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Safety Testing of AGR-2 UO_2 Compacts 3-3-2 and 3-4-2

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September 2015

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Fusion and Materials for Nuclear Systems Division

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ACRONYMS

AGR	Advanced Gas Reactor (Fuel Development and Qualification Program)
AGR-1	First AGR program irradiation experiment
AGR-2	Second AGR program irradiation experiment
BWXT	BWX Technologies
CCCTF	Core Conduction Cooldown Test Facility
CO	Carbon monoxide
DLBL	Deconsolidation leach-burn-leach
EDS	Energy-dispersive spectroscopy
FACS	Fuel Accident Condition Simulator
FIMA	Fissions per initial metal atom
HTGR	High Temperature Gas-cooled Reactor
IMGA	Irradiated Microsphere Gamma Analyzer
INL	Idaho National Laboratory
LEU	Low-enriched uranium
OPyC	Outer pyrolytic carbon (TRISO layer)
ORNL	Oak Ridge National Laboratory
PIE	Post-irradiation examination
SEM	Scanning-electron microscope
SiC	Silicon carbide (TRISO layer)
TAVA	Time-averaged/volume-averaged temperature
TRISO	Tristructural-isotropic (coated particles)
UCO	Uranium carbide/uranium oxide mixture (kernels)
UO2	Uranium dioxide (fuel kernels)

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1. INTRODUCTION

Post-irradiation examination (PIE) is in progress on tristructural-isotropic (TRISO) coated-particle fuel compacts from the Advanced Gas Reactor (AGR) Fuel Development and Qualification Program second irradiation experiment (AGR-2) [Collin 2014]. The AGR-2 PIE will build upon new information and understanding acquired throughout the recently-concluded six-year AGR-1 PIE campaign [Demkowicz et al. 2015] and establish a database for the different AGR-2 fuel designs. The AGR-2 irradiation experiment included TRISO fuel particles coated at BWX Technologies (BWXT) with a 150-mm-diameter engineering-scale coater. Two BWXT coating batches were tested in AGR-2; Batch 93085 had 508-µm-diameter uranium dioxide (UO_2) kernels and Batch 93073 had 427-µm-diameter kernels of uranium carbide mixed with uranium oxide (UCO). Data on these coating batches have been compiled [Barnes and Marshall 2009] and compared to AGR-1 UCO TRISO [Phillips, Barnes, and Hunn 2010]. AGR-2 TRISO coatings were most like the AGR-1 Variant 3 TRISO deposited in the 50-mm-diameter ORNL lab-scale coater [Hunn and Lowden 2006], where argon-dilution of the hydrogen and methyl-trichlorosilane coating gas mixture employed to deposit the SiC layer was used in both cases to produce a similar fine-grained, equiaxed SiC microstructure. In addition to the fact that AGR-1 fuel had smaller, 350-µm-diameter UCO kernels, notable differences in the TRISO particle properties included the pyrocarbon anisotropy, which was slightly higher in the particles coated in the 150-mm-diameter coater, and the exposed kernel defect fraction, which was higher in AGR-2 due to the detected presence of particles with impact damage introduced during TRISO particle handling. Both batches of BWXT AGR-2 fuel particles were compacted at ORNL with the same resinated graphite blend used for AGR-1 and a modified pressing process that incorporated a die heated to 65°C and a new computer-controlled servo-press [Hunn, Montgomery, and Pappano 2010a; 2010b]. Compared to AGR-1, the modified AGR-2 compacting process produced compacts with greater accuracy, greater reproducibility, and higher matrix density (1.6–1.7 g/cc for AGR-2 versus 1.2–1.3 g/cc for AGR-1). Compilations of the properties data for the particles and compacts are available in the AGR-1 [Hunn, Savage, and Silva 2012] and AGR-2 [Hunn, Savage, and Silva 2010] pre-irradiation characterization summary reports.

The *AGR-2 Post-Irradiation Examination Plan* [Demokowicz 2013] includes safety testing of the irradiated compacts in the Oak Ridge National Laboratory (ORNL) Core Conduction Cooldown Test Facility (CCCTF) to evaluate the effect of elevated temperature on the fuel microstructure and fission product[*] retention. The safety tests involve heating compacts to maximum temperatures of 1600–1800°C for typically 300 hours, where 1600°C is the expected maximum temperature during a high-temperature gas-cooled reactor (HTGR) depressurization conduction-cooldown event while 1700°C and 1800°C tests explore the safety margin and provide additional data on fission product interaction with the TRISO coatings. Seventeen safety tests were completed in the ORNL CCCTF and the Idaho National Laboratory (INL) Fuel Accident Condition Simulator (FACS) using irradiated AGR-1 fuel; results of these tests are summarized in the *AGR-1 PIE Final Report* and in numerous individual summary reports of the individual safety tests referenced in that report [Demkowicz et al. 2015, tables 16 and 17]. The first two AGR-2 safety tests were performed on AGR-2 UO_2 TRISO Compacts 3-3-2 and 3-4-2; both were heated to 1600°C in flowing helium for 300 hours.

Table 1 shows the AGR-2 UO_2 Compact 3-3-2 and Compact 3-4-2 calculated burnup in percent fissions per initial metal atom (FIMA), the calculated fast fluence (for neutron energies > 0.18 MeV), and the average calculated compact temperature during irradiation. The irradiation doses for these compacts were nearly identical, and Compact 3-4-2, located at the upper end of the capsule, was irradiated at a slightly lower average temperature.

[*] In this report, the term "fission product" is used in a general sense to refer to all the post-fission isotopes remaining at the end of the irradiation test. These include: isotopes directly generated by the fission process, isotopes generated by neutron activation, isotopes generated by radioactive decay, and residual uranium.

Table 1. Irradiation conditions for AGR-2 Compacts 3-3-2 and 3-4-2

Compact ID [a]	Fabrication ID [b]	Fuel Type	Average Burnup [c] (%FIMA)	Fast Fluence [c] (n/m^2)	Temperature [d] (°C)
AGR-2 3-3-2	LEU11-OP2-Z034	UO_2	10.54	3.53×10^{25}	1062
AGR-2 3-4-2	LEU11-OP2-Z150	UO_2	10.69	3.50×10^{25}	1013

[a] The X-Y-Z compact identification (ID) convention denotes the location in the irradiation test train: Capsule-Level-Stack.
[b] Physical properties data for individual compacts are available and tabulated based on fabrication ID [Hunn, Montgomery, and Pappano 2010b, pages 73–82].
[c] Burnup [Sterbentz 2014, table 6] and fast fluence [Sterbentz 2014, table 12] are based on physics calculations.
[d] Time-averaged, volume-averaged (TAVA) irradiation temperature [Hawkes 2014, table 4] is based on thermal calculations.

Compacts 3-3-2 and 3-4-2 were heated to 1600°C in the CCCTF furnace with the same methods used for AGR-1 safety testing [Baldwin et al. 2012]. The CCCTF has a water-cooled deposition cup to collect vaporized metallic elements that escape from the compact and surrounding graphite holder. Deposition cups were periodically removed and replaced with a new cup, with a maximum exchange interval of 24 h and shorter exchange intervals following heat-up, when silver release rate was expected to be higher. Gamma-emissions from the deposition cups were measured to monitor the safety test progress, with particular emphasis on collected cesium inventory that would indicate SiC failure [Hunn et al. 2014a]. Gaseous fission products were collected from the helium sweep gas as it passed through a liquid nitrogen-cooled charcoal-filled trap that was monitored for ^{85}Kr because significant and rapid krypton release would indicate complete failure of a TRISO coating [Morris et al. 2014]. After completion of each safety test, additional analysis was performed to measure the inventory of fission products on the deposition cups and other CCCTF furnace internals (graphite fuel holder, tantalum furnace liner, and tantalum gas inlet line). This allowed for the determination of an average deposition cup collection efficiency that could be used to adjust the time-dependent deposition cup data to estimate the time-dependent fission product release from the compact.

2. RESULTS OF SAFETY TESTING

Figure 1 and Figure 2 summarize the overall estimated time-dependent fission product release from Compacts 3-3-2 and 3-4-2 during the two 1600°C safety tests. The plotted data points represent the measured amount of an isotope collected on each deposition cup divided by the calculated inventory of that isotope in the compact as a result of the irradiation and adjusted for the deposition cup collection efficiency by dividing by the cumulative fraction collected on all the cups of the amount of that isotope released from the compact over the entire safety test (Table 2 and Table 3). Note that the cumulative fractions of ^{90}Sr and ^{154}Eu collected by the cups were extremely low. This resulted in a large multiplier in the adjustment from measured cup collection data to estimated compact release fraction. It can be assumed that there is a large uncertainty in the reported time-dependent fractional release for these two isotopes stemming from both the large multiplier and the unlikelihood of a constant collection efficiency over the entire test. Europium collection on the cups was so small that the low-energy gamma ^{155}Eu isotope, which is normally reported in these tests, could not be measured above the Compton continuum. The low collection efficiency for europium and strontium was not due to any failure in the CCCTF operation but was rather due to the very low release of these elements from the compacts coupled with the high retention in the graphite holder. The CCCTF graphite holder surrounds the compact during safety testing to simulate the graphite block that surrounds the compacts in a prismatic HTGR. Europium and strontium release results for these two tests were almost completely dominated by the retention in the graphite holder.

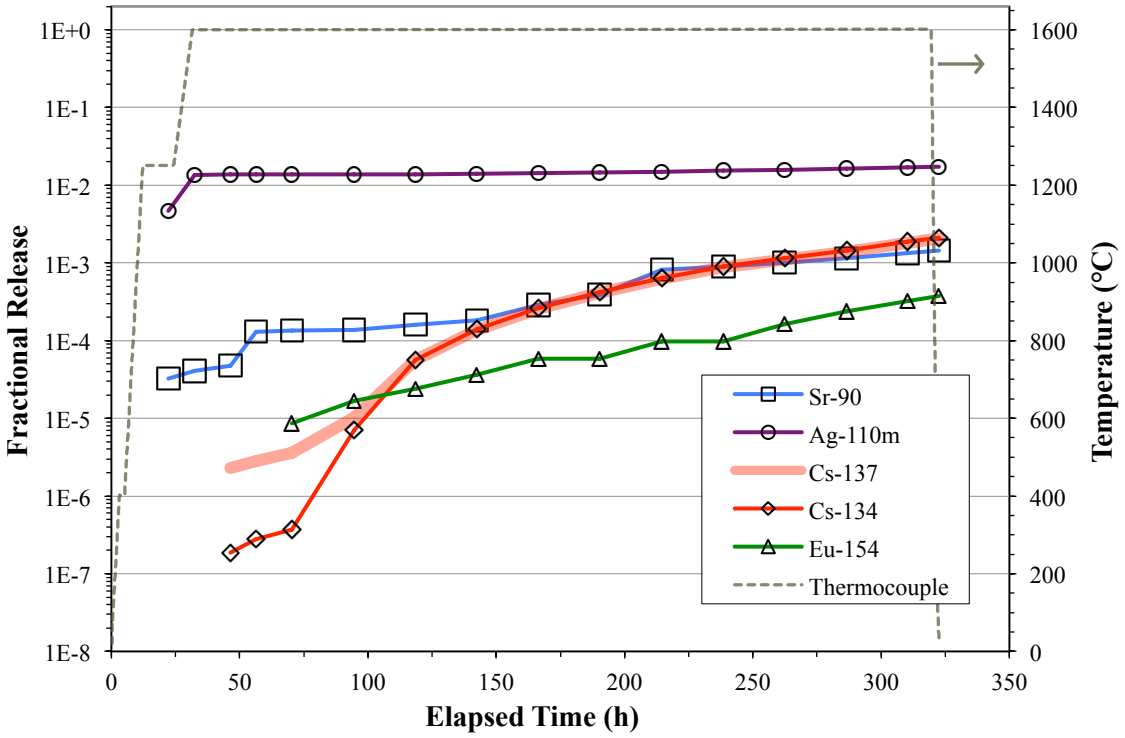

Figure 1. Release of fission products from Compact 3-3-2 during safety testing to 1600°C.

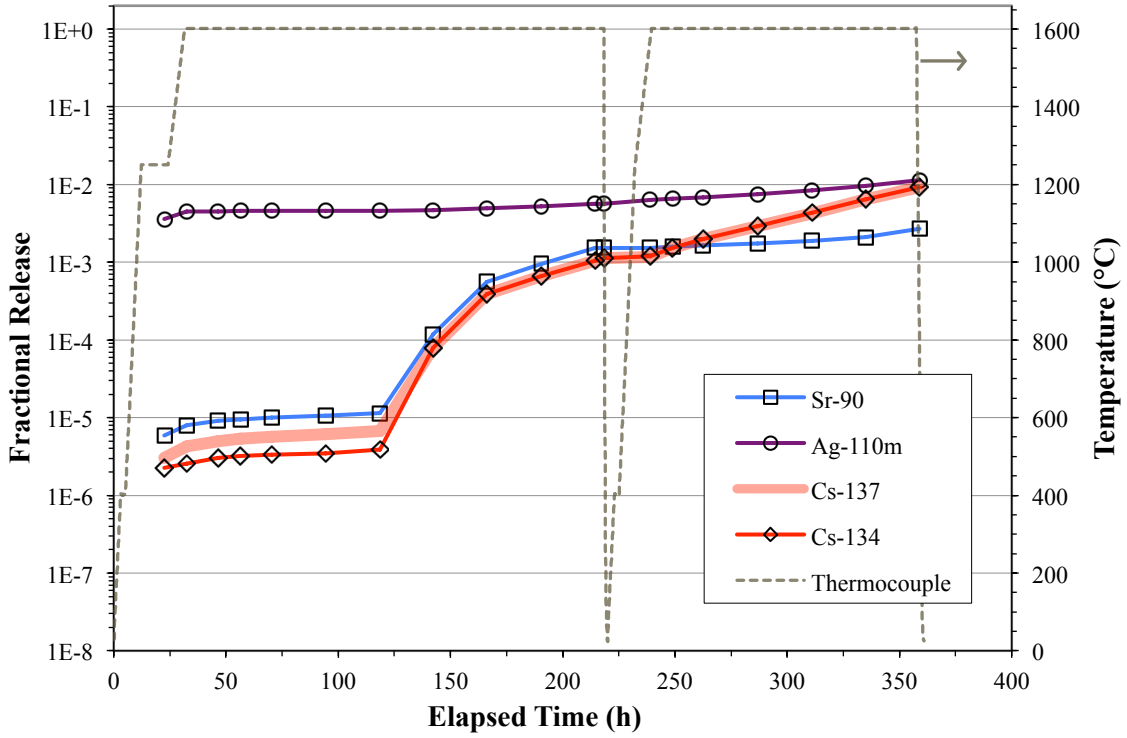

Figure 2. Release of fission products from Compact 3-4-2 during safety testing to 1600°C.

Table 2. Distribution of radioactive isotopes detected on the CCCTF furnace internal components after the Compact 3-3-2 safety test

Component	^{90}Sr	^{110m}Ag	^{134}Cs	^{137}Cs	^{154}Eu
Deposition cups	2.8%	32.2%	19.9%	19.7%	1.5%
Tantalum parts	13.5%	67.6%	79.7%	79.6%	1.6%
Graphite holder	83.7%	0.2%	0.5%	0.6%	96.9%

Table 3. Distribution of radioactive isotopes detected on the CCCTF furnace internal components after the Compact 3-4-2 safety test

Component	^{90}Sr	^{110m}Ag	^{134}Cs	^{137}Cs	^{154}Eu
Deposition cups	4.2%	35.7%	18.9%	18.7%	0.1%
Tantalum parts	0.1%	64.3%	80.2%	80.2%	2.5%
Graphite holder	95.7%	0%	0.9%	1.1%	97.4%

In contrast, silver and cesium are not expected to be retained by graphite at 1600°C, and the relative fraction of these elements detected in the graphite holder was negligible. However, more silver and cesium were deposited on the Ta gas line near the top of the furnace than were collected on the water-cooled cups. Therefore, the uncertainty in the time-dependent release for these isotopes is also elevated by a relatively-low collection efficiency, albeit not nearly as much as for the europium and strontium results. Abnormally-low collection efficiency for silver and cesium has been a persistent issue over the last several safety tests and is thought to be related to deposition cup cooling. Several corrective measures have failed to return the system to the higher collection efficiencies observed for the first eleven AGR-1 safety tests (which averaged 99% for ^{110m}Ag, 80% for low ^{134}Cs release, and 96% for higher Cs release from particles with failed SiC). After the conclusion of the Compact 3-4-2 safety test, additional diagnostic tests were conducted. A deficiency was identified in the motor used to remotely exchange the deposition cups that may have reduced the tension holding the cup in contact with the water-cooled cold-finger, thus impeding heat transfer and resulting in a higher than desired cup-surface temperature. This motor has since been replaced.

The temperature profile for each safety test is shown as a dashed line in Figure 1 and Figure 2. At the beginning of each test, the compact was held for 2 h at 400°C to allow for outgassing and then held again at 1250°C for 12 h before gently ramping at 50°C/h to the final test temperature of 1600°C. The first cup was taken at the end of the 1250°C bake and the second ~1 h after reaching 1600°C. The Compact 3-4-2 safety test was shut down after 187 h at 1600°C due to a blockage in the cooling-water line. After replacing the clogged line, the compact was reheated to 1600°C using the same ramp rates but omitting the 1250°C hold.

Figure 1 shows that most of the ^{110m}Ag released from Compact 3-3-2 was detected on the cups removed at the end of the 1250°C hold and 1 h after reaching 1600°C. This is consistent with the general silver release behavior observed during AGR-1 safety testing at 1600°C and the conclusion that residual silver in the matrix and outer pyrolytic carbon (OPyC), previously released through intact SiC during irradiation, is quickly released during initial heatup and dominates the silver release during 1600°C safety testing [Morris et al. 2014]. Compact 3-4-2 showed a similar peak in the fractional release rate of ^{110m}Ag followed by a drop of more than two orders of magnitude (Figure 3 and Figure 4). A smaller peak in the release rate was also observed when Compact 3-4-2 was reheated midway through the test; this has been

observed during AGR-1 safety testing and appears to be related to additional silver release through intact SiC that occurs at temperatures between 1075°C and 1375°C [Hunn et al. 2015].

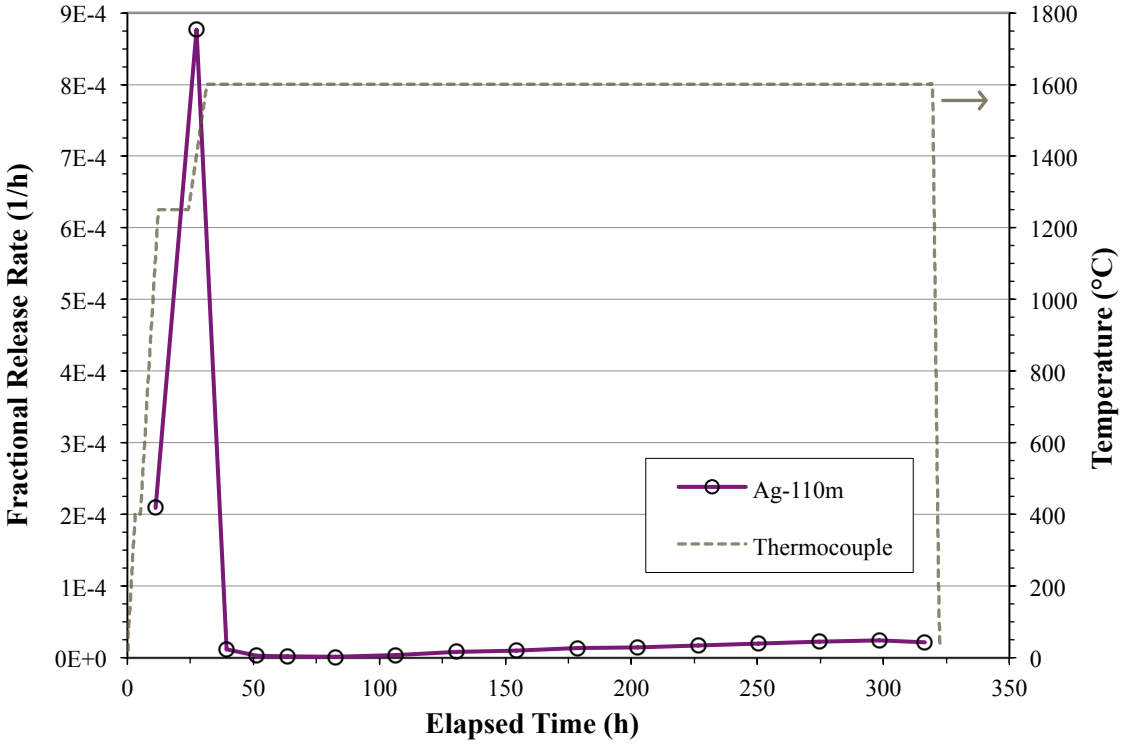

Figure 3. Rate of silver release from Compact 3-3-2 during safety testing to 1600°C.

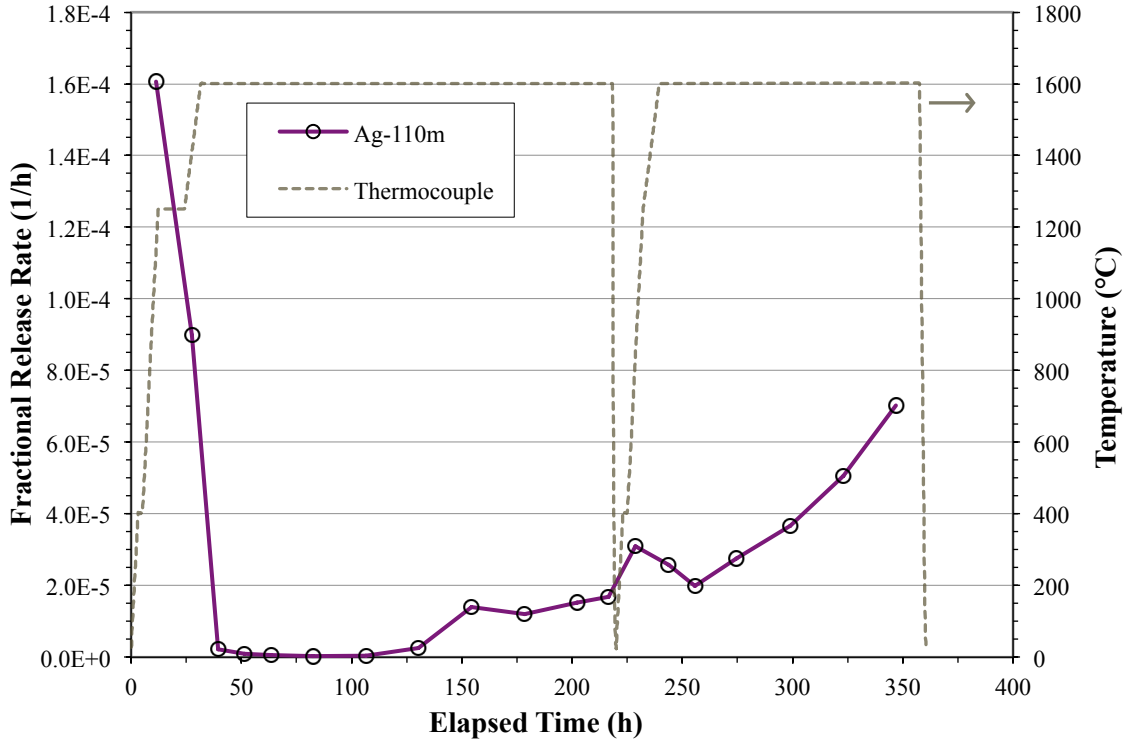

Figure 4. Rate of silver release from Compact 3-4-2 during safety testing to 1600°C.

Figure 3 and Figure 4 show that, after the initial drop in ^{110}Ag release rate, the rate slowly increased throughout the remainder of the safety tests. The amount of additional ^{110}Ag that was released during this period was equivalent to ~6 particles' inventory for Compact 3-3-2 and ~11 particles' inventory for Compact 3-4-2. This amount of release is consistent with the assumption that it may have been mostly coming from particles whose SiC layers failed during the safety test. Evidence for these SiC failures is provided by the cesium release shown in Figure 1 and Figure 2. Particles from Compact 3-3-2 have already been surveyed with the ORNL Irradiated Microsphere Gamma Analyzer (IMGA) and six low-cesium particles were identified that account for the cesium release in the CCCTF. The cesium release from Compact 3-4-2 suggests that at least 14 particles experienced SiC failure and released cesium during that safety test, but the number could be higher than 20 based on the partial cesium retention observed in the low-cesium particles from Compact 3-3-2. Note that significant cesium release was not observed immediately upon heating to 1600°C. Significant cesium release from Compact 3-3-2 began after 39–63 h at 1600°C and after 87–111 h at 1600°C for Compact 3-4-2. Cesium release from Compact 3-4-2 also appears to have accelerated after the interruption and resumption of the test. The mechanism for the SiC failure in these AGR-2 UO_2 compacts has not yet been investigated and may not be the same as observed in AGR-1 UCO fuel, where failure fractions were significantly lower ($\leq 2.4\times10^{-4}$ at 95% confidence [Demkowicz et al. 2015]). Low-cesium particles will be imaged with non-destructive three-dimensional x-ray tomography to examine the internal microstructure and then polished-cross sections will be prepared for optical and scanning electron microscopy (SEM), which will include elemental analysis of any fission product redistribution using energy-dispersive spectroscopy (EDS).

The cumulative safety-test release of cesium and the other primary radioisotopes is presented in Table 4. Unlike the time-dependent data, the deposition cup collection efficiency does not impact the uncertainty in the reported cumulative compact fractions. Europium-155 was only detected in the graphite holder, so no time-dependent data was available for that isotope, but it is included here and compares well with the cumulative measured release of ^{154}Eu, providing additional confidence in the europium release data. As was discussed for the slow release of ^{110m}Ag that was measured after the initial quick release from the matrix and OPyC, europium and strontium release may also be coming from the particles with failed SiC. Strontium-90 release, in-particular, seemed to closely track the cesium release (Figure 1 and Figure 2). No ^{85}Kr was measured in either safety test; Table 4 lists an upper bound for the ^{85}Kr release based on the minimum detection limit in the liquid nitrogen-cooled cold trap. Krypton release below this level typically indicates that no particles experienced TRISO-coating failure during safety testing.

Table 4. Cumulative releases of radioactive isotopes from AGR-2 UO_2 TRISO safety tests

Isotope	Compact 3-3-2		Compact 3-4-2	
	Compact fraction	Particle equivalent	Compact fraction	Particle equivalent
^{85}Kr	$<1\times10^{-6}$	$<2\times10^{-3}$	$<1\times10^{-6}$	$<2\times10^{-3}$
^{90}Sr	1.4×10^{-3}	2.2	2.7×10^{-3}	4.2
^{110m}Ag	1.7×10^{-2}	27	1.1×10^{-2}	17
^{134}Cs	2.1×10^{-3}	3.3	9.3×10^{-3}	14
^{137}Cs	2.1×10^{-3}	3.2	9.2×10^{-3}	14
^{154}Eu	3.8×10^{-4}	0.6	3.2×10^{-4}	0.5
^{155}Eu	4.3×10^{-4}	0.7	3.7×10^{-4}	0.6

3. CONCLUSION

Safety testing at 1600°C was completed on AGR-2 UO_2 Compacts 3-3-2 and 3-4-2. Silver release behavior was similar to that observed during 1600°C safety testing of AGR-1 UCO fuel; namely, early silver release was dominated by rapid release of silver held up in the matrix and OPyC from previous release through intact SiC during irradiation. The amount of ^{110m}Ag released from the AGR-2 UO_2 compacts during 1600°C safety testing was on the low-end of what was observed during AGR-1 safety testing [Demkowicz et al. 2015, figure 44]; this was probably due to a lower initial inventory in the matrix and OPyC. It is already known that most of the AGR-2 UO_2 compacts did not release much silver to the capsule components during irradiation [Harp 2015], and it is presumed that less silver was released through the SiC due to the lower burnup. In contrast, many of the safety-tested AGR-1 compacts released significant fractions of their calculated ^{110m}Ag inventory in the reactor [Harp 2014].

A slow release of additional ^{110m}Ag from Compacts 3-3-2 and 3-4-2 was observed after the initial rapid release of silver from the matrix and OPyC; this additional silver release may be predominantly release from particles whose SiC failed during safety testing. Based on the cesium released in the CCCTF, the SiC failure fraction for these two AGR-2 UO_2 compacts was significantly higher than what was observed during 1600°C safety testing of AGR-1 UCO fuel compacts. There were only three AGR-1 particles that exhibited cesium release due to SiC failure out of eight AGR-1 compacts safety tested at 1600°C (one in each of 3 compacts). Two of these were in compacts tested in the ORNL CCCTF, and subsequent particle survey with the IMGA successfully extracted the two failed-SiC particles for further analysis. X-ray tomography and materialographic examination showed that the SiC failure in both of these particles was related to as-fabricated defects [Hunn et al. 2012; 2014b]. All other AGR-1 failed-SiC particles subjected to microstructural analysis have shown that the failure was related to local palladium degradation of the SiC where the inner surface of the SiC had been exposed by an IPyC crack that had occurred due to interaction between the IPyC and shrinking buffer [Hunn et al. 2014a]. The fact that no SiC failure due to palladium degradation has been observed in AGR-1 compacts heated to 1600°C suggests that this failure mechanism is only significant at the higher 300-hour test temperatures of 1700°C and 1800°C or during longer-term irradiation testing.

Further PIE will be used to enumerate the actual number of failed-SiC particles in 1600°C-safety-tested AGR-2 UO_2 Compacts 3-3-2 and 3-4-2 and determine why the fuel showed poorer performance in this area. Given that the palladium-degradation mechanism did not produce significant SiC failure during 1600°C AGR-1 UCO compact safety testing, it seems unlikely that this mechanism was solely responsible for the elevated failure fraction observed in these two AGR-2 UO_2 compacts without some additional contributing factor related to the UO_2 kernel. One area to explore will be the impact of possible carbon monoxide (CO) corrosion, which can be significant in UO_2 TRISO [Minato et al. 1991] but is not an issue in UCO TRISO due to significantly reduced CO production in irradiated UCO kernels [Homan et al. 1977]. Higher CO production in the UO_2 TRISO particles may also increase the probability for internal pressure-related failure.

It is notable that no krypton release from TRISO failure was observed in conjunction with what appears to be numerous SiC failures in these two 1600°C safety-tested AGR-2 UO_2 compacts. A similar situation was observed in AGR-1 PIE, even during 1800°C safety testing where more particles exhibited cesium release from SiC failure (as high as 11 failed-SiC particles in AGR-1 Compact 3-2-3 [Hunn et al. 2014a]). In AGR-1 PIE, krypton retention in particles with failed SiC was credited to intact OPyC layers.

Europium-154 release from Compacts 3-3-2 and 3-4-2 was also on the low-end of what was observed during AGR-1 safety testing [Demkowicz et al. 2015, figure 47]. Europium can be expected to be retained more effectively in TRISO particles with 100% UO_2 kernels, compared to UCO TRISO, because the europium oxide phase is preferred over the carbide phase [Homan et al. 1977]. Strontium-90 release from

Compacts 3-3-2 and 3-4-2 was on the upper-end of what was observed during AGR-1 safety testing at 1600°C [Demkowicz et al. 2015, figure 43]. The higher strontium release from the UO_2 compacts appeared to be related to the high SiC failure fraction.

Post-safety testing PIE of AGR-2 UO_2 Compacts 3-3-2 and 3-4-2 will be presented in a future report. This will include results of the deconsolidation leach-burn-leach (DLBL) analysis for exposed fission products, results from short-counting time IMGA surveys performed on all the recovered TRISO particles, results from long-counting time IMGA measurements performed on specially-selected particles that exhibited significant cesium release or other unusual radioisotopic release, and similar IMGA measurements performed on 50–60 randomly-selected particles. Microstructural analysis using x-ray tomography and materialographic methods will be performed to investigate radiation-induced changes in the particles and elucidate the mechanisms responsible for observed fission product release.

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