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Statement of Work

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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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Idaho National Laboratory

ADVANCED REACTOR TECHNOLOGIES

Signatures

Signature and Typed or Printed Name	Signature Code	Date (MM/DD/YY)	Organization/Discipline
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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

Rev.	Date	Affected Pages	Revision Description
0	02/23/2017	All	New issue
1	11/03/2017	All	Mainly Section 3.1.1 and Table 1: FY2018 work scope
2	11/30/2018	All	Mainly Sections 3.1.1, 3.1.2 and Table 1: FY2019 work scope
3	02/05/2020	All	Mainly Section 3.1 and Table 1: FY 2020 work scope

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TABLE

1. INTRODUCTION

1.1 Background

The Advanced Reactor Technologies (ART) Program 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 facilities. 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), electron dispersive spectroscopy (EDS) and atom probe tomography (APT) on the fuel kernels of TRISO-coated fuel particles. The electron microscopy lamellae will be provided by INL and will be available at CAES or Irradiated Materials Characterization Laboratory (IMCL).

1.2 Purpose/Objectives

UF will continue to perform advanced electron microscopic examination and analysis of lamellae from the neutron-irradiated uranium oxycarbide (UCO) fuel kernels and the kernel-buffer interlayer. APT and a state-of-the-art STEM microscope (FEI ChemiSTEM) equipped with EELS will be expanded to provide more quantification of C, O and U ratios as a function of kernel diameter to determine potential stoichiometry changes with irradiation and/or position within the kernel. Work will continue to focus on determining fission product precipitate location, chemical compositions of fission product precipitates and the fuel kernel, and micro- and nanostructures. Specifically, the crystalline structure of the fission product precipitates will be determined. Baseline characterization on the lamellae from unirradiated kernel and kernel-buffer interlayers and work performed by Los Alamos National Laboratory (LANL) and INL on unirradiated kernels can be used as a fuel kernel fabrication baseline 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 facilities. 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

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

3. SCOPE

3.1 Work to Be Performed

Representatives from INL may elect to be present during the electron microscopy examinations at CAES or UF. 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 studies performed in FY2017, FY2018 and FY2019, three main research thrusts will be executed to obtain knowledge on the effect of irradiation on TRISO fuel kernels. Related Nuclear Science User Facilities (NSUF) rapid turn-around experiment (RTE) proposals may be submitted to leverage funds in support of the work topics. Progress reporting on NSU-RTE's will be reported monthly in a separate section from the main activities indicated in Table 1.

3.1.1 Determining Stoichiometry Changes in the Fuel Kernels Through Quantifying the Light Elements (C, O):

a) Conduct EELS characterization on the irradiated fuel kernels for accurately quantifying the light elements (C, O), which is critical for determining the stoichiometry changes of the UCO fuel as well as how they affect the fuel performance.

b) Conduct extensive APT characterizations for understanding the fission product behavior at a sub-nano scale. The work will complement the results from the ChemiSTEM TEM. A better APT tip sample preparation procedure will be investigated to make the sample more site-specific and contamination free.

c) Electron microscopy integration with microanalysis techniques: Although, detailed TEM/STEM/APT studies can provide significant information for the fission products local distribution, phase structure, co-precipitate behaviors at scales of sub-micro/micro, nano and atomic resolution, large area electron probe micro analyzer (EPMA) survey would offer critical complementary information for fusing the multi-scale data together. Observing the large picture from EPMA measurement with detailed information from TEM/STEM/APT studies may provide a mechanistic understanding on the fission products migration and release behavior. TEM and APT specimens shall be prepared from interesting features/phases/precipitates under the guidance of EPMA elemental maps. In addition, an algorithm will be developed to integrate the EPMA data along the fuel particle circumferential direction and to

construct fission products radial distribution profiles to better utilize EPMA measurements for drawing statistically important conclusions.

3.1.2 High Resolution Z-contrast Imaging, EDS Spectral Mapping With a Super Sensitivity and a Fast Speed, and Selected Area Diffraction/Micro-diffraction (SAD) Obtaining Multi-scale Information on the Spatial Distribution of U, O, C and Select Fission Products.

a) Perform the high-resolution STEM/EDS mapping on one or two pairs of irradiated TRISO fuel particles (in both the as-irradiated condition and after safety testing). The direct comparison will help to clearly elaborate the fission products migration behavior upon an accident scenario. The research will also cover more detailed studies on the lamellas lifted out from the interface between the fuel kernel and buffer layer.

3.1.3 Determining Comparative Baseline Microstructural Evolution of Fabrication Process and Neutron Irradiation:

a) Conduct STEM and EELS characterization on the as-fabricated kernel, as-compacted kernels and irradiated fuel kernels for accurately quantifying the light elements (C, O), which is critical for determining the stoichiometry changes of the UCO fuel as well as how they affect fuel performance. Earlier baseline work performed by LANL and INL was based on particle fabrication processes only. The compact fabrication process involves a high sinter temperature (1800 ˚C) and is understood to change the microstructure of the UCO kernels. Understanding of this process through high resolution STEM and associated diffraction patterns has not been performed previously and is necessary to complete a full understanding of irradiation effects on UCO kernel microstructure.

3.1.4 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 the start of the contract.

3.1.5 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 and 3.1.2. 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.

3.1.6 Period of Performance

The work by UF at CAES, UF and INL facilities is expected to be performed during the February 2020 through September 2020 timeframe.

3.1.7 Disposition of Lamellae

INL will dispose of the lamellae once the research work and PhD thesis are 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 is excluded. Only the experimental work as indicated in Table 1 is included.

Table 1. Lamellae details for TRISO fuel kernel and buffer advanced electron microscopy examination (Color coding indication: Green = fully completed; Yellow = partially completed, completion in FY2020; no color = new FY 2020 work)

Item	Particle(s)	Lamella Quantity	Lamella Type	Interim Deliverables	Description of Research and Preliminary* Report Delivery Date
Journal Papers and Milestone Reports					
1	Paper 1 UCO kernel examination on safety tested particle AGR1-433-004	N/A	N/A	INL internal review November 2018	**Deliverable 1:** JOU-18-51977 "Microstructure and fission products in the UCO kernel of a AGR-1 TRISO fuel particle after post-irradiation safety annealing" Date submitted to Journal: December 2018
2	Paper 2 UCO kernel examination on AGR1-632-034 and AGR2-223-RS06	N/A	N/A	INL Internal review February 2020	**Deliverable 2:** "Comparison of microstructural and micro-chemical evolutions between ATR irradiated AGR-1 and AGR-2 fuel particles" Estimated submission to Journal: March 2020
3	UCO Kernel microstructure Milestone Report	N/A	AGR-1 and AGR-2	Monthly progress report against the specific activities in table format.	**Milestone report:** UF will contribute to specific sections as determined by advanced electron microscopy lead. Estimated submission to INL: Mid July 2020 The deliverables and draft reports as shown in activities 4 to 10 will need to be delivered on time as scheduled as it contains data for input to the milestone report Estimated submission to INL: As scheduled
Electron Microscopy or Micro Analysis					

Item	Particle(s)	Lamella Quantity	Lamella Type	Interim Deliverables	Description of Research and Preliminary* Report Delivery Date
4	**Particle AGR1-632-034** (Compact 6-3-2, Mount 48, Particle 34) **Particle AGR2-223-RS06** (Compact 2-2-3, Mount D07, Particle RS06)	3 3	UCO (11.3% FIMA) UCO (12.68% FIMA)	Partially funded by NSUF-RTE **NSUF-RTE-19-1779**, awarded April 2019 Data collection completed, report to be completed Section 3.1.1 (a)	**Deliverable 3: Preliminary Report Electron Energy Loss Spectroscopy (EELS) and atomic resolution STEM:** The proposed EELS and atomic scale STEM work will complement our previously acquired characterization results by using a FEI Titan Themis 200 scanning/transmission electron microscope with ChemiSTEM. It is aimed to particularly quantify the light elements (e.g., O, C) to determine the stoichiometry of UC and UO phases and to determine the atomistic structure of the previously identified intermetallic phases. Expected draft report to INL completion date: May 2020
5	**Particle AGR1-632-034** (Compact 6-3-2, Mount 48, Particle 34) **Particle AGR2-223-RS06** (Compact 2-2-3, Mount D07, Particle RS06)	3 3	UCO (11.3% FIMA) UCO (12.68% FIMA)	Partially funded by **NSUF-RTE 18-1593** Data collection completed, report to be drafted Section 3.1.1 (c) and 3.1.1 (b)	**Deliverable 4: APT (ATP): Preliminary Report** The proposed APT work is aimed to complement previously acquired knowledge using the FEI Titan with ChemiSTEM, particularly for quantifying the light element diffusion and segregation. More specifically, the APT tips will be prepared onto the sharpened tungsten wires of the half TEM grid (100 mesh), and this will allow for both TEM characterization and APT data acquisition. This approach was not attempted or achieved previously and will provide a better understanding by direct correlation between the fuel performance and the fabrication process. Expected completion date: **April 2020**

Item	Particle(s)	Lamella Quantity	Lamella Type	Interim Deliverables	Description of Research and Preliminary* Report Delivery Date
6	**Particle AGR2-222-RS36** AGR-2 Safety tested (Safety tested (1600°C)) Mount 25D, Compact 222 (Grid To be determined)	3	UCO (12.6% FIMA)	Data collection completed; Draft report completed Section 3.1.1 (c) and 3.1.2 (a)	**Deliverable 5**: Preliminary Report High resolution EDS analysis and maps and SAD Date completed: December 30, 2019
7	**Particle AGR2-222-RS19** AGR-2 Safety tested (Safety tested 1600°C) Mount 26D, Compact 222 (Grid To be determined)	3	UCO (12.6% FIMA)	Draft presentation monthly until preliminary report due date Section 3.1.1 (c) and 3.1.2 (a)	**Deliverable 6: Preliminary Report** High resolution EDS analysis and maps and SAD (Activities described in paragraph 3.1.2 (a)) Expected completion date: May 15, 2020
8	**Particle AGR2-633-RS28** AGR-2 Mount 42D, Compact 633 (Grid To be determined)	3	UCO (7.46% FIMA)	Draft presentation monthly until preliminary report due date Section 3.1.2	**Deliverable 7: Preliminary Report** High resolution EDS analysis and maps and SAD Expected completion date: June 30, 2020
9	As-fabricated kernels and as-compacted kernels	3	UCO unirradiated	Section 3.1.3	**Deliverable 8: Preliminary report to INL** Expected completion date May 2020
10**	**Particle AGR2-633-RS09** AGR-2 Mount 43D, Compact 633 (Grid To be determined)	3	UCO (7.46% FIMA)	Draft presentation monthly until preliminary report due date Section 3.1.2	**Deliverable 9: Preliminary Report** High resolution EDS analysis and maps and SAD Expected completion date: July 30, 2020

Item	Particle(s)	Lamella Quantity	Lamella Type	Interim Deliverables	Description of Research and Preliminary* Report Delivery Date
UF will provide monthly progress bullet points with main scientific findings and a summarized technical feedback for quarterly reports.					Monthly and quarterly

*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.

** Dependent on the schedule for completion of FIB sample preparation at INL, if this work cannot be complete due to unavailability of samples, work will be replaced by the completion of a third journal paper on any of the integration of deliverables 4-9.

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 PLN-2690- INL ART Quality Assurance Plan 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. 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.

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.