CHESS Update

June 9, 2020

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Cornell High Energy Synchrotron Source (CHESS)
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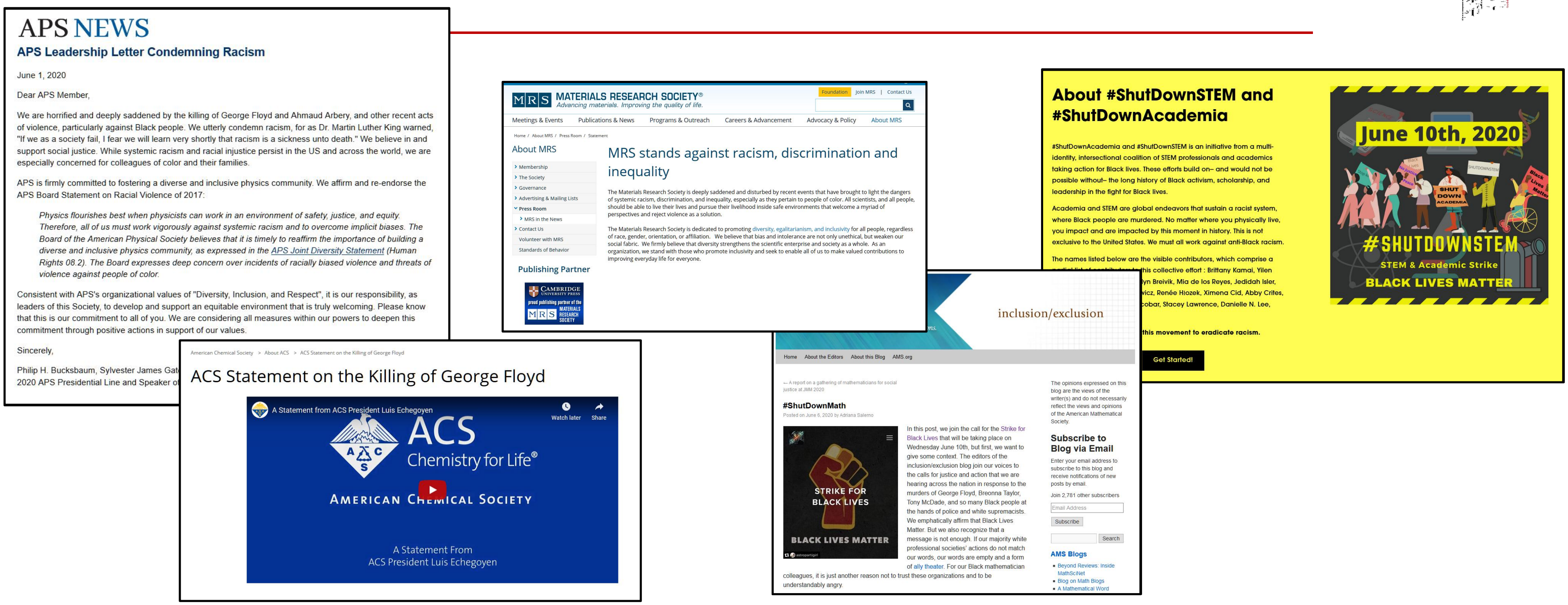
CoVID-19

Date	Event
March 16	CHESS/CESR shut down – only personnel performing “essential maintenance” allowed on Cornell University campus.
April 29	CHESS/CESR receives permission to restart for a single “essential” CoVID-19 research project. (See Cerione talk this evening!) Begin process of restarting accelerator complex.
May 15	All DoD and NIH funded research determined to be “essential.” Expand planning and preparation for additional experiments.
May 27	NYS opens Tompkins County for all research.
June 10	CoVID-19 research in 7A (Bio-SAXS) and 7B2 (FLEX). DoD research in 1A3 (FMB) and 3A (FAST). Other beamlines preparing for (remote) operations in Fall 2020.
June 29	End of Spring 2020 run, beginning of Summer down. Focus on preparing for (remote) operations in Fall 2020. (see talk by Elke Arenholz)
Labor Day	End of Summer down and beginning of Fall operations (user beam on September 23)

We expect CHESS will continue to re-open in a phased fashion: first for remote access, then local users, then national users, then international users. There is no time-table. There may be set backs. The virus will determine the rate of progress.

National Conversation on Race and Racism



We are in a time of profound societal change – change that we, as a community and a society, have the power to influence and to shape. We can, and we will, rise to this challenge. Our own consciences demand it, as do our values as a community, and our ethos as Cornellians. President Martha Pollack

2019-2020: A year of Success

- Return to operations
- 1st year of partner model
- PREM partnership
- Steady progress for CESR
- Science!
- New technology
- New Beginnings

Return to operations

1) CHESS-U upgrade to CESR
(completed January 2019 – stored e^+ beam)

2) CHESS-U upgrade to X-ray experimental floor
(completed June 2019 – x-ray beam in hutches)

3) Reorganization/restart of CHESS User Operations
(completed October 2019 – users doing science)

1st Year of Operations under Partner Model

- Center for High Energy X-ray Science (CHEXS): National Science Foundation
  - QM2, FAST, PIPOXS, HP-BIO (MX & SAXS), X-Ray R&D, Education & Outreach
- Materials Solutions Network – CHESS (MSN-C): Air Force Research Laboratory
  - SMB, FMB, ITAR
- Macro-Molecular Crystallography at CHESS (MacCHESS): National Institutes of Health and NYSTAR
  - FLEX (Room-Temperature Serial, MX, HP-MX), Bio-SAXS (SEC)

1st PREM with a National Facility as Partner

PREM CIE²M
Center for Interfacial Electrochemistry of Energy Materials

CIE²M is a partnership between
- University of Puerto Rico – Río Piedras Campus
- Universidad Ana G. Méndez – Cupey and Gurabo Campuses
- Cornell High Energy Synchrotron Source (CHESS)

2019 Summer Program

Pedro Trinidad

Marisol Figueroa

Ángel García

Kálery La Luz

Steady Progress for CESR

- Machine current during user operations increased from 50 → 75 → >100mA @ 6.0GeV – lifetime ~20 hr
- Machine current during machine studies has achieved 200mA (CHESS-U goal)
  - Must complete detailed radiation and thermal surveys in X-ray regions of lab before user operations.
- Development of novel timing modes

(See talk by Jim Shanks)

To Twin or Not to Twin: Micromechanical Response in Magnesium probed with High Energy X-Rays

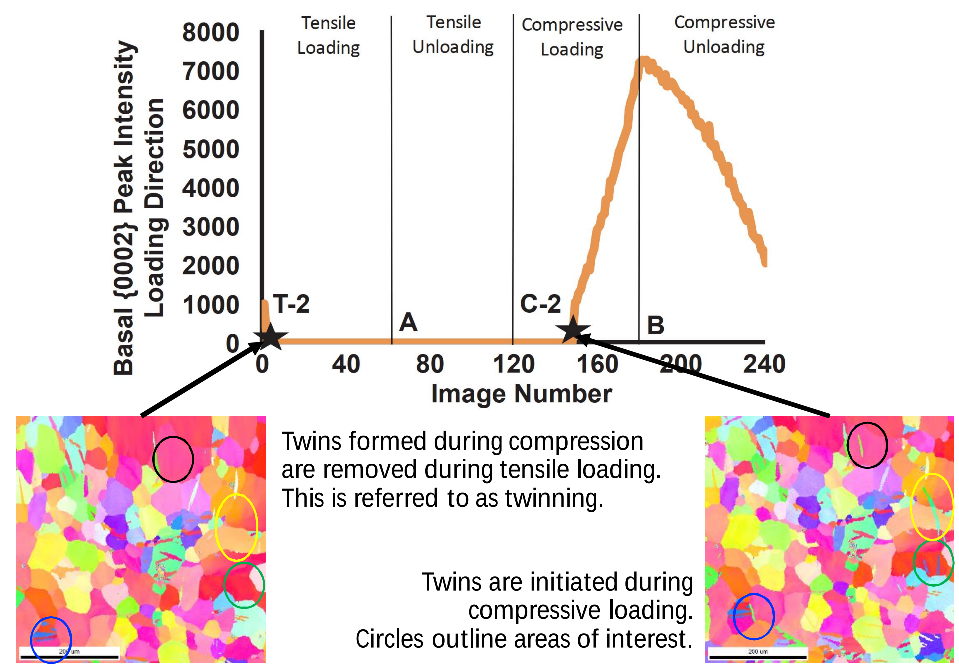
A. D. Murphy-Leonard *et al*., University of Michigan; D. Pagan, CHESS;

What did the scientists discover?

The structural evolution of extruded Mg was investigated using in-situ high energy X-ray diffraction (HEXD) at CHESS under fully-reversed low cycle fatigue conditions. At cyclic strains greater than 0.5%, twinning occurs during the compression portion of the cycle and, at early stages of fatigue, most twins are detwinned under reversed loading during the tensile portion of the cycle. As the number of fatigue cycles increases the twin volume fraction increases and the detwinning process is incomplete and a significant fraction of residual twins remains throughout an entire cycle.

Why is this important?

Reducing the weight of vehicles translates into energy conservation in transportation which is beneficial for economic and environmental reasons. Magnesium shows promise as lightweight but strong material to be used in vehicles since it has 2/3 the density of aluminum and an excellent strength-to-weight ratio. However, before it can be widely adopted, its performance during cyclic loading, i.e. fatigue, must be understood.



Evolution of diffracted intensity from basal lattice planes during low-cycle fatigue of pure magnesium. An increase in intensity from lattice planes perpendicular to loading during compression is indicative of twinning, while a decrease is related to detwinning. Grain orientation maps were collected using electron back-scatter diffraction to help visualize twinning and detwinning during cyclic loading.

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CHESS was supported by NSF under award DMR-1332208.
CHEXS is supported by NSF under award DMR-1829070.

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To Twin or Not to Twin: Micromechanical Response in Magnesium probed with High Energy X-Rays

A. D. Murphy-Leonard *et al*., University of Michigan; D. Pagan, CHESS;

What are the broader impacts of this work?

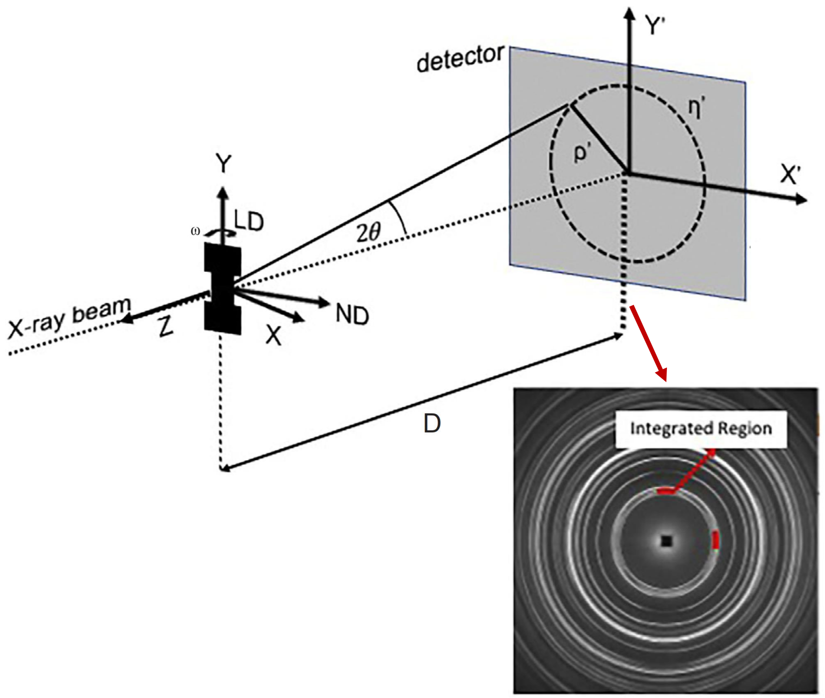
Experiments like the one presented here advance our understanding of the fatigue characteristics of magnesium and will enable its use as strong, lightweight material in vehicles and related applications.

Why did this research need CHESS?

The ability to preform high energy X-ray diffraction (HEXD) experiments during in-situ cyclic mechanical loading at the F2 Station at CHESS were crucial for this research. The sample was illuminated by a 61.332 keV X-ray beam and the diffracted intensity was measured in transmission on an area detector. A sufficient number of grains were illuminated such that nearly complete Debye-Scherer powder rings were captured on the detector. The cyclic loading was performed in displacement control with displacement end points.

How was the work funded?

CHESS was supported by NSF award DMR-1332208. A. D. Murphy-Leonard acknowledges the support of the National Science Foundation Fellowship. Part of this work is supported by DOE-BES, Division of Materials Science and Engineering under Award #DE-SC0008637.



Debye Scherer Rings

Schematic of the diffraction experiment detailing the sample geometry. An example of the continuous diffraction rings and the HEDM integration areas (Red boxes) are also shown.

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Synergistic Co–Mn Oxide Catalyst for Oxygen Reduction Reactions

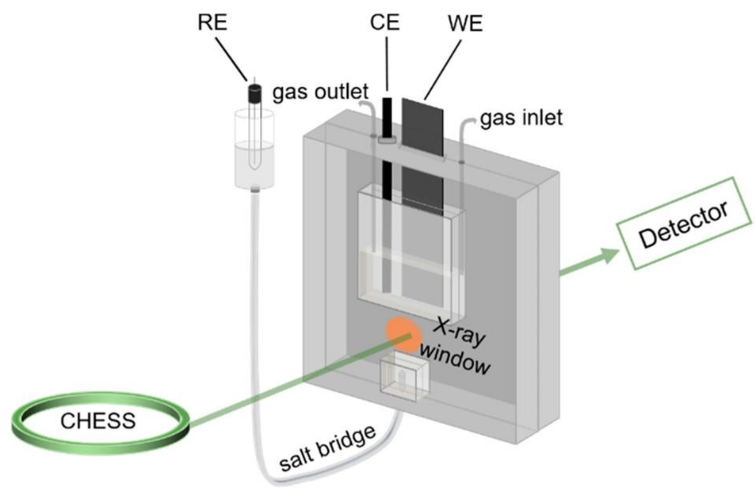
Yao Yang *et al.*, Cornell University; Wuhan University

What did the scientists discover?

Identifying the catalytically active site(s) in the oxygen reduction reaction (ORR) is critical to the development of fuel cells and other technologies. Researchers employed synchrotron-based X-ray absorption spectroscopy (XAS) at CHESS to investigate the synergistic interaction of bimetallic $Co_{1.5}Mn_{1.5}O_4$/C catalysts – which exhibit impressive ORR activity in alkaline fuel cells – under real-time *operando* electrochemical conditions. Under steady state conditions, both Mn and Co valences decreased at lower potentials, indicating the conversion from Mn-(III,IV) and Co(III) to Mn(II,III) and Co(II), respectively. Changes in the Co and Mn valence states are simultaneous and exhibited periodic patterns that tracked the cyclic potential sweeps.

Why is this important?

As an emerging candidate for energy-conversion devices, alkaline fuel cells have drawn increasing attention enabling the use of nonprecious metal electrocatalysts, rather than the expensive Pt-based catalysts. Among these, 3*d* metal oxides have garnered increasing interest as ORR electrocatalysts due to their high activity, long durability and low cost. However, the limited understanding of the complicated electrocatalytic mechanism of ORR on these materials has hindered the progress. Researchers at CHESS investigated a highly active 3*d* bimetallic electrocatalyst Co–Mn oxide and, based on *in situ* XAS measurements, and propose that Co and Mn serve as synergistic sites to catalyze ORR.



Schematic of the in situ XAS electrochemical cell. Working electrode (WE, catalyst on carbon paper) and counter electrode (CE, carbon rod) were immersed in 1 M KOH solution. The reference electrode was connected to the cell by a salt bridge to minimize IR drops caused by the resistance in the thin electrolyte layer within the X-ray window.

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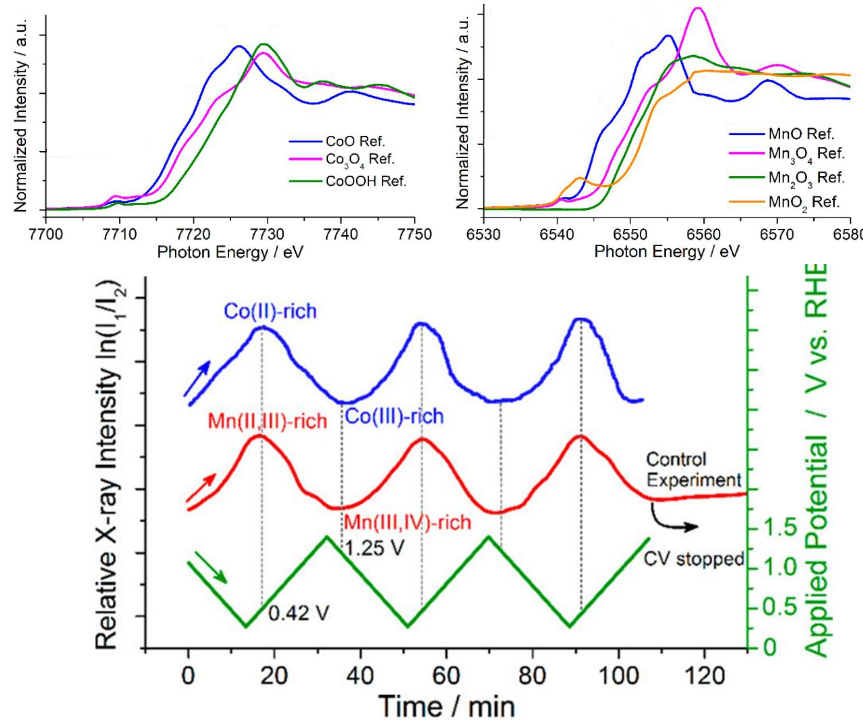
Synergistic Co–Mn Oxide Catalyst for Oxygen Reduction Reactions

Yao Yang *et al*., Cornell University; Wuhan University

What are the broader impacts of this work?
To the best of our knowledge, this represents the first study, using *in situ* XAS, to resolve the synergistic catalytic mechanism of a bimetallic oxide. Strategies developed by the research team provide a promising approach to unveil the reaction mechanism for other multimetallic electrocatalysts.

Why did this research need CHESS?
X-ray absorption near edge structure (XANES) was used to track the dynamic structural changes of Co and Mn under both steady state (constant applied potential) and nonsteady state (potentiodynamic cyclic voltammetry, CV). Rapid X-ray data acquisition, combined with a slow sweep rate in CV, enabled a 3 mV resolution in the applied potential, approaching a nonsteady (potentiodynamic) state. The PIPOXS beamline which is part of the Center for High Energy X ray Sciences (CHEXS) at CHESS will allow similar studies in the future.

How was the work funded?
This work is based upon research conducted at CHESS which was supported by NSF under award DMR-1332208 and at CCMR supported through the NSF MRSEC program (DMR-1719875). This work was also financially supported by DOE under award No. DE-SC0019445 and the National Natural Science Foundation of China (21872108, 91545205).



Top: Spectra of the Mn and Co K-edges for difference valence states indicating that high intensity at 7722.5eV and 6553eV are indicative of Co(II) and Mn (II) valance states. Bottom: Periodic changes in the relative X-ray intensities ($\ln(I_1/I_2)$) at 7722.5 eV (Co K-edge, blue) and 6553.0 eV (Mn K-edge, red) as a function of the cyclic potential sweep (green). Intensity variations at 7722.5 and 6553.0 eV reflect the conversion between Co(II) and Co(III), Mn(II,III) and Mn(IV), respectively.

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Diffuse X-ray Scattering from Correlated Motions in a Protein Crystal

Steve P. Meisburger[1,2], David A. Case[3] & Nozomi Ando[1,2]. ([1]Cornell, [2]Princeton, [3]Rutgers)

What did the scientists discover?

For many decades, X-ray crystallography has been the method of choice for determining the atomic-resolution structure of protein molecules arranged in a crystal lattice. X-rays diffract from the crystal, producing Bragg peaks on a detector that encode the structure. Interestingly, protein motions in the crystal give rise to a second signal, known as diffuse scattering, that appears between and underneath the Bragg peaks. However, this signal has been challenging to measure and interpret. Working at CHESS, the Ando group at Cornell succeeded in mapping the three-dimensional diffuse scattering from a protein crystal with unprecedented accuracy (Fig. 1). Using this high-quality map, they were able to show that lattice vibrations were responsible for most of the diffuse pattern, including the striking "halo" features near the Bragg peaks (Fig. 2a). Once these motions were accounted for, they showed that internal breathing motions of the protein contribute in a subtle but important way (Fig. 2b).

Why is this important? What are the broader impacts of this work?

It is increasingly recognized in the field of structural biology that information on protein dynamics is needed to understand function, but few techniques are sensitive to the dynamics of interest. Diffuse scattering has been proposed to fill this critical gap, since it is often observed during the course of conventional data collection and could be used to "animate" conventional crystal structures. However, diffuse scattering is routinely discarded at the first step in data processing, because it has proven exceedingly difficult to interpret. This work provides the first convincing demonstration of protein diffuse scattering data collection and analysis, opening the door to future applications in structural biology.

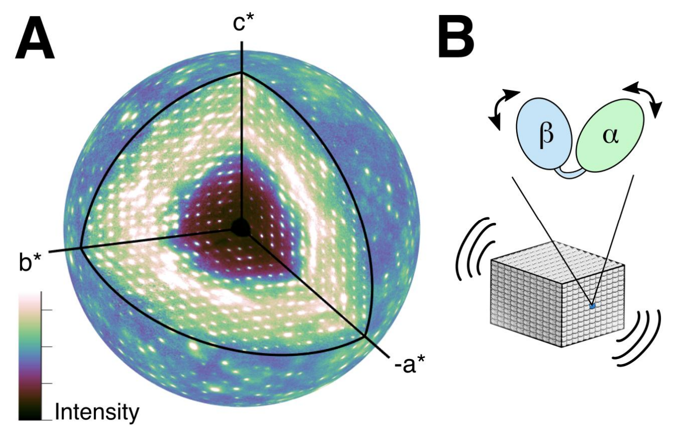

Fig. 1. Three-dimensional map of diffuse scattering from hen lysozyme crystallized in the triclinic space group. (A) The scattering includes intense "halo" features co-localized with Bragg peaks (which have been removed), as well as a continuous "cloudy" pattern that extends throughout. (B) Diffuse scattering arises from lattice vibrations as well as collective internal motions, such as the hinge-bending motion of the α and β domains.

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Diffuse X-ray Scattering from Correlated Motions in a Protein Crystal

Steve P. Meisburger[1,2], David A. Case[3] & Nozomi Ando[1,2]. ([1]Cornell, [2]Princeton, [3]Rutgers)

Why did this research need CHESS?

Many of the advances that made this work possible have occurred at CHESS, which has a long history in the field of protein diffuse scattering. Since the 1990s, Sol Gruner and his group have pioneered the use of CCDs and later, pixel-array detectors for diffuse scattering. Additionally, CHESS's unique "empty hutch" culture of experimentation makes it the ideal environment for performing non-standard experiments and developing new methods. The diffuse scattering data in this paper were collected at room temperature on the MacCHESS F1 beamline using the Pilatus 6M pixel-array detector. It was by taking full advantage of the F1 hutch and the detector's performance that the authors were able to improve data quality to the point where realistic models could be fit.

How was the work funded?

CHESS is supported by NSF Grant DMR-1332208, and the MacCHESS facility is supported by NIH/NIGMS Grant GM-103485. The authors were supported by NIH Grants GM117757 (to S.P.M.), GM100008 (to N.A.), GM124847 (to N.A.), and GM122086 (to D.A.C.) and by start-up funds from Princeton University and Cornell University (to N.A.).

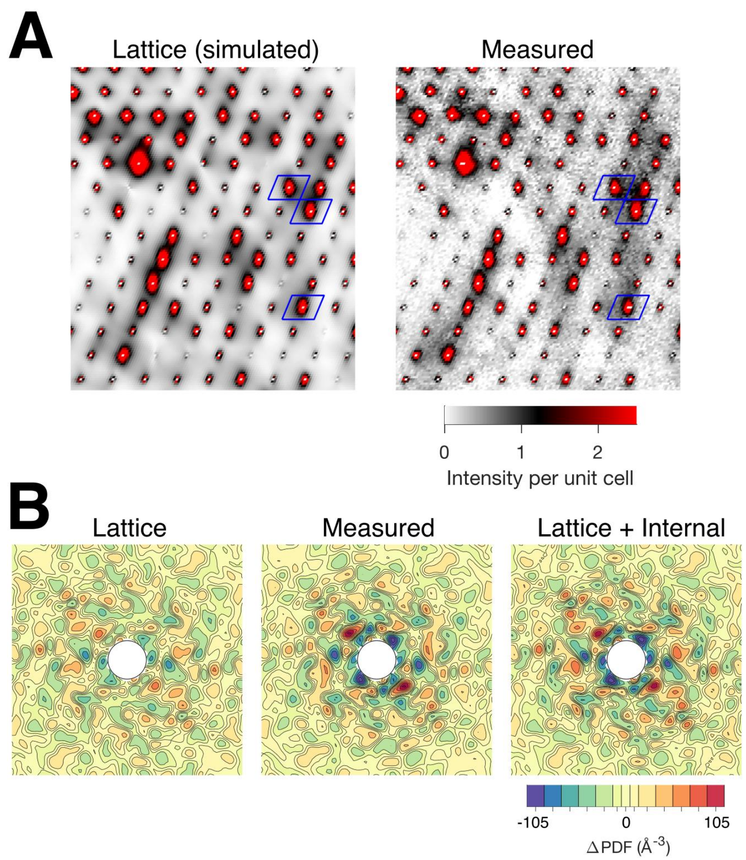

Fig. 2. Origins of the diffuse signal. (A) Slices through the three-dimensional map showing the variational part of the intensity. Most of the intense features in the measured data (right) are reproduced by a lattice vibrations model (left) that was fit to the data. Blue boxes show regions included in the fit. (B) The importance of internal motions becomes clear when the data are transformed into real space, where correlations are separated by inter-atomic vector. Lattice dynamics alone cannot explain the features near center of this map, corresponding to short inter-atomic distances (left vs. center). A realistic model for internal protein motions must be added to account for the total signal (right).

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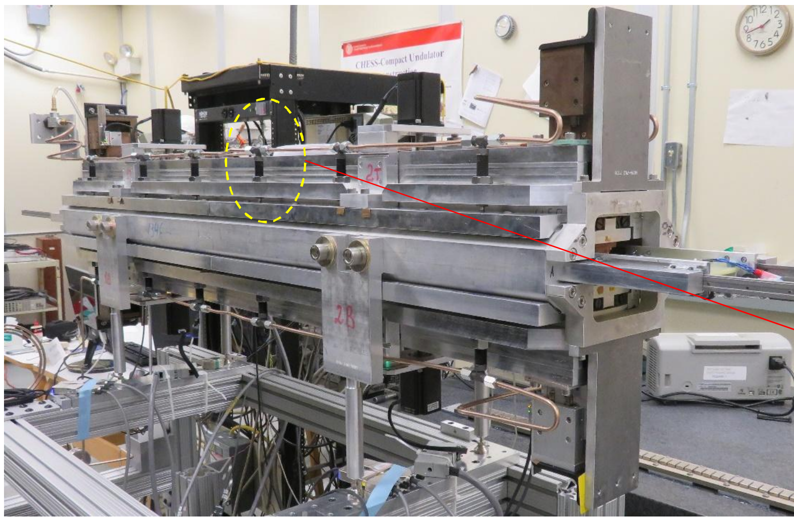
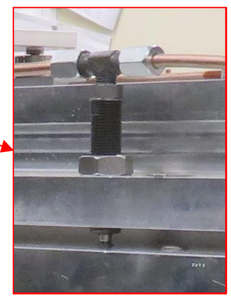
Compact Variable Gap Undulator with Hydraulic-Assist Driver

Science Mission | Develop a new insertion device technology to boost CHESS beamline performance|

Core Capabilities | Compact, lightweight, variable gap, cost efficient and easy to fabricate, with potential to boost photon flux by a factor 2x relative to the CCUs presently used at CHESS. |



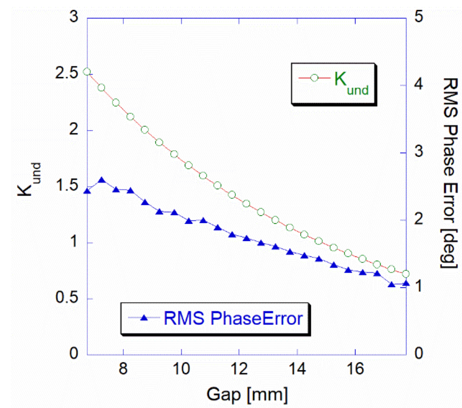
Functional Model on bench at magnetic measurement facility

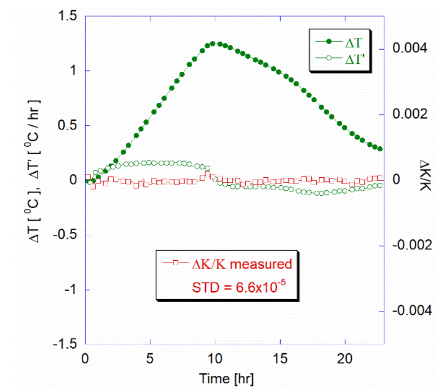


Miniature Hydraulic cylinders

Key feature: Hydraulic system compensates ~95% of magnetic forces. Simple mechanical drivers handle the rest ~5%.

Model demonstrated excellent performance





Reference: Alexander Temnykh and Ivan Temnykh. Compact variable-gap undulator with Hydraulic-Assist Driver, Nuclear Inst. and Methods in Physics Research, A 968 (2020) 163937. https://doi.org/10.1016/j.nima.2020.163937.

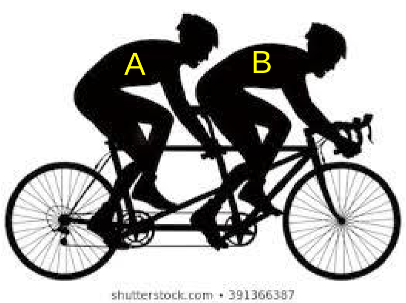
DMR-1829070

CHEXS Technology R&D | ID Program Update
CHESS Undulators “in-tandem” operating mode

Science Mission | Develop a new (for CHESS) undulator mode of operation to boost **selected** beamline performance|

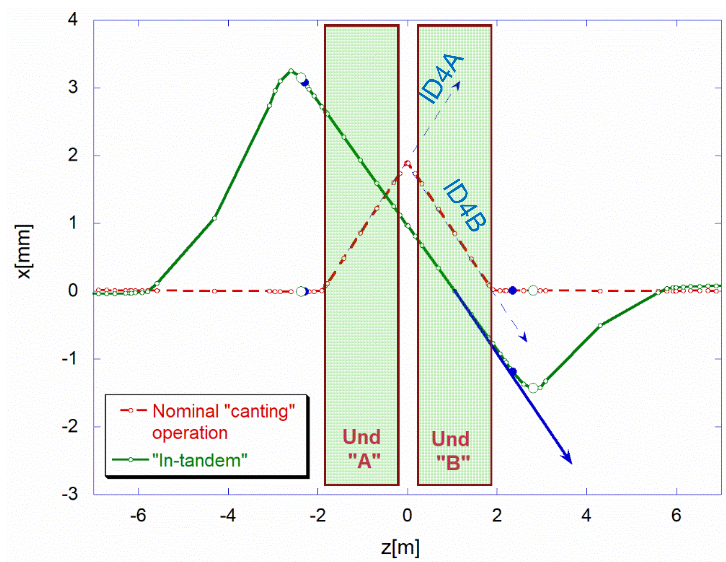
Core Capabilities | 2x increase in photon flux with no additional investment and with no impact on storage ring operation|

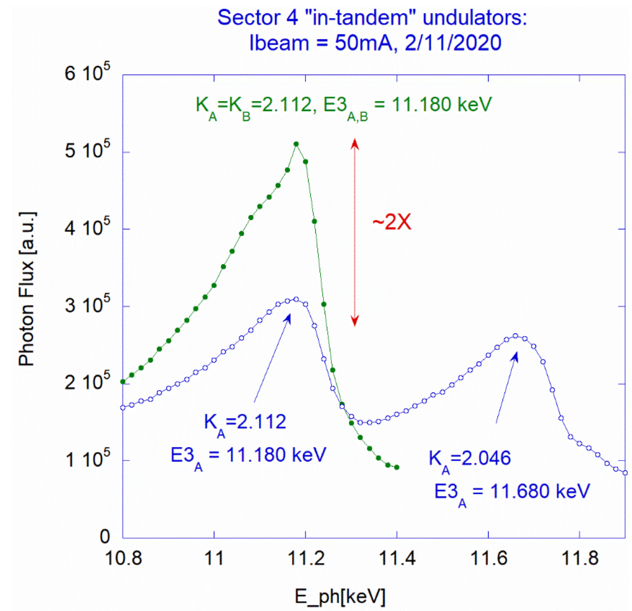
Demonstrating experiments at ID4B beam line (Jacob Ruff)



“in-tandem”

Sector 4 schematic





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DMR-1829070

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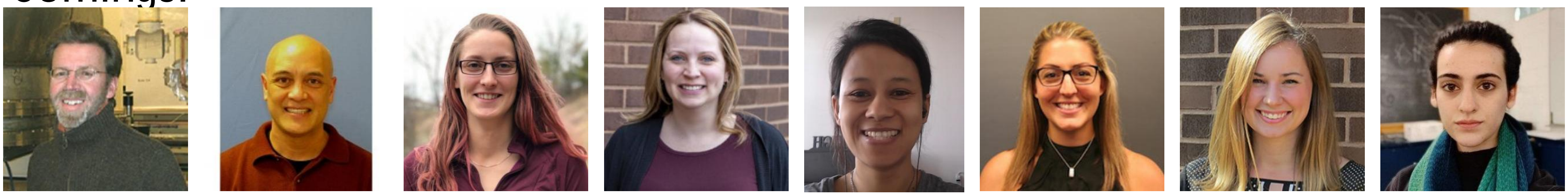
Comings and Goings

Comings:



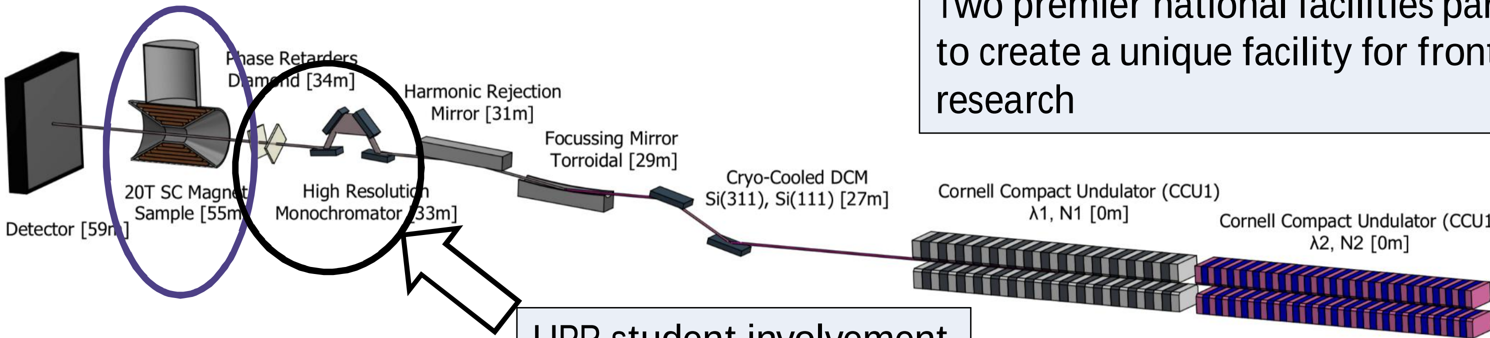
Goings:

Departing later this summer:

CHESS Mid-scale RI-2 Proposal: A first-of-its-kind X-ray facility for new science at the high magnetic field frontier.

MagLab, CHESS, and UPR-RP Team Together

Two premier national facilities partner to create a unique facility for frontier research

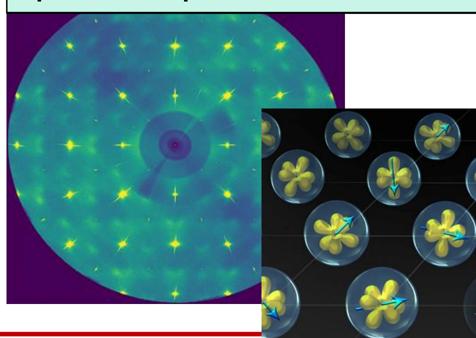


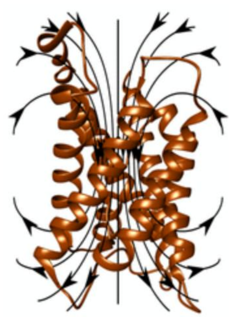
UPR student involvement

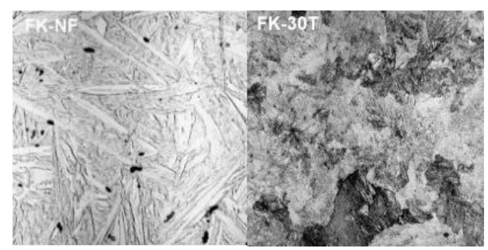
Physics: Creating and characterizing novel quantum phenomena

Biology: New tuning parameter for structural biology

Engineering: Controlling processes in engineering and chemistry

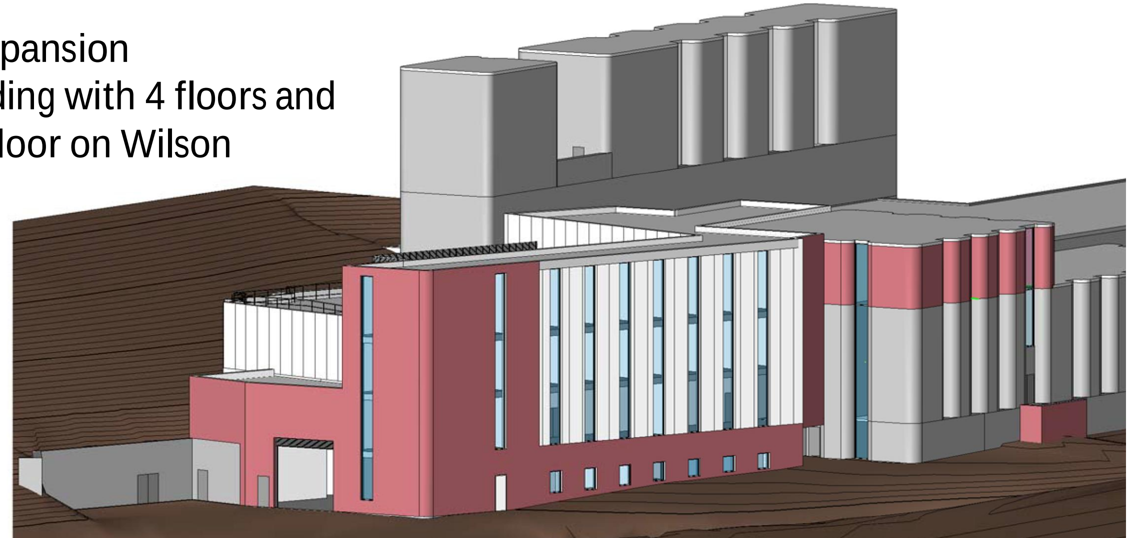




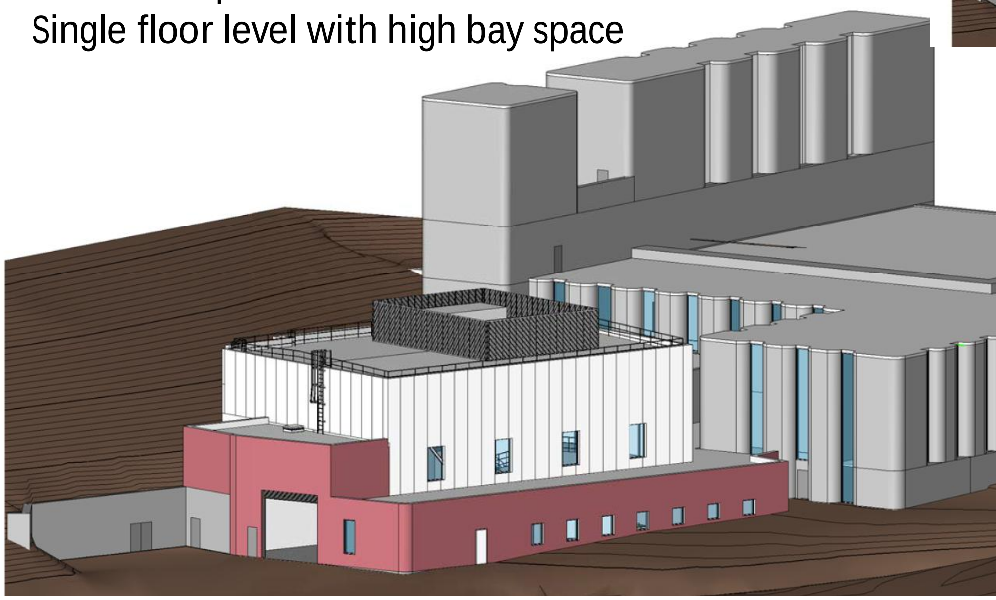


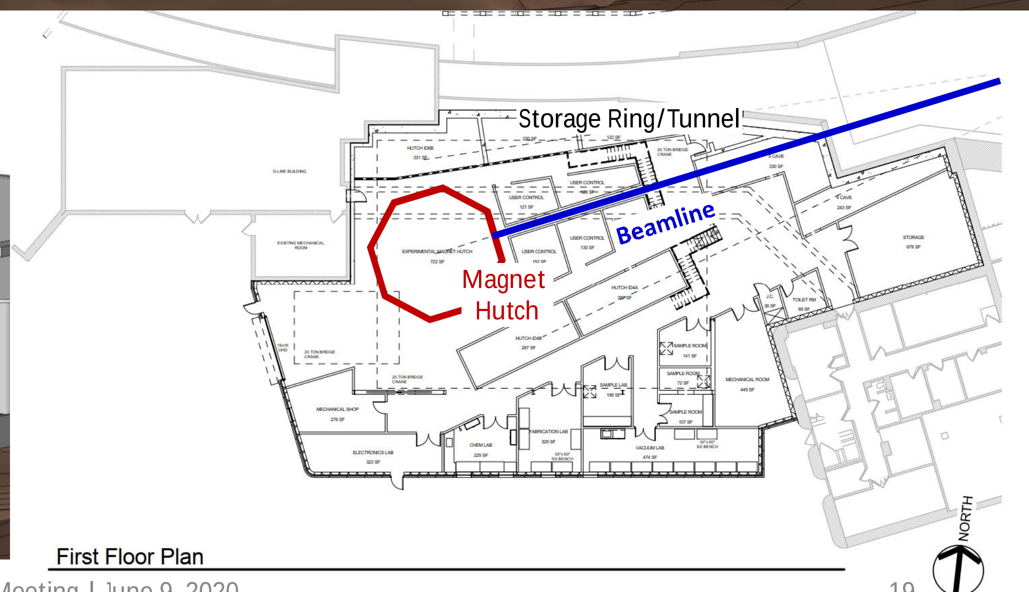
Cornell Contribution – Expansion of Wilson Laboratory

Dream expansion
New Building with 4 floors and new 4th floor on Wilson



Minimal expansion
Single floor level with high bay space





Summary and Outlook

- 2019-2020 was a very busy, productive, and exciting year with tremendous advances in capabilities, capacity, and science. Unfortunately, the CoVID-19 pandemic limited many activities.
- Although CoVID-19 continues to present challenges, 2020-2021 promises to be even more exciting, with new challenges and new opportunities.
- The HMF project is tangible evidence of the bright future for CHESS.

TUESDAY, JUNE 9, 2020

Time	Plenary Session
9:00-9:30am	Joel Brock, CHESS Director *CHESS Update*
9:30-10:00am	Elke Arenholz, CHESS Associate Director *CHESS Operations Update*
10:00-10:30am	Jim Shanks, Accelerator Physicist *Accelerator Update*
10:30-10:45am	Break
10:45am-12:00pm	Sciences@CHESS Short presentations on science at each of the CHESS beamlines
Noon - 1pm	Lunch
1:00 –4:00pm	Workshops
4:00 – 5:00pm	Poster Slam
5:00 – 6:00pm	**Online Poster Sessions**
6:00-7:00pm	Dinner Break
7pm - ?	After Dinner Session:
	Presentations by Student Paper Award Winner (10min)
	After Dinner Science Talk: Prof. Rick Cerione, Cornell *Drug Discovery At CHESS: How We Ended Up Studying COVID19*