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Plan

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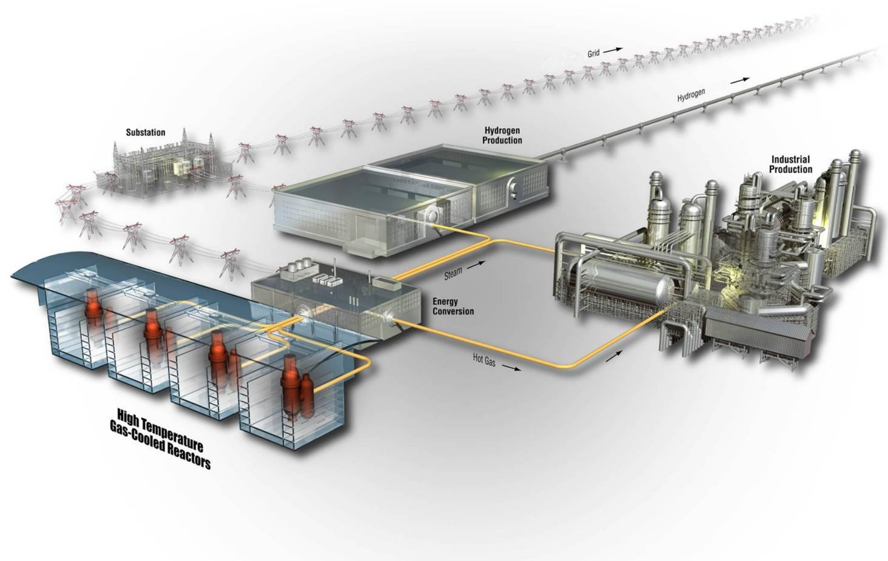
FITT Oxidation Test Plan



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REVISION LOG

Rev.	Date	Affected Pages	Revision Description
0	12/16/2020	All	New issue.
1	06/02/2021	3	Updated second bullet under Section 4.2 to include new STEM work made possible by a budget increase.

CONTENTS

TABLE

ACRONYMS

AGR	Advanced Gas Reactor
AGR-2	second AGR program irradiation experiments
DLBL	deconsolidation leach-burn-leach
IMGA	Irradiated Microsphere Gamma Analyzer
FIB/SEM	focused ion beam / scanning electron microscope
FIMA	fissions per initial metal atom
FITT	Furnace for Irradiated TRISO Testing
INL	Idaho National Laboratory
ORNL	Oak Ridge National Laboratory
PIE	post-irradiation examination
SiC	silicon carbide
TAVA	time-average, volume-average (compact irradiation temperature)
TRISO	tristructural isotropic (coated particles)
UCO	uranium carbide and uranium dioxide (multiphase kernels)

1. INTRODUCTION

This plan describes the post-irradiation examination (PIE) oxidation testing activities to be performed by Oak Ridge National Laboratory (ORNL) on individual particles from Compact 5-4-2 taken from the Advanced Gas Reactor (AGR) experiment, AGR-2. This work will be performed in accordance with the general objectives outlined in the AGR-2 PIE Plan[1] and guidance in the ORNL PIE Statement of Work.[2]

2. FUEL COMPACT DESCRIPTION

The fuel specimen contains tristructural isotropic (TRISO)-coated particles with kernels containing mixed uranium carbide and uranium oxide (UCO). The compact was irradiated in Capsule 5 of the second AGR irradiation (AGR-2) in the Advanced Test Reactor at Idaho National Laboratory (INL).[3] Table 1 shows some identifiers and irradiation conditions for AGR-2 Compact 5-4-2 and is reproduced from ORNL/TM-2018/863.[4] Particles were liberated from the compact following the typical deconsolidation leach-burn-leach (DLBL) process as described in the AGR-2 As-Irradiated UCO Compact 5-4-2 PIE Report.[4]

Table 1. Identification and irradiation conditions for AGR-2 Compact 5-4-2.

Compact ID[a]	Fuel Type	Fabrication ID	Burnup [b] (% FIMA)	Fast Fluence [b] ($\times 10^{25}$ n/m^2)	TAVA (°C) [c]
AGR-2 5-4-2	UCO	LEU09-OP2-Z059	12.03	3.14	1071

[a] The X-Y-Z compact identification (ID) convention denotes the location in the irradiation test train: Capsule-Level-Stack.[3]
[b] Physical properties data for individual compacts are available and tabulated based on fabrication ID in Hunn, Montgomery, and Pappano 2010, pages 60–69.[5]
[c] Burnup in fission per initial metal atom (FIMA) in Sterbentz 2014, Table 6 and fast fluence in Sterbentz 2014, Table 12 are based on physics calculations.[6]
[d] Time-averaged, volume-averaged (TAVA) temperature is based on thermal calculations in Hawkes 2014, Table 3.[7]

3. EXPERIMENTAL OBJECTIVES

- Explore oxidation behavior of the SiC layer in individual irradiated and unirradiated particles under various temperatures and oxidant conditions.
- Measure failure fraction of individual loose particles from irradiated AGR-2 Compact 5-4-2 as a function of oxidation conditions and exposure time.
- Analyze and quantify oxidation microstructure in individual particle samples.
- Develop insights on differences in oxidation kinetics of irradiated-TRISO SiC relative to as-fabricated, unirradiated fuel.

4. SCOPE OF WORK

4.1 Oxidation testing

A series of individual particles will be heated in oxidizing conditions in the Furnace for Irradiated TRISO Testing (FITT). Testing will consist of 10 irradiated particles isolated from Compact 5-4-2 via the DLBL process and 10 unirradiated AGR-2 UCO TRISO particles that were “burned-back” to expose the silicon carbide (SiC). The DLBL process removed the outer pyrolytic carbon (OPyC) layer to expose the underlying SiC layer of the Compact 5-4-2 particles. The unirradiated particles are from the same composite of AGR-2 UCO particles used to make Compact 5-4-2, and were similarly subjected to the compacting process, deconsolidated from a heat-treated compact, and burned in forced air at 750°C for 72 hours, which is equivalent to the burn step in the DLBL process applied to irradiated Compact 5-4-2. This process removed the OPyC layer from these unirradiated particles.

Testing will follow the standard operating procedure for FITT operation (NFM-PIE-SOG-01, Revision 2).[8] Particles will be placed in a SiC holder with a partition to isolate irradiated particles from unirradiated burned-back particles. The SiC holder will be housed in an alumina crucible and placed in the bottom of a closed-end alumina tube. The oxidant (21% O_2 balance N_2 or 2% O_2 balance He) will be delivered from a predetermined composition via compressed gas cylinder as a sweep gas at a targeted flow rate of 100 mL/min. After oxidation testing, the Compact 5-4-2 particles will be removed from the furnace and transferred to the Irradiated Microsphere Gamma Analyzer (IMGA) hot cell for visual inspection and individual particle gamma analysis to measure gamma-emitting fission product inventories following a similar process outlined in Hunn et al. 2013.[9] Particles of interest will be isolated for microanalysis to measure the oxide thickness using a dual beam focused ion beam and scanning electron microscope (FIB/SEM) located in the Irradiated Fuels Examination Laboratory. Analysis will consist of FIB milling of the external particle surface after deposition of a platinum cap to create a particle cross-section. This will allow direct analysis of the oxide layer thickness. This approach is preferred over standard materiallographic particle cross-sectioning which may cause rounding of the outer surface and compromise the analysis of the expected oxide scale. The response of the irradiated particles to oxidation conditions will be compared to the response of the unirradiated particles which serve as a reference case. This comparison includes visual inspection to observe gross failure and oxide layer analysis to determine variation in potential oxidation kinetics.

The test matrix for oxidation testing is listed in Table 2. The test matrix was developed based on the failure fractions observed for related tests reported in the literature.[10] The experiments capture conditions which would be expected to lead to complete failure (1400°C, 400 h) in the passive oxidation regime, and less aggressive tests (1400°C, < 400 h) which would explore potential progressive failure rates and support oxidation kinetics analysis. The 1400°C, 21% O_2 balance inert test condition is expected to explore an oxide thickness of 2–16 µm.[11] These layers are expected to be readily observable by the planned FIB/SEM analysis. The test matrix also includes a comparison of the impact of temperature (1200°C versus 1400°C) and oxygen partial pressure on particle stability. The difference in inert carrier gas reflects different types of mimicked accident scenarios. The lower oxygen partial pressure is expected to straddle the transition from passive to active oxidation.[12]

Table 2. Planned test matrix for oxidation testing of irradiated particles.

Temperature	Atmosphere	T1	T2	T3	T4 [a]
1200°C	21% O_2 (balance N_2)				400 h
1400°C	21% O_2 (balance N_2)	50 h	100 h	200 h	400 h
1400°C	2% O_2 (balance He)				400 h
[a.] Comparison study will depend on the observations from the 1400°C, T4 run.					

The first test to be run will be at 1400°C, 50 h, with 21% O_2 (balance N_2). Progressively more aggressive tests will follow the first test run and are subject to change based on observed oxidation behavior and failure fractions. The final two tests will be the temperature and O_2 partial pressure comparison. The specific test conditions will be selected to match the longest 1400°C, 21% O_2 (balance N_2) condition.

4.2 Data Acquisition, Analysis, and Reporting

A FITT test report will be prepared and will include a description of the experiments performed and all relevant data acquired. Overall data to be reported will include the following, as applicable:

- Initial analysis will include optical survey of particle integrity after oxidation and post-oxidation IMGA analysis of recoverable irradiated particles to determine particle failure fraction. Subsequent analysis will include inventory fraction analysis that compares measured inventory to compact average inventories in order to inform fission product release and particle failure.
- Oxide scale thickness of the outer SiC surface will be analyzed via direct measurement using FIB/SEM techniques from a minimum of three unique test conditions and will include both irradiated and unirradiated particles. Select locations will be targeted for scanning transmission electron microscopy (STEM) analysis. Analysis by STEM will be used to determine differences in oxide layer microstructure and composition between the irradiated and unirradiated particles exposed to identical oxidation conditions. Analysis will be performed on a minimum of one unique test condition. Samples for STEM analysis will be prepared using the FIB/SEM system.

5. QUALITY ASSURANCE

Activities performed at ORNL shall be performed in accordance with applicable ORNL procedures and the ORNL Quality Assurance Plan for Nuclear Research and Development Activities[13] to meet the INL Quality Assurance requirements specified in Inter-Entity Work Order #150293.

6. REFERENCES

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