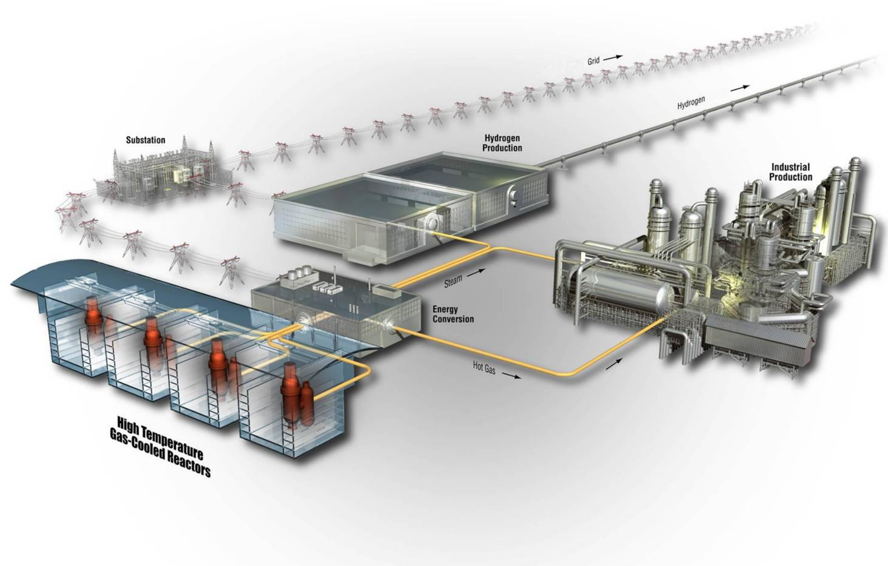
Document ID: PLN-6221
Revision ID: 0
Effective Date: 11/02/2020
INL/MIS-20-60365

Plan

Project No. 29412, 23841

AGR-3/4 Compact 7-4 Examination Plan



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

Idaho National Laboratory

Prepared by:

[signature]	11/2/2020
John D. Stempien Fuels Post-Irradiation Examination Technical Lead	Date
[signature]	11/02/2020
John D. Hunn Oak Ridge National Laboratory Technical Lead	Date

Approved by:

Paul Demkowicz [signature]	11/02/2020
Paul A. Demkowicz INL ARD TRISO Fuel Technical Director	Date
Michelle Sharp [signature]	11/02/2020
Michelle T. Sharp INL ARD Quality Assurance	Date

REVISION LOG

Rev.	Date	Affected Pages	Revision Description
0	11/02/2020	All	New issue.

CONTENTS

TABLE

ACRONYMS

AGR	Advanced Gas Reactor
AGR-3/4	third and fourth AGR program irradiation experiments
DTF	designed-to-fail (coated particles)
FIMA	fissions per initial metal atom
IMGA	Irradiated Microsphere Gamma Analyzer
INL	Idaho National Laboratory
LBL	leach-burn-leach
ORNL	Oak Ridge National Laboratory
PIE	post-irradiation examination
SiC	silicon carbide (coating layer)
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) activities to be performed by Oak Ridge National Laboratory (ORNL) on irradiated Compact 7-4 taken from the Advanced Gas Reactor (AGR) experiment, AGR-3/4. This work will be performed in accordance with the general objectives outlined in the AGR-3/4 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 driver fuel particles with kernels containing mixed uranium carbide and uranium oxide (UCO), as well as 20 designed-to-fail (DTF) particles consisting of UCO kernels with ~20 µm thick, anisotropic, high density pyrolytic carbon coatings that were expected to crack to expose the kernel during irradiation. The compact was irradiated in Capsule 7 of the AGR-3/4 test train in the northeast flux trap of the Advanced Test Reactor at the Idaho National Laboratory (INL).[3] Table 1 shows some identifiers and irradiation conditions for AGR-3/4 Compact 7-4.

Table 1. Identification and irradiation conditions for AGR-3/4 Compact 7-4.

Compact ID[a]	Container ID	Fabrication ID	Burnup[b] (% FIMA)	Fast Fluence[b] ($\times 10^{25}$ n/m^2)	TAVA (°C)[c]
AGR-3/4 7-4	AGR352	(LEU03-10T-07 DTF)-Z111	14.90	5.24	1319

a. The X-Y naming convention denotes the location in the irradiation test train—Capsule-Level.[1]
b. Fissions per initial metal atom (FIMA) and fast neutron fluence (E_n>0.18 MeV) are based on physics calculations.[4]
c. Time-average, volume-average (TAVA) temperature is based on thermal calculations.[5]

3. EXPERIMENTAL OBJECTIVES

- Radially deconsolidate the compact in a stepwise fashion and acid leach the particles and matrix debris from each discrete step using a leach-burn-leach (LBL) process, as described in Section 4.1, to measure the compact inventory of actinides and fission products not contained within intact silicon carbide (SiC) layers.
- Examine individual particles deconsolidated from the as-irradiated compact with the Irradiated Microsphere Gamma Analyzer (IMGA), as described in Section 4.2, to quantify retention of specific gamma-emitting fission products (including ^{106}Ru, ^{125}Sb, ^{134}Cs, ^{137}Cs, ^{144}Ce, and ^{154}Eu) and to identify anomalous particles, especially those with a below-average cesium inventory indicative of silicon carbide (SiC) layer failure.
- Perform microanalysis on selected particles, as described in Section 4.3, to better understand the correlation of particle microstructure with fission product retention. Microanalysis of any particles with below-average ^{144}Ce or ^{137}Cs/^{144}Ce ratio, indicative of failed TRISO or failed SiC respectively, is of particular interest, and such particles should have the highest priority for examination.

4. SCOPE OF WORK

4.1 Radial Deconsolidation and Acid Leaching

The fuel compact will be electrolytically deconsolidated in 4–8M nitric acid to break up the matrix material and free the fuel particles (AGR-3/4 UCO compacts have ~1898 TRISO driver fuel particles and 20 DTF particles[6]). The deconsolidation will be done in four segments of roughly equal volumes as shown in Table 2. The first three segments will be radially deconsolidated by rotating the compact about the cylinder axis and collecting particles and matrix debris in three stages. The fourth segment will be deconsolidated by axially deconsolidating the remaining cylinder core, which is expected to contain all the DTF particles (most of which will not be intact). Actual section volumes will be determined by a combination of video and/or still imaging with image analysis software designed for this application.[7,8]

Table 2. Target sectioning dimension for radial deconsolidation

Compact section	Inner diameter (mm)	Outer diameter[a] (mm)	Section thickness (mm)	Section Volume[b] (cm^3)
Whole compact	0.00	12.094		1.44
1st section	10.474	12.094	0.810	0.359
2nd section	8.552	10.474	0.961	0.359
3rd section	6.047	8.552	1.252	0.359
Core section	0.00	6.047	3.024	0.359

[a] Measured average diameter for irradiated Compact 7-4 was 12.094 mm.[9]
[b] Measured average length used to compute the volume for irradiated Compact 7-4 was 12.5095 mm.[9]

The four sets of deconsolidated particles and matrix debris will be individually subjected to an LBL process as described in AGR-CHAR-DAM-37[10]. Particles and matrix debris will be transferred to a Soxhlet thimble, two 24-hour nitric acid leaches in a Soxhlet extractor performed, and the leachates analyzed for actinides and fission products. After the two pre-burn leaches, further digestion of the particles and matrix debris in boiling acid will be performed to break up the matrix debris further and help remove any matrix residue from the TRISO particles. The matrix debris will be separated from the particles by washing through a sieve stack. The sieve stack will include a 1400 µm sieve to collect undeconsolidated remnants, a 600 µm sieve to collect intact driver fuel particles, a 250 µm sieve to collect particle coating fragments and intact DTF particles, and a bottom pan to collect liquids, matrix debris, and other fines. Recovered particles will be rinsed, dried, and transferred to the hot cell cubicle housing the IMGA, where they will undergo visual inspection and gamma survey as described in Section 4.2.

The separated matrix debris will be dried by distilling off the acid. The dry matrix debris will be heated at 750°C in air to burn off the carbon and oxidize metallic fission products (some metal carbides have low solubility in nitric acid). The residual ash and burn vessel will be subjected to two post-burn boiling nitric acid leaches and the leachates analyzed for actinides and fission products.

After completion of the IMGA survey as described in Section 4.2, an archive sample of about 10% will be riffled out, and the remaining 90% of the TRISO particles will be returned to the main cell for particle burn-leach. Any intact DTF particles that were identified by visually inspecting the material in the 250 µm sieve will also be held out, while the remaining particle coating fragments will be returned with the TRISO particles for burn-leach. Particles and coating fragments will be loaded back into the Soxhlet thimble used for pre-burn leaching. Similar to the matrix burn-leach, particles will be heated at 750°C in air to remove the exposed carbon and then leached twice in the Soxhlet extractor. Analysis of the particle burn-leach solutions will be performed to detect actinides and fission products not leached before the burn, including any exposed kernels from particles with failed SiC not separated out during IMGA analysis. After burn-leach, the burned-back particles will be washed, dried, and archived.

4.2 Particle Inspection and Gamma Analysis

Prior to particle burn-leach, deconsolidated particles will be inspected and imaged using the particle micro-manipulator in the IMGA cubicle to count the particles, assess the overall condition, and identify features of interest such as cracked coatings, coating fragments, or intact DTF particles. Abnormal particles or coating fragments may be selected for further examination. Any intact DTF particles will be imaged and saved for IMGA analysis.

All intact driver fuel particles will be gamma counted using a short counting time determined by the minimum time required for acquisition of sufficient counts above background to measure ^{137}Cs and ^{144}Ce. The IMGA will sort out any particles with a low ^{137}Cs/^{144}Ce ratio or low ^{144}Ce content. Below-average ^{137}Cs/^{144}Ce ratio is indicative of significant ^{137}Cs release during irradiation due to a failed SiC layer. Below-average ^{144}Ce content may indicate abnormal kernels or general fission product loss that could be due to a failed-TRISO particle. An alternate for ^{144}Ce may be selected, such as ^{106}Ru or ^{125}Sb, if counting statistics are more favorable for the alternate radioisotope.

All particles sorted out due to abnormal ^{137}Cs or ^{144}Ce inventory will undergo a longer count time (typically 3–6 hours) to more accurately measure radioisotopic inventory (^{106}Ru,^{125}Sb, ^{134}Cs, ^{137}Cs, ^{144}Ce, and ^{154}Eu). Long-count IMGA analysis may also be performed on a random riffled sample of particles (~15 particles from each segment).

4.3 Microstructural Analysis

After IMGA analysis, driver fuel particles of interest will be selected for x-ray imaging and/or materialography, with highest priority for particles with a below-average ^{137}Cs retention or low overall radioisotopic inventory that may be indicative of failed SiC or failed TRISO. If intact DTF particles are recovered, discussions with the INL Fuels PIE Technical lead will determine what to do with those DTF particles following IMGA. After discussions with the INL Fuels PIE Technical Lead, intact DTF particles could be sent to INL for reirradiation/heating testing, subjected to x-ray imaging, and/or subjected to materialography. For both TRISO particles and DTF particles, materialography may include optical imaging, scanning electron microscopy imaging, and/or elemental analysis of polished cross sections. Based on the specific results and discussions with the INL Fuels PIE Technical Lead, individual particles may be sent to INL for additional microanalysis.

X-ray imaging with tomographic reconstruction will be used to achieve non-destructive examination of the internal structure of individual particles. Particles identified with the IMGA to have below-average ^{137}Cs retention or low overall radioisotopic inventory will be subjected to x-ray tomography prior to any destructive analysis. Three-dimensional visualization of the tomographic data can provide important insight to complement and guide materialographic examination.

Particles for materialography will be mounted in epoxy and the mounts polished to inspect particle cross-sections using microscopic methods. Optical microscopy can be used to inspect the overall condition of kernels and coatings. Scanning electron microscopy can be used to perform higher resolution inspections of kernel and SiC microstructures. Energy-dispersive and wavelength-dispersive x-ray spectroscopy can be used to characterize the elemental distributions within the kernel and coating layers, as well as fission product attack of the SiC layer, where palladium or uranium clustering and interaction with the SiC are of particular interest.

4.4 Data Acquisition, Analysis, and Reporting

A compact PIE 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:

- A compact fractional inventory of fission products released during irradiation, based on as-run inventory calculations[4] and segmented in four radial segments as discussed in Section 4.1,
- Results of particle inspection and IMGA examination of individual particles,
- X-ray and materialographic images, including detailed analysis of intact DTF particles and driver fuel particles with low-cesium retention,
- Discussion of any unusual particle, kernel, or coating behavior that may be linked to fission product releases.

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 [11] to meet the INL Quality Assurance requirements specified in Inter-Entity Work Order #150293.

6. REFERENCES

1. Demkowicz, P.A., 2017, *AGR-3/4 Phase 2 Post-Irradiation Examination Plan*, PLN-5382, INL/MIS-17-41954, Rev. 0, Idaho National Laboratory, May 2017.
2. Idaho National Laboratory, 2020, *Statement of Work for the AGR-3/4 PIE at Oak Ridge National Laboratory*, SOW-12084, INL/MIS-15-34327, Revision 6, April 2020.
3. Collin, B.P., 2016, *AGR-3/4 Irradiation Test Final As-Run Report*, INL/EXT-15-35550, Rev. 1, Idaho National Laboratory, May 2016.
4. Sterbentz, J.W., 2015, *JMOCUP As-Run Daily Physics Depletion Calculation for the AGR-3/4 TRISO Particle Experiment in ATR Northeast Flux Trap*, ECAR-2753, Rev. 1, Idaho National Laboratory, July 2015.
5. Hawkes, G.L., 2016, *AGR-3/4 Daily As-Run Thermal Analyses*, ECAR-2807, Rev. 1, Idaho National Laboratory, April 2016.
6. Hunn, J.D., Trammell, M.P., and Montgomery, F.C., 2011, *Data Compilation for AGR-3/4 Designed-to-Fail (DTF) Fuel Compact Lot (LEU03-10T-OP2/LEU03-07DTF-OP1)-Z*, ORNL/TM-2011/124, Rev. 0, Oak Ridge National Laboratory, June 2011.
7. Helmreich, G.W., Montgomery, F.C., and Hunn, J.D., 2015, *Development of a Radial Deconsolidation Method*, ORNL/TM-2015/699, Rev. 0, Oak Ridge National Laboratory, December 2015.
8. Stempien, J.D., 2017, *Radial Deconsolidation and Leach-Burn-Leach of AGR-3/4 Compacts 3-3, 12-1, and 12-3*, INL/EXT-17-43182, Rev. 0, Idaho National Laboratory, September 2017.
9. Stempien, J.D., Rice, F.J., Winston, P.L., and Harp, J.M., 2016, *AGR-3/4 Irradiation Test Train Disassembly and Component Metrology First Look Report*, INL/EXT-16-38005, Rev. 1, September 2016.
10. Montgomery, F.C., and Hunn, J.D., 2020, *Data Acquisition Method for Leach-Burn-Leach of Irradiated Fuel Compacts Using a Soxhlet Extractor in the 3525 Hot Cell*, AGR-CHAR-DAM-37, Rev. 4, Oak Ridge National Laboratory, February 2020.

11. Vance, M.C., 2018, QAP-ORNL-NR&D-01, *Quality Assurance Plan for Nuclear Research and Development Activities Conducted at the Oak Ridge National Laboratory*, Rev. 1, Oak Ridge National Laboratory, October 2018.