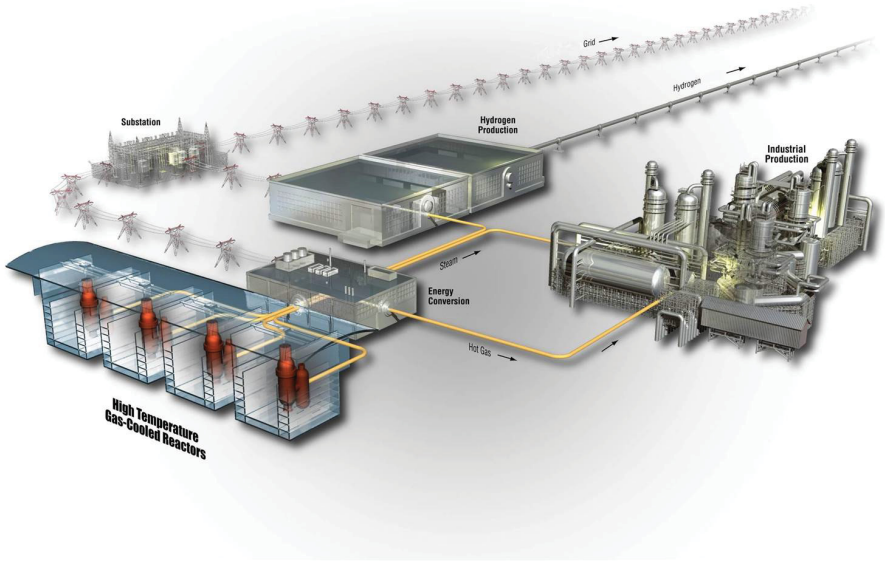
Document ID: PLN-4616
Revision ID: 0
Effective Date: 12/17/2013
INL/MIS-13-30534

Plan

Project No. 29412, 23843, 23841

AGR-2 Post-Irradiation Examination Plan



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AGR-2 POST-IRRADIATION EXAMINATION PLAN	Identifier: PLN-4616 Revision: 0 Effective Date: 12/17/2013 Page: i of vii

VHTR Program	Plan	eCR Number: 619487

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REVISION LOG

Rev.	Date	Affected Pages	Revision Description
0	12/17/2013	All	Newly issued document

SUMMARY

The AGR-2 irradiation experiment is the second in a series of test irradiations for the Very High Temperature Gas-Cooled Reactor Fuel Development and Qualification Program. The AGR-2 test includes two different types of fuel fabricated in the U.S.: uranium oxide (UO_2) and uranium oxide/uranium carbide (UCO). The tristructural isotropic coating conditions for both types of kernels are derived from the AGR-1 Variant 3 fuel. A fundamental difference compared to the lab-scale AGR-1 fuel is that that AGR-2 coatings were fabricated using an industrial scale coater and represent an important step in the establishment of an industrial scale fuel fabrication capability by the Next Generation Nuclear Plant program. One of the AGR-2 capsules (Capsule 2) contains fuel compacts that were irradiated at a significantly elevated temperature relative to the other capsules (time average peak temperature ≤1400°C) and constitutes a margin test of tristructural isotropic (TRISO) fuel irradiation performance.

Following the conclusion of irradiation in the Advanced Test Reactor, the AGR-2 test train will be removed from the reactor and shipped to the Hot Fuel Examination Facility at the Materials and Fuels Complex, located at Idaho National Laboratory, for nondestructive examination and disassembly. This will be followed by extensive post-irradiation examination (PIE) at Idaho National Laboratory and Oak Ridge National Laboratory. The PIE for this experiment will focus on:

- Evaluating the performance of the UCO fuel in the high temperature Capsule 2 and comparing to performance of identical fuel irradiated at lower temperature (AGR-2 Capsule 5)
- Comparing the performance of UCO fuel fabricated at the industrial scale (AGR-2 fuel) with UCO fuel fabricated at the laboratory scale (AGR-1 fuel)
- Comparing the performance of UCO and UO_2 fuel
- Exploring the causes of defective or failed particles.

The post-irradiation evaluation of fuel performance will focus on several key characteristics of the fuel, including (a) fission product retention of particles and compacts in-pile and during post-irradiation safety tests and (b) coating behavior and integrity (including coating fracture, coating degradation, and layer delamination), as well as any correlations between these two characteristics (i.e., how coating behavior correlates to fission product retention). The PIE activities are intended to provide information on these fuel characteristics and will include:

- Test train inspections and nondestructive analyses to determine the overall condition of the test train exterior and the condition and location of internal components
- Test train disassembly, extraction of fuel and other interior components (including the graphite fuel holders, melt wires, and flux wires), and evaluation of test train performance by characterization of melt wires and fluence wires
- Dimensional measurements of the fuel compacts and graphite holders
- Measurement of fuel compact burnup and selected fission product inventories
- Post-irradiated fission metals release analysis by measurement of fission metal inventories on metal capsule components
- Identification of any specific compacts that may contain defective or failed silicon carbide (SiC) layers by gamma scanning the graphite fuel holders to locate areas with elevated Cs levels
- Additional gamma scanning and burn-leach of graphite fuel holders and graphite spacers to quantify fission metals release

- Deconsolidation of compacts to provide particles for subsequent analyses and leach-burn-leach analysis to quantify SiC failure fractions and evaluate fission product inventories in the compact matrix
- Microanalytical characterization of fuel compacts and particles using optical metallography, electron microscopy, electron probe microanalysis, and x-ray tomography to investigate fuel microstructures, the condition of coatings, and fission product migration within the fuel
- Irradiated microsphere gamma analysis to measure fission product inventories and evaluate fission product retention for individual particles
- Safety testing to investigate release of selected fission products (including radioisotopes of Ag, Cs, Sr, and Eu) at elevated temperatures in pure He, followed by detailed post-test analysis of compacts as described above.

The PIE results will further advance the program's understanding of fuel behavior, confirm the performance of fuel particles fabricated at the industrial scale, and provide the first performance data for modern UO_2 TRISO fuel. This document presents the plan for PIE of the AGR-2 experiment and the general flow of PIE activities along with detailed descriptions of anticipated tasks.

The AGR-2 irradiation experiment also includes two capsules containing fuel manufactured internationally: Capsule 1 contains French UO_2 fuel and Capsule 4 contains South African UO_2 fuel. This plan proposes to perform examination of capsule components for these two capsules and perform non-destructive examination of the fuel compacts and graphite fuel holders from these capsules. Any destructive PIE to be performed on these fuel compacts will be governed by a separate PIE plan.

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ACRONYMS

AGR	Advanced Gas Reactor
ATR	Advanced Test Reactor
CCCTF	Core Conduction Cooldown Test Facility
DLBL	deconsolidation-leach-burn-leach
EDS	energy dispersive x-ray spectroscopy
FACS	Fuel Accident Condition Simulator
FIMA	fissions per initial heavy metal atom
HFEF	Hot Fuel Examination Facility
ICP-MS	inductively coupled plasma mass spectrometry
IFEL	Irradiated Fuels Examination Laboratory
IMGA	Irradiated Microsphere Gamma Analyzer
INL	Idaho National Laboratory
IPyC	inner pyrolytic carbon
MFC	Materials and Fuels Complex
NGNP	Next Generation Nuclear Plant
OPyC	outer pyrolytic carbon
ORNL	Oak Ridge National Laboratory
PGS	precision gamma scanner
PIE	post-irradiation examination
PNNL	Pacific Northwest National Laboratory
SEM	scanning electron microscope
STEM	scanning transmission electron microscope
TC	thermocouple
TDO	Technology Development Office
TEM	transmission electron microscope
TEV	technical evaluation
TRISO	tristructural isotropic
UCO	uranium oxide/uranium carbide
UO_2	uranium dioxide
VHTR	very high temperature gas-cooled reactor
WDS	wavelength dispersive x-ray spectroscopy

1. INTRODUCTION

1.1 Background

The Advanced Gas Reactor (AGR) Fuel Development and Qualification Program was established to perform the requisite research and development on tristructural isotropic (TRISO) coated particle high-temperature gas reactor fuel to support deployment of a very high temperature gas-cooled reactor (VHTR), which has been selected as the reactor concept for the Next Generation Nuclear Plant (NGNP) project. The work continues as part of the VHTR program. The overarching goal of the program is to provide a baseline fuel qualification data set to support licensing and operation of a VHTR. To achieve these goals, the program includes the elements of fuel fabrication, irradiation, post-irradiation examination (PIE) and safety testing, fuel performance modeling, and fission product transport (PLN-3636 Rev. 2, 2012).

A series of fuel irradiation experiments are in progress at the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL). These experiments are intended to provide data on fuel performance under irradiation, support fuel process development, qualify the fuel for operating and accident conditions, provide irradiated fuel for accident testing, and support the development of fuel performance and fission product transport models.

The first of these irradiation experiments, AGR-1, began in the ATR in December of 2006 and was completed in November of 2009, with the PIE of the AGR-1 fuel commencing in March 2010. This experiment was intended to act as a shakedown test of the multi-capsule design and to provide early data on fuel performance that will be used in fuel fabrication process refinement. This test also provided samples for post-irradiation safety testing, where fission product retention of the fuel at high temperatures is experimentally measured. The AGR-1 fuel performance was extremely good, with zero in-pile particle failures (Collin 2012) and generally very low release of key fisison products (including Cs) during the irradiation and during post-irradiation safety testing. The second irradiation experiment, AGR-2, began in June 2010 and was designed to build upon the AGR-1 experience. The AGR-2 fuel, test train, and experiment description are summarized in the AGR-2 Test Plan (Collin 2011). AGR-2 fuel performance during irradiation has been excellent, and PIE is expected to begin with capsule inspection and disassembly in February 2014.

The AGR-2 experiment includes two capsules containing French and South African fuel compacts. Details of these fuel specimens and objectives of the irradiation and PIE are not provided in this plan. However, it is anticipated that some of the basic PIE outlined for the capsules and compacts will be performed on all AGR-2 capsules.

1.2 AGR-2 Irradiation Experiment

The AGR-2 irradiation experiment was designed to provide fuel performance data for coated particles industrially fabricated on an engineering scale pilot line using a 150 mm (6 in.) diameter coater. The experiment includes fuel compacts made from particles with kernels of either uranium oxide/uranium carbide (known as UCO) or uranium oxide (UO_2). The VHTR Technical Program Plan (PLN-3636 Rev. 2, 2012) describes the AGR-2 experiment:

This test train provides irradiated fuel performance data and irradiated fuel samples for safety testing and PIE for key fuel product and process variants to broaden options and increase the prospects for meeting fuel performance requirements and to support the development of a fundamental understanding of the relationship between the fuel fabrication process, as-fabricated fuel properties, normal operation, and accident condition performance.

1.2.1 AGR-2 Fuel

Two different types of fuel kernels were used in the AGR-2 experiment: (1) low enriched UCO with mean diameter 426.7 µm and U enrichment of 14.03%; and (2) low enriched UO_2 with mean diameter 507.7 µm and U enrichment of 9.60%. Both sets of kernels were fabricated at Babcock and Wilcox Nuclear Operations Group. TRISO coatings were applied to the kernels by Babcock and Wilcox Nuclear Operations Group, and select properties of the UCO and UO_2 coated particles are given in Table (data taken from Collin 2011). The coating process conditions were derived from the production of the AGR-1 Variant 3 fuel. A list of references with detailed characterization data for the AGR-2 coated particles can be found in Collin 2011.

Table 1. Selected properties of the UCO and UO2 fuel particles (data taken from Collin 2011).

Property	Actual mean value ± population standard deviation	
Kernel type	UCO	UO_2
Kernel diameter (µm)	426.7 ± 8.8	507.7 ± 11.9
U enrichment (wt%)	14.029 ± 0.026	9.600 ± 0.010
Buffer thickness (µm)	98.9 ± 8.4	97.7 ± 9.9
IPyC thickness (µm)	40.4 ± 2.5	41.9 ± 3.2
SiC thickness (µm)	35.2 ± 1.2	37.5 ± 1.2
OPyC thickness (µm)	43.4 ± 2.9	45.6 ± 2.4
IPyC anisotropy (BAF)	1.0349 ± 0.0012	1.0334 ± 0.0027
OPyC anisotropy (BAF)	1.0263 ± 0.0011	1.0219 ± 0.0012
IPyC anisotropy post-compact anneal (BAF)	1.0465 ± 0.0049	1.0471 ± 0.0036
OPyC anisotropy post-compact anneal (BAF)	1.0429 ± 0.0019	1.0365 ± 0.0016
Particle diameter (µm)	873.2 ± 23	953.0 ± 28
Particle mass (mg)	1.032 ± 0.003	1.462 ± 0.005

The coated particles were formed into right cylindrical compacts at Oak Ridge National Laboratory (ORNL). The compact matrix material is composed of a mixture of graphite and a thermosetting epoxy resin. The same compacting process was used for the UCO and UO2 particles, and was similar to the AGR-1 process. Table presents selected properties of the AGR-2 compacts for both fuel types (data taken from Collin 2011. Figure 1 shows x-radiographs of the two types of compacts. Note that the AGR-2 compacts had much smaller fuel-free zones at the axial ends of the compacts compared to the AGR-1 compacts. A list of references with detailed characterization data for the AGR-2 compacts can be found in Collin 2011. Additional compact characterization is also available in the AGR-2 Fuel Compact Pre-Irradiation Compact Characterization Summary Report (Hunn et al. 2010).

Table 2. Selected properties of AGR-2 compacts (data taken from Collin 2011).

Property	Actual Mean Value ± Population Standard Deviation	
	UCO	UO_2
Compact mass (g)	6.294 ± 0.011	6.103 ± 0.015
Mean U loading (g U/compact)	1.257 ± 0.03	0.993 ± 0.006
Diameter (mm)	12.286 ± 0.005	12.269 ± 0.007
Length (mm)	25.141 ± 0.017	25.135 ± 0.018
Number of particles per compact [a]	3176	1543
Particle volume packing fraction (%)	37	23
Effective overall compact density [a] (Mg/m^3)	2.11	2.05
Compact matrix density (Mg/m^3)	1.589 ± 0.005	1.680 ± 0.008
U contamination fraction [b] (g exposed U / g U in compact)	$\leq 2.5 \times 10^{-5}$ [c]	$\leq 3.2 \times 10^{-5}$ [c]
U contamination fraction w/o exposed kernels (g leached U / g U in compact)	1.59×10^{-6}	1.57×10^{-6}
Defective SiC coating fraction [b]	$\leq 1.2 \times 10^{-5}$	$\leq 2.5 \times 10^{-5}$
Defective IPyC coating fraction [b]	$\leq 4.8 \times 10^{-5}$	$\leq 7.7 \times 10^{-5}$
Defective OPyC coating fraction [b]	$\leq 9.5 \times 10^{-4}$	$\leq 2.0 \times 10^{-3}$

a. Approximate calculated value derived from other characterized properties.
b. 95% confidence defect fraction.
c. Values exceed specifications: the non-conformances are documented in NCR-44791 with a disposition of "use as is."

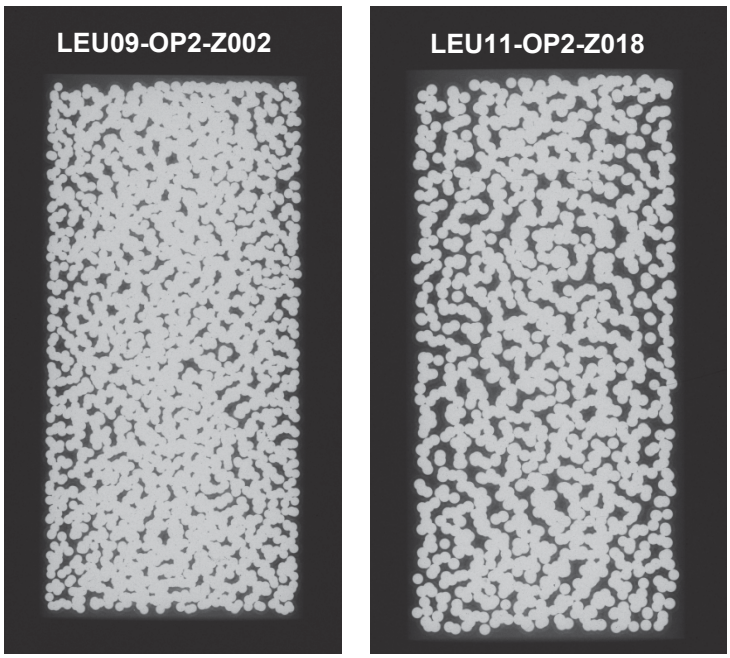

Figure 1. X-radiographs of U.S. UCO (compact LEU09-OP2-Z002, left) and UO2 (compact LEU11-OP2-Z018, right) compacts taken from the same compact lots used in the AGR-2 irradiation. Note the visibly lower packing fraction of the UO2 compact. Images taken from Hunn et al. 2010.

1.2.2 AGR-2 Irradiation Test Train

The AGR-2 test train is a multi-capsule, instrumented lead experiment, irradiated in the 38.1 mm (1.5 inch) diameter B-12 position in the ATR. The basic design of the test train is very similar to that of the AGR-1 experiment. The test train consists of six capsules, each independently temperature controlled and independently monitored for fission product release. Each capsule is approximately 150 mm (6 inches) in length and 36 mm (1.4 inches) in diameter.

Each AGR-2 capsule contains a specific fuel type. Table 3 lists the fuel in each AGR-2 capsule. The capsules are listed in Table 3 in the same physical arrangement that they were placed in the reactor (i.e. Capsule 6 is at the top of the experiment). Four of the capsules contain U.S. UCO or UO_2 fuel (described in Section 1.2.1). Capsule 4 contains UO_2 fuel compacts fabricated at ORNL using coated particles from South Africa while Capsule 1 contains 50 mm long compacts containing UO_2 coated particles and fabricated by Commissariat à l'énergie atomique (CEA) in France. These compacts are of similar diameter as the U.S. compacts. Note that this PIE Plan will not address the destructive examination of the Capsule 1 and 4 fuel.

Table 3. Type of fuel in each AGR-2 capsule.

Capsule	Fuel type	Capsule	Fuel type
6	U.S. UCO	3	U.S. UO_2
5	U.S. UCO	2	U.S. UCO
4	South African UO_2	1	French UO_2

Capsules 2 – 6 each contain a total of 12 compacts in three stacks while Capsule 1 contains 6 compacts. A cross-sectional view of an AGR-2 capsule is shown in Figure 2. The capsules consist of a graphite fuel holder with holes machined for insertion of fuel compacts, thermocouples (TCs), encapsulated melt and flux wires, and niobium through-tubes to allow gas lines and TC leads to pass through to the other capsules in the test train. The graphite fuel holders contain boron carbide (B4C) as a burnable poison to offset U-235 depletion and provide a more uniform particle power level throughout the experiment. Initial weight percentages of B4C were 4.29% in Holder 1, 5.75% in Holders 2 and 5, 4.92% in Holders 3 and 4, and 4.83% in Holder 6. The orientation of the compact stacks in the irradiation capsule places Stacks 1 and 2 closer to the reactor core than Stack 3 (see Figure 2), which would result in much higher neutron fluxes in Stacks 1 and 2. To counteract this effect, a combination of hafnium and stainless steel shrouds surround the graphite holder to provide a more uniform neutron flux during the experiment. The entire assembly is encapsulated in a stainless steel outer shell.

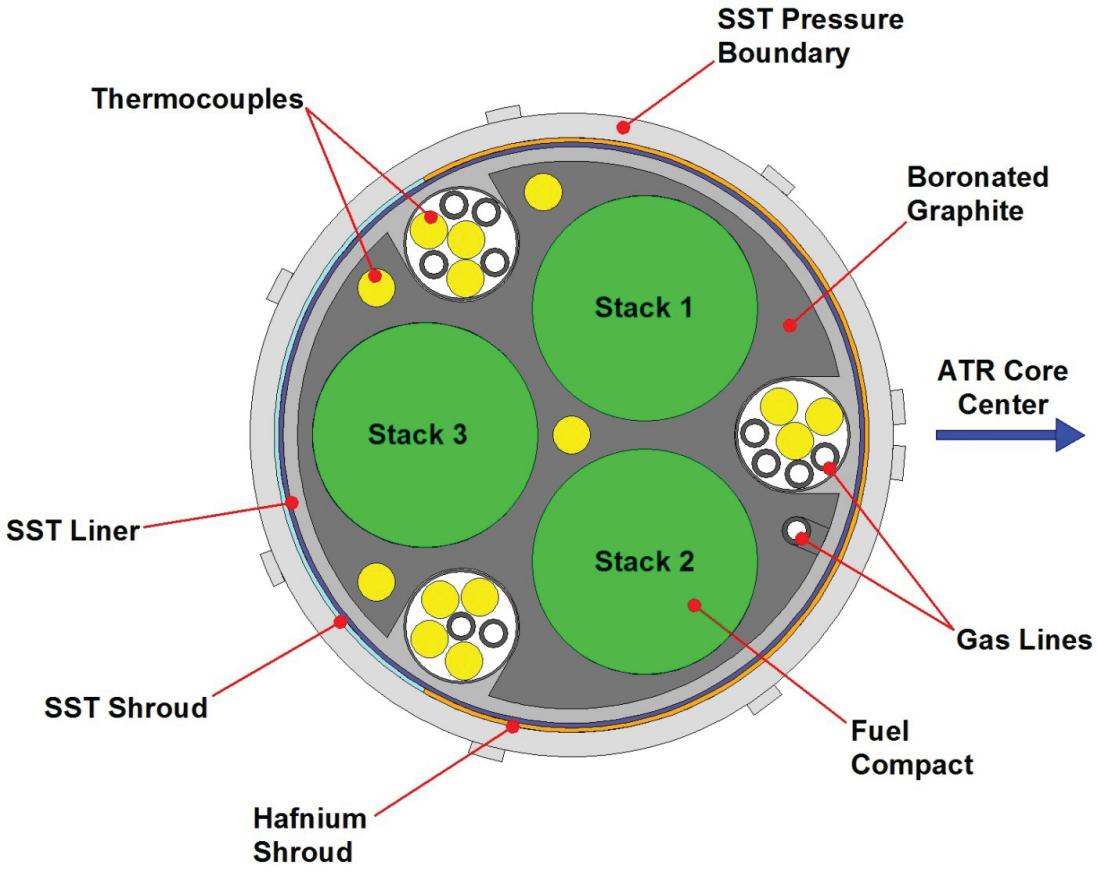

Figure 2. Cross-section diagram of an AGR-2 capsule (viewed from top).

A numbering system has been developed to uniquely identify each compact in the test train. This is based on the specific capsule, level, and stack number. Figure 2 identifies the stack and position (or level) numbers in a particular capsule. For example, Compact 6-4-1 refers to the compact in Capsule 6 at the top (Level 4) of Stack 1.

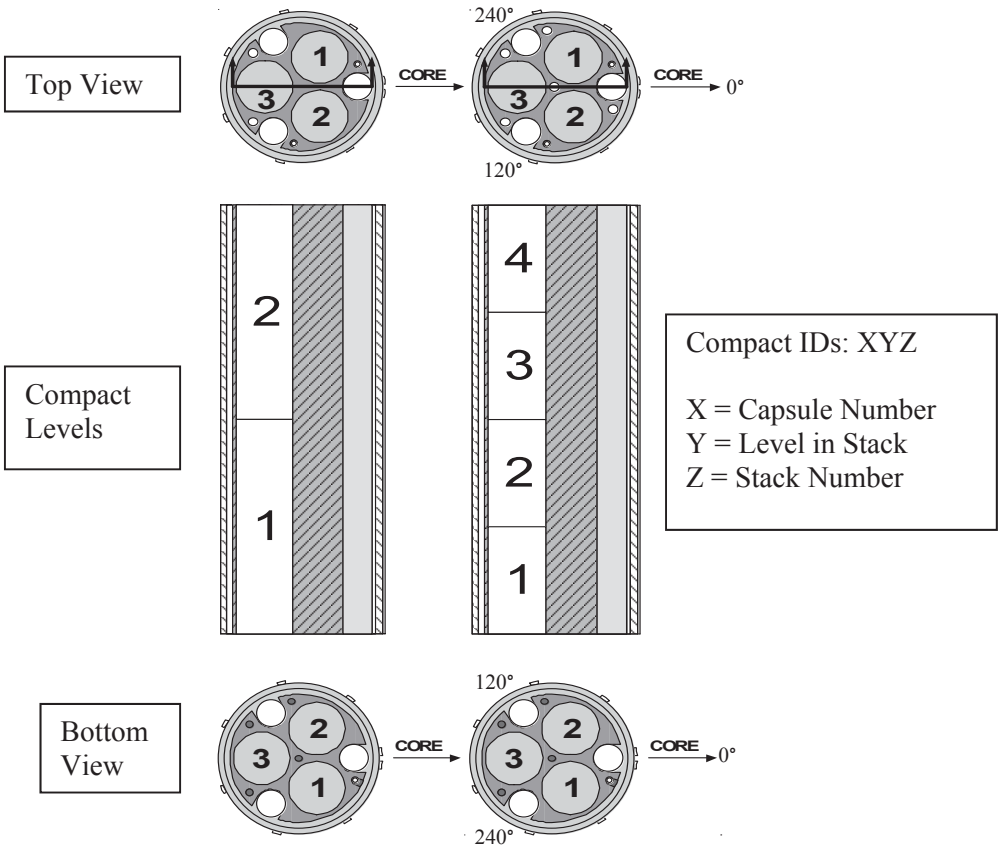

Figure 3. Numbering scheme for AGR-2 compacts in Capsule 1 (left) and Capsules 2-6 (right).

Each capsule is supplied with an inert sweep gas mixture of helium and neon. Because of the very different thermal conductivities of the gases, varying the gas mixture can act to manage the temperature in the capsule. The sweep gas from each capsule is routed to a detector that measures the quantity of fission gas present in the effluent. This provides a means of monitoring the integrity of the fuel throughout the irradiation. The AGR-2 test train is described in further detail in the AGR-2 Test Plan (Collin 2011) and a detailed description of test train assembly is given in Work Order 131971-01, AGR-2 Test Train Final Assembly.

The TCs used in the test train are Type N. The TCs are inserted into holes drilled in the graphite fuel holder at various locations. Capsules 1–5 have two TCs each, while the top capsule (Capsule 6) has five TCs. Each capsule contains a melt wire package containing two pure beryllium wires (Capsules 1, 3, 4, 5, and 6) or a single nickel wire (Capsule 2), which are encapsulated in vanadium and placed in a hole drilled at the centerline of the graphite holder. These will be used to indicate if the temperature of the capsule (at the location of the melt wires) exceeded 1287°C (beryllium wires) or 1455°C (nickel wire). Each capsule also contains three different flux wires (pure Fe, V-0.1%Co, and pure Nb), all of which are encapsulated in sealed vanadium tubes and placed around the periphery of the graphite holder. The measured activity in the wires after irradiation will be used to calculate the neutron fluence for the different neutron energy ranges covered by the three flux wires.

1.2.3 AGR-2 Irradiation

The AGR-2 irradiation started in July 2010 and was conducted in the B-12 position of the ATR. The irradiation test condition requirements for the AGR-2 experiment, which are provided in the AGR-2 Test Specification (Maki 2010), are listed in Table . A summary of predicted irradiation conditions for the AGR-2 capsules is provided in Collin 2011. A detailed report of the as-run irradiation conditions will be prepared once the AGR-2 irradiation is completed. The irradiation conditions of the fuel compacts, including compact-specific burnup, fast neutron fluence, and temperature, will be considered when identifying compacts for specific PIE activities.

Table 4. AGR-2 fuel irradiation test condition requirements (from Maki 2010).

Parameter	Specification
Instantaneous peak temperature for each capsule (°C)	≤1800
Time average, peak temperature for each capsule (°C)	≤1400 for one capsule containing UCO fuel ≤1250 for the remaining capsules containing UCO fuel ≤1150 for each capsule containing UO_2 fuel
Time average, volume average temperature goal for each capsule (°C)	≥1150 for the highest temperature UCO capsule ≥1000 for the remaining capsules containing UCO fuel ≥900 for each capsule containing UO_2 fuel
Minimum compact average burnup (% fissions per initial heavy metal atom)	>7 for UCO, U.S. UO_2 and South African UO_2 fuel >11 for French UO_2 fuel
Compact average burnup goal for majority of fuel compacts (% fissions per initial heavy metal atom)	>10 for UCO, U.S. UO_2 and South African UO_2 fuel >13 for French UO_2 fuel
Peak fast neutron fluence (n/m^2, E > 0.18 MeV)	$<5 \times 10^{25}$
Minimum peak fast neutron fluence (n/m^2, E > 0.18 MeV)	$>1.5 \times 10^{25}$
Instantaneous peak power per particle (mW/particle)	≤400

Note that the goal for one of the capsules is a time average peak temperature as high as 1400°C as a margin test for fuel performance in-pile. This high temperature capsule is Capsule 2, and analysis of the fuel performance in Capsule 2 will be a key objective during the PIE.

1.3 AGR-2 Post-Irradiation Examination Objectives

The success of the AGR-1 experiment confirmed numerous programmatic expectations regarding fuel performance prior to this experiment, including the overall high quality of the fuel, that UCO fuel effectively controls CO pressure buildup and the amoeba effect, and that the AGR-1 UCO fuel has satisfactory fission product retention under normal operation and reactor accident conditions. The primary objectives for the AGR-2 PIE are listed below.

- Evaluate the performance of the UCO fuel in the high temperature Capsule 2 and compare to performance of identical fuel irradiated at lower temperature (AGR-2 Capsule 5).

- Compare the performance of UCO fuel fabricated at the industrial scale (AGR-2 fuel) with UCO fuel fabricated at the laboratory scale (AGR-1).
- Compare the performance of UCO and UO_2 fuel.
- Explore the causes of defective or failed particles and fission product release.

All of these objectives apply to fuel performance in-reactor and during post-irradiation safety testing. The post-irradiation evaluation of fuel performance will focus on several key characteristics of the fuel, including (a) fission product retention of particles and compacts in-pile and during post-irradiation safety tests and (b) coating behavior and integrity (including coating fracture, coating degradation, and layer delamination), as well as any correlations between these two characteristics (i.e., how coating behavior correlates to fission product retention).

The basic PIE equipment and methods used to assess fuel performance and accomplish these objectives will be very similar to those used for the AGR-1 PIE. However, many of the lessons learned during the AGR-1 PIE will be applied to the AGR-2 PIE in order to focus on activities that are likely to yield the most useful data.

This plan will describe the specific activities of the AGR-2 PIE and safety testing. While a preliminary strategy concerning the number of fuel compacts to be analyzed is provided, the actual number of compacts to be analyzed and the specific compact identification will depend heavily on both the final irradiation conditions experienced by the compacts and on the results of early PIE activities that will provide valuable information regarding the in-pile performance of the fuel. This will be a collaborative effort between INL and ORNL. Initial capsule disassembly, nondestructive examinations, and capsule component analysis will be performed at INL. The destructive analysis of fuel compacts will be performed at both INL and ORNL. Specific compacts that will be shipped to ORNL will be determined based on the final irradiation conditions of the compact and the results of early PIE activities.

2. AGR-2 POST-IRRADIATION EXAMINATION ACTIVITIES

This PIE Plan will focus primarily on the U.S. fuel in Capsules 2, 3, 5, and 6. A general prioritization of each U.S. AGR-2 capsule is provided in Table 5. Note that this is a preliminary scheme only, and is not intended to strictly dictate the order in which fuel compacts or capsule components are analyzed throughout the AGR-2 PIE. It is expected that as data become available from the different capsules, the programmatic focus may shift.

A tentative approach for capsule inspection and disassembly, analysis of capsule components, and non-destructive analysis of the fuel compacts is shown in Figure. It is proposed that these activities be performed on all six AGR-2 capsules. At the completion of this scope, destructive examination of the U.S. fuel from capsules 2, 3, 5, and 6 will proceed as described in this PIE Plan. If any destructive examination on the fuel compacts from Capsules 1 or 4 will be performed, it will be governed by a separate PIE plan and will not be discussed further in this document.

2.1 Test Train Receipt and Inspection

2.1.1 Cask Transfer from ATR to HFEF

After removal from ATR and a cool down time in the reactor water canal of approximately 3 months, the test train will be sectioned just above the top of Capsule 6 and the fueled portion containing all six capsules will be loaded into a GE-2000 shipping cask according to the associated ATR procedures. The cask will then be transported by truck to the MFC Hot Fuel Examination Facility (HFEF).

HFEF routinely receives the GE-2000 shipping cask, so standard procedures will be used to mate the cask to the hot cell and open the cask. The test train will be removed from the cask and transferred to a shielded window location within the HFEF where the test train will be externally inspected.

Table 5. Preliminary AGR-2 capsule prioritization scheme.

Capsule	**Remarks**
Capsule 2	This is the high temperature capsule, and is expected to have the highest burnup and a fast fluence near the maximum for the AGR-2 experiment. It is therefore of key interest to confirm fuel performance under the most extreme conditions.
Capsule 5	Expected to have a lower temperature (more typical of expected VHTR operating conditions) and slightly lower burnup and fast fluence relative to Capsule 2. It will be an important capsule for a more direct comparison to the AGR-1 fuel performance.
Capsule 3	This is the only U.S. UO_2 fuel in the irradiation, and comparison of UO_2 and UCO fuel performance is an important objective.
Capsule 6	Expected to have similar temperature as Capsule 5 but significantly lower burnup and fast fluence. This capsule can be used to explore the independent effects of burnup and fluence, but because it will be irradiated under the least demanding conditions it will be considered secondary to the UCO fuel in Capsules 2 and 5.

2.1.2 Visual Inspection of Test Train

After unloading from the shipping cask, the exterior of the intact test train will be visually inspected to identify any significant damage or degradation. The entire test train will be inspected and photographed in segments at a macroscopic scale (approximately 6-inch field of view) with a high-resolution digital camera. Fine features of interest, such as the weld seams, can be visually inspected and photographed with a smaller field of view, if necessary. A procedure will be employed to ensure that all important features are examined, and all significant observations will be entered in an inspection log as permanent records to accompany digital photographs.

2.1.3 Gamma Scanning of Test Train

The intact test train will be examined by isotopic gamma scanning using the HFEF Precision Gamma Scanner (PGS) to identify interior components, including the fuel compacts, and determine if any damage or shifting of components within the capsules has occurred. Regions of interest on the vertically oriented test train will be raised in front of the scanner collimator slit in vertical increments equal to the adjustable slit height. Scans for fission products adjacent to the nominal fueled regions will indicate whether deterioration of graphite spacers allowed fuel compacts to shift axially.

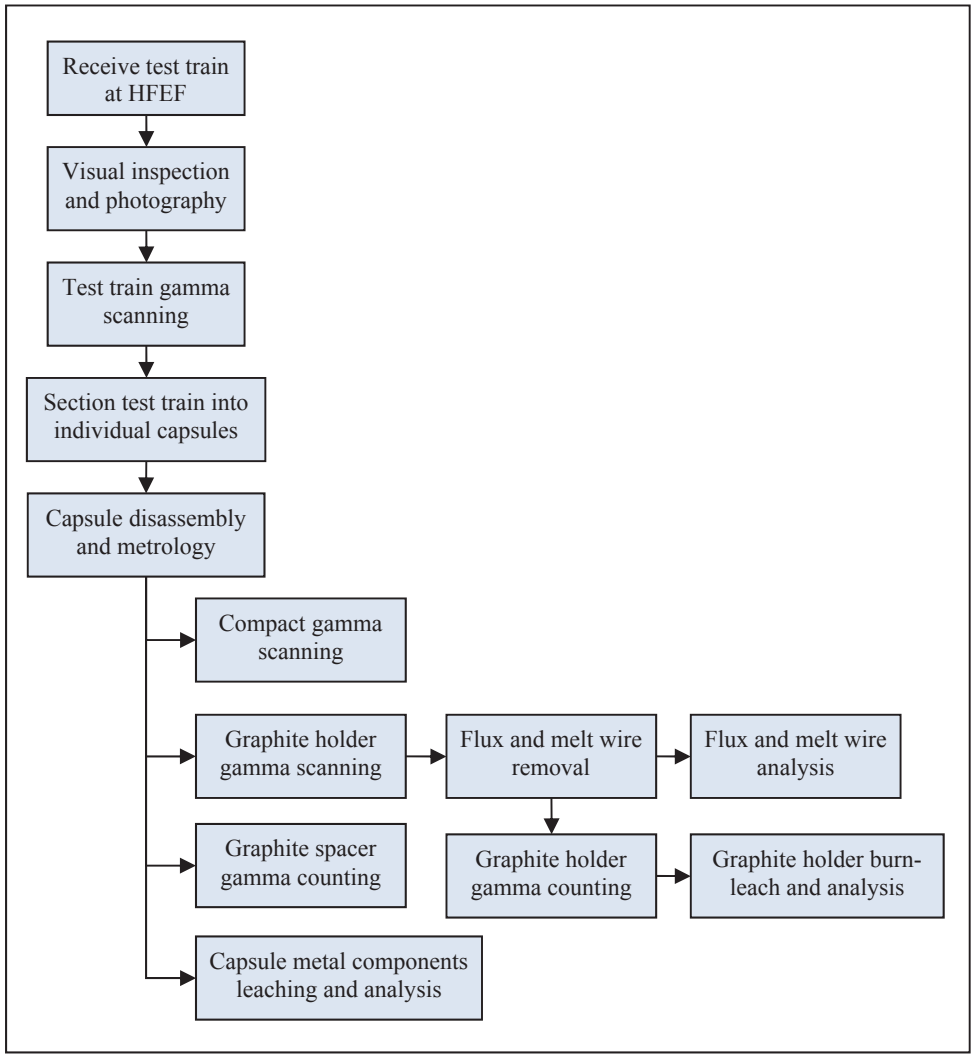

Figure 4. Flow diagram for AGR-2 test train inspection and disassembly, destructive examination of the capsule components, and non-destructive examination of the fuel compacts.

2.2 Disassembly, Inspection, and Dimensional Measurement

2.2.1 Test Train Disassembly

The test train will be disassembled by separating each capsule from the test train and then opening each capsule separately. Because the construction of the AGR-2 capsules is very similar to that of the AGR-1 capsules, the basic procedure for test train and capsule disassembly will be similar to that described in the AGR-1 Irradiated Test Train Preliminary Inspection and Disassembly First Look report (Demkowicz et al. 2011). The capsules will be separated by making circumferential cuts at the weld joint locations where the capsules were joined. The separated capsules will then be intact much as they were prior to the final assembly process to join the capsules when originally building the test assembly. The external configuration of an AGR-2 capsule is shown in Figure along with associated nomenclature. The equipment for test train and capsule disassembly will be the same as used previously for the AGR-1 experiment (Demkowicz et al. 2011), as discussed in TEV-1650 (Ploger 2012). The circumferential cuts will be made using a commercial-grade tubing cutter that has been modified for remote handling. The

tubing cutter uses a lathe-type bit designed to leave any lip or burr on the capsule head portion of the cut, rather than on the capsule body.

The test train will be disassembled in the opposite order that it was assembled, meaning that the capsules will be cut from the test train from top to bottom in descending numerical order. This is necessary because the gas lines and TCs of each of the lower capsules are routed in the through-tubes of capsules above it in the test train. Thus, the top capsule must be cut first to allow pulling each capsule free of the gas lines and TCs coming from the capsules below it.

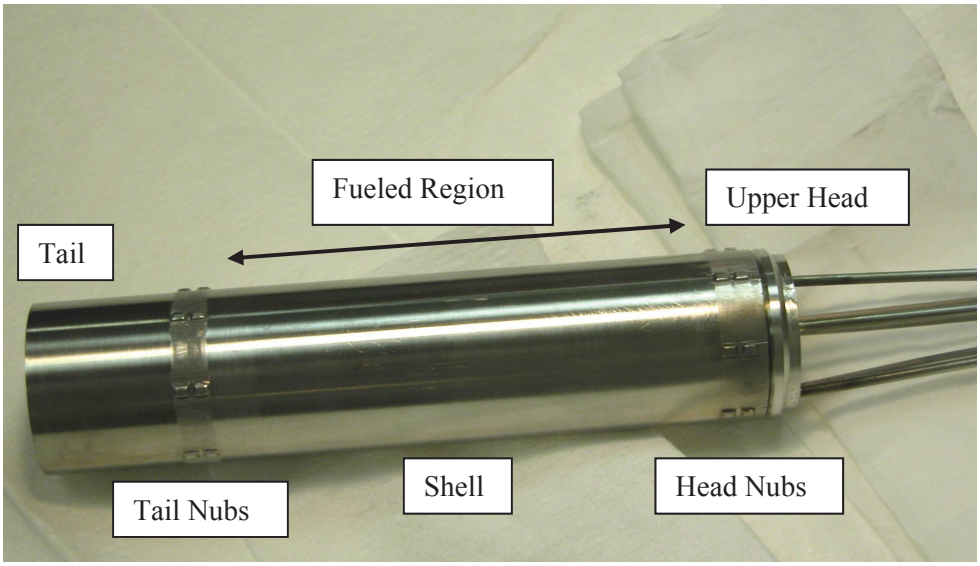

Figure 5. External configuration of an AGR-2 capsule.

Each capsule will be examined as it is separated from the test train. Along with outer capsule regions, exposed metallic capsule components (top and bottom caps, gas lines, and braze joints) will be photo-visually inspected to identify any degradation such as evidence of chemical reactions between components, cracking, or failure of the braze joints.

2.2.2 Capsule Disassembly

Following outer capsule inspection and prior to cutting the capsule head, the following cuts will be made:

- TC leads above the capsule head (these will vary in length depending on the capsule; lower capsules will have longer leads) will be cut and discarded
- The gas inlet and exhaust lines from each capsule will be cut and discarded.

The capsules will be cut open using the tubing cutter as described previously for the AGR-1 capsules (Demkowicz et al. 2011). After removal of the outer stainless steel capsule shell, the graphite holder will be removed from the upper head assembly and the fuel compacts will be removed from the graphite holder.

The irradiated graphite fuel holder and compacts may be fragile and therefore easily broken during handling operations. The disassembly tools have been designed to minimize the potential of damaging these fragile components. To the extent practicable, the components will be handled in a horizontally supported position, and sliding motions (rather than grasping and lifting) will be used. A force gauge will be used when pushing the compacts out of the graphite holder. A tentative limit of 44 N (10 lb_f) for a

single compact stack has been estalished in order to avoid damage to the compacts. If the compacts do not come out with modest force, alternative tools could be used to score and "crack" the graphite holder to free the compacts. Efforts will be made to minimize contamination of the capsule components from radionuclides present in the HFEF hot cell.

The following components will be removed from each capsule and saved for subsequent analysis:

1. Capsule body shell (1)
2. Capsule head (1)
3. Through tubes (3)
4. Capsule base (1)
5. Graphite fuel holder (1)
6. Fuel compacts (12 from Capsules 2–6; 6 from Capsule 1)
7. Graphite and Grafoil® spacers, top and bottom (4 total).
8. Melt wire package (1)
9. Flux wire packages (3)

Note that the flux and melt wire packages embedded in the graphite holders may not fall out of their locations during disassembly. In this case, they will be left in place until after graphite holder gamma scanning is complete, and then recovered from the holders. The TC ends that are embedded in the graphite holders will be removed and discarded. Similarly, the gas inlet line embedded in the graphite holder will be removed and discarded. If any of these items cannot be readily removed from the graphite holders during capsule disassembly, they will be left in place until after holder gamma scanning to avoid damaging the graphite in removal attempts.

Capsule disassembly operations will be documented by digital photography. Entire components will be inspected and photographed with a high-resolution digital camera through the hot cell window at a macroscopic scale, while fine features of interest such as cracks and corroded areas will be photo-visually inspected at a close scale. Components will be rotated as necessary to document all exterior surfaces. During component inspections, particular attention will be paid to crack formation, spallation, delamination, carbide formation, abrasion, and any other anomalous behavior. The interior surface of each capsule's stainless steel sleeve will be examined for any discoloration and deposit accumulation. Graphite holders will be inspected upon extraction from each capsule and after removal of the upper head assembly and fuel compacts to document any incremental damage during these separation steps.

All removed capsule components will be cataloged and placed in labeled containers to preserve the identity of the component and the location within the test train from which the component was removed. The capsule number, level, and stack numbers will be recorded for each fuel compact so that it can be cross-referenced to the originally assigned ORNL identification number.

Each fuel compact will be placed in a labeled, preweighed container. To the extent practicable, any loose fragments and fines associated with the compact will also be loaded into the container. The loaded container then will be weighed to the nearest milligram to determine the weight of the contents. Each graphite holder and any associated fragments will be placed in a labeled, preweighed container after separation of the upper head assembly and after unloading all compacts. The loaded holder container will then be weighed to the nearest milligram.

2.2.3 Dimensional Metrology of Internal Components

The objectives for the AGR-2 dimensional measurements are provided in TEV-1650 (Ploger 2012). Dimensional measurements will be made during PIE on the diameter of fuel compacts and graphite holders, the diameter of the three holes in each holder after removal of compacts, and the inner diameter of each stainless steel capsule sleeve. At a minimum, diameter measurements will be taken at top, middle, and bottom elevations of each component. Lengths will be measured on each AGR-2 fuel compact (excepting the 50 mm long French compacts from Capsule 1 that are problematic for the AGR-1 metrology apparatus). Diameter changes are important for assessing radial heat transfer between fuel compacts and graphite holders and between graphite holders and stainless steel capsules. Irradiation-induced dimensional changes on carbonaceous fuel compacts and graphite holders may also be used to validate assumptions on these materials used in computer models.

The same non-contact equipment that was used for the AGR-1 measurements of exterior dimensions (Demkowicz et al. 2011) will be reused for the AGR-2 fuel compacts and graphite holders as described in TEV-1650 (Ploger 2012). This system features a shielded digital camera (6.6 megapixels) and a telecentric lens for producing high-resolution images with virtually no distortion, plus measurement software proven for image analysis on cylindrical objects. Images produced by this system also will be used as inspection photographs where appropriate. Inner diameters of graphite holder holes and stainless steel capsule shells will be measured by commercial bore gauges. Both gauges use 3-point probes that can be retracted by master-slave manipulators using custom fixtures which also maintain probe shaft alignment with hole centerlines. Diameter values from conventional dial indicators will be read through the hot cell window. Fiducial marks on the extension shafts will indicate the depth inside the components at which diameters are measured.

2.3 Gamma Scanning of Graphite Fuel Holders

All of the AGR-2 graphite holders that remain intact during capsule disassembly will be gamma scanned using the HFEF PGS system in order to determine the inventory and distribution of fission products in the holders. This is a critical activity, as it can potentially provide information on specific compacts within each capsule that released abnormally elevated amounts of Cs during the irradiation, indicative of a defective or failed SiC layer. The basic methodology is based on the AGR-1 experience (Harp and Ploger 2011), but with a more refined approach for mapping the location of fission products.

Each holder will be scanned in two off axis sweeps that will provide an estimate of the total activity of fission products in the holders and help to identify holder levels of interest that contain an elevated activity of a specific fission product. Counting times in these scans will need to be adequate to establish a minimum detectable activity of Cs-137 and Cs-134 that is below the expected release of an individual defective TRISO particle. The fission product mapping method used for AGR-1 (Harp and Ploger 2011) left some ambiguity as to which compact was potentially leaking Cs or Ag. In the refined approach additional scanning angles will be utilized to create a tomographic map of the scanned levels. A new PGS fixture for the graphite holders has been developed to assist in the tomographic scanning of the holders. This fixture contains an integrated gamma ray source that will help to provide a spatial reference point and help establish a consistent coordinate system for the tomographic scanning. Any graphite holder axial regions that have elevated Cs-134 and Cs-137 activity will be a high priority for tomographic scanning, as this is generally correlated with particles with failed SiC and consequently relatively low Cs retention.

2.4 Gamma Scanning of the Fuel Compacts

All of the compacts from each AGR-2 capsule will be gamma scanned using the HFEF PGS system to quantify the inventory of gamma emitting fission products. The compacts will be scanned axially in 2.5

mm (0.1 inch) increments, and the measured inventory in each increment will be summed to determine the total inventory for an entire compact. The fission product inventories will be compared with predicted inventories from the as-run physics simulations of the AGR-2 irradiation to determine if any significant release occurred during the irradiation (particularly relevant for Ag-110m). The inventory of Cs-134 and Cs-137 will be used to determine the burnup of the compacts and these values will be compared to as-run physics simulations. The procedure for determining burnup from the spectrometric data is described for the AGR-1 fuel compacts in ECAR-1682 (Harp 2011). Experience with the AGR-1 compacts (Harp 2011) demonstrated that while quantification of most fission products can be accomplished with a relatively short live time (10 minutes per increment), quantification of Ag-110m inventory in the compacts requires much longer (several hours per increment) due to the lower inventory and gamma ray yield for energies other than 657.5 keV. Therefore counting times will need to be sufficient to quantify Ag-110m in the compacts.

2.5 Flux and Melt Wire Analysis

To verify calculations of neutron fluence and capsule temperatures achieved during the AGR-2 irradiation, each graphite fuel holder in the six test assemblies in the AGR-1 test train is instrumented with flux and melt wire packages. Each fuel holder contains three flux wires and one melt wire, for a total of 18 flux wires and six melt wires in the test train. The flux and melt wire packages will be removed from the graphite fuel holders and packaged to preserve their condition, identity, and purity as described below. The flux wire packages will be analyzed for a determination of neutron fluence by gamma spectroscopy at Pacific Northwest National Laboratory (PNNL). The melt wires will be visually inspected for evidence of melting of the beryllium indicator wires at INL.

2.5.1 Description of the Fluence and Melt Wire Packages

Each graphite fuel holder contains a cobalt-vanadium (1% Co-V), an iron (Fe), and a niobium (Nb) flux wire package, and one melt wire package containing either two beryllium (Be) wires (Capsules 1, 3, 4, 5, and 6) or a single nickel (Ni) wire (Capsule 2). All the flux and melt wires are encapsulated in sealed vanadium tubes that are nominally 5 to 9 mm long, depending on wire type. One each of the Co-V, Fe, and Nb flux wire packages are embedded at the periphery, and the one melt wire package is in the radial center of the graphite fuel holder. All four packages were inserted into axial mounting holes drilled from the bottom of the fuel holder. The specific location of the flux and melt wire packages in the graphite holders are given the drawings for each respective capsule. Details of the flux and melt wire packages for each capsule, as well as the relevant drawing numbers, are provided in Appendix A.

2.5.2 Retrieval of Flux Wire and Melt Wire Packages

After dimensional measurement and gamma spectrometry of the graphite holder is completed (described in 2.2.3, 2.3), the flux and melt wire packages will be retrieved from the graphite holder. The packages must be retrieved whole and without loss of integrity to ensure that none of the irradiated material is lost or contaminated. It is anticipated that retrieval of the melt wire packages will be more difficult than retrieval of the flux wire packages due largely to the snug fit of the melt wire capsules in the mounting holes. In addition, the AGR-1 experience demonstrated that the melt wire capsules located in the center of the graphite holder (at the hottest location in the holder) apparently experienced reaction with the graphite and became significantly embrittled, making recovery difficult.

The first attempt to remove the packages will be done by gently tapping the graphite fuel holder to dislodge the packages, exposing them sufficiently for extraction. If they will not come out, the graphite holder will be cut to facilitate retrieval of the flux and melt wire packages. A core drill fixture has been designed to facilitate extraction of the flux and melt wire packages. This will allow the small vanadium

tubes to be extracted without risk of damage, and the entire graphite core can be sent to PNNL for analysis. These operations will be documented by digital photography.

After removal, all three flux wire packages from a capsule will be placed in a single labeled, radiologically clean container while the melt wire package from each capsule will be sealed in a separate labeled, radiologically clean vial to prevent loss of material and to minimize contamination. If the retrieved packages are embedded in graphite, researchers will not be able to read the identification number stamped on each package. However, each of the three different flux wires can be identified based on its unique gamma emissions during analysis, so it is not necessary to identify the individual flux wires from a capsule.

2.5.3 Analysis of the Flux and Melt Wire Packages

The neutron fluence at the specific locations in each graphite fuel holder will be determined from the activity of the flux wires. The pertinent nuclear reactions, the neutron energy threshold, and the isotope half life for each flux wire type are given in Table 6. The neutron fluence values are calculated from the counting data, flux wire mass, the calculated effective neutron absorption cross section, the neutron energy spectrum of the reactor core, and the operating power history of the reactor. The activity of the flux wires will be determined by direct gamma counting, without opening the packages. Of the two Nb reactions, the Nb-93m is of greater interest because it yields the fluence of neutrons with >180 keV energy. Because Nb-93m decays by emission of a relatively low energy gamma, its measurement will require dissolution of the Nb flux wire and scintillation counting of the resultant solution. The vanadium components do not contribute significantly to the activity of the packages because the vanadium activation products have relatively short half-lives—generally minutes or less. For the fluence analyses to be valid, the flux wire packages must be supplied intact, with no loss of contents, and sealed in clean protective vials or counting cards. The packages do not have to be cleaned of adherent graphite or carbide because the carbon will not interfere with the sample preparation or counting processes.

Table 6. Characteristic nuclear reactions and gamma emissions for the flux wires.

Flux wire	Nuclear reaction	Threshold	Product half-life
V+1% Co	Co-59 (n,γ) Co-60	Thermal	5.27 y
Fe	Fe-54 (n,p) Mn-54 Fe-58 (n,g) Fe-59	1.0 MeV thermal	312 d 44.5 d
Nb	Nb-93 (n, γ) Nb-94	Thermal	2×10^4 y
	Nb-93 (n, n') Nb-93m	0.18 MeV	16.1 y

The flux wire packages will be analyzed by PNNL, the fabricator of the flux and melt wire packages. Both laboratories will reduce the counting data to neutron fluence values. INL will provide the neutron energy spectrum and irradiation history for data reduction. Because of the similarity of the flux wires in the AGR-2 and AGR-1 experiments, the same basic procedure that was applied to the AGR-1 flux wires (Greenwood 2012) will be applied to the AGR-2 flux wire.

The analysis of the Be melt wires requires the determination of whether or not the two Be wires in the vanadium package have melted, indicating that the capsule temperature at that location exceeded the 1287°C melting point of beryllium. This requires simply the determination of the presence or absence of two free-standing wires in the Be packages. However, this may be complicated if significant degradation of the melt wire packages has occurred during irradiation. The analysis will involve carefully cutting open the vanadium tube to inspect the contents for evidence that the Be wires melted.

2.6 Capsule Fission Product Analysis

All of the major capsule components will be analyzed to determine the inventory of fission products released from the fuel compacts, a key indicator of in-pile fuel performance. This will include an analysis of gamma-emitting fission products (including Ag-110m, Cs-134, Cs-137, Ce-144, Eu-154, and Eu-155), Sr-90, and other non-gamma-emitting fission products of interest (including isotopes of Pd). The experimental methods will be similar to those used for the AGR-1 capsule components (Demkowicz et al. 2013). The results will be compared to predicted fission product inventories to determine the fractional inventories. The data will be compared among capsules to assess the relative level of performance of the various fuel types in the AGR-2 test train and the effects of burnup and temperature.

2.6.1 Graphite fuel holders

Following gamma scanning of the graphite holders and extraction of the flux and melt wires, the graphite holders will be analyzed to quantify the total inventory of fission products. This may require that the holders be crushed so that the pieces will fit into a container suitable for pneumatic transfer from HFEF to the MFC Analytical Laboratory. The holders will first be gamma counted using the Analytical Laboratory Hot Cell 4 spectrometer. The holders will then need to be oxidized in air and leached with acid, and the leachant analyzed for Sr-90 and other non-gamma-emitting fission product isotopes (including isotopes of Pd).

2.6.2 Metal capsule hardware

The metal capsule hardware includes the stainless steel shell (along with the steel and hafnium liners), the capsule head and floor, and the niobium through tubes. Following capsule disassembly these components will be sent to the Analytical Laboratory for analysis of fission products deposited on their surfaces. This may require resizing of the various components to fit in containers suitable for pneumatic transfer from HFEF to the Analytical Laboratory. The components will be leached in acid and the leachant analyzed for fission products.

2.6.3 Graphite spacers

The thin graphite and Grafoil® disks (collectively referred to here as the graphite spacers) located immediately above and below the graphite fuel holder in each capsule will be sent to the Analytical Laboratory for analysis of fission product inventory. AGR-1 results indicated that these components accounted for a relatively small percentage of the total fission product inventory in the capsule components (Demkowicz et al. 2013).

2.7 Shipping Compacts to ORNL

During the course of the AGR-2 PIE campaign, selected compacts will be shipped to ORNL for PIE work. The shipments will be made using Model 9977 Type B shipping packages, which consist of an outer stainless steel drum and inner stainless steel containment vessel. Each compact will be packaged in an aluminum storage tube, which will then be placed inside a tungsten shielding container, which is subsequently loaded into the 9977 containment vessel. Each 9977 will contain a single AGR-2 compact. A total of four 9977 packages are available to the program for this purpose, allowing a maximum of four compacts to be transported in one shipment. Because the compacts contain accountable nuclear material, the shipments will be coordinated between INL and ORNL by the Safeguards and Nuclear Material Accountability organizations of both laboratories. Details will be coordinated with ORNL prior to these shipments, and the specific scheduling will be determined as PIE proceeds. Specific compacts will be selected for shipment based on the results of non-destructive examination of the compacts and graphite

holders (Sections 2.3 and 2.4), as well as the results of as-run physics and thermal analysis of the compacts.

2.8 Microscopy of Fuel Compacts

Selected compacts will be analyzed in cross-section at the microscopic scale to assess localized effects of ATR irradiation on the compact matrix and embedded fuel particles. It is anticipated that approximately 3 to 5 AGR-2 compacts will be analyzed in this manner. Primary features for investigation include cracks in the compact matrix, fuel kernel swelling and porosity, kernel migration, buffer layer degradation and densification, corrosion of the SiC layer by fission products, fractures in the TRISO coating layers and delaminations between them, and deleterious interactions between the carbonaceous matrix and the outer pyrolytic carbon layer. Migration of fission products within particles and from kernels into the matrix will also be examined where practical.

Selected compacts will be sectioned axially and radially, mounted, and polished. If extensive damage is observed at the saw-cut surface of the compact (for example, extensive removal of embedded particles from the cut surface), then the samples should be ground past the damaged layer such that as many intact particle cross-sections as possible are exposed on the final polished surface. Samples may be cut and mounted as slices to diminish radiation dose rates for certain analyses. However, even relatively thin cross-sectional samples of fuel compacts will be highly radioactive (~1,000 R/hr at contact for one-tenth of a compact), so analytical instruments must be heavily shielded to accommodate them. The basic approach used for sample preparation and ceramographic examination will be similar to that used for the irradiated AGR-1 compacts (Ploger, Demkowicz, and Hunn, 2012).

Some fuel compacts may be sectioned for microanalysis after safety testing. The defect types to be investigated are the same as those before safety testing, although their frequency and severity may increase appreciably at safety testing temperatures. Any compacts to be used for this purpose will be selected following safety testing.

2.9 Compact Deconsolidation-Leach-Burn-Leach

Selected compacts will be analyzed with the deconsolidation-leach-burn-leach (DLBL) process. It is anticipated that approximately six as-irradiated AGR-2 compacts will be analyzed, plus the majority of compacts that are safety tested (discussed in Section 2.13). The objectives of this analysis are to:

- Disintegrate the compact matrix and liberate particles
- Determine the inventory of fission products in the compact outside of the SiC layer (i.e., in the OPyC layer and matrix)
- Determine the number of failed particles with exposed fuel kernels, in which all three coatings have failed (determined by analysis of the pre-burn leach solutions)
- Determine the number of particles with a failed or defective SiC layer but intact inner or outer pyrolytic carbon layers (determined by analysis of the post-burn leach solutions)

The four basic steps of this process are outlined below.

1. The deconsolidation process involves the electrolytic oxidation at ambient temperature of the carbonaceous binder in the compact matrix. In the process, the compact—the anode in the electrochemical circuit—is suspended in nitric acid solution (the electrolyte) while a direct current is applied between the compact and the cathode, which is suspended in the electrolyte solution. The total power applied to the compact is maintained below 10 watts throughout the process in order to avoid damage to particles. This results in oxidation of the matrix (without

significant oxidation of the OPyC) and disintegration of the compact, liberating free particles. The deconsolidation solution may be analyzed separately for inventory of actinides and fission products, or it may be used in the first pre-burn leach and analyzed following that step.

2. The pre-burn leach is used to dissolve most of the actinides and fission products in exposed kernels (i.e., particles with all three coating layers breached) or outside intact particles due to either uranium contamination or release through one or more intact coatings The leach is performed in hot nitric acid for a period of approximately 24 hours using a Soxhlet extraction apparatus. The process for AGR-2 will be similar to that used for AGR-1 at INL (Demkowicz et al. 2012a) and ORNL (Hunn et al. 2013). This pre-burn leach is performed twice—with additional leaches if necessary—to ensure that all analytes have been effectively leached from the deconsolidated material. Each of the solutions are analyzed for actinides and fission products.
3. The burn step, performed at 750°C in air, oxidizes the carbon residue from the matrix and all exposed pyrolytic carbon coatings, including the inner pyrolytic carbon and buffer layer of particles with a defective SiC coating but otherwise intact carbon coatings. This step exposes the fuel kernel in those particles with a failed SiC (but with intact pyrolytic layers) to the subsequent post-burn leach.
4. The post-burn leach dissolves the fission products and uranium in the exposed fuel kernels exposed by the burn step, and allows for a calculation of the number of equivalent particles with defective or failed SiC coatings. It also dissolves any actinides or fission products from the oxidized carbon material that were not dissolved prior to the burn step. The post-burn leach is performed in the same manner as the pre-burn leach and is also repeated a second time. Each of the solutions are analyzed for actinides and fission products.

Minor variations on the procedure outlined above may be performed based on the specific needs for subsequent particle analysis. For example, if particles with an intact OPyC layer are needed for irradiated microsphere gamma analysis, then the process may be interrupted after the first pre-burn leach, the particles sieved to remove the matrix debris, and an additional boiling step performed on the particles to remove any additional matrix material in order to facilitate particle handling during gamma analysis (see description in Hunn et al. 2012). In addition, since gamma counting of all particles is generally effective at identifying particles with failed or defective SiC (discussed in Section 2.11), identification of these particles in the post-burn leach is not necessary.

If there are no particles with failed TRISO coatings or a failed SiC layer in the compact (and therefore no kernels exposed to acid dissolution during the DLBL process), then the cumulative inventory of actinides and fission products found in the DLBL solutions is due to original uranium contamination in the matrix and actinides and fission products that have been released from the intact particles. Since the average level of uranium contamination in the compacts is known from as-fabricated fuel analysis, the DLBL data can be used as a measure of fission products released from intact particles but retained in the compact.

2.10 Burnup Measurements

The burnup of selected compacts will be analyzed based on mass spectrometric measurements in order to compare them with the predicted values from as-run physics simulations as well as the measured values based on compact gamma spectrometry data (Section 2.4). The procedure will be similar to that used for the AGR-1 compacts (Harp et al. 2014), with modifications based on lessons learned. Small subsets of loose particles (typically 20 particles each) from the deconsolidated compacts will be crushed to expose the kernels and the kernels will be heated in air to oxidize carbide phases that can be less soluble. The oxidized material will then be leached with acid to dissolve the actinides and fission

products. The solution will be analyzed with inductively coupled plasma mass spectrometry (ICP-MS) to measure the inventory of actinides and fission products. The burnup of the particles will be calculated using the "Fission Product Monitor – Residual Heavy Atom" technique. This method is based on end of life inventory of certain actinides and fission products in the fuel and the respective fission yields. The fission products used in this technique are La-139, Ce-140, Ce-142, Pr-141, Nd-145, and Nd-146.

Selection of specific compacts to be used for this analysis will be made at the completion of the AGR-2 irradiation and will be based on several factors, including the as-run physics predictions for compact burnup (it may be desirable to select compacts that span the range of burnups), fast fluence, and irradiation temperature. It is anticipated that approximately 3 to 4 AGR-2 compacts will be analyzed. It will be beneficial if compacts selected for this analysis are also those selected for destructive PIE (including DLBL and gamma counting) based on other criteria, as loose particles will be available from these compacts as part of the PIE process and deconsolidation of additional compacts will not be required.

2.11 Particle Inspection and Gamma Counting

Individual particles will be gamma counted to quantify the inventory of selected fission products. Measured inventories will be compared with the predicted inventories based on as-run AGR-2 physics simulations to gauge the relative fission product retention in each of the analyzed particles. The data may also be used to screen particles based on radionuclide inventories prior to performing other analyses, such as destructive burnup measurements (see Section 2.10), to ensure that the kernels have remained intact after compact deconsolidation. The specific count time for a particle will be influenced by the particular radionuclides that are of interest, the burnup and age of the fuel, the level of fission product release (primarily relevant for Ag-110m), and the counting geometry.

One of the primary objectives is to screen all particles from compacts with suspected particle defects to locate any particles with abnormal fission product retention that is indicative of defective or failed coatings, particularly the SiC layer. This is typically accomplished by examining the Cs-137 inventory in the particles, since Cs is well retained by intact SiC, but is significantly released through a defective or failed SiC layer even if one or both PyC layers remain intact. Since cerium is relatively immobile in the kernels, the ratio of Cs-137 to Ce-144 activity is a useful metric to screen for particles with abnormally low Cs inventory, since it adequately adjusts for variations in initial fissle content in the kernel (due to variation in stoichiometry, density, or total kernel volume) and burnup among the particles. A particle with an abnormally low Cs-137/Ce-144 ratio most likely has released a significant inventory of Cs. As there are a large number of particles in a single compact (approximately 3200 and 1500 particles in the UCO and UO_2 compacts, respectively) the counting time for a single particle must be relatively short to enable all particles to be analyzed in a reasonable timeframe (generally several weeks). Previous experience with irradiated AGR-1 particles indicates that the Cs-137 and Ce-144 activities can usually be measured with 50 – 100 s count times. This task will be performed using the Advanced Irradiated Microsphere Gamma Analyzer (IMGA) at ORNL which was used effectively during the AGR1 PIE campaign (Hunn et al. 2013, Baldwin et al 2012).

A second objective is to analyze the fission product retention of other key gamma-emitting fission products, including Ag-110m and Eu-154. Since these fission products have relatively low fission yield (and therefore have lower inventories compared to Cs-137 and Ce-144) and also often have lower gamma yields, longer counting times (generally $\geq$ 1 hour) are required and only a subset of particles (approximately 50 to 100) will be analyzed. The measured inventory of each specific fission product analyzed will be compared to the predicted inventory, and normalized based on the relative inventory of Cs-137. Cs-137 is used here because it has a high fission yield, long half-life, it's inventory is fairly linear as a function of burnup, and it is very well retained as long as the SiC layer remains intact. This activity

will be performed on a subset of particles from selected compacts using the Advanced-IMGA at ORNL and the Hot Cell 4 gamma spectrometer at the INL Analytical Laboratory (Demkowicz et al. 2012a).

Particle gamma counting will be performed on fuel after irradiation (to examine in-pile fission metals retention) and after safety testing (to examine high temperature fission metals retention). The analysis will be performed on particles following deconsolidation-leach or full DLBL analysis. Any particles of interest identified during gamma counting can be selected for detailed microanalysis as described in the following sections of the plan. This may include particles with low Cs-137, which will be examined in an attempt to identify the cause of the defective or failed SiC layer. It may also include particles sorted based on their relative level of Ag-110m retention, which will be examined to determine if silver retention is correlated to the properties of the SiC layer.

2.12 Microanalysis of Fuel Particles

Based on the experimental results of several of the preceding PIE tasks, individual particles or groups of particles will be selected for microanalysis using an array of characterization equipment. The general objective of these fuel particle analyses is to characterize fuel kernel porosity, kernel migration, buffer layer degradation, fractures in the TRISO coating layers, delaminations between coating layers, existence of fabrication defects affecting irradiation performance, reaction of fission products (especially palladium) with the silicon carbide layer, and deposition or residual clustering of fission products outside the kernel. Because these analyses will be performed after particle gamma counting in certain cases, the gamma counting results can be factored into particle selection. In such cases, one important aspect will be relating gamma counting results on release of metallic fission products to deterioration of the SiC layer or the presence of microstructural defects. Furthermore, microstructural features observed during PIE can be compared to pre-irradiation microstructures.

This activity will primarily involve mounting one or more particles in epoxy, grinding to near the particle midplane (or alternatively, the particular plane of interest), and polishing the surface to a sufficient quality to effectively observe the features of interest. Prior to this sample preparation, selected particles may be analyzed nondestructively using x-ray radiography and three dimensional tomographic reconstruction. This can allow specific features within the particles (including coating fractures, delaminations, and SiC defects) to be observed *in-situ*. This information can then be used to focus subsequent mounting and polishing so that specific features of interest can be revealed for detailed microanalysis. The x-ray analysis will be performed using the system developed at ORNL and used successfully on irradiated AGR-1 particles (Hunn et al. 2013).

A basic analysis of polished particle cross-sections will be accomplished with optical microscopy. This will consist of a general inspection of the particle morphology, including kernel swelling and buffer densification, along with coating layer fractures and delaminations. High resolution digital images of the specimens will be acquired.

A scanning electron microscope (SEM) will be used to perform a more detailed, higher resolution examination of the particle cross-sections. This technique will be used to inspect the entire particle cross-section for features of interest, including coating damage or fracture, evidence of fission product reaction with silicon carbide, and coating delaminations. Elemental analysis using energy dispersive x-ray spectroscopy (EDS) and/or wavelength dispersive x-ray spectroscopy (WDS) will be used to identify detectable clusters of actinides or fission products in the various coating layers, with particular attention paid to the IPyC and SiC layers. The elemental data can provide important information about the migration of fission products through the coating layers and reaction of fission products with silicon carbide. The SEM and EDS/WDS analyses will be used to further focus subsequent examination to

particular areas of interest on the particle cross-section. Both INL and ORNL have SEMs with EDS/WDS capability installed in locations that will allow analysis of irradiated fuel specimens.

A transmission electron microscope (TEM) or scanning transmission electron microscope (STEM) will be used to examine small areas of the particle cross-section at very high magnification to understand microstructural behavior down to the nanometer scale. Specimens from a particular location on a polished particle cross-section can be easily prepared using a focused ion beam (FIB) to produce an electron-transparent lamella for examination with the TEM. The TEM analysis will be coupled with elemental analysis such as EDS or electron energy loss spectroscopy (EELS) to identify the elemental constituents within the observed microstructure. This will allow the location of various fission products or actinides within the microstructure to be observed, providing further information on elemental transport through the coatings layers. FIB sample preparation and TEM/EDS/EELS analysis can be performed using instruments at the INL Electron Microscopy Laboratory (EML), the Irradiated Materials Characterization Laboratory (IMCL), and the Center for Advanced Energy Studies (CAES).

Electron backscatter diffraction (EBSD) will be used to characterize the crystallographic orientation of grains and the grain boundary character within the SiC layer in order to aid the interpretation of observed fission product release behavior in the particles. In particular, the diffusive release of fission products through intact SiC may be related to the nature of the grain boundaries.

Additional advanced characterization methods, including local electrode atom probe (LEAP) tomography, may be employed to examine selected samples as well to provide further detail at the atomic or near-atomic scales. It is expected that these types of analyses will contribute to the understanding of fission product transport through coating layers.

2.13 Safety Testing

Selected AGR-2 fuel compacts will undergo testing to assess the fission product retention characteristics at high temperatures that simulate depressurized core conduction cooldown conditions. The facilities to be used for this activity are the Fuel Accident Condition Simulator (FACS) furnace system at INL (Demkowicz et al. 2012b) and the Core Conduction Cooldown Test Facility (CCCTF) at ORNL (Baldwin et al. 2012). The fuel will be heated at temperatures as high as 1800°C in a helium atmosphere while measuring fission product releases as a function of time and temperature. This will include measurement of fission gas Kr-85, as well as condensable fission products (including isotopes of Ag, Cs, Eu, and Sr). The concurrent testing of fuel at both INL and ORNL using these two furnace systems allows the program to cross-check experimental results, verify consistency, and identify any experimental biases.

Fission gases released from the compacts will be carried from the furnace in the helium sweep gas and collected in cryogenically cooled traps continuously monitored with gamma spectrometers to measure the activity throughout the tests. Water-cooled condensation plates (FACS furnace) and deposition cups (CCCTF) will be used to collect condensable fission products during the test and exchanged at regular intervals (approximately 12 to 24 hours) to get time-dependent condensable fission product release information. The plates and cups will be gamma counted in a controlled geometry to quantify the activity of gamma-emitting fission products on each plate (including Ag-110m, Cs-134, Cs-137, Eu-154, and Eu-155). The plates and cups will then be leached with acid to transfer all deposited fission products into solution, which will then be analyzed with ICP-MS to quantify the inventory of non-gamma emitting fission products (including isotopes of palladium). Aliquots of the leachant solutions will be treated with ion exchange resin to selectively extract strontium, followed by subsequent analysis of Sr-90 inventory (e.g., with gas flow proportional counting or liquid scintillation).

The measured inventory of fission products will be compared to the predicted inventory in the fuel compact based on as-run AGR-2 physics simulations to calculate the fraction of the predicted inventory released. This will also involve a collection efficiency factor, which is the fraction of a particular element released from the fuel that is deposited on the plate/cup (the remaining fraction being deposited on other internal furnace components). This collection efficiency will either be determined from previous furnace testing or by measuring the total amount of each fission product deposited on the cups and furnace internals during a specific safety test.

The majority of safety tests will consist of an isothermal step at the target temperature. Maximum temperatures of 1600 – 1800°C will be used. The temperature profile for the isothermal tests will involve the following general steps:

1. Ramp to ~400°C at a rate of 120°C/h and hold for sufficient time to eliminate adsorbed water from the fuel (typically 2 hours).
2. Ramp to the representative fuel operating temperature (e.g., 1250°C) at a rate of 120°C/h and hold for ~12 hours to establish thermal equilibrium in the fuel compact.
3. Ramp up to the target test temperature at a rate of 50°C/h[a].
4. Nominal hold time at the test temperature will be 300 hours, although tests can be shortened (for example, in the case of excessive particle failures observed in the early stages of the test based on fission gas release) or lengthened as necessary.

The temperature profiles for 1600, 1700, and 1800°C isothermal tests are shown graphically in Figure 6.

Non-isothermal heating tests will be performed to more realistically simulate the peak fuel temperature-time profiles during a postulated accident scenario. The maximum temperature of these tests and specific temperature-time profile are still to be determined. A candidate temperature profile for these tests is based upon the profile used in the 1992 safety test of the spherical fuel element Arbeitsgemeinschaft Versuchreaktor (AVR) 91/31 in the Federal Republic of Germany (details available in the FZJ report IWE-TN-17/93). The temperature profile for this test was based upon the predicted peak fuel element temperature in the HTR-MODUL reactor design during a depressurization accident with the curve extrapolated up to achieve maximum temperature of approximately 1700°C. The specific temperature profile used in the safety test of AVR 91/31 is shown in Figure 7, and it is planned that this temperature profile will be used in safety testing of AGR-1 compacts (test not yet performed). Application of this profile to the AGR-2 safety testing campaign may depend on results from AGR-1 testing or further developments in VHTR design activities.

[a] The heating rate of 50°C/h was determined based on a simple analysis of previous predictions for peak core temperatures of the 350 MWt MHTGR (HTGR-86-024) and 600 MWt GT-MHR (DOE-GT-MHR-100230) during depressurized core conduction cooldown. A rate of 50°C/h corresponds to roughly the maximum heating rate in either scenario once the temperature exceeds 1250°C.

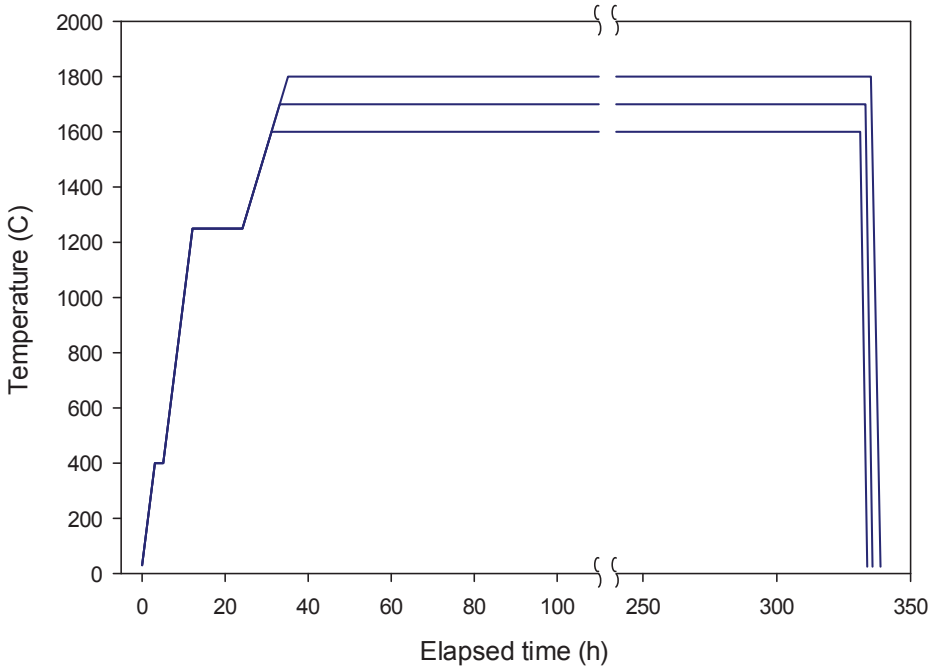

Figure 6. Temperature profiles for isothermal safety tests at maximum temperatures of 1600, 1700, and 1800°C.

Specific compacts will be selected for safety testing based on irradiation history (including burnup, fast fluence, and temperature taken from the final as-run AGR-2 physics calculations) and on PIE data including analysis of the irradiation capsule components (Sections 2.3 and 2.6) and gamma scanning of the compacts (Section 2.4). The PIE data will provide information on the relative amount of fission product release during the irradiation and the likelihood of a particular compact containing a failed or defective SiC layer. A preliminary estimate of the number of compacts from each AGR-2 capsule to undergo safety testing is presented in Table 7. For each of the fuel types (U.S. UCO and UO_2) this should include at least one test at 1700 or 1800°C.

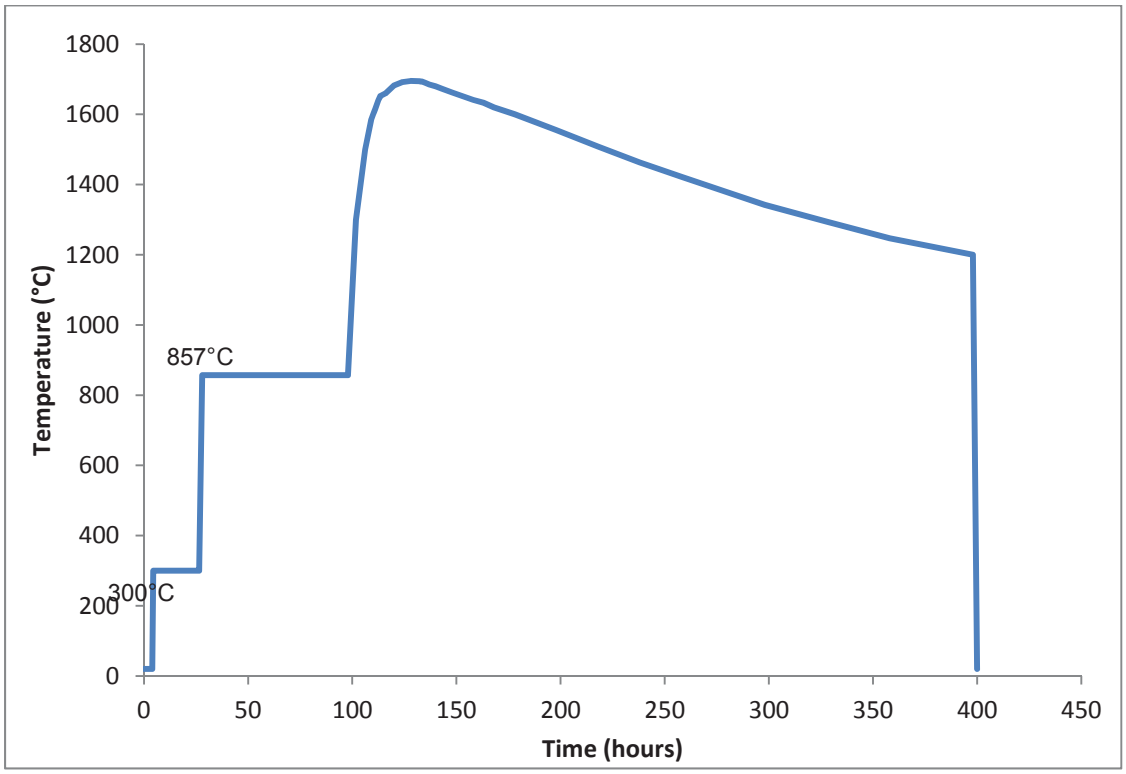

Figure 7. Proposed temperature profile for AGR-2 transient temperature accident simulation safety tests. Temperature profile based on that used for safety testing of German spherical fuel element AVR 91/31 (FZK report IWE-TN-17/93).

Table 7. Preliminary estimate of the number of AGR-2 compacts to undergo safety testing.

Capsule	Number of Compacts
2	4
3	4
5	2*

*This value assumes that the fuel in Capsule 2 performs relatively well. If Capsule 2 fuel performance is poor, an increased number of tests may be needed from Capsule 5 fuel.

Additional safety tests may be performed on loose particles. The main objective of this type of test would be to determine release from particles at elevated temperatures by removing the effects of sorption/desorption from the compact matrix. Loose particle tests may also allow for direct measurement of fission product loss by comparing gamma spectra acquired before and after the test. The scope of these tests are yet to be determined and will be based on available AGR-2 data as well as the efficacy of similar tests performed on the AGR-1 fuel (AGR-1 tests on loose particles have not yet been performed).

2.14 Reirradiation

A key data need in TRISO fuel performance evaluations is the release of radioiodine from the fuel during irradiation and during high temperature accident scenarios. To assess release during post-irradiation safety tests, fuel specimens must be reirradiated for short durations immediately prior to the safety test in order to generate short-lived I-131 (half life 8.03 days). Selected AGR-2 fuel specimens may

be reirradiated in the HFEF neutron radiography (NRAD) reactor prior to safety testing in the FACS furnace. These specimens may consist of a subset (approximately 5–30) of loose particles, particles with intentionally-induced coating fractures (can be either mechanically-induced fractures or laser-drilled holes in the coatings), or loose kernels (or buffer-coated kernels). Specific details of the fuel specimens to be used are still to be determined.

The loose kernels or particles would be placed in a specially designed container, inserted manually into the NRAD core, and irradiated for a predetermined interval deemed appropriate to generate sufficient inventory of I-131. The specimens would then be removed from the reactor, transferred to the HFEF main cell, and subjected to safety testing in the FACS furnace. Analysis of the condensation plates would focus on quantification of I-131. Analysis of the sweep gas would focus on both Kr-85 and Xe-133.

Execution of this activity will be dependent on overall program funding levels during the AGR-2 PIE campaign.

2.15 Sample Archiving and Disposal

All AGR-2 compacts not subjected to destructive analysis at INL or shipped to ORNL will be held in temporary storage in HFEF. After deconsolidation of a compact at either INL or ORNL, all particles not used for destructive analysis will be held in temporary storage at HFEF (INL) or the ORNL Irradiated Fuels Examination Laboratory (IFEL). Disposition plans for the unused fuel specimens will be determined at a later date by VHTR TDO staff.

3. WASTE HANDLING

The PIE activities will generate small amounts of radioactive waste (estimated at less than 10 ft^3 per year) that must be properly dispositioned. This waste will be generated by the disassembly, metallography, safety testing, equipment maintenance activities, and analytical laboratory activities associated with the AGR-2 examination and analysis. Typical wastes will include short sections (< 2 meters) of 1/16 to 1/8-inch diameter sheathed TCs and gas lines, turnings from the tubing cutter, condensation plates from the heating furnace, pneumatic transfer rabbits, and parts replaced on the safety testing furnaces (replacement tantalum hot zone components, metal heat shields, the graphite furnace elements, and other relatively small furnace components), and analytical laboratory solids and solidified liquids. Additionally, after analysis activities of the test train capsule components (capsule head, through tubes, outer shell, graphite holder, and graphite spacers) are completed, these components will be dispositioned as waste. Most of the waste will be classified as remote-handled low-level waste. Some of the waste, such as activated stainless steel, may be classified as greater-than-Class-C waste. These wastes will be gathered and placed into appropriate disposal containers. At INL, these wastes will be stored in the Radioactive Scrap and Waste Facility located at MFC until final disposal arrangements can be made.

The metallography preparation work will involve cutting, slicing, grinding, and polishing activities that create small volumes of highly radioactive wastes, including the grinding and polishing residuals and the unused portions of the fuel compacts. The whole compacts may have contact radiation fields as high as 10^4 R/hr 6 months after the test irradiation. The wastes associated with the fuel compact analysis and the residual compact material will be disposed of after analysis activities are complete. INL Safeguards personnel must be notified and authorize disposition activities of the accountable fuel materials, including analytical and residual material wastes, since they contain accountable materials.

ORNL plans to handle the waste generated by this work through the normal laboratory waste disposal channels. Most of the waste is expected to be low-level waste or remote handled low-level waste that falls within the current waste disposal paths. The liquid waste generated by the analytical tasks will be handled by the normal channels, either by direct disposal to the liquid waste system, drying and disposal as solid

waste, or grouting, if necessary. The remaining compacts, if any, will be dispositioned as spent nuclear fuel. Since the test train and capsule disassembly work will be done at INL, very little activated metal will be handled; most of the waste generated will be from the metallographic and analytical tasks.

4. QUALITY ASSURANCE

The VHTR Technology Development Office (TDO) Program Management Plan, PLN-2494 (Croson 2013), identifies that all VHTR TDO activities are conducted in accordance with requirements identified in the Quality Assurance Program Plan (PLN-2690). This plan identifies governing documents for:

- Records Management
- Software Quality Assurance Plan
- Data Management Plan
- Configuration Management
- Personnel Indoctrination, Training, and Qualification.

Work activities associated with this plan are conducted under a quality program implementing American Society of Mechanical Engineers NQA-1 2008, 1a 2009. Organizations or services subcontracted to support PIE quality-affecting work activities will be on the INL Qualified Suppliers List for the selected activities to be performed. Activities affecting quality include, but are not limited to, procurement, handling, shipping, storing, inspecting, testing, training, data collection, records, electronic data storage, software control for software used in data analysis, and the generation of reports from collected data. ORNL will perform PIE support services in accordance with their project AGR specific quality assurance program plan, QAP-ORNL-NR&D-01 (Vance 2013).

4.1 Data Management

INL is responsible to maintain the record copy of all data associated with the PIE and safety testing activities. This data may come from INL, ORNL, PNNL, universities, or other partners in the PIE effort. INL will work with these institutions to define the desired data formats. PIE and safety testing data that will be kept as project records will be transferred from their original source to either the Nuclear Data Management and Analysis System (NDMAS) or the INL Electronic Document Management System (EDMS). Primarily, NDMAS will be the data storage forum for machine readable data (e.g., database, spreadsheet, or tab delineated) and EDMS will be the storage forum for other types of information including pictures, evaluation reports, pdf documents, technical evaluations (TEVs), and engineering calculation and analysis reports (ECARs). Since NDMAS will have provisions that allow access to the data outside of the INL computer firewall, data that would normally be stored on EDMS may be moved to NDMAS to allow access by users outside INL. The VHTR Program Data Management and Analysis Plan (Hull 2011) details how data will be stored, controlled, categorized, and qualified.

Nuclear data from the latest Evaluated Nuclear Data File (ENDF) database (currently ENDF/B-VII.1, Chadwick et al. 2011) will be used for decay-corrections of measured radioisotopic inventories (for comparison with predicted values) and for relevant gamma ray yields used in spectral processing.

5. REPORTING

Program staff will create reports pertaining to results from the PIE and safety testing of U.S. AGR-2 capsules to ensure that pertinent data from the PIE activities are available for various programmatic decisions as necessary. These will include:

- *Test Train Inspection, Disassembly, and Metrology Report.* This report will summarize the results of preliminary PIE activities, including: gamma scanning the intact test train, test train disassembly and inspection, and compact and graphite holder metrology. The availability of these data will help support subsequent test train design and fabrication as well as support a revision of the AGR-2 capsule thermal analyses, if necessary.
- *Topical reports.* Topical reports will be prepared to provide details on specific components of the AGR-2 PIE. These will include topical reports summarizing the destructive PIE performed on specific compacts, including safety testing results as appropriate. It is envisioned that a report will be prepared for each compact that undergoes destructive PIE. Other report topics may include:
  - Gamma scanning results from the graphite fuel holders
  - Gamma scanning results from the fuel compacts
  - Results of the analysis of fission products on the AGR-2 capsule components
  - Results of compact ceramography.
- *Final AGR-2 PIE data report.* This report will be prepared at the completion of the AGR-2 PIE and when all data have been obtained from ongoing experiments and analyses. It will include data summaries taken from the relevant topical reports and present the pertinent conclusions from the AGR-2 PIE.

Regular input on PIE activities and experimental results will also be provided as needed for the VHTR TDO monthly and bimonthly reports and weekly highlights. The VHTR TDO Fuels PIE staff will make selected PIE data available to the NGNP database as it is generated and will participate in bi-weekly teleconferences, VHTR TDO fuels program meetings, and VHTR TDO annual R&D meetings to facilitate dissemination of experimental data as needed by the program and to discuss relevant issues.

All data obtained from analysis of the fuel and capsule components from Capsules 1 and 4 will be reported separately, based on agreements between the U.S. VHTR TDO program and the relevant French and South African authorities.

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Appendix A

The fluence and melt wire packages for each AGR-2 capsule are listed in Table A1, along with the identification code, the dimension of the outer vanadium package, the initial mass of the wires, and the INL drawing number corresponding to each capsule, which indicates the specific locations of the packages inside the graphite holders. Note that several pairs of fluence wire packages (color-coded in the table) share the same ID code. However, each set of three fluence wires from each capsule will be packaged together and will be identified during analysis based on the unique gamma emissions, so ID code duplication is not expected to pose a problem in package identification during capsule disassembly.

Table A1. List of AGR-2 fluence and melt wire packages, package IDs, dimensions of the package, mass of the wires, and the corresponding INL drawing number.

Capsule	Package contents	ID	Package length (mm)	Package diameter (mm)	Initial wire mass (mg)	INL Drawing #
Capsule 1	Fe fluence wire	T	7.30	1.36	7.838	600910
	Nb fluence wire	AV	8.42	1.36	9.699	
	V-1%Co fluence wire	5	4.83	1.34	0.872	
	Be melt wires	T7	8.10	1.36	0.772	
Capsule 2	Fe fluence wire	K	7.22	1.34	7.598	600911
	Nb fluence wire	0A	8.80	1.32	9.818	
	V-1%Co fluence wire	3	4.63	1.45	0.782	
	Ni melt wire	L1	8.04	1.35	8.026	
Capsule 3	Fe fluence wire	Z	7.39	1.35	7.850	600912
	Nb fluence wire	A5	8.63	1.32	9.875	
	V-1%Co fluence wire	4	4.86	1.35	0.853	
	Be melt wires	1F	8.10	1.31	0.772	
Capsule 4	Fe fluence wire	7	7.39	1.35	7.751	600913
	Nb fluence wire	AY	8.69	1.35	9.302	
	V-1%Co fluence wire	Z	4.65	1.38	0.930	
	Be melt wires	8J	8.10	1.32	0.645	
Capsule 5	Fe fluence wire	0	7.27	1.34	7.734	600914
	Nb fluence wire	A7	8.67	1.38	9.152	
	V-1%Co fluence wire	K	4.68	1.32	0.836	
	Be melt wires	2D	8.10	1.37	0.866	
Capsule 6	Fe fluence wire	4	7.17	1.31	7.486	600915
	Nb fluence wire	A9	8.65	1.28	9.204	
	V-1%Co fluence wire	T	4.90	1.33	1.080	
	Be melt wires	2Z	8.05	1.31	0.807	