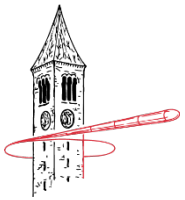
# How to write ~~a good~~ *an excellent* proposal



**Ernie Fontes**
**Technical Director, CLASSE**

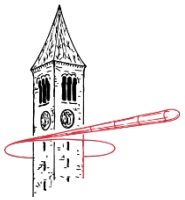
**(*many of these things are true of all writing)**

1) ***Know your audiences**
2) ***Include ONLY necessary information**
3) ***Include ALL necessary information**
4) **Details are encouraged**

This presentation was originally a round-table workshop discussion…

"Audiences":

- *CHESS User Office (**admin**)*
- *CHESS **Safety** Subcommittee*
- *External **Peer Review** Scientists*
- *Local BAC and Beamline **Scientists***



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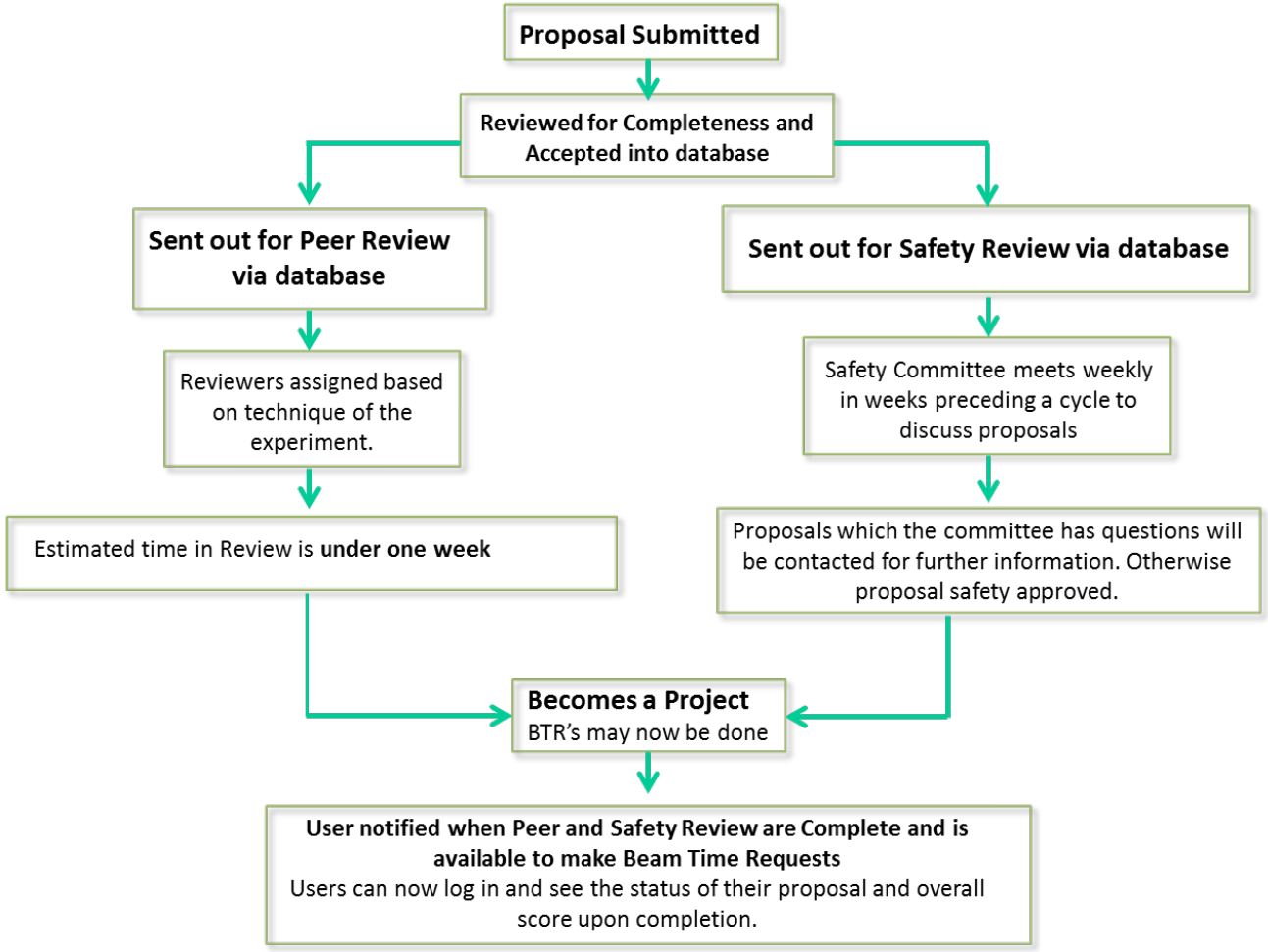

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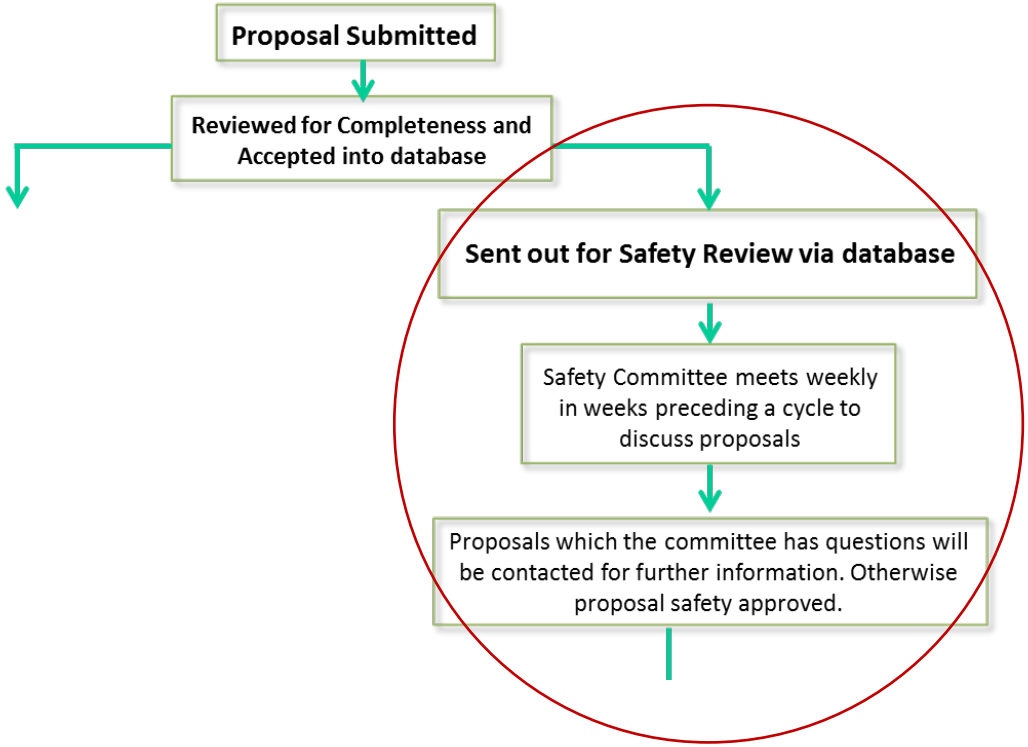

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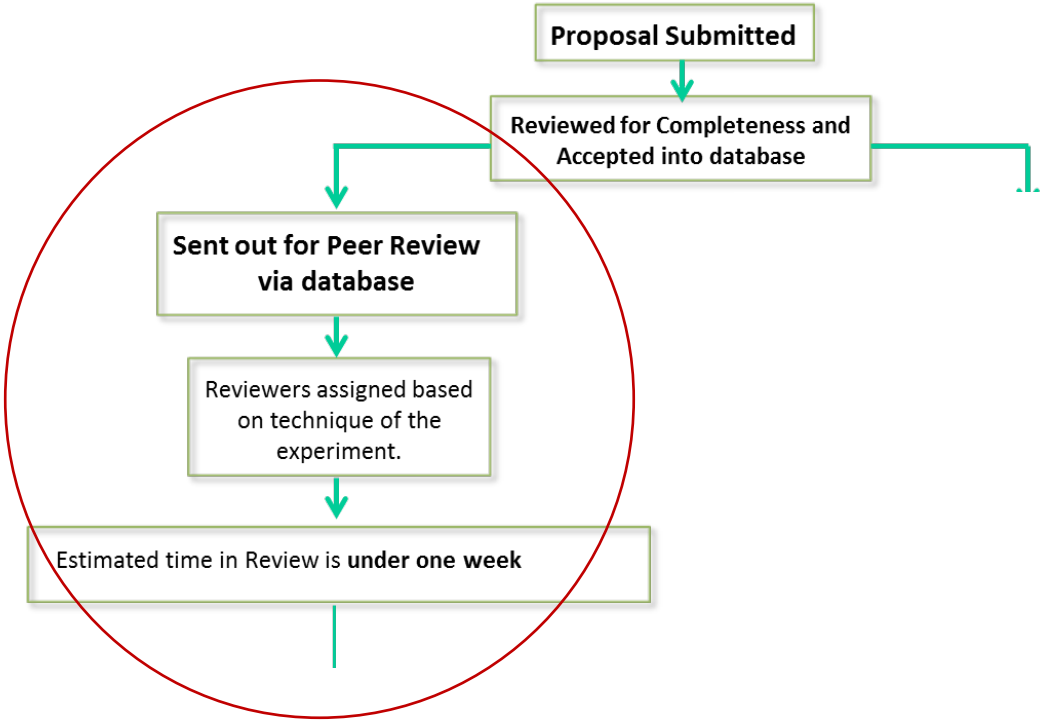

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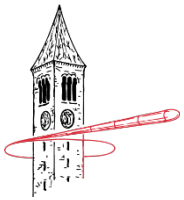


**Proposal Submitted**

**Reviewed for Completeness and Accepted into database**

**Sent out for Peer Review via database**

Reviewers assigned based on technique of the experiment.

Estimated time in Review is **under one week**

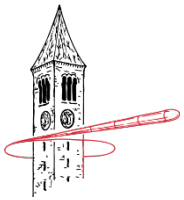
**Sent out for Safety Review via database**

Safety Committee meets weekly in weeks preceding a cycle to discuss proposals

Proposals which the committee has questions will be contacted for further information. Otherwise proposal safety approved.

**Becomes a Project**
BTR's may now be done

**User notified when Peer and Safety Review are Complete and is available to make Beam Time Requests**
Users can now log in and see the status of their proposal and overall score upon completion.



Now let's build a proposal!

CHESS
CORNELL HIGH ENERGY
SYNCHROTRON SOURCE

SCIENCE USERS PARTNERS PUBLIC ABOUT

# USERS

CHESS users come from around the world and represent both academic and corporate communities. The discoveries of these scientists and researchers not only broaden our knowledge of

Users start here:
www.chess.cornell.edu

# New User Guide

The CHESS New User Guide is a step-by-step guide to help you to apply for beamtime, to prepare for your experiments at CHESS, what to do when you arrive at CHESS and how to report publications and results after your CHESS beamtime.

## APPLY FOR BEAMTIME

- Plan your experiment and understanding beamline capabilities
- Proposal types at CHESS
- How to submit a proposal
- Proposal review and scoring
- BeamtimerRequests (BTRs)
- Beamtime allocation and scheduling

USERS

Beamline Directory

User Portal

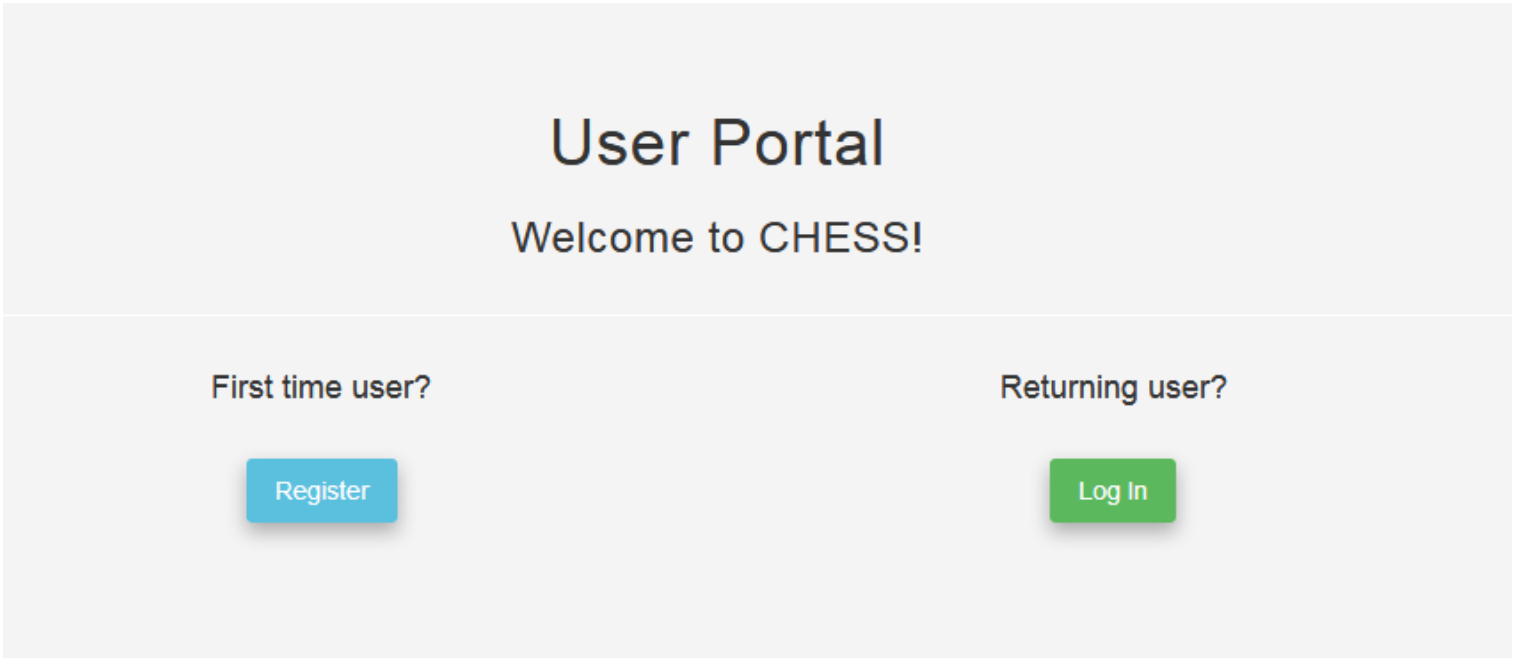
Proposal Deadlines

X-Ray Run Schedule

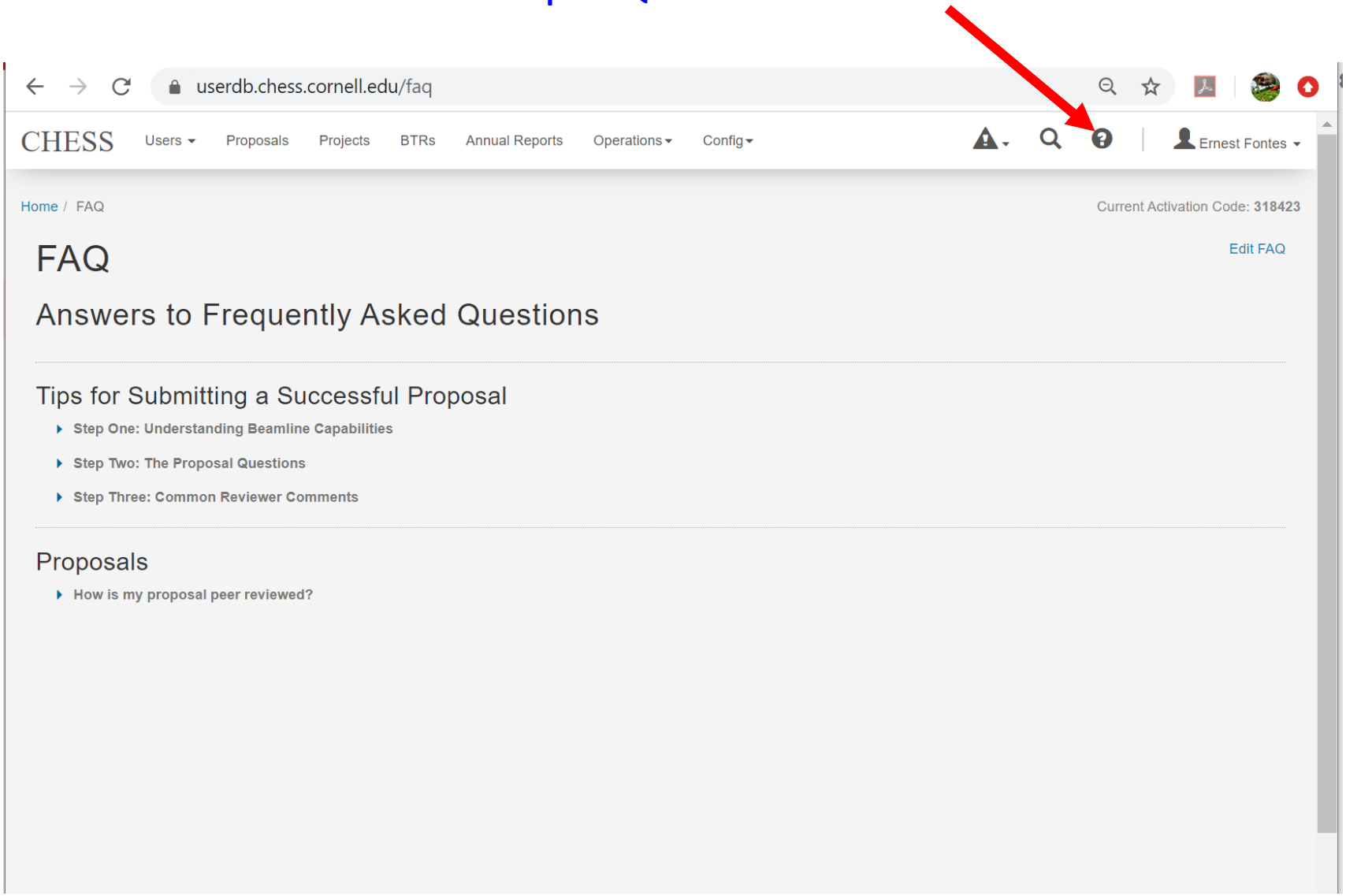
Machine Status

Acknowledgment

**Enter the User Database at:** **https://userdb.chess.cornell.edu/**



# Help! FAQ to the rescue…



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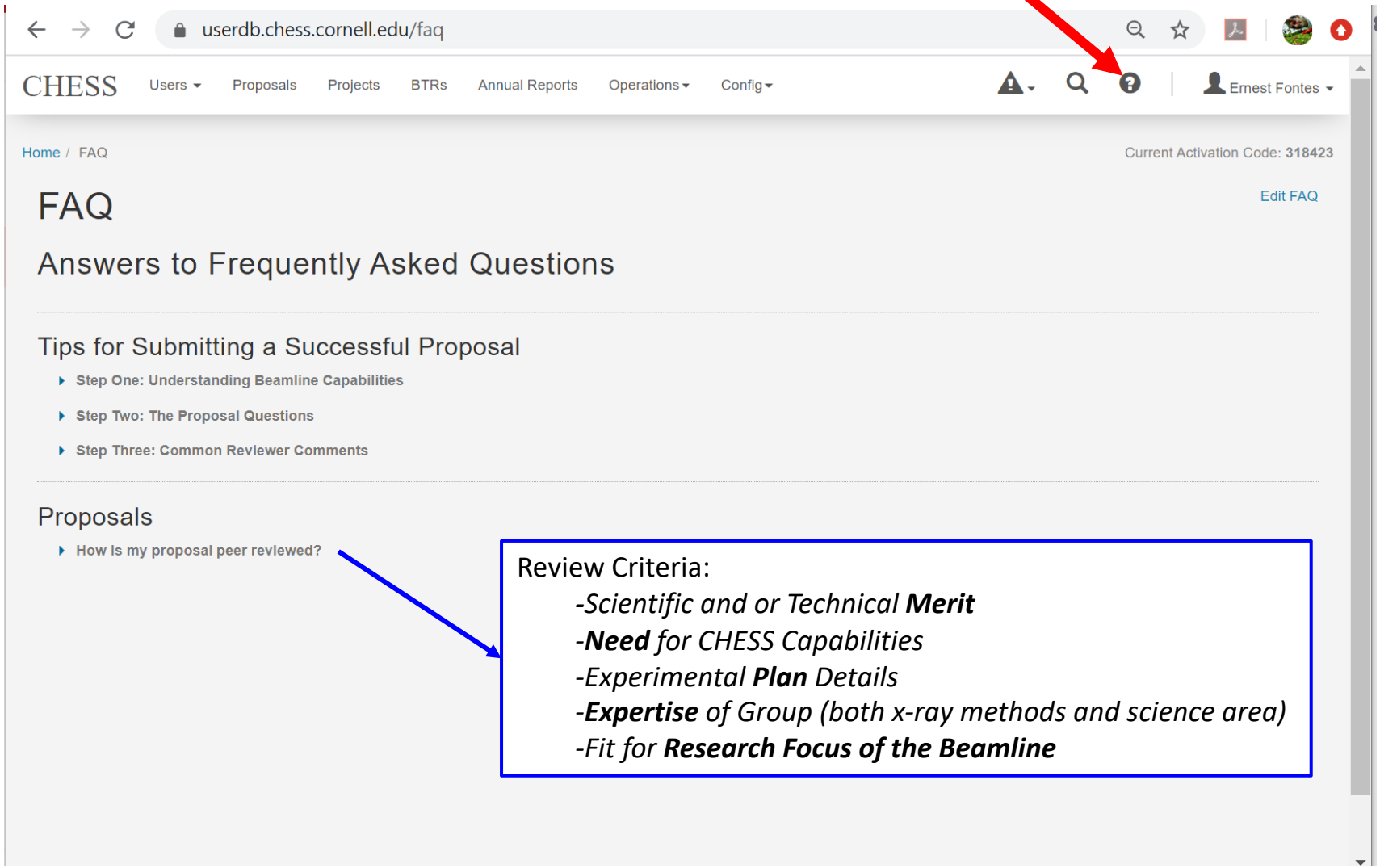

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Review Criteria:

- *-Scientific and or Technical **Merit***
- *-**Need** for CHESS Capabilities*
- *-Experimental **Plan** Details*
- *-**Expertise** of Group (both x-ray methods and science area)*
- *-Fit for **Research Focus of the Beamline***

"Know your audience, know what they're looking for…"

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| Beamline | Research Focus |
|---|---|
| BioSAXS & HP-Bio: Biological Small Angle X-ray Solution Scattering and High-Pressure Biology Beamline | Small Angle X-ray Solution Scattering (SAXS) on boiological systems;<br><br>High-pressure SAXS in diamond anvil cell<br><br>High-pressure studies in biophysics; |
| FlexX: Flexible Protein Crystallography and High-Pressure Biology Beamline | Macromolecular crystallography(MX);<br><br>Standard cryocrystallography,<br><br>Serial crystallography;<br><br>High-pressure crystallography in diamond anvil cell |
| FAST: Forming and Shaping Technology Beamline | Time-resolved studies of structural metals in manufacturing processes using high-energy diffraction microscopy and μ-computed tomography. |
| PIPOXS: Photon-In, Photon-Out X-ray Spectroscopy Beamline | X-ray spectroscopic studies of geometric and valence electronic structure in catalytic systems, functional materials and other systems. |
| QM$^2$: Q-Mapping for Quantum Materials Beamline | High-throughput characterization of quantum materials using resonant and non-resonant high energy scattering; High Dynamic Range Reciprocal Space Mapping (HDRM); Diffuse Scattering; etc. |
| FMB: Functional Materials Beamline | Time-resolved in-situ characterization of soft materials during processing (real time materials processing such as print manufacturing, solvent vapor annealing, and thermal annealing) using SAXS/WAXS. |
| SMB: Structural Materials Beamline | High-energy monochromatic and white-beam characterization of materials' structure and evolution across length-scales. |


…talk with beamline scientist(s)

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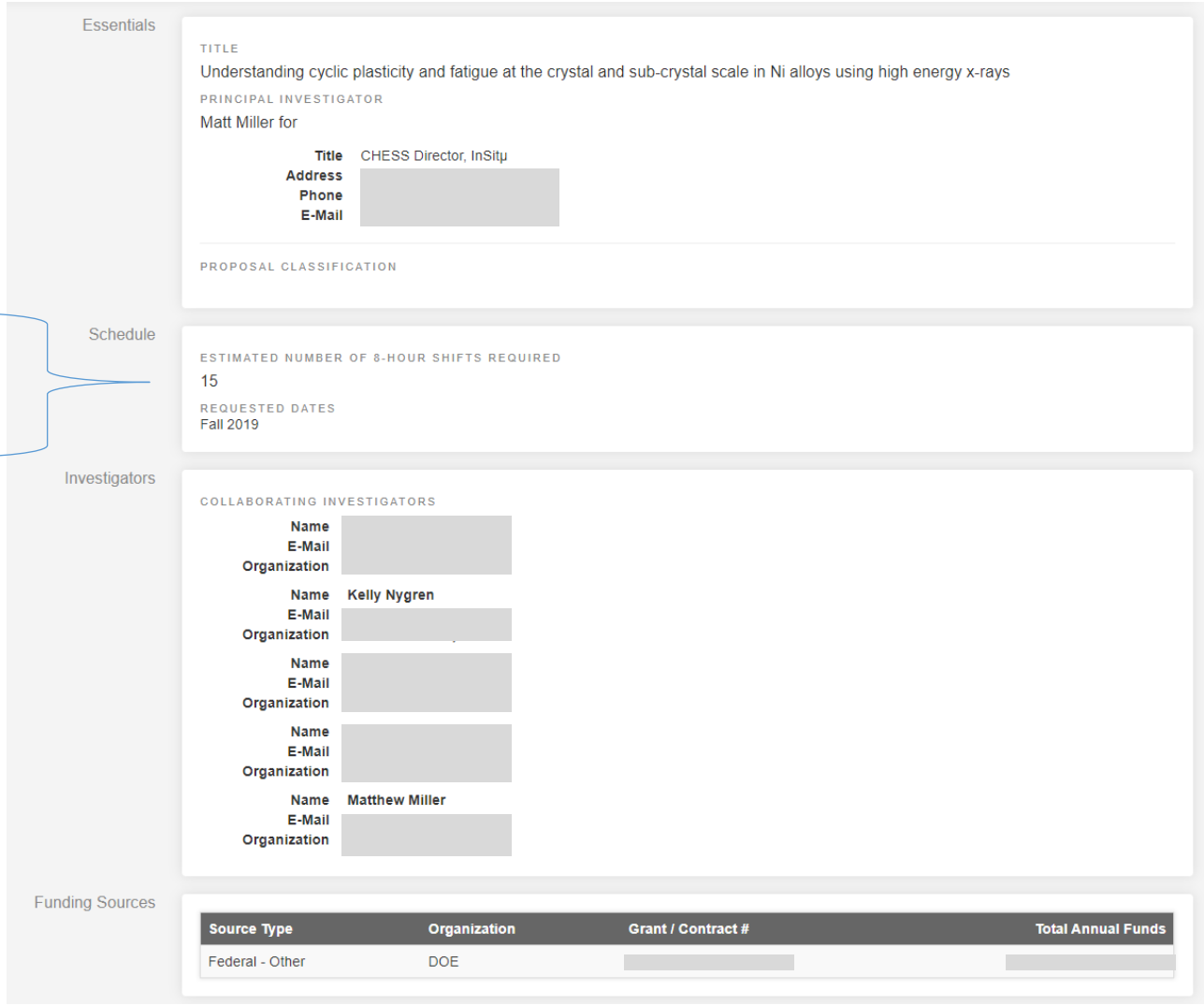

Essentials

TITLE

Understanding cyclic plasticity and fatigue at the crystal and sub-crystal scale in Ni alloys using high energy x-rays

PRINCIPAL INVESTIGATOR

Matt Miller for

Title CHESS Director, InSitμ
Address
Phone
E-Mail

PROPOSAL CLASSIFICATION

Schedule

ESTIMATED NUMBER OF 8-HOUR SHIFTS REQUIRED

15

REQUESTED DATES

Fall 2019

Investigators

COLLABORATING INVESTIGATORS

Name
E-Mail
Organization

Name Kelly Nygren
E-Mail
Organization

Name
E-Mail
Organization

Name
E-Mail
Organization

Name Matthew Miller
E-Mail
Organization

Funding Sources

| Source Type | Organization | Grant / Contract # | Total Annual Funds |
|---|---|---|---|
| Federal - Other | DOE | | |

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…talk with beamline scientist(s)

More details are welcomed

Must declare hazards or non-hazardous

Stations & Technique

EXPERIMENTAL TECHNIQUE

X-ray Diffraction

REQUESTED EXPERIMENTAL STATION(S)

| Station | Beamline | Techniques | |
|---|---|---|---|
| 3A | FAST | Near field high-energy diffraction microscopy, Far-field high-energy diffraction microscopy, µ-Computed tomography, Area detector-based powder diffraction | **Energy:** Energy Range: 30-70keV<br>**Spot Size:** |

Specimens & Materials

SPECIMENS AND MATERIALS TO BE STUDIED

Direct aged [redacted] superalloy [redacted] samples, [redacted] high yield strength which will allow for grain stress data collection.

DOCUMENTS

| Filename | Size | |
|---|---|---|
| [redacted].pdf | 57 KB | ⬇ |

Materials Declaration

Only non-hazardous materials on this Proposal

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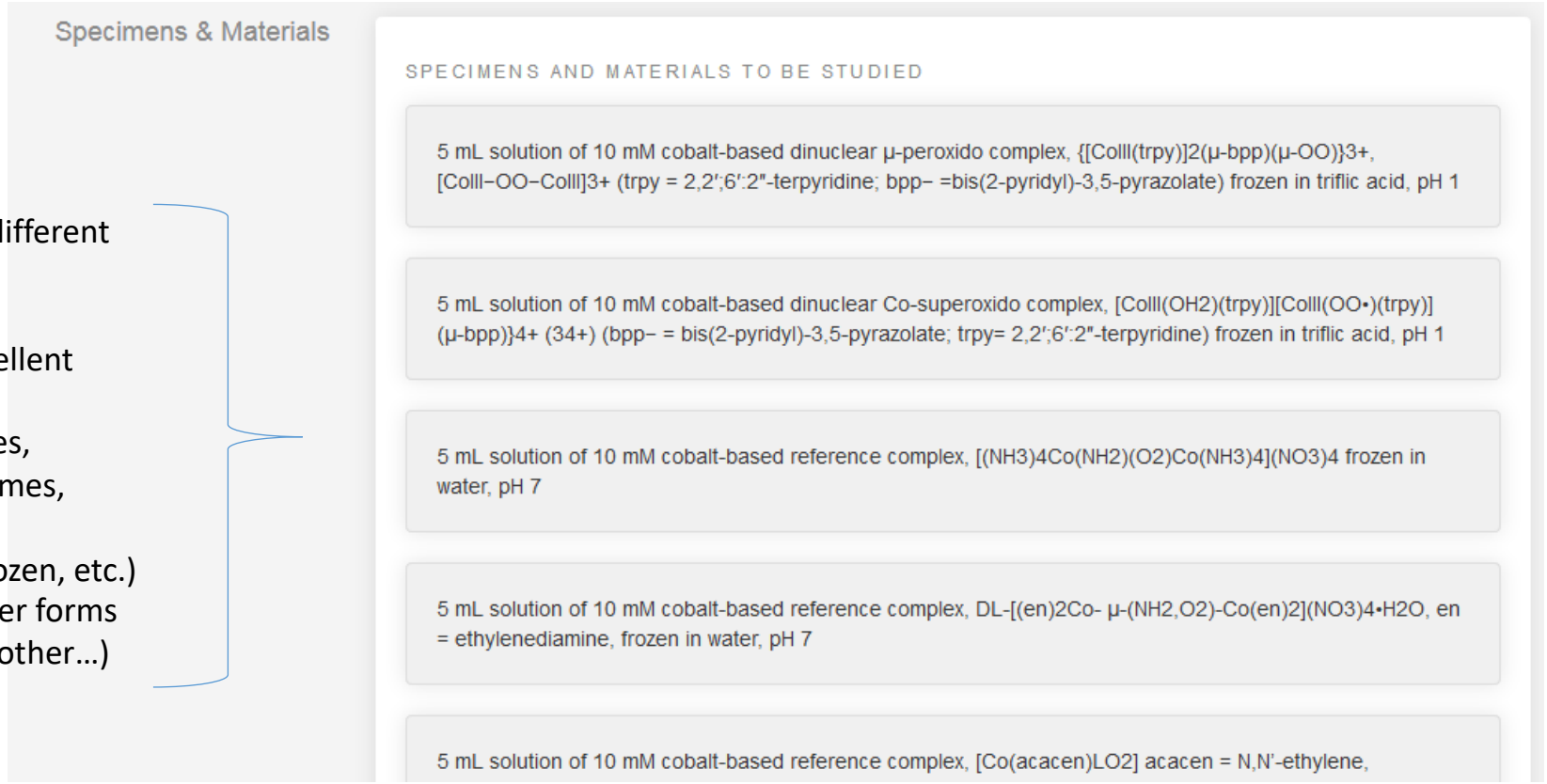
…a very different proposal.

Note excellent details:
-quantities,
-exact names,
-pH,
-state (frozen, etc.)
-SDS, other forms
-(upload other…)



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Excellent intro/summary

Really compelling conclusion!

Every scientist, lightsource and funding agency wants to enable "unique opportunities"

Scientific Justification

The aim of the work is two-fold: (1) to develop a fundamental understanding of low cycle fatigue (LCF) and the cyclic
lead to formation of slip bands, localizations, and crack initiation; and (2) perform a proof of concept for using HEDM
very high cycle fatigue (VHCF). These studies both require identifying "signatures" in the HEDM data relating to the p

1

Low cycle fatigue (LCF):
This work builds on previous LCF experiments on conducted at CHESS F2 as part of a previous versic

The proposed experiment will be performed in two parts. Part one consists of a uniaxial, in-situ, cyclic tension-compre

2

Very High Cycle Fatigue (VHCF):
Part two consists of performing ex-situ HEDM measurements on samples provided thro

Both parts of the experiment are one step in a unique opportunity to couple HEDM experiments with TriBeam EBSD

SCIENTIFIC FIELD

Engineering (Chemical/Civil/Electrical/Mechanical)

Details are good (I've said), but work to keep it easy to read.

Identify system of interest in the introduction

Important question(s) of interest

Challenges that x-rays might overcome

Critical question(s) will be answered

Great: provide figures and tables in separate document

## Scientific Justification

Determination of the structure and electronic configurations of the critical intermediates of water oxidation by catalytically active first row transition metal complexes is an important area of research for the development of cheap and efficient water oxidation catalysts (WOCS). The molecular cobalt complexes, hereby described, are the best systems for mechanistic studies due to the possibility to control via ligand design the stability of the reactive intermediates. This proposal particularly serves to resolve the valence orbital composition, ligand identities and metal-to-ligand bond strength of the key intermediate catalyzed by a cobalt peroxo-bridged WOC ({[CoIII(trpy)]2(μ-bpp)(μ-OO)}3+ (bpp- = bis(2-pyridyl)-3,5-pyrazolate; trpy = 2,2';6':2"- terpyridine)) for the 4 electron oxidation of water to O2 through Kβ XES (3p to 1s) and valence to core X-ray emission spectroscopy (VtC XES). Kβ XES mainlines represent the electric dipole allowed 3p to 1s transition after ionization of 1s electron, and VtC XES represents the ligand np/ns to metal 1s transitions providing a direct signature for the metal coordinated ligands (Fig. 1).[1-3 ]

Previous studies [4] have shown that oxidation of the peroxo derivative can generate a bridging or end-on superoxo complex which upon further oxidation generates O2 (Scheme 1). However, although the proposed superoxo complex in the rate limiting step of the reaction was characterized by Resonance Raman (RR), Electron Paramagnetic Resonance (EPR), X-ray absorption absorption near edge (XANES) and Extended X-ray absorption fine structure (EXAFS) spectroscopies, the exact arrangement of terminal O-O groups and determination of a bridging versus end-on geometrical arrangement is still unknown.

While RR was impactful in identifying the vibrational states of a superoxo and peroxo complex through isotopic labeling experiments (Fig.2), differentiation between a bridging and an end-on superoxo intermediate involving cleavage of the O-O peroxo bridge (scheme 1) was challenging due to the small energy difference between their Raman bands (theory predicts a shift of 1-3 cm-1 for related end-on and bridging superoxido complexes). Moreover, XANES analysis (Fig. 3) revealed a shift of around 0.4 eV in the rising energy edge of the superoxo vs the peroxo complex together with a more pronounced multiplet pre-edge feature showing an increased mixing of the valence 3d states with the ligand N/O p orbitals in the superoxo intermediate. However, although, theoretical XANES simulations of an end-on superoxo derivative reproduced the experimental energy shift and pre-edge features well, similar characteristics were noticed in the case of a bridging superoxo derivative (Fig.4). Lastly, EXAFS spectroscopy has limitations in distinguishing between light-atom scatterers and cannot easily differentiate between an end-on and a bridging complex.

Thus, while XANES and EXAFS represents the average contribution of the entire coordination environment displaying limited ligand sensitivity, use of Kβ mainline and VtC XES exhibit an increased sensitivity to the ligand identity, hybridization, protonation and metal-to-ligand bond strength providing the possibility to distinguish between a bridging and end-on superoxo intermediate. In this context, we note that VtC XES has previously been used to establish the extent of O-O and N-N bond activation in Mn[5] and Fe[6]complexes, respectively. Critical questions regarding the shortening of the O-O bond in the superoxo complex will be answered, and known peroxo and superoxo complexes (Table 1), previously documented in literature, will be used for comparisons. Theoretical XES simulations, currently in progress, will be used to support experimental results.

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Publications

PUBLICATIONS FROM PREVIOUS WORK DONE AT CHESS

(Since 2017)

Wielewski, E., Boyce, D. Park, J-S, Miller, M.P. and Dawson, P.R. A methodology to determine the elastic moduli of crystals by matching experimental and simulated lattice strain pole figures using discrete harmonics. Acta Materialia, 126:469 – 480, 2017.

Dawson,P.R., Boyce,D.E., Park,J-S, Wielewski, E. and Miller, M.P., Determining the strengths of hcp slip systems using harmonic analyses of lattice strain distributions, Acta Materialia, 14, 92–106, 2018.

Daniel Banco, Eric Miller, Matthew P. Miller, and Armand Beaudoin "Sparse modeling of space- and time-varying diffraction response of a progressively loaded aluminum alloy, " Materials Characterization, 145, 713 – 723, 2018.

Miller, M.P., Budrow, C., Long, T. and Obstalecki, M, "Understanding the evolving state of deforming polycrystals using synchrotron x-rays," proceedings of Risoe 2019, September 2019.

Might be OK to mention work not done at CHESS, but please distinguish it as such.

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Experimental Plan & Facility Needs

Really nice read here:

- Consistent format with scientific justification
- Gives explicit calculations as basis of estimate for time requested
- Conveys plan for efficient use of CHEES beamtime

…talk with beamline scientist(s)

EXPERIMENTAL PLAN

Summary:
4 days for in-situ cyclic testing and data collection (Part I),
1 day for ex-situ data collection (Part II).
The RAMS2 load frame will be required with the Dual Dexela detector configuratio
configuration for near-field measurements.

Part I: Low cycle fatigue (LCF):
Part one will test 2 undeformed samples. Each sample will require near-field meas
shape, and evolution. Far-field measurements will be collected inside the hysteres
the evolution of misorientation. The specific cycles will range from cycle 1 to cycle
hysteresis loops collections, scans will be performed at the maximum tensile and
Time calculation: ((near-field x 2 = 16 hours) + (far-field loops = 1 hours/ loop (10
= 6 hours) + (margin = 3 hours )) x 2 samples = 74 hours minimum

Part II: Very high cycle fatigue (VHCF):
Ex-situ near-field/ far-field measurements on 4 VHCF deformed samples.
Time calculation: ((near-field x 4 = 32 ) + (far-field x 4 = 20 minutes)) = 32 hours n

ADDITIONAL NEEDS

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Experimental Plan & Facility Needs

Experimental plan:

Expected results:

Team experience:

Additional Needs:

EXPERIMENTAL PLAN

Experimental plan: X-ray emission measurements will be measured at beamline C-1 of CHESS. All samples will be measured in ~10 mM concentrations in water or in powder form, and kept at the lowest temperature in the helium displex cryostat available at beamline C-1. The incident energy at CHESS will be set to around 8000 eV, using a pair of Mo/B4C multilayers7 for approximately 1% bandpass, with a storage ring electron current of 85 mA. A beam spot size of around 1mm x 2mm (Horizontal x Vertical) will be used.
The DAVES (Dual Array Valence Emission spectrometer) high resolution spectrometer developed by Finkelstein, Lyndaker and Krawczyk will be used. The flight path of the emitted X-rays will be filled with He gas with a helium balloon to minimize signal attenuation. Additionally, for all experiments, the XES spectrometer will be calibrated using the well known Kβ emission features of cobalt(II) and cobalt(II,III) oxides.

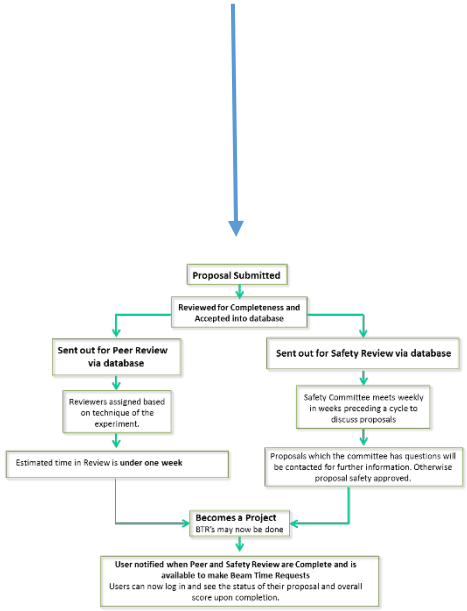
Expected Results: As mentioned in the scientific justification section, use of Kβ mainline and VtC XES exhibit an increased sensitivity to the ligand identity, hybridization, protonation and metal-to-ligand bond strength providing the possibility to distinguish between a bridging and end-on superoxo intermediate. In this context, we note that VtC XES has previously been used to establish the extent of O-O and N-N bond activation in Mn [5] and Fe[6]complexes, respectively. (Please see Figures_Ref_beamtime proposal for adequate references)

Team experience: Our team has participated in beamtimes at several of the world's synchrotron facilities including the Advanced Photon Source (APS) at Argonne, the Advanced Light Source at Berkeley, the Linac Coherent Light Source (LCLS) at Stanford SLAC, ALBA in Barcelona, Spain, as well as CHESS. Our experimental team consists of experts in the field of X-ray emission spectroscopy as well as an interdisciplinary background in X-ray absorption spectroscopy and synthetic chemistry.

ADDITIONAL NEEDS

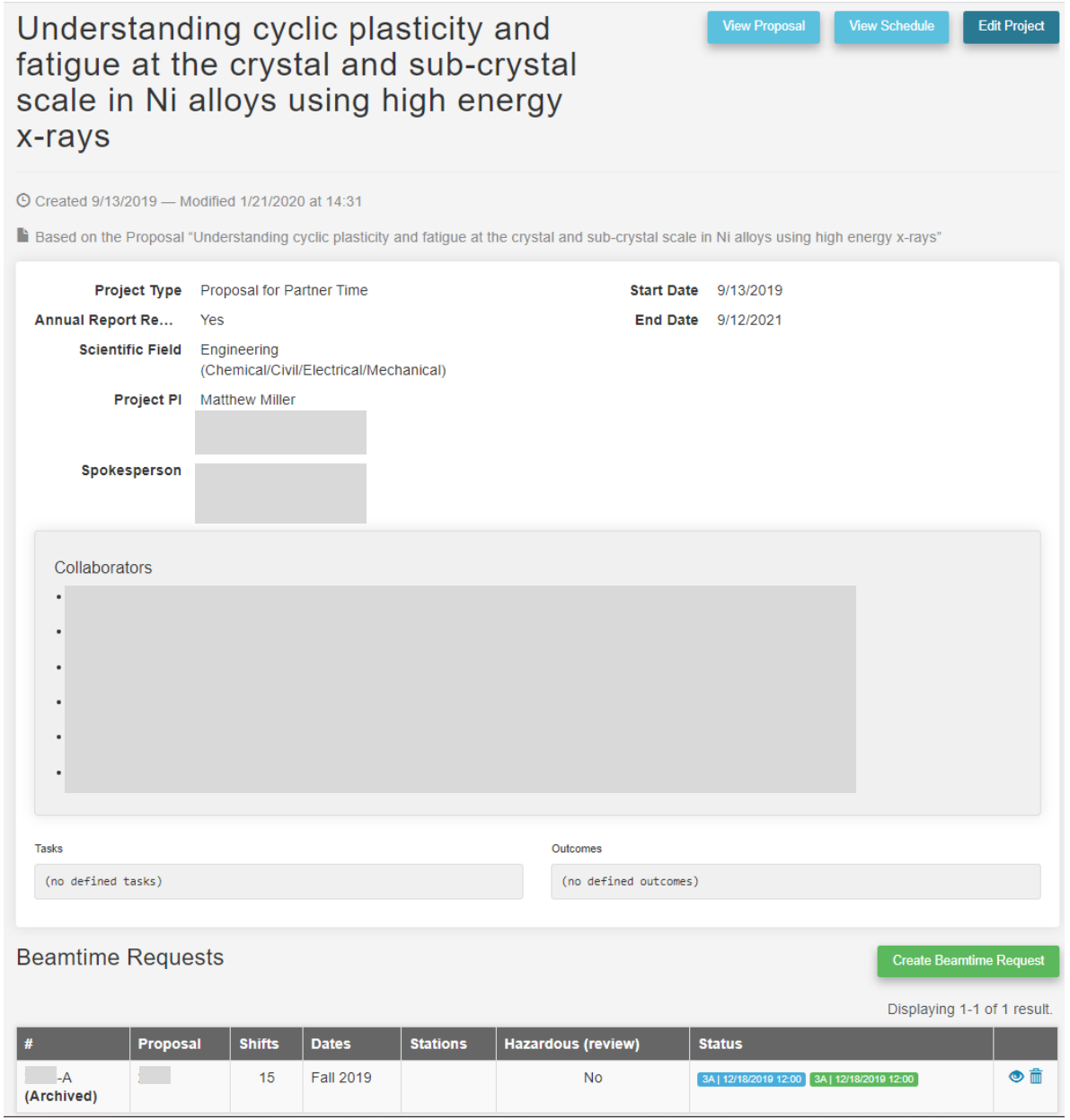
- Chemistry Room Ventilation Hood
- Bench Space in Chemistry Room
- -80 Freezer

# Your proposal is done…



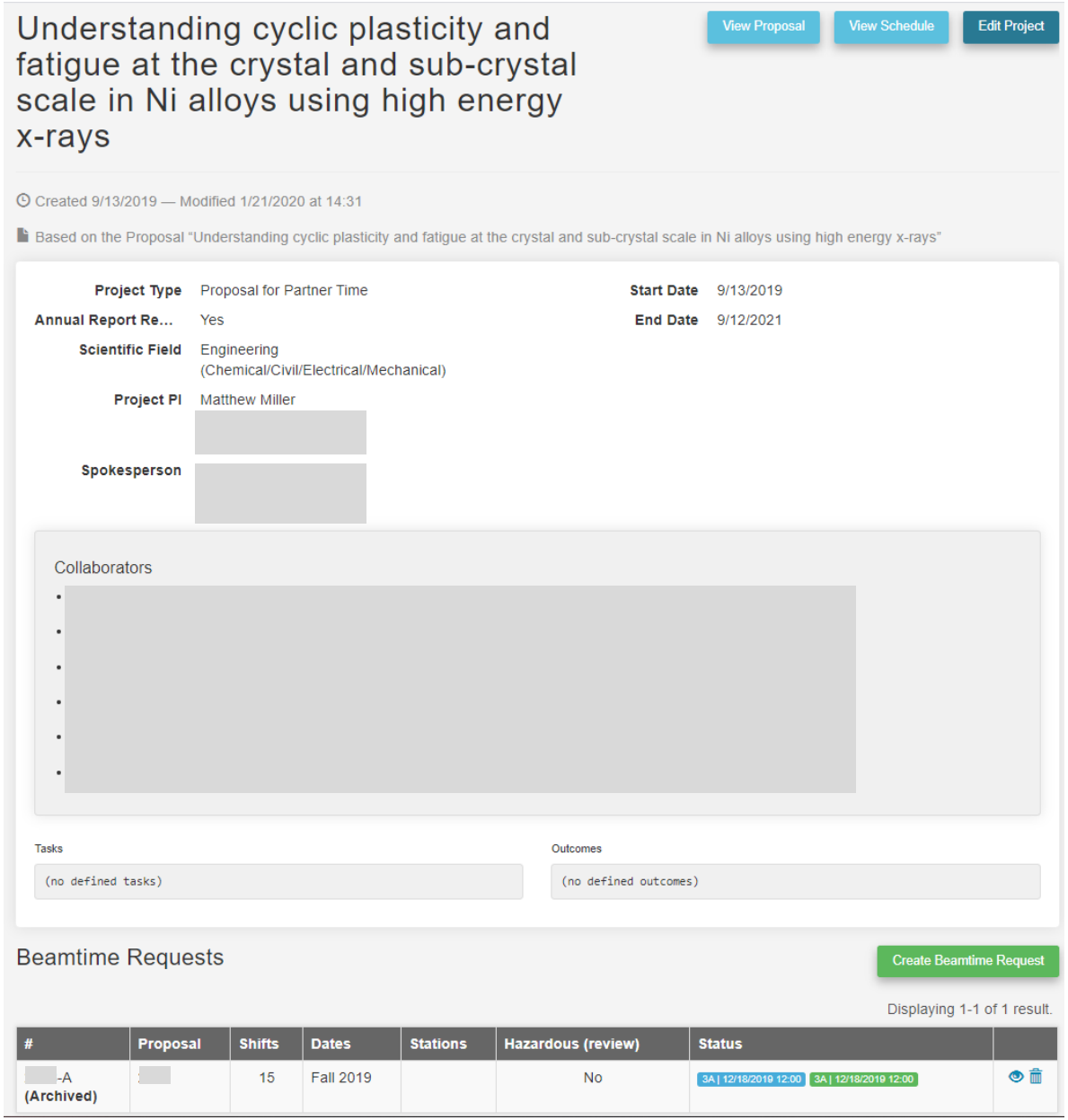
After grinding through the Safety and Peer Review, your proposal becomes a

# Project



Now you can schedule X-ray beamtime using “Beam Time Request”

BTR



CHESS
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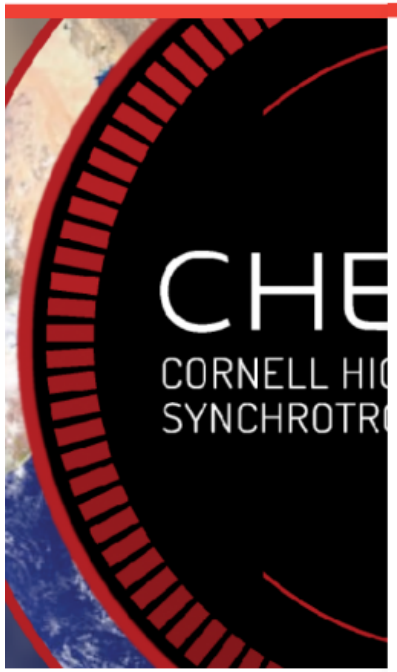
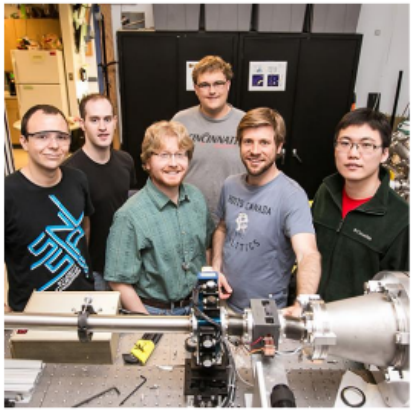
SCIENCE USERS PARTNERS PUBLIC ABOUT



## Users

CHESS users come from around the world and represent both academic and corporate communities.

- Beamline Directory
- User Portal
- Proposal Deadlines
- X-Ray Run Schedule
- Machine Status
- Acknowledgment
- ^ New User Guide
- ^ Resources
- ^ Safety
- Travel & Lodging
- Shipping
- 2020 Users' Meeting