working at synchrotrons, x-ray absorption spectroscopy and time-resolved x-ray powder diffraction in his studies. He has published over 210 articles.

Xiao-Qing Yang is a material scientist and PI for the Applied Battery Research for Transportation (ABRT) and Batteries for Advanced Transportation Technologies (BATT) projects at BNL funded by the Office of Vehicle Technologies, EE&RE, U.S. Department of Energy (USDOE). The goals of these projects include synthesis and characterization of new cathode, anode, and electrolyte materials with improved calendar and cycling life, better abuse tolerance for lithium batteries. Together with his colleagues at Brookhaven National Lab., he has developed several new synchrotron based x-ray techniques to do in situ characterization of battery materials. Using these techniques, many new structures, new phase transitions, and new observations about the structural changes of battery materials during charge-discharge cycling and under other operating conditions have been reported by his research group. He was awarded several US patents. Dr. Yang's research on lithium battery materials is well recognized internationally. He has been invited to give presentations at international conferences and world class institutions and industrial companies in US and abroad. Dr. Yang organized and co-organized several international conferences, such as IBA2007 in Shenzhen and CIBF2006 in Beijing. He is one of the organizers and the general secretary for the 14th International meeting of Lithium Battery (14th IMLB) in Tianjin, China, June 22, 2008. Dr. Yang was elected as a member of the board of directors of the International Battery Materials Associate (IBA) in December of 2008.

Steven Ehrlich is currently a physicist at the National Synchrotron Light Source at Brookhaven National Laboratory. He serves as User Liaison and Experimental Operations Manager of the NSLS. He coordinates operations with internal and external research groups and facility users, troubleshoots operational problems at beamlines, runs the weekly X-ray/VUV users meetings, and creates and distributes weekly X-ray/VUV users meeting minutes. He also provides NSLS experimental users an interface with machine operations and NSLS and BNL staff. Steven is also the spokesperson for beamline X18A. He is in the process of completing an upgrade of the X18A beamline, part of which adds EXAFS and quick EXAFS capabilities to the scattering and diffraction line. He has worked with the Synchrotron Catalysis Consortium (SCC) to optimize beamline X18A for EXAFS, quick EXAFS and combined EXAFS/diffraction measurements. His

research includes structural studies of long alkane films, noble gases confined in porous media and liquid systems.

John B Parise, Distinguished Professor at Stony Brook University, has been involved in the synthesis of novel materials and their characterization using x-ray and neuron powder and single crystal crystallography for the last 30 years. His research program addresses fundamental problems in solid-state chemistry, environmental science, and mineralogy and his group specializes in characterization under extreme environments incluing time resolved studies at high *p*T.

Trevor A. Tyson is a professor in the Department of Physics of the New Jersey Institute of Technology. He has over twenty years of experience in synchrotron based research and is a member of the X11A and X14A PRT groups. His research involves studies of nanoscale materials and complex transition oxide systems. This involves modeling, synthesis and synchrotron based x-ray spectroscopic and scattering studies. The x-ray measurements are complemented by IR and UV synchrotron experiments. His research focuses on understanding the mechanisms in correlated electron systems such as thermoelectricity, magnetoresitance, magnetism, ferrroelectricity, multiferroicity, and superconductivity and the exploring the effect of nanostructuring on atomic and electronic properties. His interests include studies of the high temperature formation of these materials (atomic and electronic structure) as polycrystals, single crystals and films. In parallel with the experiments, he is involved in the development and testing of instruments (detectors and spectrometers and high field magnets) related to scattering and spectroscopic studies at synchrotron facilities.

Jun Wang has 17 years of experience in synchrotron x-ray diffraction/ reflectivity/scattering, protein crystallography and powder diffraction; she is currently leading TXM project, and coordinating industrial research and energy research at NSLS.

Camden R. Hubbard is the principal leader of the ORNL research group responsible for the activities in the Residual Stress, Diffraction, and Thermophysical Properties User Centers within the High Temperature Materials Laboratory. These activities all contribute to laboratory-industry-university collaborative research aimed at improving advanced materials processing and properties with particular emphasis on high temperature structural ceramics and alloys. He is the PI for the multidisciplinary project of "Development and Demonstration of Residual Stress Mapping by Neutron and X-ray Diffraction Methods" and the spokesperson of Beamline X14A.

Jose A. Rodriguez is a chemist whose main research interests are in the areas of catalysis and surface sciences. His research is supported by US-DOE. He is spokesperson for beamline X7B and contact person for beamline U7A at the NSLS. He has more than 15 years of experience working at synchrotrons, using photoemission, x-ray absorption spectroscopy and time-resolved x-ray powder diffraction in his studies. He has published over 250 articles and has given many invited seminars about synchrotron-based techniques for catalyst characterization.

F. Suggestions for BAT Membership

In addition to the members of the proposal team, Dr. Andrew Payzant, Dr. Yan Gao, Prof. Scott Misture and Prof. Angus Wilkinson have agreed to serve in the Beamline Advisory Team of IXD.

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G. The list of appendixes

Appendix A1 Battery Studies at BNL Chemistry
Appendix A2 Stress Measurements
Appendix D A list of user groups that will be benefited by IXD
Appendix E The Biographical Sketches of the members of the proposal team.

List of letters of support:

1, Letter of Support from JPSI
2, Letter of Support from Prof. Meilin Liu of Georgia Tech
3, Letter of Support from Prof. Yet-Ming Chiang of MIT
4, Letter of Support from Dr. Yan Gao of GE Global Research