INL/EXT-21-64857-Revision-0

In-Situ SEM Irradiation Enhanced Creep Studies of 14 YWT

October 2021

David M Frazer, Joshua White, Tarik Saleh, Stuart Maloy

Changing the World's Energy Future

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October 2021

Idaho National Laboratory
Idaho Falls, Idaho 83415

http://www.inl.gov

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Email completed template and images (up to four figures) as separate attachments to tiera.cate@inl.gov

2021 Annual Report Short Communications Template

ALL FIELDS IN THIS TEMPLATE MUST BE FILLED OUT

Finished reports must include figures and graphics.

Questions? Contact Simon Pimblott (simon.pimblott@inl.gov, 208-526-7499) or Tiera Cate (tiera.cate@inl.gov, 208-526-4828).

1. **Project Title** (from your proposal) In-Situ SEM Irradiation Enhanced Creep Studies of 14 YWT
2. **Principal Investigator** David Frazer
3. **PI Email** David.frazer@inl.gov
4. **NSUF Technical Lead** Click or tap here to enter text. **NSUF Project ID** Click or tap here to enter text.
5. **NSUF Technical Lead** Click or tap here to enter text. **NSUF Project ID** Click or tap here to enter text.
6. **NSUF Technical Lead** Click or tap here to enter text. **NSUF Project ID** Click or tap here to enter text.
7. **Team Member/Collaborator Information** Click or tap here to enter text.

For All Team Members/Collaborators			If a Student		
Name	**Role (Team Member or Co-Principal Investigator)**	**Institution**	**Level in School**	**Field of Study/ Degree Seeking**	**U.S. or Non-U.S.**
Tarik Saleh	Co-Principal Investigator	Los Alamos National Laboratory	Click or tap here to enter text.	Click or tap here to enter text.	☐ U.S. ☐ Non-U.S.
Stuart Maloy	Co-Principal Investigator	Los Alamos National Laboratory	Click or tap here to enter text.	Click or tap here to enter text.	☐ U.S. ☐ Non-U.S.
Joshua White	Co-Principal Investigator	Los Alamos National Laboratory	Click or tap here to enter text.	Click or tap here to enter text.	☐ U.S. ☐ Non-U.S.
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8. **Degrees granted as a result of this research.** (Please list any degrees from the start of your project award, even those from previous years.)

Name	Institution	Degree	Field of Study/Degree Seeking	Date
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9. **NSUF Facility used (check all that apply)** *NOTE: If facilities other than those specified below were used, they can be mentioned in the body text of the accomplishments section.)*

Argonne National Laboratory:

☐ The Intermediate Voltage Electron Microscopy (IVEM) – Tandem Facility

Belgian Center for Nuclear Research (SCK/CEN)

☐ Belgian Reactor 2

☐ Laboratory for High and Medium Activity

Brookhaven National Laboratory:

☐ National Synchrotron Light Source II

Center for Advanced Energy Studies:

☐ Microscopy and Characterization Suite (MaCS)

Idaho National Laboratory:

☐ Advanced Test Reactor (ATR)

☐ Advanced Test Reactor Canal

☐ Advanced Test Reactor Critical (ATRC) Facility

☐ Analytical Laboratory

☐ Electron Microscopy Laboratory (EML)

☐ Experimental Fuels Facility

☐ Fuel Conditioning Facility

☐ Fuel Manufacturing Facility

☐ Fuels and Applied Science Building

☐ High Performance Computing

☐ High Temperature Test Laboratory

☐ Hot Fuel Examination Facility (HFEF)

☐ INL Research Center

☐ Irradiated Materials Characterization Laboratory (IMCL)

☐ Neutron Radiography Reactor (NRAD)

☐ Stress Corrosion Cracking Lab

☐ Test-Train Assembly Facility

☐ Transient Reactor Experiment and Test Facility (TREAT)

Lawrence Livermore National Laboratory:

☐ Center for Accelerator Mass Spectrometry

Los Alamos National Laboratory:

☐ Chemical and Metallurgical Research Facility (Wing 9)

☐ Los Alamos Neutron Scattering Center - Lujan Center Beamlines

☐ Plutonium Surface Science Laboratory

Massachusetts Institute of Technology:

☐ Nuclear Reactor Laboratory

☐ Reactor

North Carolina State University:

☐ PULSTAR Reactor Facility

Oak Ridge National Laboratory:

☐ High Flux Isotope Reactor (HFIR)

☐ Irradiated Fuels Examination Laboratory (IFEL)

Hot Cells

☐ Irradiated Materials Examination and Testing Facility (IMET) Hot Cells

☐ Low Activation Materials Design and Analysis Laboratory (LAMDA)

The Ohio State University:

☐ The Ohio State University Nuclear Research Laboratory

Pacific Northwest National Laboratory:

☐ Materials Science and Technology Laboratory (MSTL)

☐ Radiochemical Processing Laboratory (RPL)

Purdue University:

☐ Interaction of Materials with Particles and Components Testing (IMPACT)

Sandia National Laboratory:

☐Annular Core Research Reactor

☒SNL Ion Beam Laboratory

☐ Gamma Irradiation Facility

☐ Sandia Pulse Reactor Facility Critical Experiment (Burnup Credit Critical Experiment)

Texas A&M University:

☐ Accelerator Laboratory

University of California, Berkeley:

☐ Nuclear Materials Laboratory

University of Florida

☐ Nuclear Fuels and Materials Characterization Facility

University of Michigan:

☐ Irradiated Materials Testing Laboratory

☐ Michigan Ion Beam Laboratory

University of Wisconsin:

☐ Characterization Laboratory for Irradiated Materials

☐ Ion Beam Laboratory

Westinghouse:

☐ Churchill Laboratory Services

10. **Keywords**

In-situ Mechanical Testing, Small Scale mechanical testing, 14 YWT, nano-structured alloys

11. **Funding**: List the entities that provided research and development funding for this project.

Fiscal Year (Oct. 1 – Sep. 30)	Funding Source	Percent
2021	Other	50
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12. **Project Hypothesis or Goal:** Succinctly describe the hypothesis or goal of the research project (50-100 words)

It was the goal of this project to evaluate the effects of irradiation on the creep properties of 14 YWT. Two microscopes have the potential to perform these measurements at sandia national laboratory the I3SEM and the I3TEM. They both allow for the simultaneously mechanical testing will be ion beam irradiated including at elevated temperature. In this case micro-compression experiments were performed in the I3TEM at temperature and under irradiation to evaluate the effects on the creep rates of the 14 YWT.

13. **Experimental or Technical Approach:** Describe the experimental or technical methods of the research project (150-200 words)

A sample of 14 YWT was first mechanical polished to 50 μm thick. A focused ion beam instrument was then used to mill the excess material away until and 10s of nano pillars where manufactured. The dimensions of the nano pillars were 280 nm thick X 280 nm wide X 650-700 nm in height and were spaced 5 μm apart. The in-situ TEM experiments were performed on the I^{3}TEM at Sandia National Laboratory. The I^{3}TEM allows for imaging of the sample while being irradiated with ions. In addition, the Hysitron PI-95 picoindenter enables instrumented indentation in the TEM to allow mechanical testing. Room temperature (RT) compressive creep experiments were performed with ion beam irradiation and no irradiation. The nanopillars were irradiated with 2.8 MeV Au^{4+} ions. Using SRIM with composition of 14 YWT and displacement energy of 40 eV, with thickness of the pillars at 25° and 2.8 MeV Au^{4+} ions the peak displacement damage in the material was calculated at 0.028 dpa/s. For the compression creep experiments, the PI-95 was used in load control mode. The pillars were loaded up to approximately 450 MPa (50 μN load) and held for 300 seconds to measure the change in displacement with time. For these experiments, 3 nanopillars were tested in each condition.

14. **Results**: Summarize what was measured, the readings taken, and observations made (150-200 words)

The strain rate increased under irradiation going from negligible levels to $6x10^{-4}$ s^{-1}. While this is higher than typical creep rates of 14 YWT there are some experimental challenges with regard to sample geometry, dose rate, and other potential contributing factors. It should be noted there was the ability to see a difference in the creep strain rate between the two conditions which show promise for in-situ experiments for the I3TEM and the I3SEM. There are some challenges with sample preparation and use in the TEM with magnetic samples that would make elevated temperature experiments more challenging. The stability of the measurements and the ability could allow for more sophisticated experiments such as nano-tensile, varying the damage rate or strain rate jump tests to gather more information out of these experiments.

15. **Discussion/Conclusion**: Describe the impact and meaning of the results of your project (75-100 words)

With the ability to see a difference in the irradiated and non irradiated test conditions there is the possibility for elevated temperature experiments and moving to large specimens in the I3SEM. There were some challenges with sample shape and geometry in the I3TEM but there is the possibility to use the I3SEM located at Sandia National Laboratory with larger specimens enable the measurement of macro-scale creep properties. This could allow for the rapid evaluation of materials properties in irradiation environments.

16. **References**

    Click or tap here to enter text.

17. **Publications:** Include conference, journal, textbook and special workshop reports. **List the citations in text form here and use the "Load a Publication" link on the website**. Publications are tracked electronically to be included in the publications archive on the NSUF website and used for metric analysis. Only publications from the current fiscal year will be included in the annual report, but all publications need to be loaded on the website so they are included in the archive.

    Riley J. Parrish, Daniel C. Bufford, David M. Frazer, Caitlin A. Taylor, Jacob Gutierrez-Kolar, Daniel L. Buller, Brad L. Boyce, and Khalid Hattar "Exploring Coupled Extreme Environments via In-situ Transmission Electron Microscopy" (2021) Microscopy Today, 29(1), 28-34. doi:10.1017/S1551929520001595

    Under preparation:

"In-situ Irradiation creep and small scale creep testing of 14 YWT"

18. **Patents applied for because of this research.**

    No Patents

19. **Principal Investigator Bio Photo.** Please provide a professional bio photo that may be used to accompany the article.

    Please make sure to also send this photo as a separate file to tiera.cate@inl.gov

20. ***(Optional)*** **Graphics (only high-resolution, 300 dpi, .jpeg, .tif, .pdf, and eps file formats only).** Please include pertinent, high-quality graphics with captions. Please also provide a high-resolution image (300 dpi or higher) showing research being done by team members using an NSUF capability for possible inclusion in an Annual Report highlight graphic. If you need assistance with your figures, please contact Tiera Cate (tiera.cate@inl.gov). **Limit of one graphic for short communications.**

    Along with uploading the individual high resolution .jpeg, .tiff pdf or eps files, please also send the file separately to tiera.cate@inl.gov.

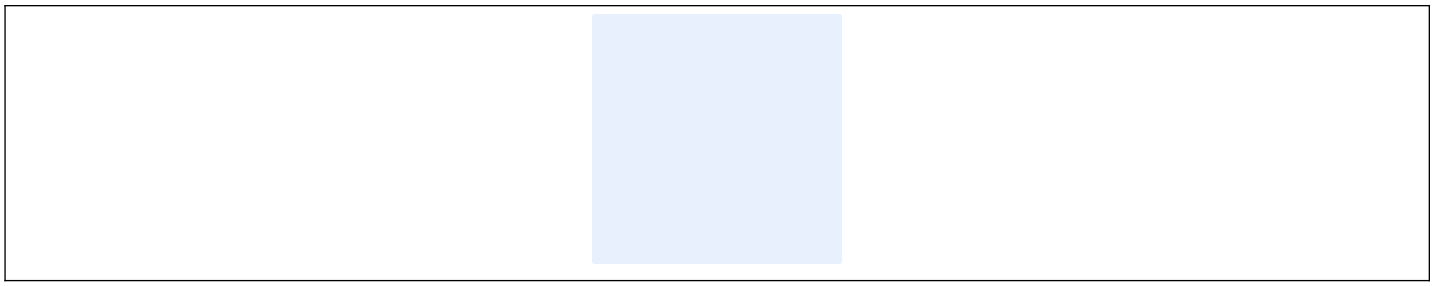

Figure 1. Enter Caption.