DOE-ID NEPA CX DETERMINATION
Idaho National Laboratory

CX Posting No.: DOE-ID-INL-21-180

SECTION A. Project Title: Phase Stability of Yttrium Titanium Oxides

SECTION B. Project Description and Purpose:

This proposed study aims at adding new contributions to nuclear material research on two major fronts: 1) expanding INL's material characterization capabilities to provide holistic microstructural and microchemical characterization ranging from atomic scale to millimeter-scale, and 2) filling the knowledge gap on the effect of microstructural evolution on material phase stability at extreme conditions resembling those inside nuclear reactors, using Yttrium-Titanium-Oxide (YTO) as a model material. Previous research indicated the kinetics of phase transition of YTO materials varies between bulk samples and powder samples, suggesting that microstructural characteristics such as defect, micro-strain, and crystallite size could play a significant role in phase transition. The effect of crystallite size on the thermodynamics of phase transition has been well documented for other ceramic materials at nanometer crystallite size, with potential impacts from surface and strain energy and defects. In this proposed study, we will perform microstructural (micro-strain, crystallite size, defects, phase) and microchemical (elemental mapping) analyses through in situ high temperature measurements using X-ray diffraction (XRD); at millimeter-scale for powder and bulk samples) and transmission electron microscopy (TEM), at nanometer-scale and atomic scale for powder and thin foil samples), to provide a comprehensive view of the effect of microstructural and microchemical evolution on phase transition and phase stability of YTO materials. These findings will also establish a roadmap and provide new insights on the phase stability of other materials that are critical to nuclear energy and other renewable energy infrastructure. Besides advancing our understanding of this fundamental material science question, this project will also enable the development of state-of-the-art material characterization capabilities at Idaho National Laboratory (INL)/Materials and Fuels Complex (MFC) for microstructural and microchemical analysis at high temperature conditions. The new collaboration between the High-Resolution Materials Characterization group at INL and the Electron Microscopy group at the University of Florida (UF) in this project will open the possibilities for more fruitful and synergetic cutting-edge nuclear material research in the future.

To test the hypotheses about the effects of micro-strain, defects and crystallite size on the phase stability of pyrochlore Y_2TiO_5 at high temperature conditions, we will develop correlative XRD/TEM characterization techniques at MFC, and examine the relationships between micro-strain, crystallite size, defects and phase stability at various temperatures. XRD will provide information for bulk and powder samples at millimeter-scale whereas TEM will provide the information on thin foil and powder sample at atomic scale.

Task 1 In situ XRD (INL, Pu): The bulk and powder YTO samples will be heated and cooled at very slow rates (1-5°C/min), followed by high-resolution XRD scans to be collected at each temperature interval, within a range of 100 to 800 °C, during both heating and cooling. These results will be correlated to lattice parameter and phase fraction results and provide information on the phase transformation kinetics as a function of temperature and microstructural properties at bulk level (millimeter-scale), with the TEM analysis providing information at nanometer and atomic scales.

Task 2 In situ TEM (INL, He & UF, Kim): To complement the XRD in situ analysis on bulk and powder samples, the TEM analysis will examine thin foil and powder samples. Scanning transmission electron microscopy (STEM) imaging will be used to characterize microstructure and defects in YTO. In addition to imaging techniques, STEM will be used to obtain atomic-resolution elemental mapping. For the study of microstructural evolution in YTO at high temperatures, we will use an in-situ heating TEM holder, which is capable of heating the focused ion beam (FIB)-prepared samples and powder samples up to 1,200 °C with minimal loss of spatial resolution. Videos will record the evolution of atomic structure of YTO and capture phase transitions, if any, through tracing atomic motions.

Products and Deliverables

The proposed research will last for 12 months. Upon completion of this project, the in situ correlative XRD/TEM characterization and atomic scale imaging/chemical analysis capabilities will be available to internal and external users, and the mechanistic understanding of phase stability of pyrochlore Y_2TiO_5 in extreme environments will be advanced. This will strengthen INL's leadership position on R&D of nuclear fuels and materials for advanced reactors. A list of deliverables is defined below.

- In situ XRD characterization of micro-strain and phase transformation in YTO materials;
- In situ TEM characterization of micro-strain, defect evolution and phase transformation in YTO materials;
- Atomic-resolution STEM imaging/elemental mapping analysis for point defects and micro-strain;
- Strengthened the research partnership between UF and INL on advanced characterization of nuclear materials through a UF visiting student working at INL;
- Preparation of at least two peer-reviewed journal publications, focused on the microstructural evolution during phase transition of YTO material, and the new development on correlative microscopy. Target journals include Acta Materialia and Science Advance.

SECTION C. Environmental Aspects or Potential Sources of Impact:

Air Emissions

N/A

Discharging to Surface-, Storm-, or Ground Water

N/A

Disturbing Cultural or Biological Resources

N/A

Generating and Managing Waste

Small amounts industrial waste may be generated from personal protective equipment, wipes or sample debris.

Releasing Contaminants

N/A

Using, Reusing, and Conserving Natural Resources

All materials will be reused and recycled where economically practicable. All applicable material will be diverted from disposal in the landfill where conditions allow.

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 research and development, laboratory operations, and pilot projects."

Justification:

The project is consistent with B3.6 Small-scale research and development, laboratory operations, and pilot projects. 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); and 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 contiguous to a previously disturbed or developed 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) ☐ Yes ☒ No

Approved by Jason L. Anderson, DOE-ID NEPA Compliance Officer on: 04/18/2022