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CX Posting No.: DOE-ID-INL-20-233

SECTION A. Project Title: Stable High-Temperature Molten Salt Reference Electrodes

SECTION B. Project Description and Purpose:

Revision 1:

The revision identifies new scope and changes the EC from a tiered EC to a new ECP due to off-site work at the University of Utah (UofU). The approach for this current Phase II project is to design, develop, and demonstrate a High-Temperature Reference Electrode (HTRE) that uses a near-net shape fluoride ion transport membrane or a controlled porosity membrane with seals engineered for this demanding high-temperature molten salt application. The challenges associated with improving reference electrodes for molten salts applications are 1) to select the membrane and container materials that are chemically and mechanically stable in higher temperature fluoride-based molten salts, and 2) to develop robust HTRE and seal designs that are chemically compatible, can withstand thermal cycling, are cost effective, and are manufacturable (designed for manufacturing and assembly).

Project experiments will be performed at INL's Materials and Fuels Complex (MFC), specifically the Engineering Design Facility (MFC-789), a non-radiological facility.

Task 1. Design HTRE

HiFunda, UofU, and INL will establish a prototype HTRE design that builds upon the strengths and weaknesses of proof-of-concept (POC) HTRE developed during Phase I. INL will provide inputs on existing HTREs and will host HiFunda during visit(s) to observe INL's molten salt electrochemical experimental setups. INL will also provide technical consultation and participate in design reviews.

Task 2. Evaluate HTRE Materials & Fabrication Processes

HiFunda will lead Task 2 to develop and evaluate alternative options for robust near-net shape porous and dense membrane materials. INL will provide technical consultation for this task.

Task 3. Fundamental Molten Salt Electrochemical Measurements & Analysis

UofU will lead Task 3 to perform short and long-term fundamental electrochemical analyses and testing of metal working electrodes including: tungsten, platinum, silver, and nickel in molten FLiNaK at 500, 600, 700, and 800°C for 30 to 180 days. INL will provide technical consultation for this task.

Task 4: Fabricate, Assemble, and Characterize HTRE

HiFunda will lead Task 4 to fabricate, assemble, and characterize HTREs. INL will provide technical consultation for this task.

Task 5: Perform HTRE Electrochemical Performance Testing

INL will lead Task 5 to perform short (30 days) and long-term (up to 180 days) electrochemical testing of HTREs that HiFunda makes as part of Task 4.

Task 6: HTRE Failure Analyses

HiFunda will lead Task 6 to perform failure analyses on the HTREs, and INL will provide X-ray computed tomography (XRCT) support to nondestructively image HTREs before and after testing prior to destructive cross sectioning.

Task 7: Establish Phase III Commercialization Plan and Business and Cost Models

HiFunda will lead Task 7 to develop cost models as well as commercialization and transition plans to deploy the HTRE technology to specific target markets.

Task 8: Reporting

HiFunda will lead Task 8 to prepare the final technical report and presentation to DOE. INL and the UofU will provide technical input (slides, data, emails) to support HiFunda's reporting requirements.

Original EC:

This EC covers research on stable high-temperature molten salt reference electrodes that will take place at the Material Fuels Complex (MFC), in the argonatmosphere gloveboxes inside building MFC-789.

HiFunda, LLC (HiFunda), located in Salt Lake City, Utah, is a technology incubator that identifies and develops new materials and energy related technologies to advance the state-of-the-art in energy harvesting, energy storage, and energy efficiency. They also specialize rapid, focused early stage technology risk reduction to commercialize new energy and materials-related technologies. HiFunda has developed custom electrochemical processes for a wide range of applications and has performed electrochemical testing in aqueous, nonaqueous, molten salts, and solid electrolyte media.

INL is the nation's lead laboratory for nuclear energy research, development, demonstration, and deployment. Molten salt research has been continuously performed at INL since 1985. INL also has extensive experience in pyrochemical processes and has a full suite of world-class capabilities to support all aspects of molten salt electrochemistry research.

HiFunda and INL were recently awarded funding by the Small Business Innovation Research (SBIR) program for a proposal entitled, "Stable High-Temperature Molten Salt Reference Electrodes." Under the award, HiFunda and INL will develop a high-temperature thermodynamic reference electrode suitable for use in fluoride molten salt systems that are candidates for molten salt reactor applications.

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Conventional nuclear reactors are water cooled, operate at temperatures of $\approx 300^{\circ}$ C, and drive steam turbines with efficiencies of $\approx 30^{\circ}$. Molten fluoride salts enable next-generation nuclear reactors to operate at ambient pressure in the temperature range of 600°C to 700°C with efficiencies of 60%. These salts offer advantages as fuel salts, coolants, and electrolytes but also present challenges because of their aggressive and corrosive nature. Because of this nature, the salt redox potential must be monitored, and the salt chemistry controlled to prevent oxidation and corrosion of structural materials. A stable thermodynamic reference electrode, or an electrode that has a known, stable potential, is critical for this work. The important attributes of a reference electrode are stability (during an experiment), reproducibility (between experiments), compactness of construction, comparability among laboratories, and chemical compatibility with the system of interest.

The U.S. Department of Energy's Office of Nuclear Energy (DOE-NE) desires improved reference electrodes for molten salt-cooled reactors and fuel processing equipment. Unfortunately, robust thermodynamic, high-temperature reference electrodes (HTREs) for this challenging application are not commercially available and HTREs that have been evaluated are custom designs with lifetimes measured in days. The operational lifetimes of existing HTREs are limited by corrosion and instability of containment cells, ionic membranes, or frits of a thermodynamic reference electrode due to chemical incompatibility, stress due to thermal cycling, and exposure to high temperature.

The approach for this current Phase I project is to design, develop, and demonstrate an HTRE that uses a near-net shape fluoride ion transport membrane or a controlled porosity membrane with seals engineered for this demanding high-temperature molten salt application. The challenges associated with improving reference electrodes for molten salts applications are 1) to select the membrane and container materials that are chemically and mechanically stable in higher temperature fluoride-based molten salts, and 2) to develop robust HTRE and seal designs that are chemically compatible, can withstand thermal cycling, are cost effective, and are manufacturable (designed for manufacturing and assembly).

Project experiments will be performed inside the argon-atmosphere gloveboxes in MFC-789, a non-radiological facility. No gaseous emissions will be generated during the experiments.

Tasks

Task 1. Design HTRE

HiFunda and INL will establish a proof-of-concept HTRE design that builds upon the strengths and weaknesses of designs in literature. INL will provide inputs on existing HTREs and will host HiFunda during visit(s) to observe INL's molten salt electrochemical experimental setups. INL will also provide technical consultation and participate in design reviews.

Task 2. Develop and Evaluate New Membrane Materials and Fabrication Processes

HiFunda will develop and evaluate alternative options for robust near-net shape porous and dense membrane materials. INL will provide technical consultation for this task.

Task 3. Fabricate, Assemble, and Characterize HTRE

HiFunda will use the designs from Task 1 and the membranes from Task 2 to fabricate, assemble, and characterize the different configurations of HTREs. HiFunda has access to the University of Utah's User Facility where additional inspection using scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) will be performed (separate from this agreement). INL will provide technical consultation for this task.

Task 4: Perform HTRE Voltage Stability Testing

INL will be the technical lead on Task 4 efforts and will perform short (hours) and long-term (days) electrochemical testing of HTREs. INL and HiFunda will jointly establish designed experiments that INL will perform to evaluate the stability and reproducibility of HTREs as a function of time, temperature, and melt composition.

Task 5: Establish Phase II Prototype HTRE Design and Business and Cost Models

The results from testing and evaluation of the HTRE samples in Task 4, and the HTRE designs from Task 1, will be used to establish a Phase II prototype HTRE design. In Task 5, INL will evaluate conceptual prototype designs and cost models. After completing the evaluation, INL will provide HiFunda input and recommendations regarding molten salt applications.

Task 6: Reporting

During the project, HiFunda will periodically update the program manager(s) at DOE on progress.

INL and HiFunda will prepare and submit a final Phase I progress report at the end project.

SECTION C. Environmental Aspects or Potential Sources of Impact:

Air Emissions

N/A

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Disturbing Cultural or Biological Resources

N/A

Generating and Managing Waste

Industrial waste will be generated. Namely, FLiNaK salt (about 500 g) in glassy carbon crucibles with a few electrodes made of tungsten. FLiNaK is a mixture of LiF, NaF, and KF. Some PPE may also be generated. Waste generated at the UofU will be handled through normal university procedures.

Releasing Contaminants

Whenever chemicals are used there is a potential for releases.

Using, Reusing, and Conserving Natural Resources

All applicable waste will be diverted from disposal in the landfill when possible. Project personnel will use every opportunity to recycle, reuse, and recover materials and divert waste from the landfill when possible.

SECTION D. Determine Recommended Level of Environmental Review, Identify Reference(s), and State Justification: Identify the applicable categorical exclusion from 10 Code of Federal Regulation (CFR) 1021, Appendix B, give the appropriate justification, and the approval date.

For Categorical Exclusions (CXs), the proposed action must not: (1) threaten a violation of applicable statutory, regulatory, or permit requirements for environmental, safety, and health, or similar requirements of Department of Energy (DOE) or Executive Orders; (2) require siting and construction or major expansion of waste storage, disposal, recovery, or treatment or facilities; (3) disturb hazardous substances, pollutants, contaminants, or Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)-excluded petroleum and natural gas products that pre-exist in the environment such that there would be uncontrolled or unpermitted releases; (4) have the potential to cause significant impacts on environmentally sensitive resources (see 10 CFR 1021). In addition, no extraordinary circumstances related to the proposal exist that would affect the significance of the action. In addition, the action is not "connected" to other action actions (40 CFR 1508.25(a)(1) and is not related to other actions with individually insignificant but cumulatively significant impacts (40 CFR 1608.27(b)(7)).

References: 10 CFR 1021 Appendix B to subpart D, Item B3.6, "Small-scale research and development, laboratory operations, and pilot projects."

Justification: The proposed R&D activities are consistent with CX B3.6 "Siting, construction, modification, operation, and decommissioning of facilities for small-scale research and development projects; conventional laboratory operations (such as preparation of chemical standards and sample analysis); small-scale pilot projects (generally less than 2 years) frequently conducted to verify a concept before demonstration actions, provided that construction or modification would be within or continuous to a previously disturbed area (where active utilities and currently used roads are readily accessible). Not included in this category are demonstration actions, meaning actions that are undertaken at a scale to show whether a technology would be viable on a larger scale and suitable for commercial deployment."

Is the project funded by the American Recovery and Reinvestment Act of 2009 (Recovery Act)

Approved by Jason L. Anderson, DOE-ID NEPA Compliance Officer on: 06/30/2021