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Irradiated AGR 2 Compact 6 2 3 Examination Plan

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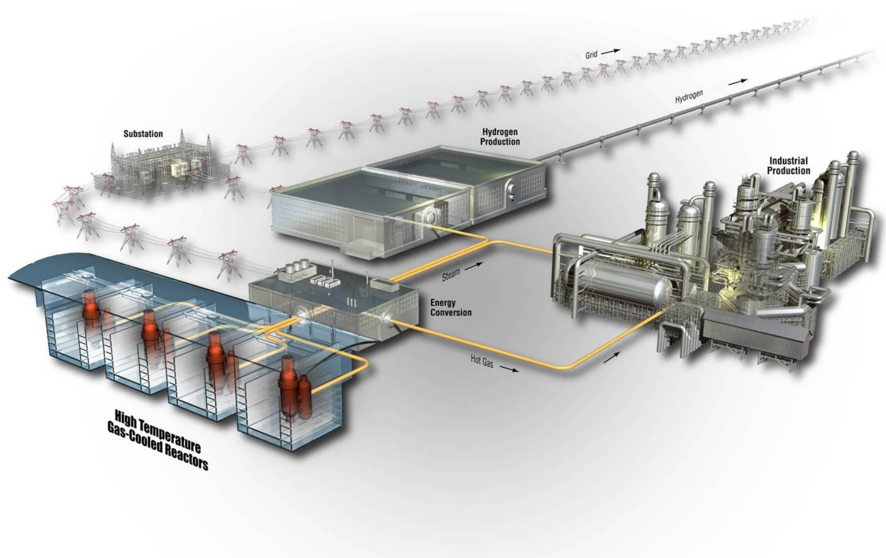
Irradiated AGR-2 Compact 6-2-3 Examination Plan



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REVISION LOG

Rev.	Date	Affected Pages	Revision Description
0	05/23/2017	All	New issue.

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ACRONYMS

AGR	Advanced Gas Reactor
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/uranium oxide (kernels)

1. INTRODUCTION

This plan describes the post-irradiation examination (PIE) activities to be performed by Oak Ridge National Laboratory (ORNL) on irradiated Compact 6-2-3 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 both uranium carbide and uranium oxide (UCO) and was irradiated in Capsule 2 of the AGR-2 test train in the B-12 position in the Advanced Test Reactor at Idaho National Laboratory (INL).[3] Table 1 shows some properties and irradiation conditions for AGR-2 Compact 6-2-3.

Table 1. Identification and irradiation conditions for AGR-2 Compact 6-2-3.

Compact ID[a]	Compact Container ID	Fabrication ID	Fuel Type	Average Burnup (% FIMA[b])[c]	Fast Fluence $\times 10^{25}$ (n/m^2)[c]	Irradiation Temperature (°C)[d]
AGR-2 6-2-3	AGR248	LEU09-OP2-Z104	UCO	8.22	2.30	1095

[a] The X-Y-Z naming convention denotes the location in the irradiation test train: Capsule-Level-Stack.[1]
[b] Fissions per initial metal atom.
[c] Based on physics calculations.[4]
[d] Time-average, volume-average (TAVA) temperature based on thermal calculations.[5]

3. EXPERIMENTAL OBJECTIVES

- Measure the inventory of uranium and fission products outside of intact silicon carbide (SiC) layers but retained in the compact matrix or outer pyrolytic carbon. These measurements will be done by electrolytic deconsolidation of the compact and acid leaching of the particles and matrix debris using a leach-burn-leach (LBL) process, as described in Section 4.2.
- Examine individual particles deconsolidated from the as-irradiated compact with the Irradiated Microsphere Gamma Analyzer (IMGA) to quantify retention of specific gamma-emitting fission products (including ^{106}Ru, ^{110m}Ag, ^{134}Cs, ^{137}Cs, ^{144}Ce, and ^{154}Eu) and to identify anomalous particles, especially those with a below-average cesium inventory indicative of SiC failure. Concentrated regions of ^{134}Cs activity were not detected in the graphite holder adjacent to this compact, suggesting that it does not contain particles that released significant amounts of cesium through defective or failed SiC layers. Short-duration gamma scanning of every particle recovered from the compact will provide additional data to confirm the absence of particles with SiC failure in this compact.
- Perform microanalysis on selected particles 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, is of particular interest and such particles should have the highest priority for examination. Particles with below-average retention of other fission products (e.g., ^{110m}Ag or ^{154}Eu) and particles that are representative of average fission product retention will also be examined. Based on the specific results and discussions with the Fuels PIE Technical Lead, particles may be sent to INL for additional microanalysis.
- Archive remaining particles for possible later use.

4. SCOPE OF WORK

4.1 Receipt Inspection

The compact shipping drum will be unpacked and the individual aluminum compact storage container removed. The compact will be removed from the storage container and inspected for any damage prior to proceeding with subsequent analysis. The condition of the compact and any features of interest will be photographically documented.

4.2 Deconsolidation and Acid Leaching

The fuel compact will be electrolytically deconsolidated in nitric acid to break up the matrix material and free the TRISO fuel particles (AGR-2 UCO compacts have ~3176 particles[3]). Particles and matrix debris will then be subjected to an LBL process. Particles and matrix debris will be collected in a Soxhlet thimble, two 24-hour nitric acid leaches in a Soxhlet extractor performed, and the leachates analyzed for uranium and fission products. After the two pre-burn leaches, further digestion of the particles and matrix debris in boiling acid will be performed to help remove any matrix residue from the TRISO particles and break up the matrix debris further. The matrix debris will be separated from the particles by washing through a sieve. Recovered particles will be rinsed and dried and transferred to the hot cell cubicle housing the IMGA, where they will undergo gamma survey as described in Section 4.3.

The separated matrix debris will be dried by distilling off the acid and rinse water. 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 nitric acid leaches and the leachates analyzed for uranium and fission products.

After completion of the IMGA survey as described in Section 4.3, 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. Particles 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 uranium 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.

4.3 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 assess overall condition and identify features of interest such as cracked coatings or coating fragments. Abnormal particles or coating fragments may be selected for further examination.

All intact particles will be gamma counted using a short counting time (typically 50–100 seconds) to measure ^{137}Cs and ^{144}Ce and 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 may indicate a failed-TRISO particle.

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 (^{95}Zr, ^{106}Ru, ^{110m}Ag, ^{134}Cs, ^{137}Cs, ^{144}Ce, and ^{154}Eu) and determine distributions for ^{154}Eu and ^{110m}Ag. Long-count IMGA analysis will also be performed on a random riffled sample of about 50 particles.

4.4 Microstructural Analysis

After IMGA analysis, particles of interest will be selected for materialography and/or x-ray imaging, especially any particles with a below-average $^{137}Cs/^{144}Ce$ ratio or low overall radioisotopic inventory that may be indicative of failed SiC or failed TRISO. 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 Fuels PIE Technical Lead, individual particles may be sent to INL for additional microanalysis.

In addition to identifying particles that released cesium due to failed SiC, another goal of this PIE is to identify any mechanisms that may explain why some particles release silver and europium to a greater extent than others. Features of interest will include structure of the coatings and kernels, particularly changes induced by radiation. Also of interest are location and composition of fission products or actinide inclusions within the coating layers, primarily in the inner pyrolytic carbon and SiC layers.

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 a below-average $^{137}Cs/^{144}Ce$ ratio or low overall radioisotopic inventory will always 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.

4.5 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:

- Compact fractional fission product inventories outside of SiC, as determined by the deconsolidation and acid leaching and based on as-run inventory calculations[4]
- Results of particle inspection and IMGA gamma analysis of individual particles
- X-ray and materialographic images, including detailed analysis of 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

PIE activities performed at ORNL shall be performed in accordance with the AGR-2 PIE Plan, applicable ORNL procedures, and the ORNL Quality Assurance Plan for Nuclear Research and Development Activities[6] to meet the INL QA requirements specified in Inter-Entity Work Order (IEWO) #150293.

6. REFERENCES

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

2 SOW-11467, *Statement of Work for the AGR-2 PIE at Oak Ridge National Laboratory*, Rev. 1, October 2015.

3 INL/EXT-14-32277, *AGR-2 Irradiation Test Final As-Run Report*, Rev. 2, August 2014.

4 ECAR-2066, *JMOCUP As-Run Daily Depletion Calculation for the AGR-2 Experiment in the ATR B-12 Position*, Rev. 2, April 2014.

5 ECAR-2476, *AGR-2 Daily As-Run Thermal Analyses*, Rev. 1, August 2014.

6 QAP-ORNL-NR&D-01, *Quality Assurance Plan for Nuclear Research and Development Activities Conducted at the Oak Ridge National Laboratory*, Oak Ridge National Laboratory, Rev. 0, May 2013.