Title

An Analysis of Nuclear Fuel Burnup in the AGR-1 TRISO Fuel Experiment Using Gamma Spectrometry, Mass Spectrometry, and Computational Simulation Techniques

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Abstract

AGR-1 was the first in a series of experiments designed to test US TRISO fuel under high temperature gas-cooled reactor irradiation conditions. This experiment was irradiated in the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL) and is currently undergoing post-irradiation examination (PIE) at INL and Oak Ridge National Laboratory. One component of the AGR-1 PIE is the experimental evaluation of the burnup of the fuel by two separate techniques. Gamma spectrometry was used to non-destructively evaluate the burnup of all 72 of the TRISO fuel compacts that comprised the AGR-1 experiment. Two methods for evaluating burnup by gamma spectrometry were developed, one based on the Cs-137 activity and the other based on the ratio of Cs-134 and Cs-137 activities. Burnup values determined from both methods compared well with the values predicted from simulations. The highest measured burnup was 20.1 %FIMA (fissions per initial heavy metal atom) for the direct method and 20.0 %FIMA for the ratio method (compared to 19.56% FIMA from simulations). An advantage of the ratio method is that the burnup of the cylindrical fuel compacts can be determined in small (2.5 mm) axial increments and an axial burnup profile can be produced. Destructive chemical analysis by inductively coupled mass spectrometry (ICP-MS) was then performed on selected compacts that were representative of the expected range of fuel burnups in the experiment to compare with the burnup values determined by gamma spectrometry. The compacts analyzed by mass spectrometry had a burnup range of 19.3 % FIMA to 10.7 % FIMA. The mass spectrometry evaluation of burnup for the four compacts agreed well with the gamma spectrometry burnup evaluations and the expected burnup from simulation. For all four compacts analyzed by mass spectrometry, the maximum range in the three experimentally determined values and the predicted value was 6% or less. The results confirm the accuracy of the nondestructive burnup evaluation from gamma spectrometry for TRISO fuel compacts across a burnup range of approximately 10 to 20 % FIMA and also validate the approach used in the physics simulation of the AGR-1 experiment.

Keywords

Text

1 Introduction

AGR-1 (Advanced Gas Reactor-1) was the first in a series of experiments designed to test US tristructural isotropic (TRISO) fuel under high temperature gas-cooled reactor irradiation conditions (Grover, 2010; Collin, 2012). This experiment was irradiated in the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL) and is currently undergoing post-irradiation examination (PIE) at INL and Oak Ridge National Laboratory. One component of the AGR-1 PIE is the experimental evaluation of the burnup of the fuel by two separate techniques. The measured burnup data is needed in order to validate the models used in physics simulations of the AGR-1 experiment (Sterbentz, 2011), which provide depletion, burnup, and fast neutron fluence values, as well as power generation rates, which are subsequently used in thermal models of the experiment to predict fuel and capsule component temperatures.

Gamma spectrometry was initially used to non-destructively evaluate the burnup of all 72 of the TRISO fuel compacts that comprised the AGR-1 experiment. Burnup evaluation based on gamma spectrometry was performed by two methods. The first method involved relating the total activity of a particular fission product (Cs-137) in a

compact to the compact average burnup, and the second method used the activity ratio of a shielded and direct fission product (Cs-134 and Cs-137) to determine both compact average burnup and local burnup in a small axial region of the compact. Gamma scanning TRISO fuel has some advantages over scanning monolithic fuel forms like UO₂ pellets or metallic fuel slugs. Most notably, the fission products (with the exception of Ag-110m) in TRISO fuel stay where they are generated. Post irradiation examination of the AGR-1 test train components confirms that the fractional release of Cs from the AGR-1 fuel was on the order 10⁻⁵ or less (Harp et al., 2012). Conversely Cs in UO₂ and metallic fuel is volatile and migrates during irradiation.

Destructive chemical analysis by inductively coupled mass spectrometry (ICP-MS) was also performed on selected compacts that were representative of the expected range of fuel burnups in the experiment to compare with the burnup values determined by gamma spectrometry. The results of both experimental techniques were compared to detailed depletion simulations. (Sterbentz, 2011)

The AGR-1 fuel consisted of TRISO coated particles based on 350 µm diameter kernels comprised of a mixture of uranium oxide and uranium carbide (referred to as UCO) with 19.7% U-235 enrichment. The particles were pressed into cylindrical fuel compacts (25.1 mm long x 12.4 mm diameter) each contained an average of 4126–4150 particles. The compact consists of TRISO particles in a graphite matrix to contain the particles. The packing fraction for TRISO particles in the compacts was approximately 37% (Maki, 2009; Phillips, 2010). Figure 1(a) contains an image of a typical AGR-1 compact, and Fig. 1(b) is a radiograph of a single AGR-1 compact. The dark circles in the radiograph are the high density (10.4 g/cm³) UCO kernels. Each end of the compact has a graphite endcap (1-2 mm thick) which were deliberately added to protect the TRISO particles in the compact during sintering. The particle packing fraction was approximately 37%. The fuel was irradiated for 620 effective full power days in the ATR located at the INL, and is estimated to have reached a peak burnup of 19.5% FIMA (fissions per initial heavy metal atom).; (Sterbentz, 2011). In the AGR-1 experiment, the fuel compacts were split into 6 different capsules that each contained 12 compacts, with the capsules welded end-to-end to create the fully assembled test train. The temperature in each capsule was controlled by changing the helium-neon gas flow based on temperature feedback from a designated thermocouple in the capsule. The compacts were arranged in a triangular array and stacked 4 high in each capsule (see Fig. 2). The stacks were held in place by a graphite cylinder that contained holes for the three fuel stacks. The combination of all the AGR-1 fuel compacts having the same enrichment and the chopped cosine shaped axial neutron flux profile in the ATR core resulted in variable burnup across the AGR-1 fuel compact stacks (Grover, 2010). Two of the three stacks of fuel compacts (Stack 1 and Stack 3 in Fig. 2) were directly facing the ATR core at the same angle creating very similar burnup profiles, while the third stack (Stack 2 in Fig. 2) was shielded by the other two stacks from the ATR core, resulting in lower burnup. Graphite spacers were placed at either end of the graphite holders to prevent contact between the fuel and the metal components of each capsule. See Fig. 3 for an illustration of the major components of the AGR-1 capsules. See Grover (2010) and Maki (2009) for more details on the compacts, capsules, and experiment design. The X-Y-Z nomenclature used for the AGR-1 compacts indicates the capsule number, the axial level, and stack number. For example, Compact 6-3-2 is taken from Capsule 6, Level 3, Stack 2.

Fig. 1 (a) Image of typical fuel compact (fractured to show the interior structure) and (b) a x-ray radiograph of an AGR-1 TRISO fuel compact.

Fig. 2. Numbering system used to identify AGR-1 compacts.

Fig. 3. Major components of a representative AGR-1 capsule.

2 Experimental Methods

2.1 Gamma Scanning of AGR-1 Fuel

A comprehensive effort was made to develop an integrated summation of isotope activity for all of the components of the AGR-1 experiment (Harp et al., 2012). This included doing detailed gamma scans of the individual fuel compacts and graphite holders. All 72 fuel compacts were individually gamma scanned using the Precision Gamma Scanner (PGS) system located in the Hot Fuel Examination Facility (HFEF) at INL. The PGS system consists of a collimator that penetrates the HFEF hot cell wall, a high purity germanium (HPGe) gamma spectrometry system with Compton suppression, and an in-cell stage used to position samples for examination. The hot cell side of the collimator consists of tungsten alloy blocks that can be adjusted to create a slit height varying from 0.254 to 0.00254 cm (0.1 inches to 0.001 inches). The width of the collimator opening is fixed at 2.22 cm (0.875 inches) creating a rectangular viewing area for the PGS system. An assembly drawing of the PGS collimator is shown in Fig. 4. Gamma-rays entering the collimator travel through a thin aluminum window (right side of Fig. 4), pass through the initial tungsten collimator blocks (Aperture Insert and Upper Cam in Fig. 4), through a second tungsten alloy beam scraper, and then to the HPGe detector. The PGS collimator can be rotated from a horizontal slit orientation 90 degrees to a vertical slit orientation when needed for specific scanning procedures.

HFEF-N Collimator Assembly

Fig. 4. Assembly drawing of PGS collimator

The HPGe detector is coupled with a bismuth germanate (BGO) detector for Compton suppression. The BGO crystal surrounds the HPGe detector with the exception of a single hole to allow gamma-rays from the collimator to directly strike the HPGe. The Compton suppression uses a unique logic that allows it to operate in a relatively higher background area such as the HFEF main floor without voiding an unacceptably high number of true full energy photon absorptions in the HPGe. In the system, Compton suppression only occurs when a true Compton event (where a photon Compton scatters from the HPGe crystal into the BGO crystal) has occurred. Using this logic, random events in the BGO crystal from background do not suppress the HPGe signal. The PGS uses an INL developed control software interface that controls both the collection of gamma-ray spectra and the movement of the sample in the hot cell. Spectroscopic control is achieved by communicating with an ORTEC 921 multi-channel buffer (MCB). Once the sample is positioned, the PGS collects a spectrum until the live time clock on the MCB reaches the desired limit. The contents of the buffer are then stored on the PGS control computer as a uniquely named ORTEC .CHN binary spectrum file. Additionally a corresponding .DAT file that contains position and system status information related to the PGS during spectrum collection is also saved after each spectrum is collected. The control software then moves the sample to the next desired position and begins counting again.

For the AGR-1 PGS examination, each compact was loaded into an individual aluminum container. The containers containing the fuel compacts were then stacked axially in an external aluminum tube connected to the PGS positioning stage and progressively scanned in 0.254 cm intervals with the collimator horizontally oriented and the slit height set to 0.254 cm. The slit was oriented perpendicular to the compact axis such that each scan captured a 0.254 cm axial "slice" of the compact. A single scan measured all 12 fuel compacts from one of the six AGR-1 capsules. Well-characterized Eu-152 calibration sources (Drigert, 2010) were inserted into the external tube preceding and following the fuel compacts to diagnose any potential shifts in detection system behavior during a scan. The container preceding the fuel compacts contained a single Eu-152 source, and the container following the fuel compacts contained two Eu-152 sources. The two sources were located a precise distance apart inside the container to allow confirmation of the precision of the movement of the positioning stage.

2.2 Calibration and Characterization of PGS system

The gamma-ray spectrum for each incremental scan was analyzed to determine the activity of the fission products present in the fuel compacts. The collected gamma-ray spectra were analyzed using the PC-GAP software

package developed at INL. The PCGAP software performs a peak search for each gamma-ray spectrum, fits the area of each identified peak, characterizes the likely source isotope for each peak and calculates a detected activity for the spectrum based on the detected gamma-ray peaks and user-provided efficiency calibration. The energy calibration was performed and confirmed using the spectra collected from the three Eu-152 sources as they were scanned during each PGS scan. Comparison of peak channel positions from the top Eu-152 source and the bottom two Eu-152 sources confirmed that the PGS detector system was stable throughout the entire PGS scan of 12 fuel compacts from an AGR-1 capsule, which consisted of about 420 individual steps (approximately 75 to 80 hours including dead time in the detector; empty space between compacts was also scanned).

The creation of a gamma-ray efficiency calibration for the AGR-1 compacts was complicated by the lack of a calibrated standard that could faithfully replicate the geometry present in a fuel compact. To account for the geometry effects of the TRISO fuel compact, a Monte Carlo N-Particle (MCNP) model was created of the PGS collimator, the PGS detector, the Eu-152 calibration source geometry, and a representative AGR-1 fuel compact. In this work MCNP5 v1.60 was used (X-5 Monte Carlo Team, 2003). Initially the simulated geometry represented the case of a Eu-152 source being viewed by the PGS collimator. The MCNP model was refined until efficiency curves derived from the simulation closely matched measured efficiency curves from the in cell Eu-152 sources. The MCNP source geometry was then changed to the AGR-1 geometry. The AGR-1 compact model included particles with the exact geometry of the AGR-1 particles with all their corresponding layers (Maki, 2009). In the model, spheres of coated particles were surrounded by a cube of graphite large enough to replicate the AGR-1 particle packing fraction of approximately 37%. An MCNP URAN card was applied to the coated sphere so that it was randomly located in the surrounding graphite. Source photons were initiated inside kernels of the coated spheres to represent fission products trapped in the fuel. For energies above 300 keV, the resulting simulated efficiency was consistent with the measured Eu-152 source efficiency. The derived efficiency was applied to the collected spectra.

With the energy calibration and efficiency calibration it was possible to analyze the collected spectra to determine the activity per scanned axial slice of each compact. There were 11 prominent fission products detected in the spectra: Y-91, Zr-95, Ru-103, Rh-106 (the daughter of Ru-106), Sb-125, Cs-134, Cs-137, Ce-141, Ce-144, Eu-154, and Pr-144 (daughter of Ce-144). Because of the large number of scans and isotopes of interest, a PYTHON script was written to automatically process the output data from each scan and organize it in an easily interpreted format. More detailed information is available in (Harp, 2011).

2.3 Mass Spectrometry of Select Compacts

After non-destructive examination of the fuel compacts, four select compacts were designated for destructive burnup examination in addition to other tests such as electrolytic deconsolidation, irradiated microsphere gamma analysis (IMGA) and particle microscopy (Demkowicz et al., 2012a; Demkowicz, 2010). Burnup measurements were performed on loose particles by using the mass spectrometry measured actinide and fission product inventory in the kernels. The four compacts selected for destructive burnup (6-3-2, 3-2-1, 5-3-1, and 1-3-1) were specifically chosen because they roughly spanned the burnup range of the AGR-1 test. Additionally, the compacts were only taken from level 2 or 3 of the capsule where the axial flux profile varied less across a compact, and three of the compacts were from the same stack in the experiment. This was done to assist in the comparison of experimental burnup results to simulation.

Solutions for mass spectrometry analysis were prepared using the following general procedure. The compacts were electrolytically deconsolidated, leached in nitric acid, oxidized to remove the outer pyrolytic carbon layer and acid leached again. Typically three sets of 20 deconsolidated particles were selected, mechanically crushed to expose the fuel kernels, oxidized to convert insoluble carbides to soluble oxides and the residue leached in an acid solution. However there were several variations in the leaching method used for the four different compacts, as one of the objectives of this work was to optimize the process for this specific fuel form.

For Compact 6-3-2, four sets of 20 particles were randomly selected for burnup analysis. The first set of particles was crushed and immediately leached in nitric acid. However the measured gamma activity of the leached residue revealed incomplete dissolution of fission products. The three subsequent batches of particles were crushed, rinsed out of the crushing device with nitric acid, oxidized in an air atmosphere in a muffle furnace for 24 hours at 400°C to convert any carbide compounds to oxides, and then leached in boiling nitric acid for 1 hour. The leachate solutions were then analyzed by ICP-MS (Demkowicz et al., 2012b). A very similar procedure to the final Compact 6-3-2 procedure was followed for Compact 3-2-1. However, there were concerns about full oxidation of the crushed kernels with a temperature of only 400°C, and the oxidation temperature was increased to 750°C for 24 hours.

When comparing the amount of Pu recovered from the particles from Compacts 6-3-2 and 3-2-1 to the expected inventory from simulations, the ratio of measured to predicted inventory was significantly less than 1, suggesting that either the simulation was over-predicting Pu in the fuel or that the Pu was not being fully recovered from the

crushed particles. In order to address the possibility of incomplete recovery of Pu from the particles, one sample of 20 particles from Compact 5-3-1 was processed using the same procedure as the sample from Compact 3-2-1, while three samples were varied slightly by the addition of a small amount of hydrofluoric acid to the 24 hour hot acid leach. For Compact 1-3-1, three techniques were applied in an attempt to further explore the optimum approach. Three sets of particles were oxidized at 400°C and leached in nitric acid, three sets of particles were oxidized at 400°C and leached in nitric acid with a small amount hydrofluoric acid added, and three sets of particles were oxidized at 750°C and leached in nitric acid with a small amount of hydrofluoric acid added. Use of 3 groups of particles for each technique was intended to ensure that it would be possible to verify repeatability, to distinguish inadvertent variations in technique, or detect possible cross-contamination from specific results. The variations in sample preparation technique are summarized in Table 1.

Table 1. Summary of ICP-MS Sample Preparation Techniques

Compact	Sets of 20 Particles	Burn	Acid
6-3-2	1	None	HNO ₃
6-3-2	3	400°C	HNO ₃
3-2-1	3	750°C	HNO ₃
5-3-1	1	750°C	HNO ₃
5-3-1	3	750°C	HNO ₃ + HF
1-3-1	3	400°C	HNO ₃
1-3-1	3	400°C	HNO ₃ + HF
1-3-1	3	750°C	HNO ₃ + HF

The preparation of the samples for ICP-MS was performed in a hot cell. After preparation the samples were diluted and aliquots were transferred out of the hot cell for ICP-MS analysis. The samples were analyzed for both fission products and actinides. In addition to the burnup calculation, the quantity of each measured actinide was compared to the expected actinide inventory from simulations (Sterbentz, 2011). These comparisons influenced some of the changes in sample preparation technique. Overall the agreement between the measured quantity of uranium (U-235 and U-238) was in good agreement with the expected inventory in 20 particles. However, there was consistently less measured plutonium (Pu-239 and Pu-240) than expected relative to the simulated predictions. Unfortunately, the addition of HF to the leaching step did not conclusively result in consistently higher Pu recovery for all samples. The various iterations in sample preparation and several iterations modifying the simulations were unable to resolve the discrepancies between recovered Pu and the expected inventory or positively identify if the bias is in the experimental measurements, the simulations, or both. Due to the difficulty of performing measurements on a small mass, the precise mass of the 20 kernels analyzed in each case was not known prior to dissolution. Variations in the as-fabricated AGR-1 kernel size (kernel mean diameter was 350 µm with a standard deviation of 9.0 µm) could bias the comparison of predicted actinide inventory with the measured values. However, the mass of fuel leached is not required for the burnup evaluation techniques discussed in the next section. Future burnup measurements planned for subsequent AGR irradiation tests will continue to investigate this disagreement. Fortunately a 20% Pu difference has little impact on the burnup evaluation at this initial U-235 enrichment (19.7 weight percent U-235) and burnups up to 20% FIMA.

3 Burnup Evaluation Techniques and Results

3.1 Measured Activities from Gamma Spectrometry Compared to Simulation

Total activity for each compact and each isotope of interest was collected by summing the slice activities derived from spectra that corresponded to a certain AGR-1 compact. A typical spectrum collected from an AGR-1 compact can be seen in Fig. 5. The major peaks are labeled with their source fission product. While the energy window in Fig. 5 focuses on the major peaks in the spectra, additional data were collected out to 1600 keV. Detailed tables containing all the measured activities are available in (Harp, 2011). Because of their importance in this work, the total decay corrected activity per compact measured for Cs-137 and Cs-134 is shown in Table 2. In Fig. 6, the measured activities of several AGR-1 compacts are compared with the calculated activities from Sterbentz (2011). The Calculated to Experimental (C/E) ratio for Cs-137 (test average C/E 1.00 (±0.03)), indicates that its behavior was captured very well by simulation and is a reliable link between measured activity and burnup.

The simulated activity of Cs-134 also closely matches the measured activities with a test average C/E of 1.0190 (± 0.0506) for all the compacts. The test average C/E for Eu-154 is 1.1835 (± 0.0525), indicating a bias between the simulation and the measurement. The simulated activities of Ce-144 also agree well with PGS measurements. The average C/E for Ce-144 as measured by Pr-144 activity is 0.9939 (± 0.0367). The measured Ce-144 activity from its own gamma is excluded in favor of the activity measured from its daughter Pr-144. Pr-144 is the daughter of the longer lived Ce-144 and is in secular equilibrium with Ce-144. Cerium-144 has a low energy gamma-ray (133.5 keV) that is difficult to detect and properly convert into an activity, but Pr-144 produces two easily detectable gamma-rays.

Fig. 5. Typical spectrum collected from an AGR-1 compact (Compact 4-2-2) with major peaks labeled

Fig. 6. Calculated to Experimental (C/E) activity ratio for several different fission products for each fuel compact in the AGR-1 test train. Fuel compacts are plotted by vertical position in the AGR-1 test train, with the top of the test train at the left and the bottom at the right. There are three compacts at each axial location.

Table 2. Decay corrected, measured activity in each AGR-1 compact for the cesium fission products

Compact ID	Cs-137 Ci	Cs-134 Ci	Compact ID	Cs-137 Ci	Cs-134 Ci	Compact ID	Cs-137 Ci	Cs-134 Ci
AGR-1 643	0.37	0.27	AGR-1 543	0.48	0.53	AGR-1 443	0.55	0.75
AGR-1 633	0.38	0.30	AGR-1 533	0.48	0.54	AGR-1 433	0.54	0.74
AGR-1 623	0.42	0.36	AGR-1 523	0.49	0.58	AGR-1 423	0.55	0.76
AGR-1 613	0.45	0.45	AGR-1 513	0.52	0.67	AGR-1 413	0.55	0.77
AGR-1 642	0.29	0.17	AGR-1 542	0.40	0.37	AGR-1 442	0.47	0.55
AGR-1 632	0.30	0.19	AGR-1 532	0.40	0.36	AGR-1 432	0.46	0.52
AGR-1 622	0.34	0.24	AGR-1 522	0.41	0.39	AGR-1 422	0.45	0.52
AGR-1 612	0.38	0.32	AGR-1 512	0.44	0.48	AGR-1 412	0.47	0.58
AGR-1 641	0.36	0.26	AGR-1 541	0.47	0.51	AGR-1 441	0.49	0.66
AGR-1 631	0.38	0.29	AGR-1 531	0.47	0.52	AGR-1 431	0.48	0.62
AGR-1 621	0.41	0.35	AGR-1 521	0.48	0.56	AGR-1 421	0.48	0.64
AGR-1 611	0.45	0.44	AGR-1 511	0.51	0.64	AGR-1 411	0.51	0.75
Compact ID	Cs-137 Ci	Cs-134 Ci	Compact ID	Cs-137 Ci	Cs-134 Ci	Compact ID	Cs-137 Ci	Cs-134 Ci
AGR-1 343	0.55	0.81	AGR-1 243	0.53	0.76	AGR-1 143	0.46	0.58
AGR-1 333	0.53	0.76	AGR-1 233	0.52	0.70	AGR-1 133	0.46	0.51
AGR-1 323	0.56	0.80	AGR-1 223	0.50	0.66	AGR-1 123	0.44	0.47
AGR-1 313	0.55	0.81	AGR-1 213	0.51	0.69	AGR-1 113	0.44	0.47
AGR-1 342	0.47	0.60	AGR-1 242	0.45	0.53	AGR-1 142	0.42	0.44
AGR-1 332	0.45	0.54	AGR-1 232	0.47	0.52	AGR-1 132	0.39	0.37
AGR-1 322	0.44	0.52	AGR-1 222	0.43	0.46	AGR-1 122	0.37	0.33
AGR-1 312	0.45	0.57	AGR-1 212	0.45	0.50	AGR-1 112	0.38	0.33
AGR-1 341	0.50	0.71	AGR-1 241	0.51	0.69	AGR-1 141	0.47	0.55
AGR-1 331	0.52	0.71	AGR-1 231	0.50	0.63	AGR-1 131	0.45	0.47
AGR-1 321	0.51	0.69	AGR-1 221	0.49	0.61	AGR-1 121	0.43	0.43
AGR-1 311	0.51	0.72	AGR-1 211	0.50	0.63	AGR-1 111	0.43	0.43

3.2 Burnup Evaluation from Gamma Spectrometry

To convert a measured fission product activity into a burnup value, a relationship between the activity of an isotope or the ratio between activities of specific isotopes and the burnup must be established through calculation or simulation. Burnup assessment by both the direct activity measurement method and the activity ratio method were explored in this work. The absolute activity needed in the direct method requires careful evaluation of the efficiency of the detector system. The activity ratio method requires only a relative detector system efficiency calibration which is much easier to perform. However, for high burnup experiments, such as AGR-1, the inventory of a fission product is complicated by the depletion of U-235, the breeding of Pu-239, and changes in the neutron energy spectrum as burnup increases. These same complicating factors also render the use of shielded fission product-to-direct fission product activity ratios difficult. (Hsue et al., 1978) The production of shielded fission products relies on a neutron capture reaction taking place in a previously produced fission product. For a system greater than 5% FIMA, the uncertainty of shielded fission product production makes development of a function to determine burnup as a ratio of a shielded fission product (e.g. Cs-134 or Eu-154) and a direct fission product (e.g. Cs-137, Sb-125) in high burnup systems problematic. However, the simulations (Sterbentz, 2011) appear to have

faithfully reproduced the end of irradiation activities for several isotopes including Cs-134, making it is possible to use the relationship between the measured Cs-134 to Cs-137 ratio as an accurate indicator of burnup.

A usable fission product for burnup analysis needs to increase monotonically in activity during the entire course of an irradiation. As stated in Section 2.2, 11 fission products were consistently detectable in the PGS scans. During the course of the AGR-1 irradiation of 620 effective full power days that spanned nearly 3 calendar years, several of these isotopes (e.g. Y-91, Zr-95) achieved an equilibrium activity during the irradiation, rendering them unsuitable for burnup measurements. Of all the fission products detected in the PGS scans, Cs-137 is the optimum direct burnup indicator isotope for several reasons. It is a direct fission product, meaning its production in the fuel is roughly proportional to neutron fluence which is proportional to burnup. It is sufficiently long-lived ($T_{1/2}=30.1$ years) to keep increasing in activity linearly with burnup throughout the course of the long AGR-1 irradiation. It does not have a significant neutron absorption cross section that would complicate the simulation of its inventory. It also has a similar fission yield between U-235 and Pu-239 which also reduces the complexity of simulation for this isotope.

Detailed production/depletion calculations of the AGR-1 experiment were performed in which all of the as-run reactor power conditions were considered (Sterbentz, 2011). The results of this simulation can be used to link the measured compact activities to burnup. The simulation coupled Monte Carlo neutron transport in MCNP5 (X-5 Monte Carlo Team, 2003) with depletion calculations in ORIGEN2.2 (Croff, 1980) using the INL developed code JMOCUP (Jim's MCNP-ORIGEN2 Coupled Utility Program; Sterbentz, 2000). JMOCUP uses data from MCNP to calculate one-group cross sections for use in ORIGEN depletion calculations. In this simulation each fuel compact was divided perpendicular to the axial center line into two calculation cells for improved inventory resolution predictions. The results of this simulation included actinide inventories and fission product inventories. The actinide inventories were used to calculate burnup in FIMA using Equation 1.

$$BU = \frac{N_{U-235,i} + N_{U-238,i} - N_{U-235,f} - N_{U-238,f} - N_{Act,f}}{N_{U-235,i} + N_{U-238,i}} \quad (1)$$

Where $N_{U-235,i}$ is the initial number of U-235 atoms, $N_{U-238,i}$ is the initial number of U-238 atoms, $N_{U-235,f}$ is the end-of-irradiation number of U-235 atoms, $N_{U-238,f}$ is the end-of-irradiation number of U-238 atoms, and $N_{Act,f}$ is the final number of actinide atoms created by neutron reactions with U-235 and U-238, and includes other U isotopes (U-234 and U-236), Pu isotopes, Am isotopes, Cm isotopes etc. The initial values of U-234 (0.3% U total) and U-236 (324 ppm U total) were not considered in the burnup and were not present in quantities significant enough to impact the resulting burnup values. An advantage of calculating burnup in FIMA by Equation (1) rather than by atom percent of U-235 consumed is that it accounts for fission from Pu and other minor actinides. The FIMA burnup values are then directly comparable to the burnup values found by mass spectrometry. The calculation cell burnup values from Equation 1 were averaged for each fuel compact to get compact average burnups that are used for comparison to burnup determined from the measured compact fission product inventory.

Using the fission product inventory results of the simulation (Sterbentz, 2011), a relationship was developed between the final predicted Cs-137 activities of the different calculation cells and the burnup of each cell. A linear regression was fit to the data to create a function relating fuel compact averaged Cs-137 inventory to fuel burnup in % FIMA. This linear relationship is given by Equation 2.

$$BU = 1.020(A_{Cs-137}) - 0.8356 \quad (2)$$

Where BU is the burnup of the fuel in % FIMA, and A_{Cs-137} is the total compact activity of Cs-137 in GBq.

The activity-to-burnup relationship for Cs-137 can also be expressed through a series of analytical expressions set out by Freis et al. 2006 and repeated here in Equations (3) to (6). Equation (3) is the Bateman equation for Cs-137 where N_{Cs-137} is the number of Cs-137 atoms, λ_{Cs-137} is the decay constant of Cs-137, γ_{Cs-137} is the fission yield of Cs-137, $\bar{\sigma}_f$ is the spectrum averaged fission cross section for the system under consideration, $\bar{\phi}_n$ is the neutron flux, and N_{HM} is the number of heavy metal atoms in the system.

$$\frac{dN_{Cs-137}}{dt} = -\lambda_{Cs-137}N_{Cs-137} + \gamma_{Cs-137} \cdot \bar{\sigma}_f \bar{\phi}_n N_{HM} \quad (3)$$

Assuming no initial Cs-137 activity in the system, Equation (3) can be solved to find Equation (4) assuming the number of fissile atoms remains roughly constant in the system which is true for AGR-1 (Maki and Sterbentz, 2010). Using the same fissile atom assumption from Equation (4), an equation for burnup can be derived in the form of Equation (5). Equations (4) and (5) can be combined to get Equation (6), which directly relates burnup to Cs-137 activity.

$$N_{Cs-137}(t_{irr}) = \frac{\gamma_{Cs-137} \bar{\sigma}_f \bar{\phi}_n N_{HM}}{\lambda_{Cs-137}} \left(1 - e^{(-\lambda_{Cs-137} t_{irr})}\right) \quad (4)$$

$$BU = \frac{(\bar{\sigma}_f \bar{\phi}_n N_{HM}) t_{irr}}{N_{HM}} \quad (5)$$

$$BU = A_{Cs-137}(t_{irr}) \frac{t_{irr}}{\gamma_{Cs-137} N_{HM}} \left(1 - e^{(-\lambda_{Cs-137} t_{irr})}\right)^{-1} \quad (6)$$

The irradiation time in the AGR-1 experiment is short enough compared to the half-life of Cs-137 that Equation (6) can be simplified by Taylor series to a linear relationship between BU and Cs-137 activity. Using these equations, the burnup in %FIMA as a function of Cs-137 activity is shown in Equation (7) where the activity of Cs-137 is in GBq. The nuclear data ($\lambda_{Cs-137}=7.3021 \times 10^{-10} \text{ s}^{-1}$ $\gamma_{Cs-137}=0.06188$) in Equation (7) are from ENDFB-VII.1 (Chadwick, 2011), and the N_{HM} of 2.28×10^{21} is from Sterbentz (2011).

$$BU = \frac{A_{Cs-137}}{\lambda_{Cs-137} \gamma_{Cs-137} N_{HM}} = 0.9706(A_{Cs-137}) \quad (7)$$

This burnup relationship compares favorably with the relationship derived from the coupled Monte Carlo / ORIGEN calculations shown in Equation (2). This example shows Cs-137 to be a robust burnup indicator. The simple analytical solutions to the relationship between Cs-137 and burnup were also shown to match other sophisticated simulations for different fuel systems. (Freis et al., 2006)

A relationship between burnup and the ratio of the activities of Cs-134 and Cs-137 can also be derived from the results of the coupled MCNP / ORIGEN calculations. Only certain fission product inventories change as a function of burnup. For example, a ratio of Cs-137 and Ce-144 would not change with burnup since the inventory of both fission products increases linearly with neutron fluence for irradiation times that are small compared to their half life. Shielded fission products like Cs-134 increase quadratically with neutron fluence for irradiation times that are small compared to their half life. Therefore, the ratio of a shielded-to-direct fission product will still increase linearly with neutron fluence and burnup under the proper conditions. By using a similar technique to the derivation of Equation (2), a linear regression was fit to the calculated compact-averaged activity ratio of Cs-134 to Cs-137 values taken from (Sterbentz, 2011). This relationship between activity ratio and burnup for the AGR-1 experiment is shown in the equation below, where BU is burnup in % FIMA, A_{Cs-134} is the activity of Cs-134 and A_{Cs-137} is the activity of Cs-137. Because the relationships in Equations (2) and (8) were derived from end of irradiation values, they are only applicable to measured activities from fuel with a burnup that is near the burnup range of the simulations (approximately 10-20 % FIMA).

$$BU = 10.4239 \left(\frac{A_{Cs-134}}{A_{Cs-137}} \right) + 4.5053 \quad (8)$$

The compact-averaged burnup for each AGR-1 compact can be estimated from the measured Cs-137 activities and the measured Cs-134/Cs-137 activity ratio. The results of the PGS scans were converted to a measured burnup value using Equations (2) and (8). The measured burnups using the activity of Cs-137 were then compared to the burnup calculated by simulation in (Sterbentz, 2011) in Fig. 7. Fig. 7 shows the calculated and measured burnup values for each compact in the AGR-1 test train from top (left) to bottom (right). As the irradiation capsules consisted of a triangular array of fuel compacts, there are three compacts at each of the 24 axial levels presented in Fig. 7. The burnup is higher in Stacks 1 and 3 since they were facing the core as is seen in Fig. 2. Note that the simulated burnups in Stacks 1 and 3 are nearly identical (data points for Stack 1 are hidden behind Stack 3 data). There were some measured burnup differences between Stack 1 and Stack 3 (that face the core) that is best seen in Capsules 1, 2, 3, and 4. In these capsules Stack 3 has a consistently higher measured burnup than the adjacent compact from Stack 1. This split may be due to power variations in the different ATR lobes and differences in the ATR outer shim control cylinder banks on either side of the AGR-1 test train. The power variations from outer shim control cylinder rotations were accounted for in the simulation but did not produce a variation in the two stacks as wide as was seen in the measurements. Stack 2 was shielded from the core by the other fuel compacts resulting in a lower measured burnup which was also predicted by simulation. The majority of uncertainty on the measured burnup is propagated from uncertainty on the efficiency calibration and uncertainty of the Eu-152 calibration sources. The relative uncertainty of the measured burnup values from these sources is approximately 5.3%, and is represented by the error bars in Fig. 7. The peak measured burnup is 20.1% FIMA for Compact 3-2-3, and the

minimum measured burnup is 10.1% FIMA for Compact 6-4-2. A plot similar to Fig. 7 using the Cs-134 to Cs-137 activity ratio is shown in Fig. 8. The relative uncertainty on the burnup values in Fig. 8 is approximately 3.1%, and is represented by the error bars in Fig. 8. The lower relative uncertainty results from the simpler efficiency calibration needed by the ratio technique. In the Fig. 8 data, the minimum measured burnup is 10.7% FIMA for Compact 6-4-2, and the maximum burnup is 20.0% FIMA for Compact 3-1-3. For reference in the simulations, the highest burnups were 19.56% FIMA for Compact 3-4-3 and 19.53% FIMA for Compact 3-1-3. The minimum burnup was 11.30% FIMA for Compact 6-4-2. The numeric burnup values for all 72 compacts by both techniques and by simulation can be found in (Harp, 2011).

Fig. 7. Comparison of burnup derived from Cs-137 measurements to burnup calculated from simulations (Sterbentz, 2011).

Fig. 8. Comparison of burnup derived from Cs-134/Cs-137 measured activity ratio to burnup calculated from simulations (Sterbentz, 2011).

Because of the random packing of particles in an AGR-1 compact, the precise starting inventory of fissile material in any particular 0.254-cm axial slice is not known. Therefore the burnup of each slice cannot be accurately determined by the absolute Cs-137 activity method. However, since the ratio method for burnup does not depend on any assumptions regarding the initial fissile inventory in the slice but only on the Cs-134/Cs-137 ratio, the burnup of each 0.254 cm slice can be determined by applying Equation (8), and therefore the axial burnup variation across the compact can be determined. The burnup data obtained in this manner for all 6 capsules are shown in Fig. 9. There is some uncertainty in activity per scanned axial slice at the edges of compacts due to the aperture effect. The derived efficiency accounts for aperture effects in the central region of the fuel compacts where there is a small contribution to the gamma signal from the portions of the fuel compact above and below the slice directly viewed by the collimator. This effect along with interference from background complicated the identification of the exact start and end of each compact in the scans and limits this techniques applicability at the compact edges. A systematic culling criterion was applied to the measurements used in the creation of Fig. 9 to eliminate spurious burnup estimates taken from the activity ratio. If the measured activity of Cs-137 or Cs-134 in a particular slice was 10 times lower than the average slice activity for that compact, the slice was not included in the burnup estimate plots. In Fig. 9, the space between each vertical grid line represents an axial compact level. The lower set of U-shaped profiles is from Stack 2, and the upper 2 profiles are from Stacks 1 and 3. Note that the Stack 1 and 3 burnup values overlap closely in Capsules 5 and 6 and tend to separate slightly in Capsules 1-4. The error bars in Fig. 9 represent the uncertainty burnup derived from uncertainty in the activity determination for each slice and are equivalent to the 3.1% shown in Fig. 8.

The data indicate that burnup could exhibit significant axial variation over the ~25 mm compact length, with the variation exceeding 2% FIMA in certain instances, particularly for compacts at the axial ends of a capsule. For example, Compact 5-1-2 has a minimum measured slice burnup of 15.0% FIMA and a maximum measured slice burnup of 17.2% FIMA. It is also interesting to note the burnup peaking phenomena at the axial ends of each capsule (Fig. 9). This peaking is due to capsule design. The borated graphite holders do not extend above or below the compact stacks and the graphite spacers above and below the compacts do not contain neutron absorbers (see Fig. 3). This design allows an increase of thermal neutron in-leakage from the core between capsules thus increasing the burnup at the axial ends of the capsules and creating the U-shaped axial burnup profiles. Overall, the compact-average burnups in the AGR-1 test train peaks near the middle of the ATR core (between Capsules 3 and 4) with a slight asymmetrical peak shift below midplane due to ATR hafnium safety rods placed 7.62 cm into the top of the active core above the AGR-1 irradiation test train (the capsule-average burnup is highest in Capsule 3).

The measured burnup values are compared to the results of the detailed inventory calculations from Sterbentz 2011 in Fig. 9. Neutronics and inventory were tracked over both calculation cells, so each compact has two sets of simulated burnup values, both of which are plotted in Fig. 9 for each compact. The data from gamma spectrometry and the simulations generally agree well over the entire experiment. There is some burnup disagreement at the top (left side of Fig. 9) of Capsule 6 (Stacks 1 and 3). The disagreement probably results from relatively stronger local thermal neutron absorption in the experiment relative to the simulation. While the ultimate cause is not known at this time, such stronger absorption could be due to a number of effects, including non-uniform loading of the boron-10 in the graphite holder, slight variation in the hafnium shroud thickness, ATR safety rods penetrating into the core slightly more than the designated 7.62 cm, or a combination of these effects. The previously mentioned consistently higher burnup in Stack 3 relative to Stack 1 can be seen in finer detail in Fig. 9 compared to Fig. 7 and Fig. 8. The higher Stack 3 burnup is attributed to the standard core flux tilt and slight variations in the outer shim control cylinder rotations on either side of the AGR-1 irradiation position in the ATR beryllium reflector.

Fig. 9. Burnup derived from the Cs-134 / Cs-137 activity ratio for each measured PGS slice for all capsules compared to AGR-1 burnup simulations. The spaces between the vertical tick marks represent separate levels (i.e. vertical compact locations) in each capsule.

3.3 Mass Spectrometry Burnup Method

The mass spectrometric data for the four AGR-1 compacts discussed in Section 2.5 were used to calculate the burnup for each compact. Burnup was calculated using the measured mass of a specific fission product in the fuel, the cumulative fission yield of that specific fission product, and the total mass of actinides present in the sample. This method is sometimes referred to as the "Fission Product Monitor - Residual Heavy Atom" technique. (Maeck et al., 1973) Ideally, the fission products used in the calculations should have a small neutron absorption cross section, a high cumulative fission yield, and a similar fission yield for that nuclear isobar between U and Pu fission. Chemically the fission product must also readily dissolve during the leaching process. This technique uses the following formula to calculate burnup based on a specific fission product detected in the ICP-MS spectrum.

$$BU = \frac{\left(\frac{N_{fp}}{y_{fp}} \right)}{\left(\left(\frac{N_{fp}}{y_{fp}} \right) + N_{Act} \right)} \times 100 \quad (9)$$

Where N_{fp} is number of atoms of a specific fission product fp measured in the sample, y_{fp} is the cumulative fission yield of fission product fp , and N_{Act} is the number of atoms of actinides in the sample. All fission yields were taken from ENDF/B-VII.1 (Chadwick, 2011). In the AGR-1 test, enough fission occurred in actinides other than

U-235 that the burnup would be biased if only the U-235 cumulative fission yield was used in Equation (9). To account for the different fission yields of actinides like Pu-239, the results of detailed simulations of the AGR-1 experiment (Sterbentz, 2011; Maki and Sterbentz, 2010) were used to create effective cumulative fission product yields. These effective yields are a weighted average of the different fission product yields based on the relative amount of fission that occurred in different actinides over the course of the experiment. The correction due to effective fission product yields can be as much as 5% for some isotopes which is on the order of the uncertainty in the measurements.

There are six isotopes that work reliably for the ICP-MS technique in the AGR-1 fuel: La-139, Ce-140, Ce-142, Pr-141, Nd-145, and Nd-146. These isotopes occur on the higher atomic number peak of the bimodal fission product distribution. They are all lanthanides that will readily oxidize during the oxidation step and readily dissolve in nitric acid. The differences between U-235 yield and Pu-239 yield are fairly small for these isotopes as well. All these isotopes are nonradioactive and have relatively small neutron absorption cross section with the exception of Nd-145. Because of its cross section, the number of Nd-145 and Nd-146 atoms in the samples and their respective yields are summed in the calculation of burnup. In this calculation, the burnup measurement for each sample of particles was found by taking the average of the individual isotope results from Equation (9) for La-139, Ce-140, Ce-142, Pr-141, and the Nd-145 + Nd-146 combined result. The burnup for a compact was then determined from the average of a set of burnup measurements from the separate particle samples.

For Compact 6-3-2, the burnup was derived from the three sets of particles that were oxidized at 400°C and leached in nitric acid. The burnup for Compact 3-2-1 was derived from the three sets of particles that were oxidized at 750°C and leached in nitric acid. The three sets of particles that were oxidized at 750°C and leached in nitric and hydrofluoric acid were used for burnup determination of Compact 5-3-1. Three burnup values for the three different methods from Compact 1-3-1 were 15.6 % FIMA for the 400°C oxidation nitric acid leach method, 16.2 % FIMA for the 400°C oxidation nitric and hydrofluoric acid leach method, and 16.3% FIMA for the 750°C oxidation nitric and hydrofluoric acid leach method. All three of these burnup evaluations are within the experimental relative uncertainty of 5%. Based on these results there appears to be no statistical difference in burnup derived from the three methods. However it is anticipated that the 750°C oxidation nitric and hydrofluoric acid leach method will be used when preparing burnup solutions for future irradiations in the AGR series of experiments. For this reason, 16.3 % FIMA was used as the Compact 5-3-1 burnup.

Burnup values for each compact evaluated by mass spectrometry, both gamma spectrometry techniques and simulation are shown in Table 3. The agreement between the three experimental measurements and the simulation is quite good. For all four compacts, the maximum range in the four burnup values is approximately 6% or less. The agreement between the mass spectrometric values and those derived from gamma spectrometry for these four compacts provides a level of confidence in the gamma spectrometry based measurements on the remaining compacts in the AGR-1 experiment. In addition, the agreement between all experimental values and the simulations validates the AGR-1 physics model.

Table 3. Mass spectrometry burnup data compared to other techniques in % FIMA.

Compact	Mass Spectrometry	Gamma Spectrometry Direct	Gamma Spectrometry Ratio	Simulation
6-3-2	10.7(±0.5) %	10.7(±0.5) %	11.0(±0.3) %	11.31%
3-2-1	19.3(±1.0) %	18.2(±0.9) %	18.6(±0.6) %	18.98%
5-3-1	16.3(±0.8) %	16.9(±0.8) %	15.9(±0.5) %	16.88%
1-3-1	16.3(±0.8) %	16.0(±0.8) %	15.6(±0.5) %	15.98%

4 Conclusions

The burnup of fuel compacts from the AGR-1 experiment has been determined experimentally using two different methodologies: non-destructive gamma spectrometry and destructive mass spectrometry. Detailed gamma scans of all 72 compacts from the AGR-1 irradiation test were used to determine the total activity of different fission products in each compact. The measured inventory of Cs-137 and Cs-134 compared well the calculated inventories from a detailed physics-depletion simulation of the AGR-1 experiment. The total measured compact inventory of the direct fission product Cs-137 was used to calculate the compact average burnup. Additionally, the measured activity ratio of the shielded fission product Cs-134 to the direct fission product Cs-137 was also used to determine the compact average burnup and the local burnup for each 0.254 cm axial compact slice. Both of these burnup

measurements compared well to the predicted burnup from simulations. The highest measured burnup was 20.1 %FIMA for the direct method and 20.0 %FIMA for the ratio method (compared to 19.56% FIMA from simulations). Four compacts that spanned the burnup range in the AGR-1 experiment were then analyzed for burnup by dissolving fuel kernels and measuring isotopic inventory in the solutions with mass spectrometry. These compacts had a burnup range measured by mass spectrometry of 19.3 % FIMA to 10.7 % FIMA. The mass spectrometry evaluation of burnup for the four compacts agreed well with the gamma spectrometry burnup evaluations and the expected burnup from simulation. The results confirm the accuracy of the nondestructive burnup evaluation from gamma spectrometry for TRISO fuel compacts across a burnup range of approximately 10 to 20 % FIMA and also validate the approach used in the physics simulation of the AGR-1 experiment.

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