

Statement of Work

Project No(s): 23841, 29412

Effects of Neutron Irradiation on the Micro/Nano Scale Structure and Fission Product Distribution of TRISO Coated Particle Fuel Kernels from AGR Experiments

The INL is a
U.S. Department of Energy
National Laboratory
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Battelle Energy Alliance



Idaho National Laboratory

**EFFECTS OF NEUTRON IRRADIATION ON
THE MICRO/NANO SCALE STRUCTURE
AND FISSION PRODUCT DISTRIBUTION OF
TRISO COATED PARTICLE FUEL KERNELS
FROM AGR EXPERIMENTS**

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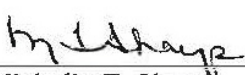

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INL ART TDO Program

Statement of Work

Signatures

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P For Preparer of the document.**A** For Approval: This is for non-owner approvals that may be required as directed by a given program or project.**C** For documented review and concurrence.**Note:** Applicable QLD Level 3 (QL3)

REVISION LOG

[illegible]

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1. INTRODUCTION**1.1 Background**

The Advanced Reactor Technologies (ART) Technology Development Office (TDO) at Idaho National Laboratory (INL) includes the Advanced Gas Reactor (AGR) fuel development and qualification program that consists of fuel fabrication, experiment irradiations, post-irradiation examination (PIE) and safety testing to assess tristructural isotropic (TRISO) fuel performance during normal irradiation and under potential accident conditions. Advanced microscopy work on selected AGR-1 and AGR-2 unirradiated and irradiated fuel specimens is performed as part of the PIE effort. PIE work on fuel from the first experiment irradiation, AGR-1, began at INL in April 2010, and AGR-2 PIE began at INL in July 2014. This work scope includes University of Florida (UF) performing advanced electron microscopy examination and analysis using facilities at the Center for Advanced Energy Studies (CAES), INL or UF's electron microscopy facility. Electron microscopic examination and analysis may include scanning transmission electron microscopy (STEM), transmission electron microscopy (TEM), selected area diffraction (SAD), electron energy loss spectroscopy (EELS) and electron dispersive spectroscopy (EDS) on the fuel kernels of TRISO-coated fuel particles. The electron microscopy lamellae will be provided by INL and will be available at CAES.

1.2 Purpose/Objectives

UF will continue to perform advanced electron microscopic examination and analysis of lamellae from the neutron-irradiated uranium oxycarbide (UCO) kernels and the kernel-buffer interlayer. Characterization techniques will be expanded to include Atom Probe Tomography (APT) and a state-of-the-art STEM microscope (FEI ChemiSTEM). Work will focus on determining fission product precipitate location, chemical compositions of fission product precipitates and the fuel kernel, and micro- and nanostructures. Baseline characterization on the lamellae from unirradiated kernel and kernel-buffer interlayers may also be included, although results from work performed by Los Alamos National Laboratory (LANL) and INL on unirradiated kernels can be used as a baseline and unirradiated reference.

1.3 Anticipated Benefits

The anticipated results from the UF work scope will contribute to the knowledge of fuel kernel response to different burnups and fuel fabrication processes. The results will also be compared against those from similar microscopy performed on other specimens from the same experiments at other facilities. Advanced electron microscopy results may be used as part of a UF PhD candidate's experimental project with the INL TRISO fuel advanced microscopy lead as a PhD supervisor. At least one journal publication will be prepared during this contract's timeframe.

2. APPLICABLE CODES AND REFERENCES

ASME NQA-1-2008/-1a-2009, "Quality Assurance Requirements for Nuclear Facility Applications"

PLN-2828, "AGR-1 Post-Irradiation Examination Plan," March 2010, Rev. 1

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PLN-4616, "AGR-2 Post-Irradiation Examination Plan," December 2013, Rev. 0

3. SCOPE

3.1 Work to Be Performed

3.1.1 Microscopic Examination

Representatives from INL may elect to be present during the electron microscopy examinations at CAES. UF shall perform the examination, analyze the results, and issue a report for each lamella or series of lamellae describing the procedure used, the equipment used, analysis of the results obtained, and the findings as related to fission product precipitate locations, kernel structure, chemical composition and the items described earlier in this document as well as in Table 1.

Based on the results from the previous study performed in FY2017, three work thrusts will be executed to conclude the investigations on the irradiated TRISO fuel kernels:

- a. Perform multiple EDS spectrum mapping on the items 1 to 3 in Table 1 (8 lamellae examined by mainly point and/or line EDS analysis in FY2017 of Particles AGR2-223-RS06, AGR1-632-034 and AGR1-433-004) and item 4 (three unexamined TEM lamella from Particle AGR2-222-RS036) for obtaining multi-scale information on the spatial distribution of U, O, C and select fission products. Conduct detailed selected area diffraction of targeted areas within different lamellae. For each lamella, at least two areas of interest (AOIs) should be surveyed. For the isolated product precipitates, a spot EDS spectrum and selected area diffraction or micro-diffraction (at least two near zone axis) should be obtained.
- b. APT studies should be conducted on tips prepared from Particle AGR1-433-004 lamellae. To date, there is no reported data of the APT measurements on irradiated UCO fuel kernels. This pioneering work would provide more accurate measurement of the chemical composition of the fission products precipitates. The exact content of each isotope of a given element in a precipitate can also be obtained. The APT tip specimens will be selectively prepared from the TEM lamellae where possible. However, if it is deemed not practical to use the TEM lamellae for APT tip fabrication, APT tips will be prepared from the mounted TRISO-coated particles, already available at Electron Microscopy Laboratory (EML), at relevant locations identified from previous research information as determined by the project PIs.
- c. More advanced EDS studies using the FEI Titan with ChemiSTEM at the Irradiated Materials Characterization Laboratory (IMCL) at INL will be conducted as it is expected the facility access will be granted through a submitted rapid turn-around experiment (RTE) proposal. The study will combine the techniques of high resolution Z-contrast imaging, EDS spectral mapping with a super sensitivity and a fast speed, and selected area diffraction/micro-diffraction. The TEM lamellae will be re-thinned for better

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STEM image and diffraction, and if needed, new specimens will be prepared from the fuel Particles AGR2-223-RS06 and AGR1-632-034.

3.1.2 Procedures and Lessons Learned

Procedures used and lessons learned from previous high-resolution transmission electron microscopy (HRTEM) examinations at INL and LANL will be used during the examination of these specimens. Previous draft reports, presentations and results will be made available at start of the contract.

3.1.3 Number of Examinations to Be Performed

Focused Ion Beam (FIB) lamellae listed in Table 1 will be examined or further examined as part of this work scope. Additional TEM specimens and APT tips will be prepared from selected fuel particles as described in 3.1.1. Depending on the results obtained, the INL advanced microscopy lead may direct further investigation be performed on particular specimens. Decisions will be made jointly between UF representatives and the INL advanced microscopy lead as to the extent of additional investigation and additional specimens to be examined. UF will prepare preliminary monthly PowerPoint slides for INL to support monthly and quarterly report input.

3.1.4 Period of Performance

The work by UF at CAES, UF and INL facilities is expected to be performed during the November 2017 through September 2018 timeframe.

3.1.5 Disposition of Lamellae

INL will dispose of the lamellae once the research work and PhD thesis is complete and awarded or as determined by the INL advanced microscopy lead.

3.2 Work Excluded

3.2.1 PhD thesis

Full completion of PhD thesis during the timeframe November 2017 to September 2018 is excluded. Only the experimental work as indicated in Table 1 is included.

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Table 1. Lamellae details for TRISO fuel kernel and buffer advanced electron microscopy examination

Item	Description	Quantity of Lamella	Lamella Type	Interim Deliverables	Preliminary* Report Delivery Date
1	AGR-2 Mount D07 Compact 2-2-3 (Grid 1) Particle AGR2-223-RS06	3	UCO (10.8% FIMA)	Draft presentation monthly until preliminary report due date	Deliverable 1: EDS map and SAD Report on Particles AGR2-223-RS06, AGR1-632-034, AGR1-433-004, and AGR2-222-RS036 (Activities described in paragraph 3.1.1 (a)) due March 30, 2018
2	AGR-1 Mount 34 Compact 6-3-2 (Grid 1) Particle AGR1-632-034	2	UCO (11.3% FIMA)	Draft presentation monthly until preliminary report due date	Deliverable 2: APT measurement on Particle AGR1 433-004 Report(Activities described in paragraph 3.1.1 (b)) due July 30, 2018
3	AGR-1 (Safety tested (1600°C)) Mount 36V Compact 4-3-3 (Grid 1) Particle AGR1-433-004	3	UCO (Safety tested)	Draft presentation monthly until preliminary report due date	Deliverable 3: High resolution EDS analysis of Particles AGR2-223-RS06 and AGR1-632-034 (Activities described in paragraph 3.1.1 (c)) due September 15, 2018
4	AGR-2 Safety tested (Safety tested (1600°C)) Mount 25D Compact 222 (Grid To be determined) Particle AGR2-222-RS036	3	UCO (12.6% FIMA)	Draft presentation monthly until preliminary report due date (Pending on NSUF-RTE award)	
At least one journal article (contents to be confirmed with mutually agreement between UF and INL PhD supervisors, but potentially a comparison between particles AGR1-632-034 and AGR2-223-RS06).					Paper submitted January 2018 and August 2018
*Preliminary report will include all the results obtained during the electron microscopy examination as well as preliminary interpretation and conclusions. It is understood that full interpretation may not be completed and will still take place during the thesis writing, which is not a deliverable under this contract. The results reported in these deliverables may be used in INL integrated report(s), papers and presentations, in collaboration with UF.					

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3.3 Requirements**3.3.1 Environmental**

Work will be performed in accordance with applicable CAES and INL requirements.

3.3.2 Safety and Health

Work will be performed in accordance with applicable CAES and INL requirements.

3.4 Quality Assurance

Work performed at INL and CAES will be in accordance with an approved CAES and INL Quality Assurance program to NQA-1-2008/-1a-2009. Electron Microscopy performed at UF will be reviewed and approved by the INL Advanced Microscopy Lead. The quality level for this work is QL-3.

3.5 Place of Performance

The work scope identified in this SOW will be performed at the Electron Microscopy Laboratory at CAES, IMCL or UF.

3.6 Interfaces

Interfaces will be between INL and UF technical representatives.

3.7 Miscellaneous**3.7.1 Presentations and Reports**

Preparation of presentations, reports for publication, and travel will be included within this work scope in order to share the results. This will be charged to the appropriate activity as approved by the INL advanced microscopy lead. All data, reports, and/or presentations related to this activity must be reviewed and approved by the INL advanced microscopy lead or a designee prior to issuance or distribution.

4. DELIVERABLES

UF will prepare and issue the reports at the dates indicated in Table 1. These reports shall describe the examinations performed, the procedures used, the equipment used, the observations made, analyses performed, and results obtained.

All raw data (including but not limited to images and chemical analyses) will be provided to INL electronically in addition to the reports described above.

Preliminary PowerPoint presentations will be sent to INL to support monthly and quarterly report input.

5. SCHEDULE AND MILESTONES

See the deliverables due and dates for submittal to INL in Table 1.

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6. COMPLETION CRITERIA AND FINAL ACCEPTANCE

Reports will be reviewed and approved by the INL advanced microscopy lead. All comments made during review of the reports will be incorporated into the report or rationale provided for not including them.

7. APPENDIXES

None.

8. ATTACHMENTS

None.