Form B: IGEM-HERC Full Proposal Cover Sheet

Idaho State Board of Education

PROPOSAL NUMBER: (to be assigned by HERC)	TOTAL AMOUNT REQUESTED: $250,000

Proposal Track (select one):

- Initial Startup

TITLE OF PROPOSED PROJECT: **High Energy/Power Layered Oxide Cathode Materials for Sodium Ion Batteries**

SPECIFIC PROJECT FOCUS: Our patented layered transition metal oxide cathodes have demonstrated improved capacity and cycling stability in sodium-ion batteries for large-scale energy storage applications with > 70% cost savings. We aim to move up the current Technology Readiness Level (TRL) level (TRL 3) to TRL 5-6 through the Initial Startup track where we continue research and development in optimization of the materials composition, structure, and electrochemical properties as well as utilize industry processes to achieve highly reproducible and mass production for large-scale pouch cell manufacture.

PROJECT START DATE: 8/15/2025	PROJECT END DATE: 8/14/2027
NAME OF INSTITUTION: Boise State University	DEPARTMENT: Office of Sponsored Programs
ADDRESS: 1910 University Dr., Boise, ID, 83716	
E-MAIL ADDRESS: osp@boisestate.edu	PHONE NUMBER: 208-426-4420

	NAME:	TITLE:	SIGNATURE:
PROJECT DIRECTOR/PRINCIPAL INVESTIGATOR	Hui (Claire) Xiong	Professor	

Authorized Organizational Representative	NAME: Ella Christiansen Assistant Director, Pre-Award	SIGNATURE:

1. **Name of Primary Idaho public institution:** Boise State University
2. **Project Title: High Energy/Power Layered Oxide Cathode Materials for Sodium Ion Batteries**
3. **Name and Institution of Principal Investigator(s) and Key Personnel:** Dr. Hui (Claire) Xiong is the PI of this proposal, and she is a professor in the Micron School of Materials Science and Engineering at Boise State University. She joined Boise State University in 2012. Her current research focuses on the synthesis, characterization, and development of advanced functional materials for sustainable energy systems. Xiong has expertise and track records in materials synthesis and advanced characterizations of electrode materials for sodium ion batteries. She is one of the first researchers who reported sodium ion battery materials. She has over 12 years' experience in Na-ion batteries and published many high impact peer-reviewed articles on Na-ion battery materials. She holds two patents on electrode materials for Li-ion and Na-ion batteries.
4. **Total Amount requested:** $250,000
5. **Significance of project and project objectives**

Executive Summary: **<u>Our patented layered transition metal oxide cathodes have demonstrated improved capacity and cycling stability in sodium-ion batteries for large-scale energy storage applications with > 70% cost savings</u>**. We aim to move up the current Technology Readiness Level (TRL) level to TRL 5-6 through the **Initial Startup** track where we continue research and development and move towards scaling up our technology as shown in **Fig. 1**.

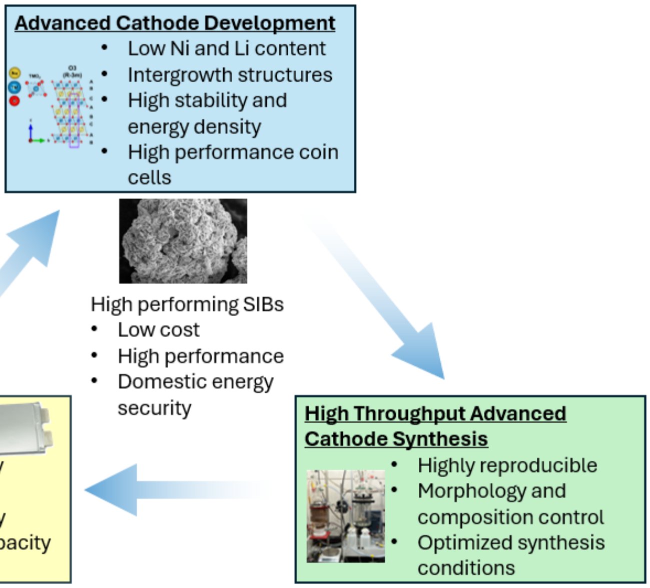


Figure 1: Overview of Project Plan.

<u>Value Propositions</u>:

1) Our advanced cathode uses **low-cost** materials for high performing electrical storage competing with established lithium-ion battery (LIB) cathode materials.
2) Our advanced cathode material will enable **improved safety** compared to current LIBs.
3) **High energy density and stability** for battery systems used for large-scale energy storage.
4) **Domestic energy security** will be achievable by utilizing non-critical materials readily available in the US.
5) **Utilization of current industry processes and facilities** with no adjustment offers a smoother transition to commercialization.

5.1. Background

Operating Principles of Sodium Ion Battery (SIB): SIBs have similar working principles as LIBs, in which Na ions are shuffled back and forth between anode and cathode repeatedly through insertion/extraction in host materials upon battery cycling (**Fig. 2**). During discharge, Na ions flow from anode (low voltage) to cathode (high voltage) within an ion-conducting and electron-impeding non-aqueous

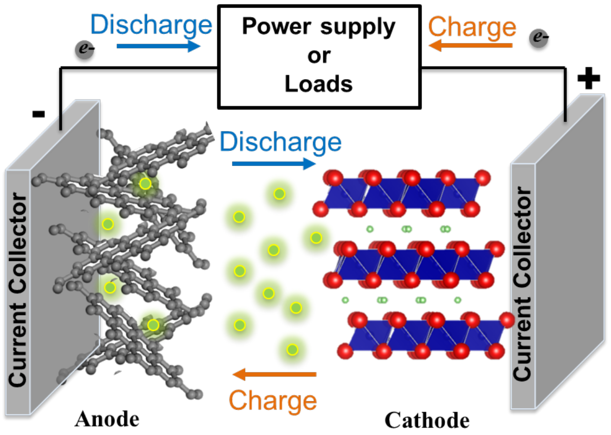


Figure 2. Schematic of the configuration and principle of rechargeable Na-ion batteries.

electrolyte and electrons flow through the external circuit to provide electricity to the load. During charge, Na ions are driven back from cathode to anode by the external power sources to store energy. Accompanied with Na ion insertion/extraction, the redox changes in metal ions to enable the charge balance within electrode materials during cycling.

Global Issue: Research and development (R&D) towards electrochemical energy storage (EES) technologies has been fueled by the ever-growing global population and the increased demand for electricity production. Estimates project nearly triple the amount of electricity used today will be required to power the world by 2050[1,2]. With artificial intelligence (AI) currently experiencing a significant surge in popularity across various industry sectors such as healthcare, finance, and customer service, energy storage systems like batteries are considered a key solution for data center operations to support the electric grid and improve resilience. The industry standard for EES systems are Li-ion batteries (LIBs) that deliver high energy density and power density reliably, but use costly and unsustainable (critical) elements/materials such as cobalt and graphite (**Fig. 3a**)[3]. These materials often have geopolitical issues with sourcing and refining which became exacerbated since 2020 during the pandemic when the US realized the large reliance on China for the manufacturing and supply of LIBs. Additionally, many reports have concluded that long term solutions for EES systems utilizing these elements will be challenged by diminishing resources[4,5]. Taking advantage of earth abundant elements in the next generation of EES systems is of great importance if we hope to keep up with increasing electricity demands[4].

Technological Solutions and Challenges: Due to the high abundance and low cost of sodium, sodium ion batteries (SIBs) have been considered an attractive alternative to LIBs for large-scale EES[3,6]. Current cathode solutions for enabling reliable and efficient SIBs include Prussian blue analogs (PBAs), polyanionic compounds, and layered transition metal oxides (LTMOs). **Of these materials, LTMOs are the most promising cathodes for SIBs due to their highly competitive energy/power densities and range of tunability (Fig. 3b)**[3,7–9]. PBAs are low cost, have good rate performance, and have an adjustable working voltage, but are unstable under normal working conditions. Polyanionic compounds have good thermal and cycling stability but suffer from low operating potentials and poor electronic conductivity. Compared to the other two candidates, LTMOs have the following benefits: (1) **higher volumetric energy density** compared to PBAs and polyanionic compounds; (2) **a broad range of tunability** from element substitution and **easy incorporation of earth abundant elements** (e.g., Fe, Mn, Ti, Mg, and Al) in its Na_xMO_2 (M= Ni, Mn, etc.) framework to afford **synergistic effects of different metals for improved performance;** (3) **a "drop-in" solution to the current LIB industrial practices** due to their fundamental similarities.

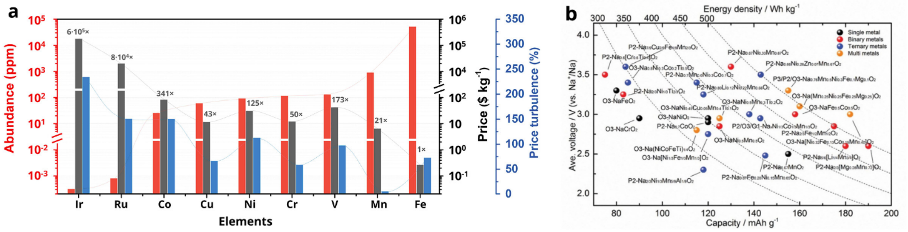


Figure 3: a) Comparison of abundance, price, and price turbulence of elements, and b) comparison of voltage, capacity, and energy density of various layered oxide sodium-ion cathode materials with different transition metals.

Challenges of LTMOs: Despite their promises, LTMOs have a major challenge to overcome for their practical applications which are the irreversible phase transitions that lead to capacity fade and structural instability, and poor kinetics.

Materials Engineering Solution: One strategy to limit the irreversible phase transitions occurring in LTMOs

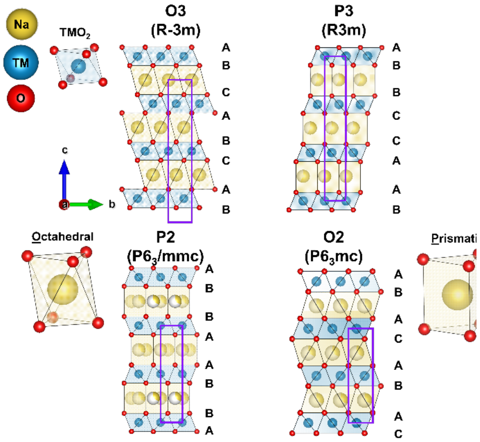


Figure 4: The common LTMO structures with the unit cells indicated by the purple lines.

and enable superior stability with improved capacity retention is to design cathode materials with intergrowth structures. The layered structures can be denoted by Delmas notation[10] with Na occupancy sites defined by an O or P for octahedral or prismatic sites, respectively, followed by a number designating periodicity. Different from the Li system, which can only form a select few layered structures with the first-row transition metals (TMs) such as Co, Ni, Mn, Ti due to the similarity in the ionic size of Li (0.76 Å) and TMs (0.5 – 0.7 Å), Na (1.02 Å) can form a variety of layered structures with almost all 1st row TMs. Among layered Na_xTMO_2, four types of stacking sequences are frequently seen: O3-, P3-, P2-, and O2-type. The most commonly observed forms of layered Na_xTMO_2 in their pristine state (before charge/discharge) are the O3, P2, and P3 structures (**Fig. 4**)[11].The stacking sequence can vary depending on Na concentration[10] during electrochemical cycling, which leads to complex phase transitions. These phase transitions are often partially or completely irreversible and may cause significant strain on the crystal structure, leading to poor reversibility in extended cycling. Compared to their lithium-ion counterparts, the sodium based LTMOs suffer from accelerated capacity decay due to phase transition because of the larger size of the Na^+ ion compared to the Li^+ ion. The study of methods to mitigate the detrimental phase transitions which occur during charge/discharge is of great importance for the development of practical sodium ion batteries. One strategy is to design intergrowth structures that integrate multiple phases at the atomic level through *in situ* synthesis methods. The intergrowth structure can provide significant enhancements to the electrochemical properties by combining the benefits of individual phases while mitigating their weaknesses. The intergrowth structure could potentially induce chemomechanical coupling at the phase interface that can mitigate mechanical stress occurring during cycling. The strategy of engineering cathode materials with intergrowth structures allows for the possibility of a broad wealth of discovery and innovation to achieve higher performing advanced cathode materials at low costs.

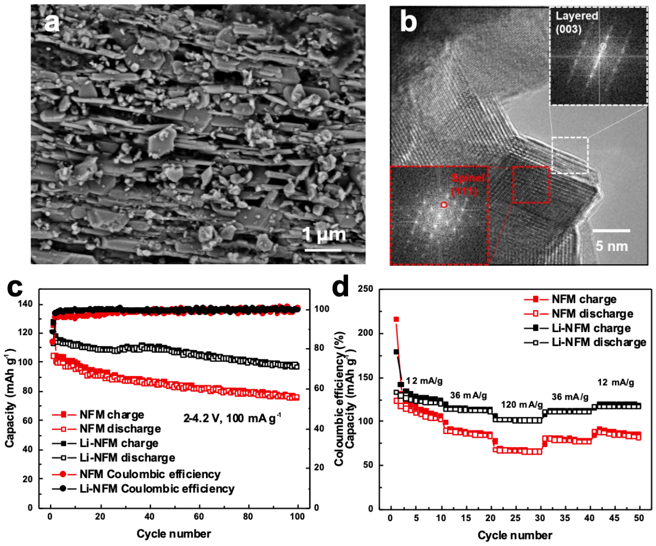


Figure 5: a) and b) Scanning electron microscope and transmission electron microscope images of LS-NFM pristine material; c) cycling performance and d) rate capability of LS-NFM and NFM cathodes.

5.2. Innovative Approach

Our patented low-cost layered transition metal oxide cathode $Na_xLi_yNi_{0.4}Fe_{0.2}Mn_{0.4}O_2$ (LS-NFM) (**Fig. 5a** and **5b**)*, can provide enhanced power performance and cycling stability for large-scale energy storage by preventing structural degradation and enhancing Na-ion mobility*[12,13]**.** The Li-substituted layered-tunneled $Na(Ni_xFe_yMn_z)O_2$ cathode material utilizes Li doping and layered-tunneled intergrowth phase design strategies to combat structural and cycling instability.

a) Li substitution has been reported to improve overall electrochemical performance of layered cathodes[14]. Li ions have a similar ionic radius to transition metals whereby favoring transition metal sites. By ensuring the stoichiometric ratio of Na over the sum of transition metals and Li equals 1, intergrowth structures (layered-tunneled) can be made to suppress undesirable phase transitions in LS-NFM at high voltages.

b) The cathode demonstrates a 94% layered and 6% spinel intergrowth structure with great structural compatibility and connectivity. The intergrown structures allow for the synergistic effects of two or more phases to obtain improved performance[15].

c) The intergrown structure LS-NFM exhibits high reversible capacity ~ 125 mA h/g at a rate of 12 mA/g and high capacity retention of ~ 86% after 100 cycles with superior rate capability (**Fig. 5c** and **5d**)[13].

5.3. Objectives

Our patented LS-NFM cathode demonstrates a proof of concept that can offer many benefits as a novel cathode material for SIBs, but structure and performance can still be improved with further research and development. The following objectives have been chosen to enhance our LS-NFM cathode material performance, lower initial material costs, and enable scaling up manufacturing to reach TRL 5/6.

Objective 1. *Research and development in reducing the amounts of Li and Ni in current LS-NFM with optimization of the transition metal composition, dopants (Al, Mg, Ti, and Nb) and crystal structure to realize lower cost, superior energy density and cycling stability.*

Objective 2*. Additional research towards optimization of microstructures and morphologies developed further via a coprecipitation method using our custom-made continuous stirred tank reactor (CSTR) with increased volume to improve batches at a large scale with high reproducibility.*

Objective 3. *Assembly of pouch-cell full cells with hard carbon anode and an advanced electrolyte for electrochemical testing and optimization of performance for large scale production.*

All electrochemical testing will be completed according to practical conditions set by the Department of Energy (DOE) and the US Advanced Battery Consortium (USABC) protocols. These objectives are specifically laid out to **enable a cathode material for SIBs that provides higher capacity, stability, safety, and cost savings overall, leading to a more competitive viable product for scaling up**. **Table 1** in the project management section provides a timeline for completion of the objectives outlined above.

6. Detailed project plan and timeline

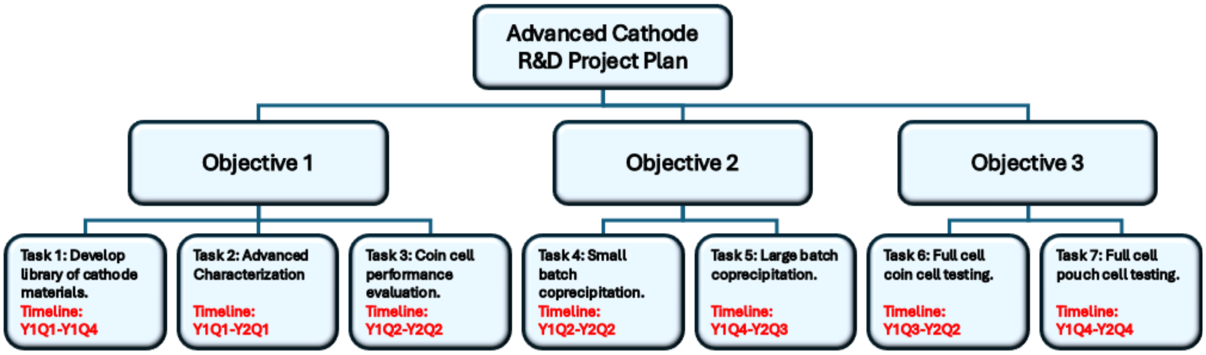


Figure 6: Project plan map including objectives and relative tasks associated with each objective.

6.1. Project Plan

6.1.1 Objective 1. *Research and development in reducing the amounts of Li and Ni in current LS-NFM with optimization of the transition metal composition, dopants (Al, Mg, Ti, and Nb) and crystal structure to realize lower cost, superior energy density and cycling stability.*

Our LS-NFM material provides an intergrowth structure utilizing Li, Na, Ni, Fe, and Mn in its composition. Li and Ni are great TMs for electrochemical performance but inevitably increase materials costs. Limitation of Li and Ni will allow for a more cost-effective cathode material, though challenges with high Fe and Mn content may become more apparent. Cathode materials with high Fe and Mn content have undesirable irreversible phase transformations at high voltages leading to degradation if not properly designed due to TM dissolution. By altering Na content and introducing dopants, intergrowth structures can be formed, suppressing irreversible phase transformations. To complete this work, a systematic study on the cathode composition will be completed to limit Li and Ni content within the material with increased Fe and Mn. The cathode composition will be altered through Fe:Mn ratios ranging from 1:1 to 1:4. These studies will be completed by utilizing our LS-NFM cathode as a baseline and varying the composition. This will also include investigation of dopants such as Al, Mg, Ti, and Nb (0.5% - 10%), which are known to be beneficial for high capacity and stability. Na content will be varied based on the charge compensation of the transition metals. Calcination/sintering conditions as well as choice of heating medium will be optimized for reproducibility ranging from temperatures at 500 – 1000 °C. Material characterization will be completed with X-ray diffraction (XRD), inductively coupled plasma mass spectrometry (ICPMS), and finally X-ray absorption spectroscopy (XAS) to understand advanced cathode structure, composition, and charge compensation, respectively. Finally, electrochemical performance with optimized laminates and advanced electrolytes will be evaluated using standard testing procedures at the coin cell level to study the capacity and stability of the advanced cathode materials. All cells at this stage will be completed in a half-cell setup vs Na metal to determine baseline performance metrics. Galvanostatic cycling, electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV), and galvanostatic intermittent titration technique (GITT) will be utilized to determine the samples' charge storage capacity and transport kinetics. Cells will be cycled at potential windows from 2V to different cutoff voltages (4V, 4.2V, and 4.4V) to determine their stability windows. Studies to determine cycle life will be completed with 3 – 10 initial cycles at C/10 rate followed by C/3 rate reaching up to 1000 cycles with incremental checkpoints every 100 – 250 cycles to track progress. The electrochemical testing will allow us to determine appropriate voltage windows, ideal cycling protocols, expected failure points, diffusion properties, and optimal electrolytes. Objective 1 will be broken up into 3 tasks: **Task 1**: Develop a vast library of cathode compositions limiting Li and Ni content with varied Na content and dopants for high capacity, enhanced stability, and safe cathode materials; **Task 2**: Characterize the physiochemical properties of the materials to determine structure, composition, and charge compensation; **Task 3**: Electrochemically evaluate the performance of the advanced cathode materials screened from Task 1 & 2.

Preliminary work: We have made an intergrowth cathode material $Na_wM_xFe_yMn_zO_2$ (for IP protection, M, w, x, y, and z are not defined here) and cycled it in a half cell vs Na. Results show higher capacity than our previous LS-NFM material with improved stability over 25 cycles at about 150 mAh/g (**Fig. 7a** and **7b**). When compared to our LS-NFM baseline, this material has no Li or Ni content and was made via a coprecipitation method that has yet to be optimized. Our preliminary work demonstrates significant promise of the cathode materials with the capability to improve further by R&D in this objective.

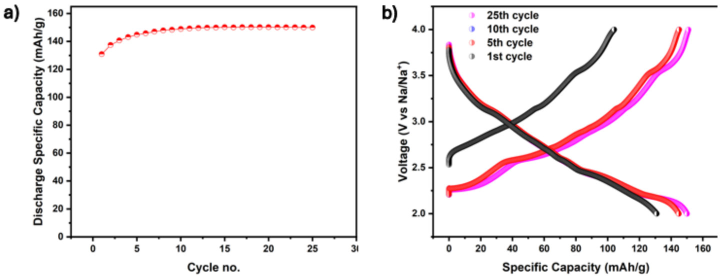


Figure 7: Preliminary work with intergrowth cathode material $Na_wM_xFe_yMn_zO_2$ with no Li or Ni.

Expected outcomes: Cathodes with ideal compositions that have high performance at the coin cell level will be identified. Though coin cell level performance will not exactly scale to pouch level performance it is a representative benchmark that is valued in the battery industry. Additionally, coin cell results will allow for high impact work to be published and IP to be filed.

6.1.2 Objective 2*. Additional research towards optimization of microstructures and morphologies developed further via a coprecipitation method using our custom-made CSTR with increased volume to improve batches at a large scale with high reproducibility.*

The preferred method for large-scale synthesis of LTMO hydroxide precursors is coprecipitation. Coprecipitation is a well-established method utilized for synthesis of commercial lithium-ion electrode materials today such as lithium nickel manganese cobalt oxides (NMC) and can be utilized to produce SIB cathode materials. It offers a simple scalable method for providing homogenous mixing at the atomic scale and tunability over product yield, composition, and particle morphology[16,17]. We will complete small batch coprecipitations (<100 g) for cathode compositions screened in ***Objective 1*** to produce high product yield, desired particle morphology, and electrochemical performance. Adjustments to reactor stirring rate, pH level, chelating agent, salt reagents, precipitating agent, and addition rate will be made for ideal secondary particle morphology control and desired composition. Stir rate will be tested between 400 – 1000 rpms with focus on higher rpm levels as they promote more uniform and spherical secondary particles. Different chelating agents (ammonia, oxalate, citrate, etc.) will be experimented with a focus on compatibility. With high Fe and Mn content, ammonia often forms impurities with Fe and is not ideal for these compositions. With this consideration in mind, alternative chelating agents will be prioritized. Finally, larger scale batches

(>100 g) will be completed with optimizations from small batch reactions. Additional adjustments to account for the larger reactor size such as pH level, addition rate, and stirring rate will have to be made for optimization of cathode precursor synthesis. Physical characterization of particles produced will be completed throughout *Objective 2* with XRD, scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS) to determine structure, morphology, and TM distribution, respectively. Objective 2 will be split into 2 phases: **Task 4**: Complete small batch coprecipitations on cathode compositions found in Objective 1 to determine conditions for optimized product yields, correct compositions, and varied particle morphologies. Modifications for stir rate, addition rate, pH, chelating agent, salt reagents, and precipitating agent will also be made at this phase; **Task 5**: Scale up coprecipitation synthesis of precursors based on the optimizations from Task 4 for highly reproducible production of advanced cathode materials.

Preliminary work: Our custom large-scale reactor has been properly set up and tested on a well-known sample batch as a controlled test seen in **Fig. 8a**. The morphology of the secondary particles of the sample by large-scale synthesis is shown to be "spherical-like" (**Fig. 8b**) during our control test batch previously run. Though this first attempted batch did not produce the ideal perfect sphere morphology we targeted, it is a good proof-of-concept that our large batch reactor works. The structure and composition of this batch came out consistent with what we were expecting, and the morphology seems to be relatively consistent from particle to particle, seen in **Fig. 8c**.

Expected outcomes: Work towards optimizing the conditions for ideal secondary particles will still need to be improved upon, though as can be seen in **Fig. 8b** and **8c** the morphology is already relatively close. Proper testing with small batches will allow for smoother transition to large batch production. We expect to be able to obtain spherical morphology cathode precursors with optimized conditions that are reproducible across multiple batches. Additionally, our custom-made reactor setup allows for future optimization and modification based on our needs. Therefore, if situations arise where we may need to adjust certain parameters, these changes should be made with relative ease.

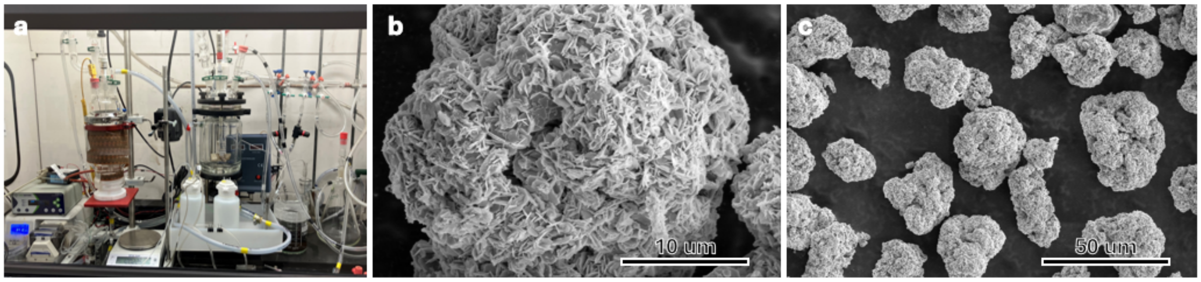


Figure 8: Preliminary work with large-scale synthesis (>100 g). a) large scale reactor setup. b, c) Large scale batch produced precursors showing consistent spherical-like morphology.

6.1.3 Objective 3. *Assembly of pouch-cell full cells with hard carbon anode and an advanced electrolyte for electrochemical testing and optimization of performance for large scale production.*

To gain realistic data on our material performance, full cells will be made with hard carbon as the anode material and our advanced LTMO as the cathode. Full cells will enable testing to be completed to mimic testing parameters and conditions that would normally be seen when operating a battery energy storage system (BESS). The full cells will initially be tested on coin cells and optimization for N:P ratio (ratio of negative electrode to positive electrode) will be determined. To start, anode and cathode half cells will initially be assembled to determine capacities. Ideal N:P ratio is essential for high-capacity full cells. Subsequently, pouch cells with varying N:P ratios will be assembled and tested. The pouch cells will be electrochemically tested in the same manner as completed on the half cells in ***Objective 1***. Pouch cell assembly is currently being set up in our lab, and we anticipate obtaining our first operating pouch cell by this summer 2025. Objective 3 will be split into 2 parts: **Task 6**: Assemble full cells at the coin cell level to determine proper N:P ratio; **Task 7**: Assemble full cells at the pouch cell level and complete electrochemical studies mimicking those completed in Task 3.

Expected outcomes: Following this step, we expect a minimum viable product to be completed in our lab that can be used as a tool to garner further funding and gain partnerships with individuals in the private and public sector. The electrochemical data and N:P ratio data we obtain will allow for future optimization of materials for second generation deployment of our optimized cathode materials. We will collaborate with

Dr. Pete Barnes at Idaho National Laboratory (INL), Energy Storage & Electric Transportation Department, to evaluate the pouch cell performance following DOE and USABC protocols (see Letter of Collaboration in the appendix).

6.2. Plan to move along the TRL spectrum

Our patented work has already achieved proof of concept and has completed the research stage on the TRL continuum (TRL 3) where our work has been completed at the coin cell level. We are currently in the development phase at TRL 4 where we need to continue to do bench scale research and move towards pilot scale testing. With funding support from IGEM HERC we believe we can reach TRL 5/6 where we can begin producing/testing on an application scale in realistic environments. Optimization of the material synthesis process as well as material performance will enable us for a more competitive edge against benchmark cathodes used in LIBs (e.g., $LiFePO_4$). Further testing as pouch cell full cells with hard carbon anodes will provide realistic use parameters for advancing TRL and will produce a minimum viable product.

7. POTENTIAL ECONOMIC IMPACT

7.1. Commercial Viability

Technological Advantage: Our patented LS-NFM is synthesized using a coprecipitation process commonly used commercially for today's leading LIB cathodes. This allows for an easy "drop-in" solution that can be made at scale without major modifications for cost savings. Additionally, phase transformations leading to structural degradation are suppressed by our novel structures enabling high performing batteries with improved cycling stability required for large-scale energy storage. This applied research was designed from its inception to use sustainable elements and materials to solve the demand for electrification and support the transition away from fossil fuel and non-domestic EES dependence.

Cost Advantage Analysis: Our patented LS-NFM cathode provides a significant cost advantage when compared to LIB cathodes. Especially with the rise in demand for electric vehicles (EVs), price instability for Ni, Co, Li and graphite in LIBs becomes more severe. LIBs and SIBs differ dramatically in starting material price where **Li_2CO_3 ($78/kg) is 190 times more expensive than Na_2CO_3 ($0.4/kg)**[3]. When comparing a high-performance LIB cathode such as NMC622 to our SIB LS-NFM, **we see a > 70% cost savings potential** based on our preliminary cost analysis. Additional research to limit the Li and Ni content will enable an even greater yield for savings potential with improved performance. This price difference will enable our product to be highly competitive with current outgoing technology.

7.2. Economic Impact for Idaho

This work is the result of fundamental research on energy storage technology that was completed at Boise State University directly aligning with the ID HERC funding priorities. The project allows for translational research that can move past ideas and demonstrate a viable product where if supported can change the energy landscape. With IGEM HERC's support the foundation for this technology will be established, enabling Idaho to stand as a leader of the sodium-ion battery industry**.** Our innovative technology and project align with all four goals outlined in the HERC strategic plan for 2023-2027. Support for our innovation will enable further research to be completed at BSU with collaboration from Idaho National Laboratory (INL) to verify our results independently at their state-of-the-art testing facility. If funded, the project will help take our proven concept to scale up for commercial products. As of this proposal date, there is only one battery manufacturer headquartered in Idaho with a focus on current LIB technology. With IGEM HERC support we hope to contribute to the economic development of Idaho with jobs, expertise, and capital through our novel SIB technology. IGEM HERC support would allow for new job opportunities and provide a hub in Idaho for energy storage to be established domestically, analogous to Micron with the semiconductor industry. With continued success, additional students from Idaho institutions will be recruited to bring this product to the market and a workforce pipeline on battery technology in Idaho to be established.

7.3 Path to Profitability

Market Analysis/NSF ICorps Participation: Our team participated in a 4-week entrepreneurial training program in the NSF ICorps at Boise State University as part of the Desert and Pacific Region. During the program our team interviewed more than 22 customers targeting two different customer segments: utility providers (Idaho Power) and battery system manufacturers (Prevalon, Powin, etc.). Our team found that our major beachhead would be battery manufacturers who source materials and are involved with providing battery energy storage systems (BESS) to major utility providers such as Idaho Power. Interviews with decision makers allowed us to determine that their primary stressor was cost followed by cell performance and safety. Our current strategy towards profitability will be to 1) maximize our cost savings and 2) provide material performance and safety that can rival current industry cathodes.

During the interviews we also found the consensus being that there is a dramatic need for batteries in large scale utility energy storage applications. Idaho Power explained to us that they use their Integrated Resource Plan (IRP) to predict use and demand of resources within a 20-year projection[18]. The IRP is prepared through a model every 2 years and was last updated in 2023 projecting use from 2024 – 2043. According to the 2023 IRP, Idaho Power's demand is expected to grow dramatically with increases of 80 MW per year or 1500 MW per decade. They estimated 1453 MW of battery storage is required to meet the demand caused in large part by heat waves and large population influx. Most recently Idaho Power is building two BESS, an 80 MW battery energy storage system in Owyhee County and a 40 MW battery energy storage system in Elmore County with plans to expand storage capacity in the near future.

Lastly, in our interviews with battery system manufacturers, they explained to us that technology adoption is relatively easy so long as the economics involved behind the switch are beneficial. Our technology is already designed in such a way that materials can be made in the same fashion as current outgoing LIB cathode materials. Additionally, during a discussion with Idaho Power we discovered that the technology (SIB vs LIB) used in the BESS is of almost no concern compared to the cost and performance specs. With our preliminary estimated cost savings, our targeted performance objectives, and our easily adopted technological design, we believe adoption of our technology will occur with ease. Our targeted objectives outlined above align well with our strategy towards profitability and continued innovation will allow for a minimum viable product to assist in moving up in TRL.

Strategic Partnerships: Additionally, during our interviews completed for the NSF ICorps we found that scaling from pilot scale to production scale can take many years and require large financial investment. Our strategy to expedite this process and streamline our work will be to partner with existing established companies to mitigate risk and lessen the learning curve to reach large scale production and profitability sooner. Our technology is perfectly aligned to be adapted into current industrial practices for producing cathode materials and our biggest limitation will be acquiring expensive equipment, learning the nuances of scaling with a reliable supply chain, and building efficient checkpoints for highly effective quality control. Larger established companies will already be perfectly versed for handling these challenges. In the case where partnerships with companies cannot be made, we have already established collaboration with INL, Energy Storage & Electric Transportation Department, who houses the Battery Test Center (BTC) to routinely evaluate commercial batteries. In addition, Pacific Northwest National Laboratory (PNNL) and Argonne National Laboratory (ANL) both have battery facilities that are purpose built to bridge the gap between battery research and commercial-scale battery development. PNNL has the Advanced Battery Facility and The Innovation Center for Battery500 Consortium; ANL has the Argonne Collaborative Center for Energy Storage Science. These facilities do come with user fees, but the equipment used is representative of commercial scale production and testing, and the staff are all composed of highly knowledgeable individuals with extensive experience in battery scaling. Completion of the milestones below to achieve a TRL level of 5/6 will enable the development of a minimum viable product for negotiations on partnerships to occur.

8. Criteria for measuring success

Success for this work will be measured through milestone achievements. These milestones will represent significant points within the anticipated timeline that will provide additional strength on the path to profitability stated above. The achievement of these milestones will not only be used to measure success but also to pursue additional funding and collaboration.

Milestone 1: Develop a collection of optimized high performance cathode materials with low Li and Ni contents, of various compositions, microstructures, and morphologies. Demonstrate reproducibility with <3% composition variation among large scale batches with optimized spherical secondary particles and >80% product yield. Achievement of milestone 1 is expected to occur following ***Objective 1*** and ***2***.

Milestone 2: Analysis of the electrochemical performance of cathode compositions with an advanced electrolyte, optimized cycling conditions, and evaluated pouch cell performance with standardized testing procedures resulting in a minimum viable product. Performance metrics for electrochemical cells with >500 Wh/kg (cathode basis) and >160 Wh/kg (full cell basis) and exceeding 1000 cycles with <20% capacity fade. Demonstration of full-cell pouch cell with cell capacity $\geq$ 0.1 Ah. Achievement of milestone 2 is expected to occur following ***Objective 3***.

9. Anticipated development challenges/barriers

Though the advancement of SIB cathodes has come a long way and offers much promise, there exists several challenges that must be overcome to achieve commercialization. Reactivity with moisture is a major concern with advanced cathode materials for SIBs. Water and carbon dioxide in the air will readily react with our cathode materials without extra precautions of utilizing inert gases such as nitrogen and argon. Our lab has currently operated at a small batch level when dealing with this undesirable reactivity and large batch production may introduce new factors when continuing to deal with it. Though this may seem like a complicated issue to deal with, our prior experience has prepared us to adapt to these issues and identify when oxidation of our cathode material is occurring. In the case where we cannot adapt with our current setup, dry chambers/rooms are available and are commonly used in industry to mitigate these undesirable reactions.

An additional concern that may arise when scaling from pilot to commercial scale is the inability to create partnerships to mitigate some of the risk and time that it takes to deploy battery materials. If this may occur, further funding will be applied to such as SBIR/STTR programs in NSF and DOE or venture capital funding to pursue scaling at the national laboratory facilities as stated in the path to profitability section.

Lastly, our project is in a particularly advantageous situation where PI Xiong is one of the first scientists investigating advanced intergrowth SIB cathodes and has more than 12 years of experience developing high performing battery materials. Much of the academic research completed at PI Xiong's lab investigates the fundamental problems plaguing these types of materials. Our prior knowledge and experience will dramatically improve our chances at problem solving any research and developmental issues we may encounter along the way.

10. Budget (See Form D)

11. Budget Justification

LINE ITEM REQUEST	JUSTIFICATION	TOTAL REQUEST
Personnel (salary and fringe)		
PI (Xiong)	Summary salary ($12,300 + fringe) for 0.75 mo	$32,400
Research Scientist (TBD)	Salary ($70,000 + fringe) for 1 year for a Ph.D. level materials scientist to direct the proposed work	$98,600
Ph.D. student (Cyrus Koroni)	Salary ($32,000 + fringe) for 1 year to support the project. Cyrus is a senior graduate student in MSE, has participated in the 4-week NSF ICorp program and has extensive experience in electrode materials synthesis and characterization	$33,800
Undergraduate student (TBD)	Salary ($7,200 + fringe) for 2 years to assist the materials synthesis	$7,700
Equipment		
Pouch cell assembly	Pouch cell assembly line is requested for pouch cell fabrication	$35,000
Other Direct Costs		
Materials and supplies	Consumable materials and supplies for materials	$16,400

	synthesis, coin cell/pouch cell parts, gases, electrochemical testing parts, parts for co-precipitation reactor.	
GRA health insurance	GRA health insurance is included as part of their hiring package.	$3,200
GRA tuition	GRA tuition is included as part of their hiring package.	$11,900
Recharge center services	User fees for use of equipment with BSU recharge service centers. These include the Boise State Center for Materials Characterization, which includes an X-ray diffractometer and a field-emission scanning electron microscope.	$11,000
		$250,000

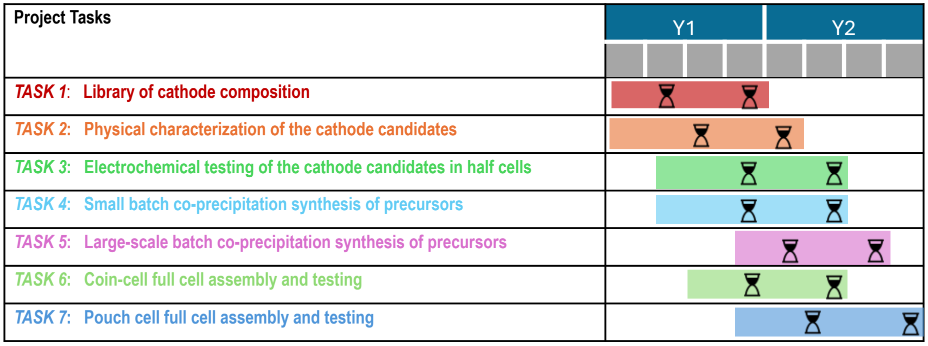
12. Project management

PI Xiong will manage the project, overseeing research and development goals. The team will use the Slack platform for rapid communication. The PI will hold weekly individual meetings with students, and a research scientist as well as weekly project meetings. These meetings will include project updates and topical presentations that solicit discussion, suggestions, and critiques. Project timeline and milestones are shown in **Table 1**.

13. Additional institutional and other sector support

INL has a long history of being a leader of research in LIBs and SIBs. They are an active member of the battery 500 consortium including various national laboratories and universities to pursue achieving a battery cell with 500 Wh/kg. The team at INL has published in many high-impact peer-reviewed journals and have established methods for validating and testing battery materials[19–24]. Our team hopes to collaborate with INL to perform independent validation of our battery materials at their state-of-the-art facilities. Independent validation of the product will enable us to obtain objective performance metrics to optimize our materials, verify our findings, and garner further investment and support for our technology.

Table 1: Project timeline.



Project Tasks
TASK 1: Library of cathode composition
TASK 2: Physical characterization of the cathode candidates
TASK 3: Electrochemical testing of the cathode candidates in half cells
TASK 4: Small batch co-precipitation synthesis of precursors
TASK 5: Large-scale batch co-precipitation synthesis of precursors
TASK 6: Coin-cell full cell assembly and testing
TASK 7: Pouch cell full cell assembly and testing

14. Future funding

Investor interest in the next generation of high-performance batteries will elevate SIBs as a powerful alternative due to their cheap sustainable materials and similarity with the existing LIB supply chain practices. The prospects for future funding from both public and private sources are optimistic. Currently, the established battery suppliers are almost entirely Asian manufacturers, including Panasonic, LG Chem, Samsung, BYD, Guoxuan, Lishen, and Contemporary Amperex Technology; meanwhile, the US has less than 7% of the global battery production. In recent years the US government has actively supported domestic manufacturers to build a domestic supply chain for energy security. Additional funding will be sought after the completion of this project such as Seed Fund from National Science Foundation (NSF) SBIR/STTR and Department of Energy SBIR/STTR, as well as venture capital.

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Appendix A: Facilities, Equipment and Other Resources

Boise State PI Laboratory Facilities

- **Electrochemical Energy Laboratory (Xiong)**

In support of the proposed research, this 1000 sq. ft laboratory has been fully committed by the College of Engineering to supporting the research in synthesis and electrochemical characterization of intergrowth electrode materials. The lab includes a 6 ft. x 6 ft. walk-in thermal/chemical fume hood and two standard 5 ft. fume hood.

- **Boise State Center for Materials Characterization**

The Boise State Center for Materials Characterization (BSCMC) was established by the Idaho State Board of Education in August 2006 to provide a state-of-the-art characterization facility in order to attract more students into science and engineering careers, to improve science and engineering education at the undergraduate and graduate levels, and to foster leading research and interaction with local industry. As such, it provides the organization and infrastructure to make various materials characterization tools available for academia and regional companies. The BSCMC is available for this project.

- **Surface Science Laboratory**

The SSL consists of a 600 sq. ft lab specifically engineered to provide a constant temperature, low noise environment for optimal AFM and SEM operation. Individual systems are located on isolated concrete slabs, and all 4 Bruker AFMs are situated atop vibration isolation tables and housed in acoustic dampening enclosures. A Herzan VA-2 3-axis accelerometer is available for characterizing and monitoring the vibrational noise spectrum.

- **Trace Analytical Laboratory**

The facility is equipped with a suite of analysis tools for interrogating aqueous and solid phase environmental samples. Specific capabilities include: aqueous major, minor and trace metal analysis to the ppt level (Quadrupole ICP-MS, Ion Chromatography), investigation of solids (ranging from bird feathers to mineral grains) using laser-based methods (LA-ICP-MS), soil and other solid characterization of carbon and nitrogen content (Flash EA), and nutrient quantification using automated wet chemistry methods (Lachat Auto Analyzer). The facility is also able to perform a suite of solid phase digestions and leaches using microwave, flux and Parr bomb methods.

- **Machine Shop (College of Engineering)**

The 2400 sq. ft. Machine Shop consists of a professionally staffed shop as well as a staff-supported student shop.

- **Personnel**

Supported by the Micron School of Materials Science & Engineering, a PhD-level staff scientist manages the Surface Science Laboratory, which houses Boise State's existing suite of AFMs and Raman microscope along with the newly acquired eAFM/SECM. He is currently assisted in his duties by two undergraduate research assistants. These personnel maintain the instruments and train students. The Micron School of Materials Science & Engineering, College of Engineering will support a PhD graduate student with assistantship to participate in the proposed research.

- **Student Desks and Offices**

A common office space at Boise State will be available to all graduate students and undergraduate students working on this project. Each student will be provided with a desk, chair, basic office supplies, telephone, and Internet connection. On-campus computing sites with printer capabilities are also available to all students.

- **Administrative Support**

Administrative staff in the Micron School of Materials Science & Engineering at Boise State University will assist with the administrative duties of this project, including logistics, purchasing, and filing.

National Laboratory Facilities

Idaho National Laboratory, Energy Storage & Electric Transportation Department

The Energy Storage Technology Group is involved in multiple federally sponsored programs and projects to develop and enhance the energy, power, and improve diagnostics, prognostics, and predictive capabilities of next generation batteries. The group has key capabilities in the evaluation and understanding of electrochemical processes across temporal and spatial domains. This includes characterization and analysis using physics-based methods for technologies spanning low TRL to near commercial. The group also has key expertise and capabilities in understanding how interfacial processes including mass and electron transfer are impacted as design and surface parameters change. The group collects, manages, and coordinates complimentary analyses (chemical, electrochemical, morphological, and mechanical) related to electrochemical systems. The group also develops advanced and complementary machine learning and data science techniques for life, performance, and safety predictions along with identification of underlying failure pathways of existing and next-generation batteries. INL has established strong expertise and capability in discovering and developing novel technologies, and electrode architecture design and interfacial engineering for Li-S and Li-NMC batteries. These technologies—and associated electrode active-material and architecture characterizations using physicochemical and electrochemical techniques—are aiding the Battery500 Consortium to achieve project goals.

Boise State PI Equipment

The following equipment will be utilized to perform the proposed research. The equipment is listed by the facility in which is it located.

Electrochemical Energy Materials Laboratory (Xiong)

- *MBraun (LabStar) Glove Box:* This Ar-filled glove box contains O_2 and H_2O at <0.5 ppm. Water- and air-sensitive materials preparation such as coin cell assembly will be done with this equipment.
- *ThermoScientific Vacuum Oven:* Affixed with Edwards RV 8 vacuum pump. This oven offers maximum flexibility, with a maximum temperature of 220°C (428°F), two control configurations and display options. Radiant warm-wall heating system optimizes uniformity and conserves chamber space for drying, curing, vacuum embedding and plating applications. Samples in this project will be vacuum baked prior to battery testing.
- *Maccor Model 4200 Battery Cycler:* This is a complete turnkey system with Tester PC and powered by Maccor's standard test system software and utilizing the high-precision Automated Test System. It is supplied with a PowerPole® interface for easily connecting cables or cell holders. Each desktop cabinet is available with up to 16 test positions. It can be configured with multi-current range channels, which have a current control range of 300 ηA to 5 A. These multi-current range channels can be configured for voltage ranges of -2V to +8V, 0V to +5V, or 0V to +10V as standard. The accuracy for current and voltage on these channels is ± 0.02% FSR. Alternately, the system can be configured with single current range channels for higher voltages and currents, and having a current accuracy of ± 0.05% FSR and a voltage accuracy of ± 0.02% FSR. This equipment will be used to test the electrochemical properties of irradiated TiO_2 in a Li system.
- *Arbin BT2043 Battery Tester:* This battery tester provides state of the art hardware with the highest level of specifications available in the market. The BT-2043 series consists of specially designed potentiostat/galvanostat testing stations for testing batteries and electrochemical research. This product is intended to provide economical entry level testing solutions for use in quality control. The product contains 40 channels per chassis.
- *Bio-Logic Electrochemical Workstations (VMP-300, SP-240):* These potentiostat / galvanostat will be used to run electrochemical testings such as battery cycling, cyclic voltammograms, GITT, electrochemical impedance spectroscopy.
- *CHI 660D Potentiostat:* This potentiostat will be used to obtain cyclic voltammograms and electrochemical impedance spectra for the samples.
- *CHI 440C 400C Time-Resolved Electrochemical Quartz Crystal Microbalance (EQCM):* The model instrument contains a quartz crystal oscillator, a fast digital function generator, high resolution and high-speed data acquisition circuitry, a potentiostat, and a galvanostat (Model 440B only). The QCM is integrated with the potentiostat and galvanostat, to facilitate simple and convenient EQCM studies.
- *ESPEC environmental chamber*: This Criterion model has a 19.5 inch wide interior, which is 3.5 inches more than traditional benchtops, yet the exterior is 20% narrower. The extended temperature ranges of the Criterion series make testing at extreme

conditions possible without buying a larger, stand-alone chamber. This unit can go in the temperature range of -70 to 180°C.

- *Mettler Toledo XS105 Analytical Balance:* The minimum display is 0.01 mg. It has a fully automatic calibration program for precise measurement.
- *1200°C Compact Split Tube Furnace with Vacuum Flanges:* The furnace can be adopt a 2" O.D quartz tube with a pair of stainless steel vacuum sealing flanges. The 1/4" barb fitting for rubber hose connection allows heating sample in vacuum or flowing gas. Built-in precision temperature controller can provide up to 30 segments of heating and cooling steps with +/- 1 °C accuracy.
- *Vulcan 3-550 NEY Benchtop Digital Multi-Stage Programmable Furnace*: This furnace is a digital, multi-stage programmable furnace. Its three-stage digital programmable controller features adjustable rate of temperature climb, nine multi-stage programs, a hold temperature program, and a delay start option which allows a program's start time to be delayed for up to seven days. It has a maximum temperature of 1000 Celsius or 2012 Fahrenheit. The Vulcan 3-550 features High Performance Hybrid Muffle Technology exclusive to Vulcan benchtop furnaces for faster heating and cooling than full firebrick furnaces. It is constructed with a heavy gauge steel cabinet finished with a durable baked enamel coating. It will be used for solid state reactions for cathode synthesis.
- *Retsch Planetary Ball Mill PM 100*: powerful and quick grinding down to nano range reproducible results due to energy and speed control; suitable for long-term trials; 2 different grinding modes (dry and wet); optional pressure and temperature measuring system PM GrindControl; wide range of materials for contamination free grinding; Safety Slider for safe operation; innovative counter weight and imbalance sensor for unsupervised operation; power failure backup ensures storage of remaining grinding time; jars with O-type sealing for safe operation, pressure tight.
- *Eppendorf® Centrifuge 5804:* Three centrifuges in one: a high-capacity, general-purpose centrifuge for materials separation. It allows for molecular applications in tubes up to 250 mL and offers additional wing-bucket and fixed-angle rotors as well as deep well plate capacity for increased versatility.
- *Grinding and Polishing Instruments*: A full suite of specimen preparation equipment is available for this project. Instruments include a low-speed saw with diamond wafering blade, belt sander, grinding and polishing wheels, and vibratory polisher.

Equipment within Boise State Shared-Use Facilities

- ***Boise State Center for Materials Characterization***

The Boise State Center for Materials Characterization (BSCMC) is available for this project. The suite of microscopy and characterization tools at BSCMC that will be used in this project are":

- *JEOL JEM-2100 High Resolution Analytical Transmission Electron Microscope (TEM):* The imaging camera has 0.23 nm point resolution and 0.14 nm line resolution. In scanning (STEM) mode, resolution is 1.0 nm. Chemical analysis through energy-dispersive x-ray spectroscopy (EDX) is enabled with the Thermo Scientific Noran

  System 7, and through electron energy-loss spectroscopy (EELS) with the Gatan Model 776 Enfina 1000.

  - *FEI Teneo Field Emission Scanning Electron Microscope (FESEM):* The SEM resolution is as low as 0.8 nm at 30 keV STEM, 1.0 nm at 15 keV, and· 1.4 nm at 1 keV. It is also equipped with EDS and EBSD.
  - *Bruker AXS D8 Discover X-Ray Diffractometer (XRD):* The XRD will provide high-resolution determination of crystallographic structure, phase composition, and texture over a range of temperatures between -180°C and 1600°C.
  - *Rigaku Miniflex 600 bench-top X-ray diffractometer:* One dimensional D/teX Ultra high-speed detector (very quick scans). Scintillating point detector with graphite monochromator (best for resolution). Multiple-sample loading capability
  - *CAMECA SXFIVE ELECTRON MICROPROBE (EPMA):* The electron microprobe analyzer (EPMA) is particularly well suited for determining the distribution of small elemental concentrations with excellent spatial resolution.

- ***Shared facilities in MSE faculty's labs***
  - *PHI 5600 Electron Spectroscopy for Chemical Analysis (ESCA) X-ray Photoelectron Spectroscopy (XPS) system:* Monochromated Al x-ray source with a hemispherical energy analyzer, ion gun for sputtering with argon, neon, and xenon, motorized sample stage, sample neutralizer gun, and turbo-pumped load-lock sample entry. Glove box sample transfer vessel allows transfer of samples without air exposure.
  - *TA Instruments SDT Q600*: Simultaneous Thermal Analyzer (STA) - TGA and DSC.
- ***Surface Science Laboratory***

The SSL comprises a 600 ft^2 lab specifically engineered to provide a constant temperature and a low noise environment for optimal AFM and SEM operation. The instruments in the SSL include:

  - Horiba LabRAM HR Evolution systems: This Raman system is equipped with 442, 633, and 532 nm excitation wavelengths. It is ideally suited to both micro and macro measurements, and offers advanced confocal imaging capabilities in 2D and 3D. The true confocal microscope enables the most detailed images and analyses to be obtained with speed and confidence.
  - *Bruker Dimension Icon/FastScan AFM in an MBraun Glove Box:* 64-bit AFM system capable of quantitative nanomechanical mapping, Kelvin force probe microscopy, tunneling microscopy, and scanning electrochemical microscopy with sample heating and cooling. The system includes a turbo-pumped antechamber for fast sample exchange.
  - *Bruker Dimension Icon/FastScan Bio AFM:* Same capabilities as above with additional fuild cells and movie mode high speed sample scanning.
  - *Bruker MultiMode 8 AFM*: High-resolution ultra-low noise floor AFM with same imaging modes as above. Additional capabilities include scanning tunneling microscopy and electrochemical-AFM mode.

  - *Bruker Dimension 3100 AFM*: AFM capabilities with nanomechanical measurements, including a Hysitron TS 75 Triboscope Nanoindentation System.
  - *FEI Phenom Tabletop SEM*: 5 kV source SEM with 525-240,000x variable electron magnification
  - *PHI 5600 ESCA XPS system:* Monochromated Al x-ray source with a hemispherical energy analyzer, ion gun for sputtering with argon, neon, and xenon, motorized sample stage, sample neutralizer gun, and turbo-pumped load-lock sample entry
- ***Trace Analytical Laboratory***
  - *Quadrupole ICP-MS:* Trace element analysis of solids (ppt level).

APPENDIX B: Biographical Sketches

1. Hui (Claire) Xiong, PI

IDENTIFYING INFORMATION:

NAME: Xiong, Hui (Claire)

POSITION TITLE: Professor

PRIMARY ORGANIZATION AND LOCATION: Boise State University, Boise, Idaho, United States

Professional Preparation:

ORGANIZATION AND LOCATION	DEGREE (if applicable)	RECEIPT DATE	FIELD OF STUDY
Argonne National Laboratory, Lemont, IL, USA	Postdoctoral Fellow	03/2009 - 08/2012	Mater. Sci. & Eng.
Harvard University, Boston, MA, USA	Postdoctoral Fellow	01/2008 - 02/2009	Mater. Sci. & Eng.
University of Pittsburgh, Pittsburgh, PA, USA	PHD	12/2007	Anal. Chem./Electrochem.
East China U. of Sci. & Tech., Shanghai, n/a, China	MS	12/2000	Inorganic Chemistry
East China U. of Sci. & Tech., Shanghai, n/a, China	BENG	06/1998	Applied Chemistry

Appointments and Positions

2023 - present	Professor, Boise State University, Boise, Idaho, United States
2019 - 2022	Associate Director, Graduate Program, Mater. Sci. & Eng., Boise State, Boise, Idaho, United States
2018 - 2023	Associate Professor, Boise State University, Boise, ID, United States
2012 - 2018	Assistant Professor, Boise State University, Boise, ID, United States
2009 - 2012	Postdoctoral Fellow, Argonne National Laboratory, Lemont, Illinois, United States
2008 - 2009	Postdoctoral Fellow, Harvard University, Boston, Massachusetts, United States

Products

Products Most Closely Related to the Proposed Project

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Certification:

I certify that the information provided is current, accurate, and complete. This includes but is not limited to current, pending, and other support (both foreign and domestic) as defined in 42 U.S.C. § 6605.

I also certify that, at the time of submission, I am not a party to a malign foreign talent recruitment program.

Misrepresentations and/or omissions may be subject to prosecution and liability pursuant to, but not limited to, 18 U.S.C. §§ 287, 1001, 1031 and 31 U.S.C. §§ 3729-3733 and 3802.

Certified by Xiong, Hui (Claire) in SciENcv on 2025-02-12 14:56:02

APPENDIX C: Senior Personnel

N/A

APPENDIX D: Other

1. Letter of Support - Pete Barnes, INL

Idaho National Laboratory

Prof. Hui (Claire) Xiong
Micron School of Materials Science and Engineering
Boise State University
1910 University Dr.
Boise, ID, 83725
clairexiong@boisestate.edu

via Email

February 7, 2025

SUBJECT: Letter of support for testing of sodium cathode material

Dear Prof. Xiong

If your application entitled, "**High Energy/Power Layered Oxide Cathode Materials for Sodium Ion Batteries**", is selected for funding by the IGEM-HERC, it is my intent to collaborate in this research by investigating the performance of the pouch cells made by your electrode materials under practical conditions set by the Department of Energy (DOE) and the US Advanced Battery Consortium (USABC) protocols.

Regards,

Pete Barnes, PhD, Research Scientist
Energy Storage and Transportation
Idaho National Laboratory
208-358-0659
pete.barnes@inl.gov