



GAS-COOLED REACTOR
ADVANCED REACTOR TECHNOLOGIES PROGRAM

July 16, 2024

AGR-5/6/7 Safety Testing and Compact Destructive Exams at ORNL

William F. Cureton, PhD

TRISO PIE Specialist- Oak Ridge National Laboratory

DOE ART GCR Review Meeting

Hybrid Meeting at INL

July 16–18, 2024



ORNL Coauthors and Contributors

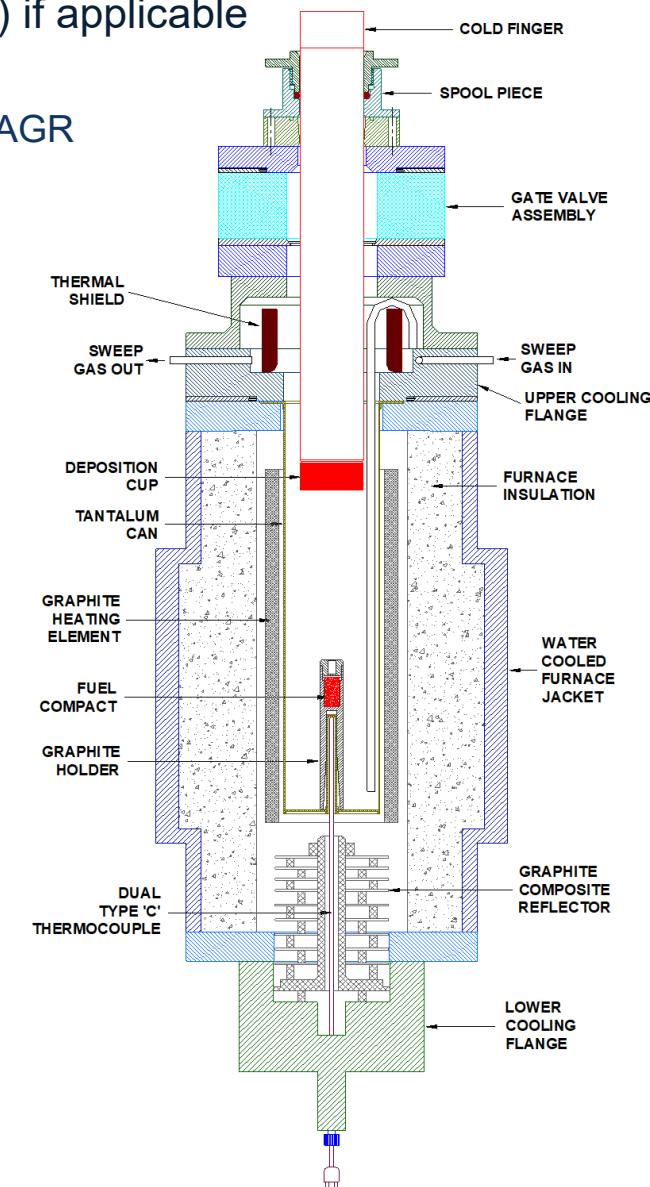
- John Hunn — ORNL Fuels Lead
- Tyler Gerczak — Electron microscopy and Furnace for Irradiated TRISO Testing (FITT)
- Will Cureton — Safety testing in the Core Conduction cooldown Test Facility (CCCTF)
- Stephen Trewitt — Safety testing in the CCCTF and particle heating in FITT
- Fred Montgomery — Deconsolidation Leach-Burn-Leach (DLBL) and burnup analysis
- Martino Hooghkirk — Irradiated Microsphere Gamma Analyzer (IMGA) and materialography
- Grant Helmreich — X-ray Computed Tomography (XCT)
- Jesse Werden — Electron microscopy
- Katherine Montoya — Electron microscopy, analysis of oxidation of FITT specimens
- Bob Morris (consulting) — IMGA, gamma scanning, and CCCTF systems
- Chuck Baldwin (consulting) — IMGA, gamma scanning, materialography, and CCCTF systems
- Irradiated Fuels Examination Laboratory (IFEL) hot cell operators
- Radioactive Materials Analytical Laboratory (RMAL) chemists and supporting staff



Standard ORNL PIE Process



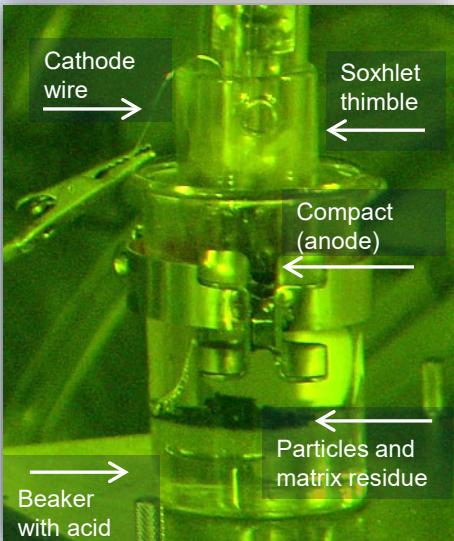
- Perform safety test in ORNL Core Conduction Cooldown Test Facility (CCCTF) if applicable
 - modernized version of the same in cell furnace used for TRISO fuel tests predating AGR
 - cylindrical fuel compact is placed in graphite holder
 - compact in holder positioned on thermocouple finger
 - copper-plated deposition cup screwed on water-cooled cold finger at top
 - compact heated in flowing helium up to 1800°C
 - ^{85}Kr release monitored in He exhaust
 - metallic fission products (FP) collected on cup
 - ^{90}Sr , $^{110\text{m}}\text{Ag}$, ^{134}Cs , ^{137}Cs , ^{154}Eu , ^{155}Eu
 - deposition cup exchanged every 12–24 h to monitor FP release
 - Ta furnace liner, Ta gas inlet tube, graphite holder, and compact matrix are analyzed after the test to provide information on compact release and TRISO performance



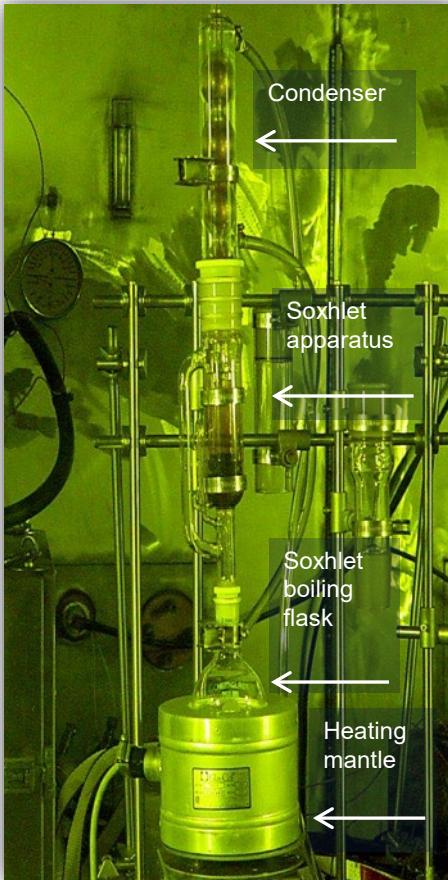
Key Personnel: Stephen Trewitt, Will Cureton
Safety Testing in the CCCTF

Standard ORNL PIE Process

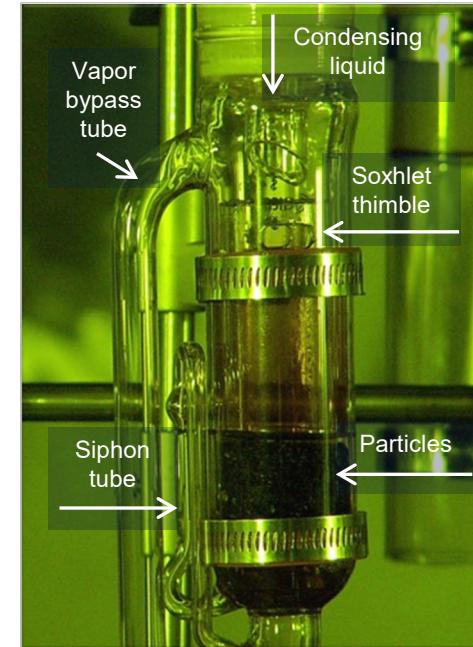
- Perform safety test in ORNL Core Conduction Cooldown Test Facility (CCCTF) if applicable
- Deconsolidate and leach (DL) compact



Deconsolidation Rig



Soxhlet extractor for LBL



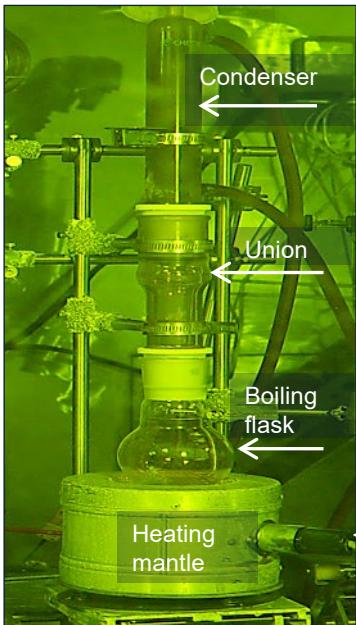
Particles leached in
Soxhlet extractor

Key Personnel: Fred Montgomery
Deconsolidation Leach-Burn-Leach and Burnup Analysis



Standard ORNL PIE Process

- Perform safety test in ORNL Core Conduction Cooldown Test Facility (CCCTF) if applicable
- Deconsolidate and leach (DL) compact
- Further digest matrix in boiling acid, wash and sieve out TRISO particles, burn-leach (BL) matrix



Digestion and Matrix BL Rig

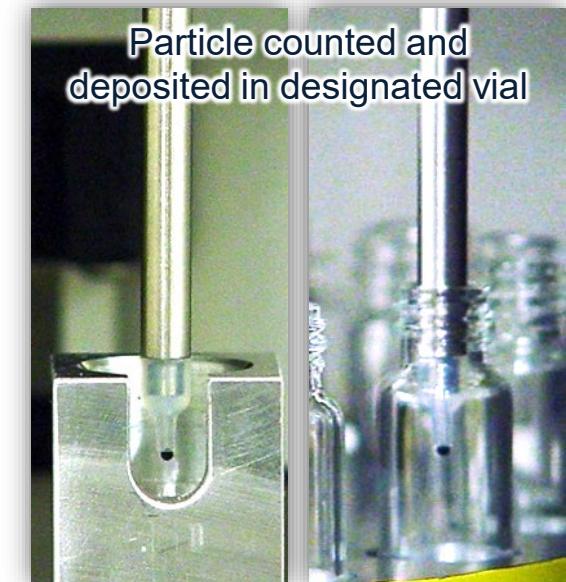
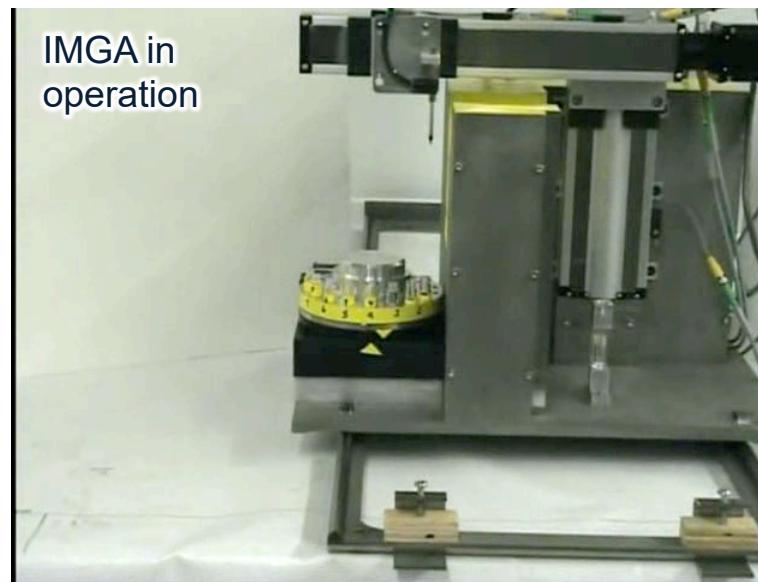
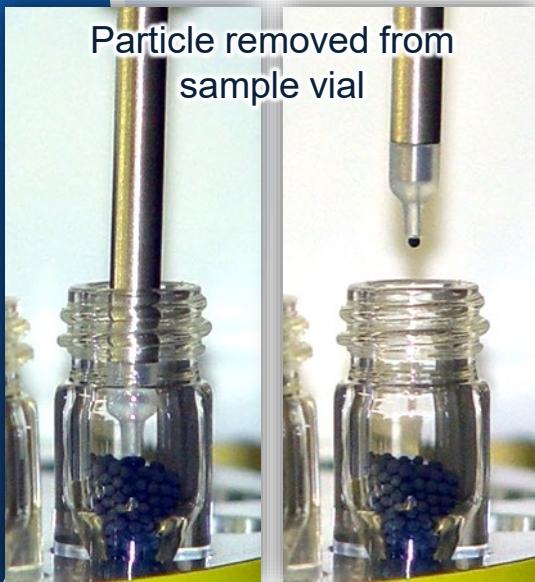


Clean TRISO particles

Key Personnel: Fred Montgomery
Deconsolidation Leach-Burn-Leach and Burnup Analysis

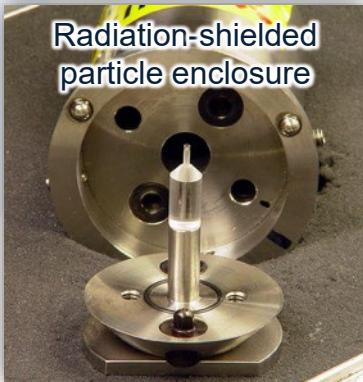
Standard ORNL PIE Process

- Perform safety test in ORNL Core Conduction cooldown Test Facility (CCCTF) if applicable
- Deconsolidate and leach (DL) compact
- Further digest matrix in boiling acid, wash and sieve out TRISO particles, burn-leach (BL) matrix
- Gamma scan TRISO particles with Irradiated Microsphere Gamma Analyzer (IMGA)
 - 1–5-minute quick survey of all particles to find low-Ce and low-Cs particles
 - 4–6-hour extended scans to measure particle inventories (^{106}Ru , $^{110\text{m}}\text{Ag}$, ^{125}Sb , ^{134}Cs , ^{137}Cs , ^{144}Ce , ^{154}Eu)

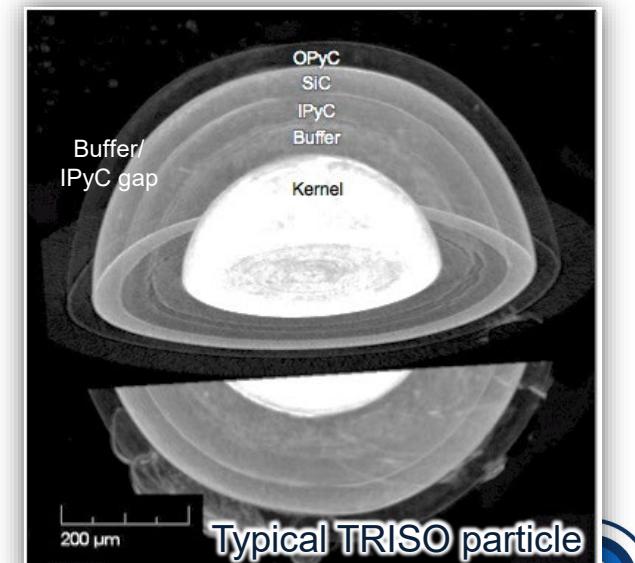
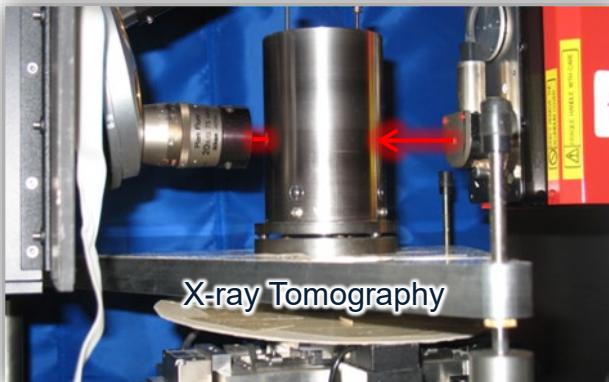


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 - Burn-leach 90% of the particles after IMGA survey, saving 10% unburned TRISO as an archive
- Analyze select particles with nondestructive 3D x-ray computed tomography (XCT)
 - XCT of particles with low-Ce or low-Cs that may have failed TRISO or failed SiC
 - XCT of particles with varied inventories (e.g., high vs low Ag or Eu retention)



Key Personnel: Grant Helmreich—XCT



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 - XCT of particles with low-Ce or low-Cs that may have failed TRISO or failed SiC
 - XCT of particles with varied inventories (e.g., high vs low Ag or Eu retention)
- Perform materialographic examination (optical and electron microscopy of polished sections)
 - guided sectioning for targeted examination of regions of interest observed with XCT
 - random midplane cross sections of particles with varied inventories
 - scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) for microstructural and elemental information, as well as 3D SEM using focused ion beam (FIB) technology



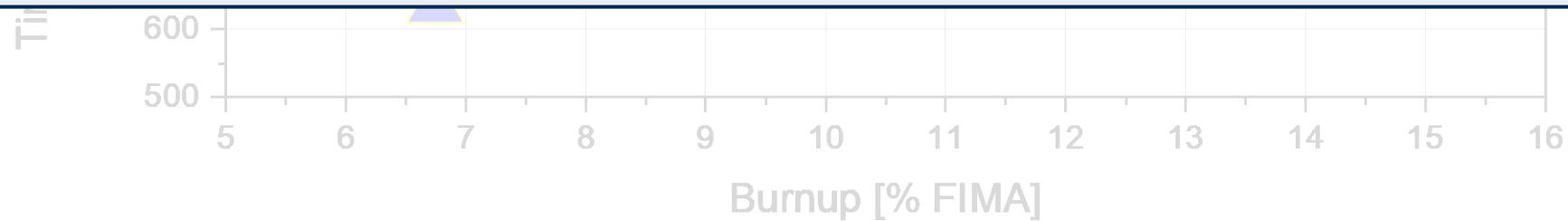
FY22Q3–FY24Q3 AGR-5/6/7 Progress at ORNL

- 12 Safety-tested compacts to date
- 7 As-received compacts deconsolidated to date



FY23/FY24 ORNL PIE related DOE Milestones

Fiscal Year	DOE Level	Activity	Status
FY23	Level II	DLBL on 4 compacts	✓ complete
FY23	Level II	Safety Tests on 4 compacts	✓ complete
FY24	Level II	DLBL on 5 compacts	On track
FY24	Level II	Safety Tests on 4 compacts	On track



- Fissions per initial metal atoms (FIMA) burn-up from p. 42 of Sterbentz, J.W., "JMOCUP Physics Depletion Calculations for the As-Run AGR-5/6/7 TRISO Particle Experiment in ATR Northeast Flux Trap," ECAR-5321, Rev. 0.
- Temperatures from Hawkes, G.L., "AGR-5/6/7 Daily As-Run Thermal Analyses," ECAR-5633, Rev. 0



Cumulative Safety Test Releases

Compact	^{110m}Ag	^{134}Cs	^{154}Eu	^{90}Sr	^{104}Pd	^{85}Kr
AGR-5/6/7 2-2-2 @1600°C	2.6E-2 59	8.3E-6 0.019	6.5E-3 15	2.8E-3 6.3	1.8E-3 4.2	<MDA --
AGR-5/6/7 2-2-4 @1600°C	2.1E-2 48	7.2E-6 0.016	6.9E-3 16	3.0E-3 6.8	9.4E-4 2.1	<MDA --
AGR-5/6/7 2-3-2 @1800°C	9.9E-3 22	1.1E-3 2.5	1.0E-1 232	7.1E-2 160	3.1E-2 70	2.4E-4 0.53
AGR-5/6/7 2-5-1 @1600°C	6.3E-2 137	9.3E-6 0.02	2.7E-3 5.9	1.2E-3 2.6	3.0E-4 0.66	<MDA --
AGR-5/6/7 3-1-2 @1600°C	5.0E-3 11	4.8E-4 1.1	9.5E-3 21	1.3E-2 30	9.1E-4 2.1	3.4E-5 0.076
AGR-5/6/7 4-1-2 @1600°C	2.0E-1 444	3.9E-5 0.085	2.0E-3 4.5	1.4E-3 3.0	4.5E-4 0.99	<MDA --
AGR-5/6/7 5-2-2 @1800°C	3.3E-1 1128	5.3E-4 1.8	4.0E-3 13	1.7E-3 5.8	3.0E-2 103	4.8E-4 1.6
AGR-5/6/7 5-5-3 @1600°C	1.8E-3 6.2	1.3E-5 0.045	6.6E-5 0.22	8.3E-6 0.028	4.8E-4 1.6	<MDA --
AGR-5/6/7 5-6-2 @1600°C	1.7E-2 56	2.0E-4 0.66	1.4E-4 0.48	7.0E-5 0.24	3.2E-4 1.1	5.5E-5 0.19

Values are presented as compact fraction and particle-equivalent highlighted

(relative highlight: green – lower release, red – higher release)

Estimated uncertainty in solute analysis is $\pm 10\%$ and less than values for ^{110m}Ag are estimated from first leach.

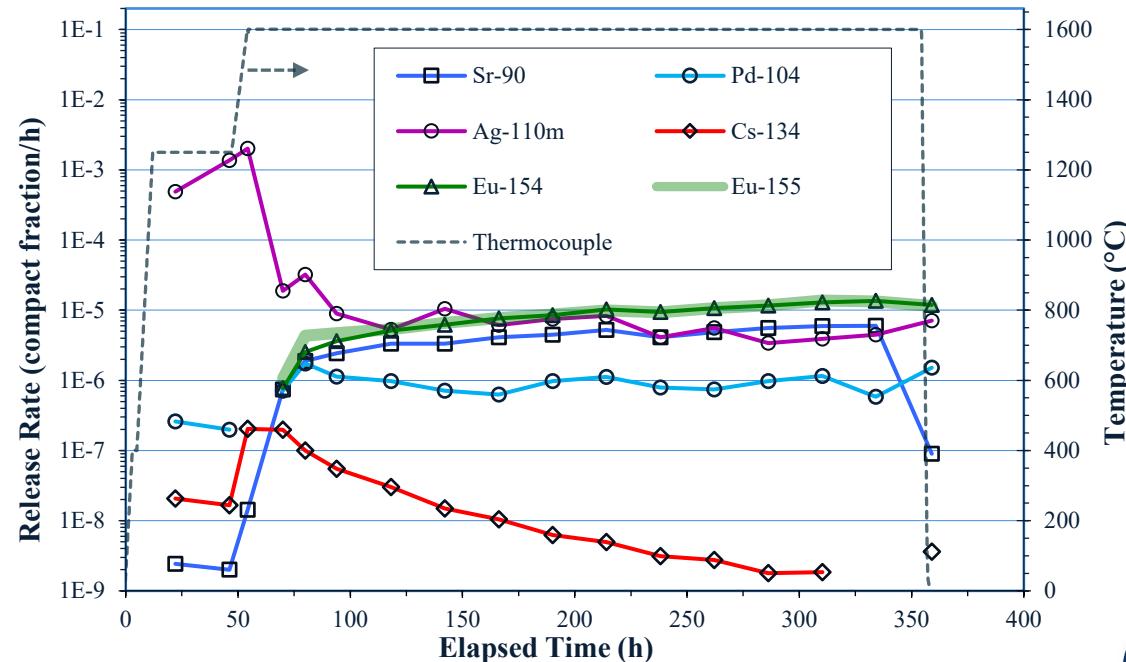
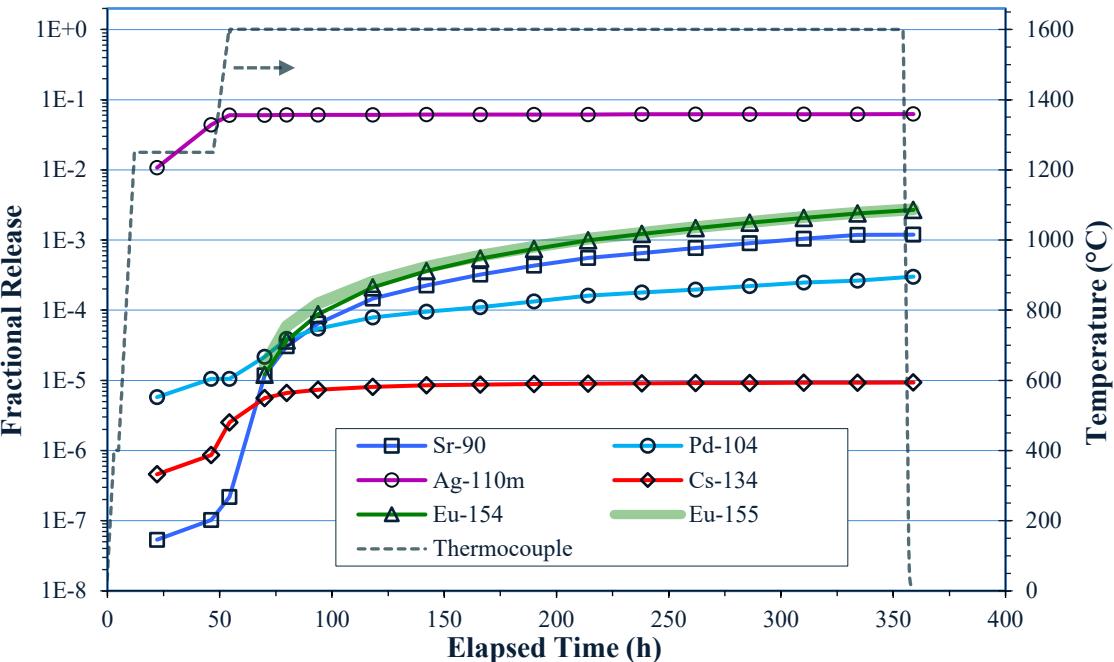


1600°C Safety Test of Compact 2-5-1

(14.8% FIMA, 851°C TAVA Temperature)

	^{110m}Ag	^{134}Cs	^{154}Eu	^{90}Sr	^{104}Pd	^{85}Kr
Compact Fraction	6.3E-2	9.3E-6	2.7E-3	1.2E-3	3.0E-4	<MDA
Particle Equivalents	137	0.02	5.9	2.6	0.66	--

- Liquid nitrogen supply issues for ^{85}Kr traps caused longer pause at 1250°C.
- No indication of TRISO failure — ^{85}Kr below MDA of 5.5E-7 (<0.0013 particle equivalents).
- No indication of SiC failure — ^{134}Cs release of <0.02 particle equivalents.
- ^{154}Eu , ^{90}Sr , and ^{104}Pd release similar to other Capsule 2 compacts tested at 1600°C.
- Moderate ^{110m}Ag release at the beginning of the safety test related to a high inventory outside the SiC at the end of the irradiation test.

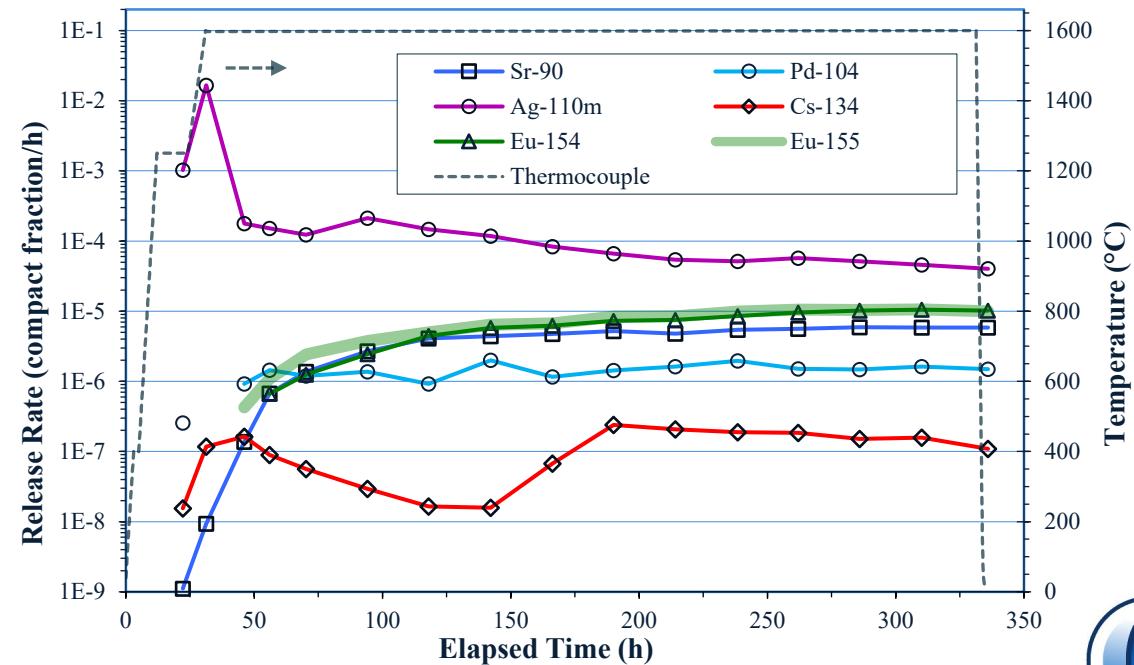
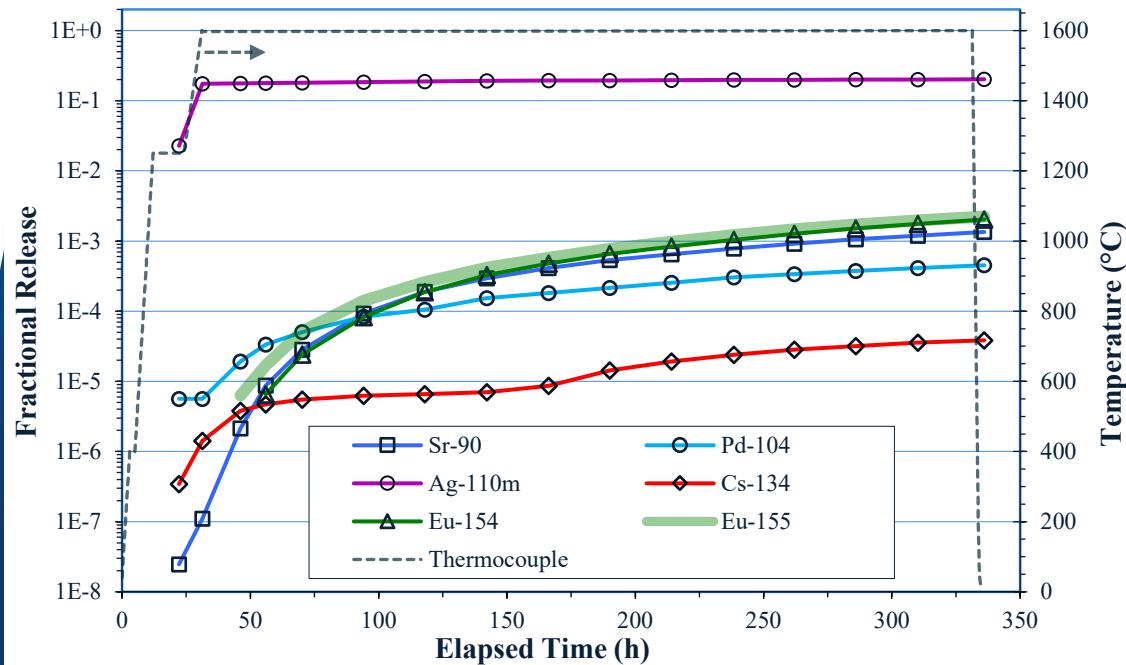


1600°C Safety Test of Compact 4-1-2

(13.7% FIMA, 774°C TAVA Temperature)

	^{110m}Ag	^{134}Cs	^{154}Eu	^{90}Sr	^{104}Pd	^{85}Kr
Compact Fraction	2.0E-1	3.9E-5	2.0E-3	1.4E-3	4.5E-4	<MDA
Particle Equivalents	444	0.085	4.5	3.0	0.99	--

- No indication of TRISO failure — ^{85}Kr below MDA of 5.5E-7 (<0.0013 particle equivalents).
- No indication of SiC failure — ^{134}Cs release of <0.085 particle equivalents.
- ^{110m}Ag release highest observed among all AGR-5/6/7 1600°C safety-tested compacts at ORNL. Low irradiation temperature causes higher retention in the matrix post irradiation.
- ^{154}Eu , ^{90}Sr , and ^{104}Pd release similar to Capsule 2 compacts tested at 1600°C.

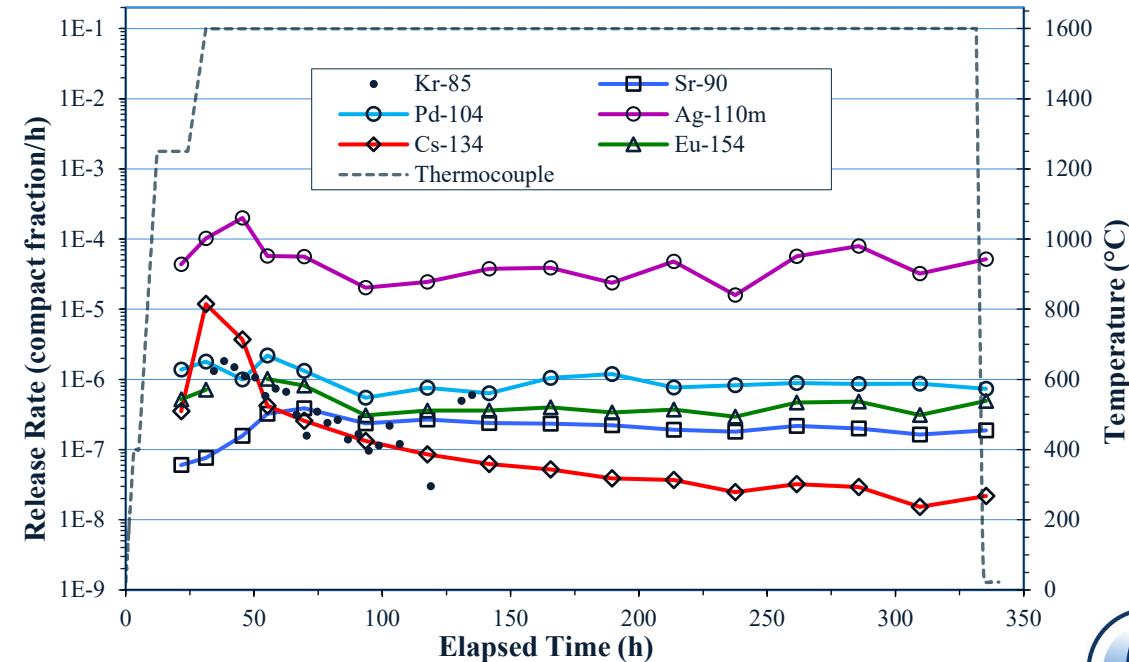
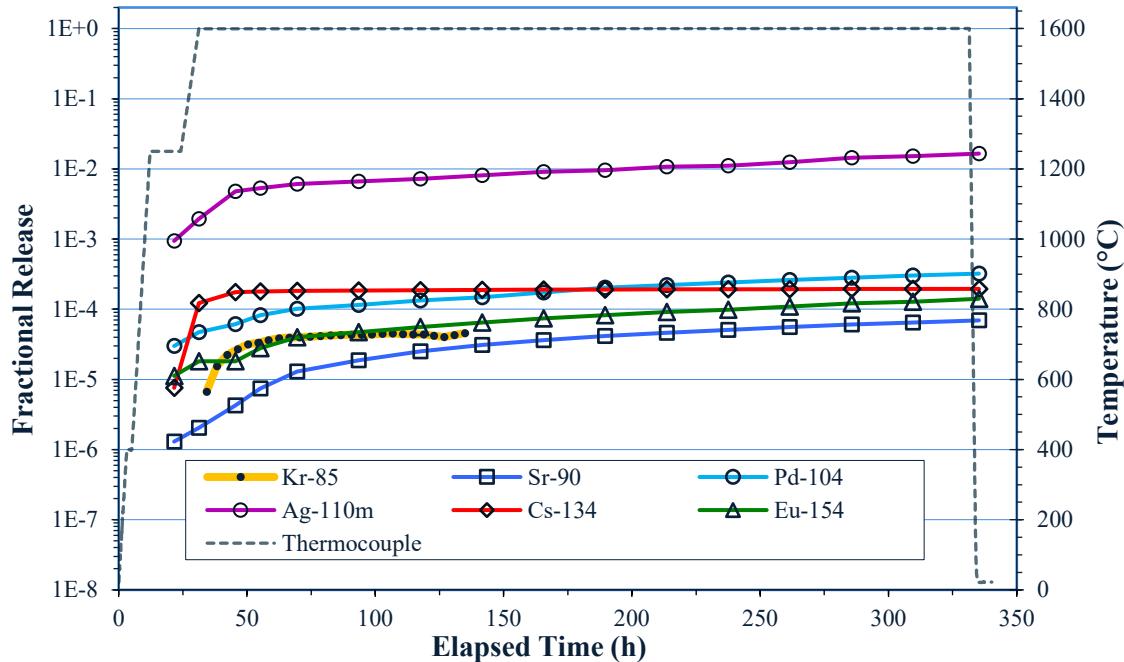


1600°C Safety Test of Compact 5-6-2

(6.8% FIMA, 634°C TAVA Temperature)

	^{110m}Ag	^{134}Cs	^{154}Eu	^{90}Sr	^{104}Pd	^{85}Kr
Compact Fraction	1.7E-2	2.0E-4	1.4E-4	7.0E-5	3.2E-4	5.5E-5
Particle Equivalents	56	0.66	0.48	0.24	1.1	0.19

- Initial ^{134}Cs release of 2/3 particle equivalent indicates degraded or defective SiC, likely from a single particle given time-dependent rate
- ^{85}Kr accumulation of 5.5E-5 (0.19 particle equivalents) indicative of degraded or defective TRISO.
- Early ^{110m}Ag release dominated by early release of matrix inventory. Relatively constant rate of release through the rest of the test.
- ^{154}Eu , ^{90}Sr , and ^{104}Pd had typical rate trends related to slow transport to cups and some diffusive release from particles.

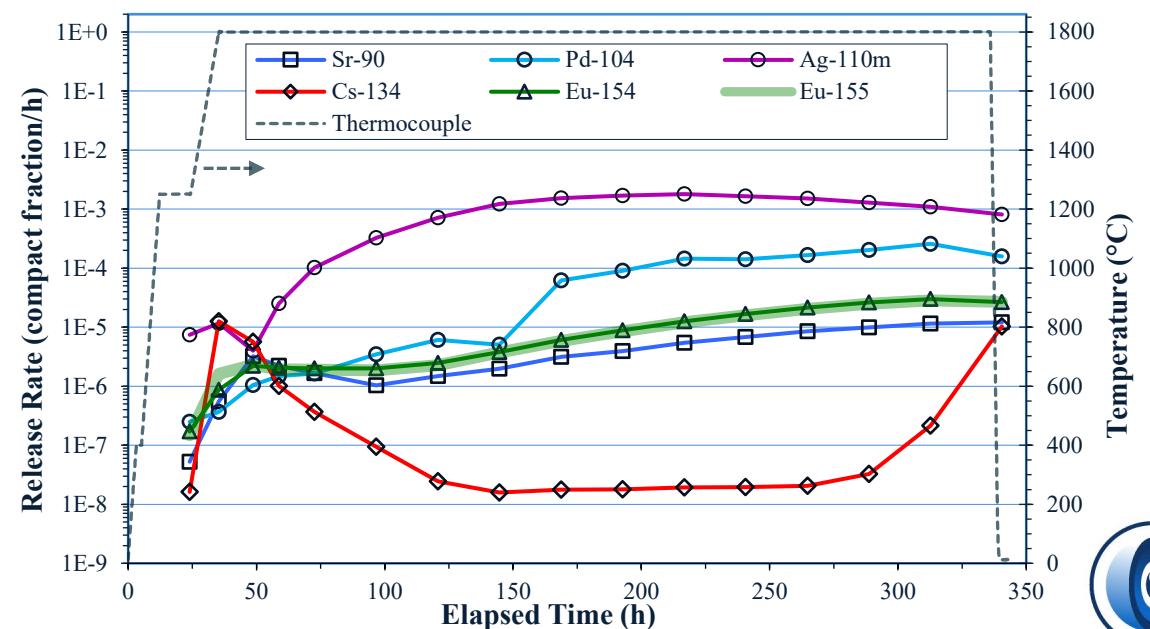
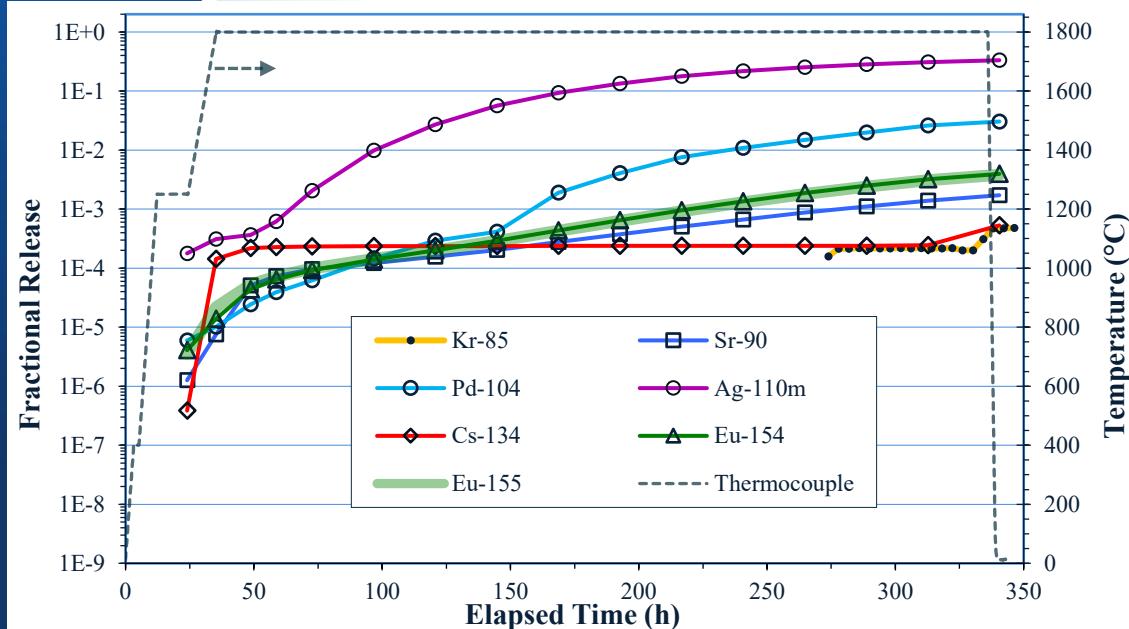


1800°C Safety Test of Compact 5-2-2

(8.8% FIMA, 789°C TAVA Temperature)

	110mAg	134Cs	154Eu	90Sr	104Pd	85Kr
Compact Fraction	3.3E-1	5.3E-4	4.0E-3	1.7E-3	3.0E-2	4.8E-4
Particle Equivalents	1128	1.8	13	5.8	103	1.6

- ^{85}Kr accumulation of 4.8E-4 (1.6 particle equivalents) indicative of degraded or defective TRISO.
- Initial ^{134}Cs release of ~one particle equivalent indicates degraded or defective SiC, likely from a single particle given time-dependent rate. Uptick in ^{134}Cs at end of test may be from a second particle and likely related to the observed ^{85}Kr release.
- Cumulative $^{110\text{m}}\text{Ag}$ release of 33% was mostly from diffusive release through SiC during the safety test and higher than observed in 1800°C test of Compact 2-3-2 because of higher fractional inventory retained in irradiated particles.
- Uptick in ^{104}Pd release at ~170 hours possibly indicating microstructural evolution at 1800°C. Further PIE necessary.



1600°C Simultaneous Safety Test of Compacts 5-2-1, 5-2-4, 5-3-2

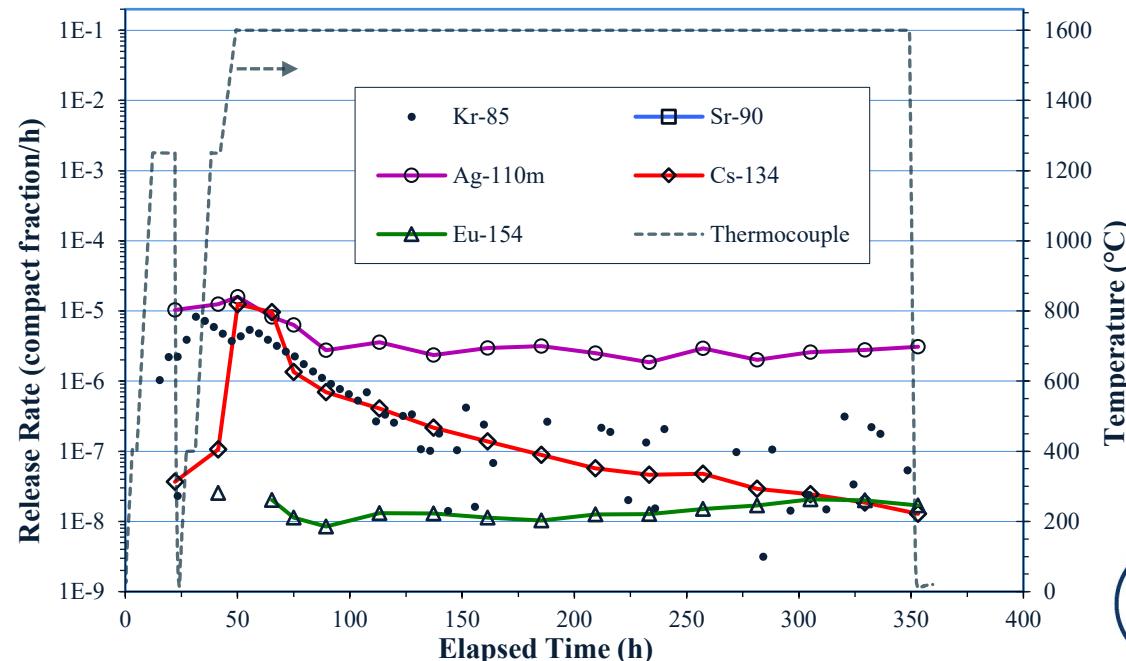
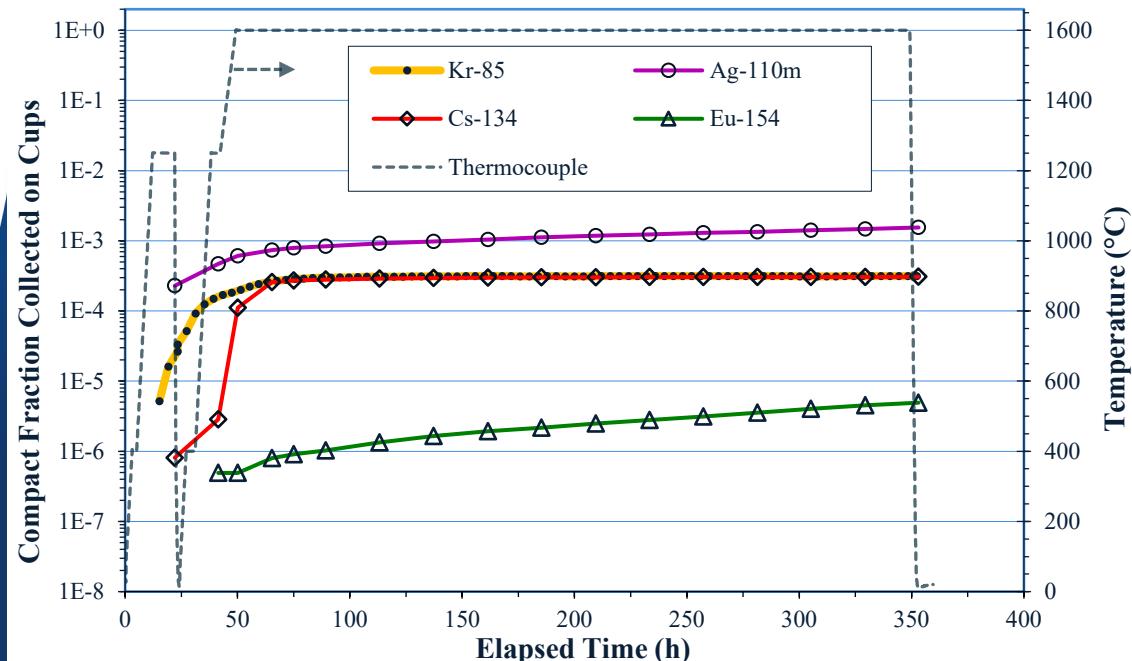
(8.8% FIMA, 790°C TAVA Temperature)

(9.0% FIMA, 801°C TAVA Temperature)

(8.4% FIMA, 800°C TAVA Temperature)

	^{110m}Ag	^{134}Cs	^{85}Kr
Compact Fraction	1.6E-3	3.1E-4	3.1E-4
Particle Equivalents	16	3.1	3.3

- Cold finger chiller malfunction forced automatic shutdown during 1250°C hold.
- ^{85}Kr release observed during 1250°C hold and accumulated to 3.1E-4 (3.3 particle equivalents) indicative of multiple degraded or defective TRISO.
- ^{134}Cs release of 3.1E-4 (3.1 particle equivalents) indicates particles with degraded or defective SiC.
- ^{110m}Ag showed initial release from matrix plus a steady release throughout test of roughly 16 particle equivalents on par with other Capsule 5 compacts safety-tested at 1600°C.



Preliminary: no efficiency correction



Cumulative Safety Test Releases

Compact	^{110m}Ag	^{134}Cs	^{154}Eu	^{90}Sr	^{104}Pd	^{85}Kr
AGR-5/6/7 2-2-2 @1600°C (845°C TAVA, 14.0% FIMA)	1.5	1.0E-3	1.5	1.5	1.2	1.0E-3
AGR-5/6/7 2-2-4 @1600°C (856°C TAVA, 14.3% FIMA)	2.1E-2	7.1E-3	3.9E-3	3.1E-3	2.4E-4	1.0E-3
AGR-5/6/7 2-3-2 @1800°C (874°C TAVA, 14.4% FIMA)	9.9E-3	1.1E-3	1.0E-1	7.1E-2	3.1E-2	2.4E-4
AGR-5/6/7 2-5-1 @1600°C (851°C TAVA, 14.8% FIMA)	22	2.5	232	160	70	0.53
AGR-5/6/7 3-1-2 @1600°C (1193°C TAVA, 13.8% FIMA)	1.8	1.0E-3	1.5	1.2E-3	3.0E-4	1.0E-3
AGR-5/6/7 4-1-2 @1600°C (774°C TAVA, 13.7% FIMA)	1.37	1.0E-3	1.5	1.2E-3	3.0E-4	1.0E-3
AGR-5/6/7 5-2-2 @1800°C (789°C TAVA, 8.8% FIMA)	3.3E-1	5.3E-4	4.0E-3	1.7E-3	3.0E-2	4.8E-4
AGR-5/6/7 5-5-3 @1600°C (773°C TAVA, 7.6% FIMA)	1128	1.8	13	5.8	103	1.6
AGR-5/6/7 5-6-2 @1600°C (634°C TAVA, 6.8% FIMA)	1.0E-3	1.0E-3	1.0E-3	1.0E-3	1.0E-3	1.0E-3

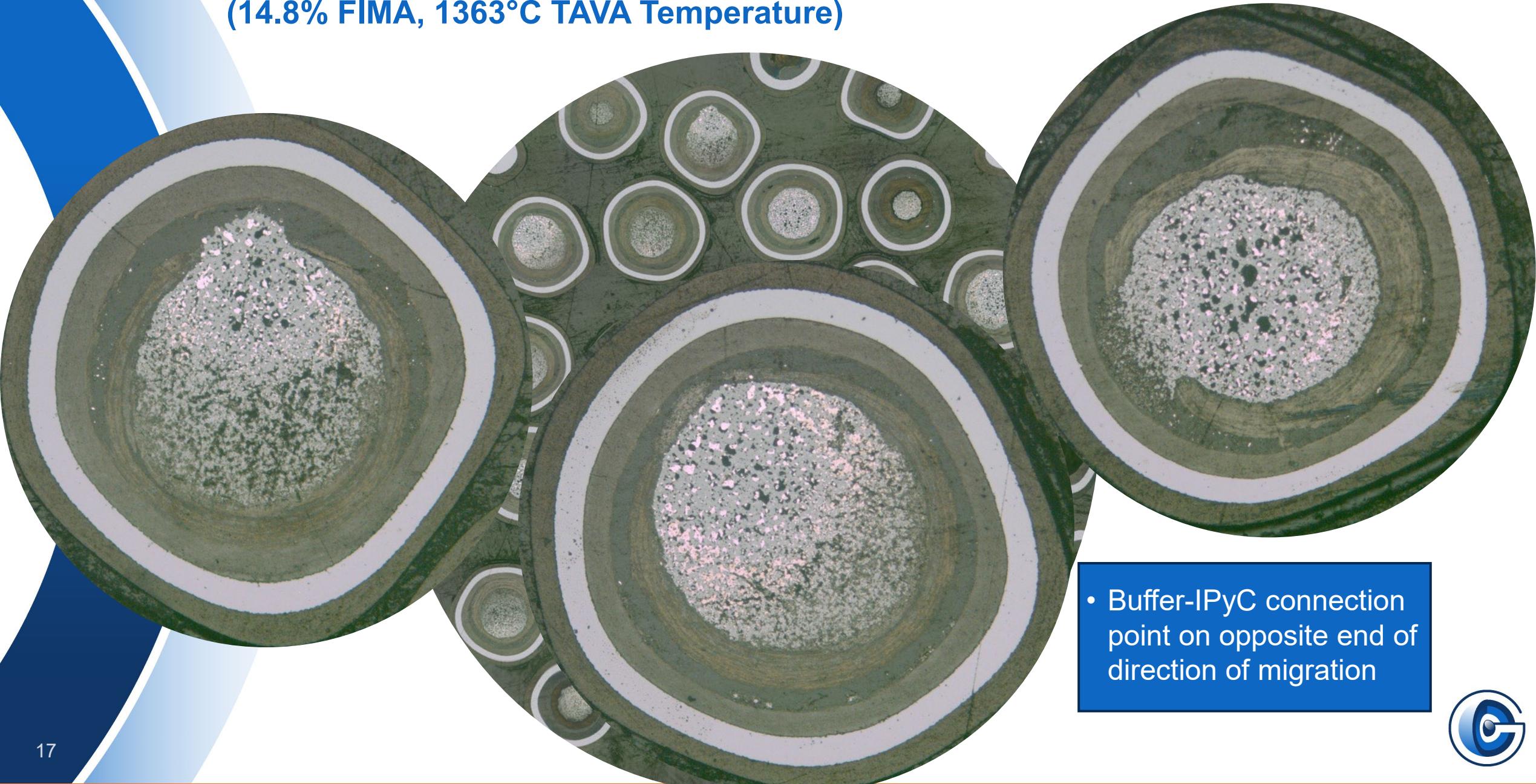
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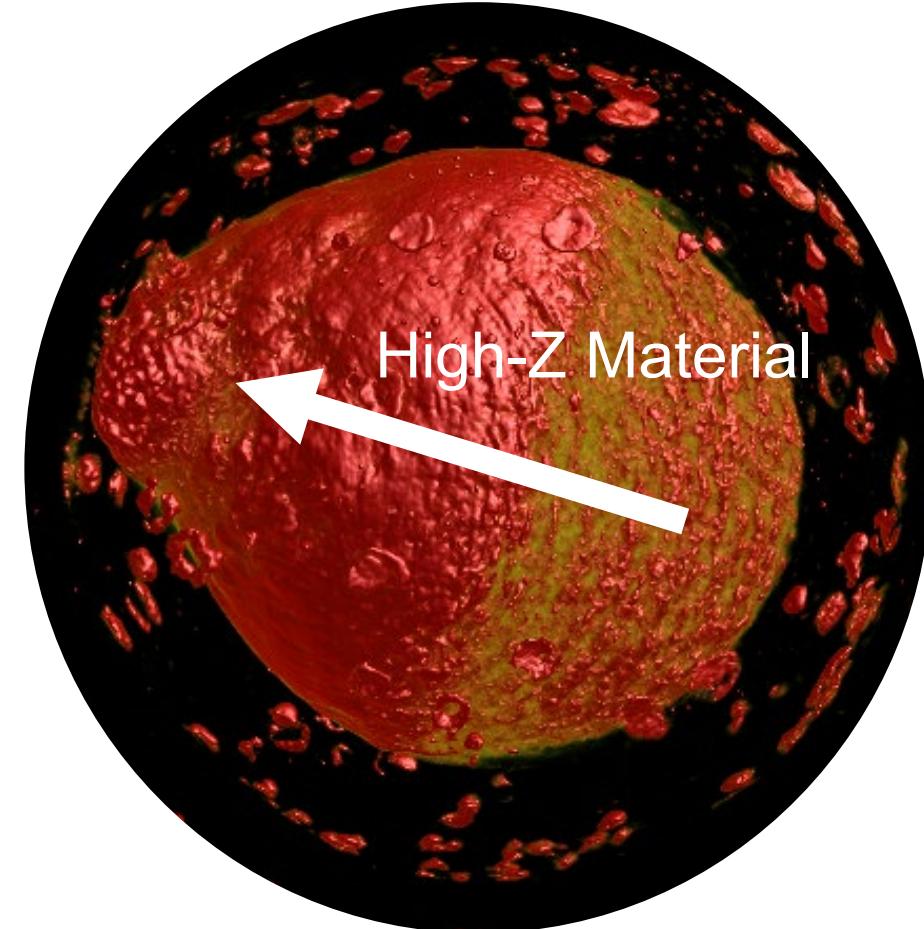
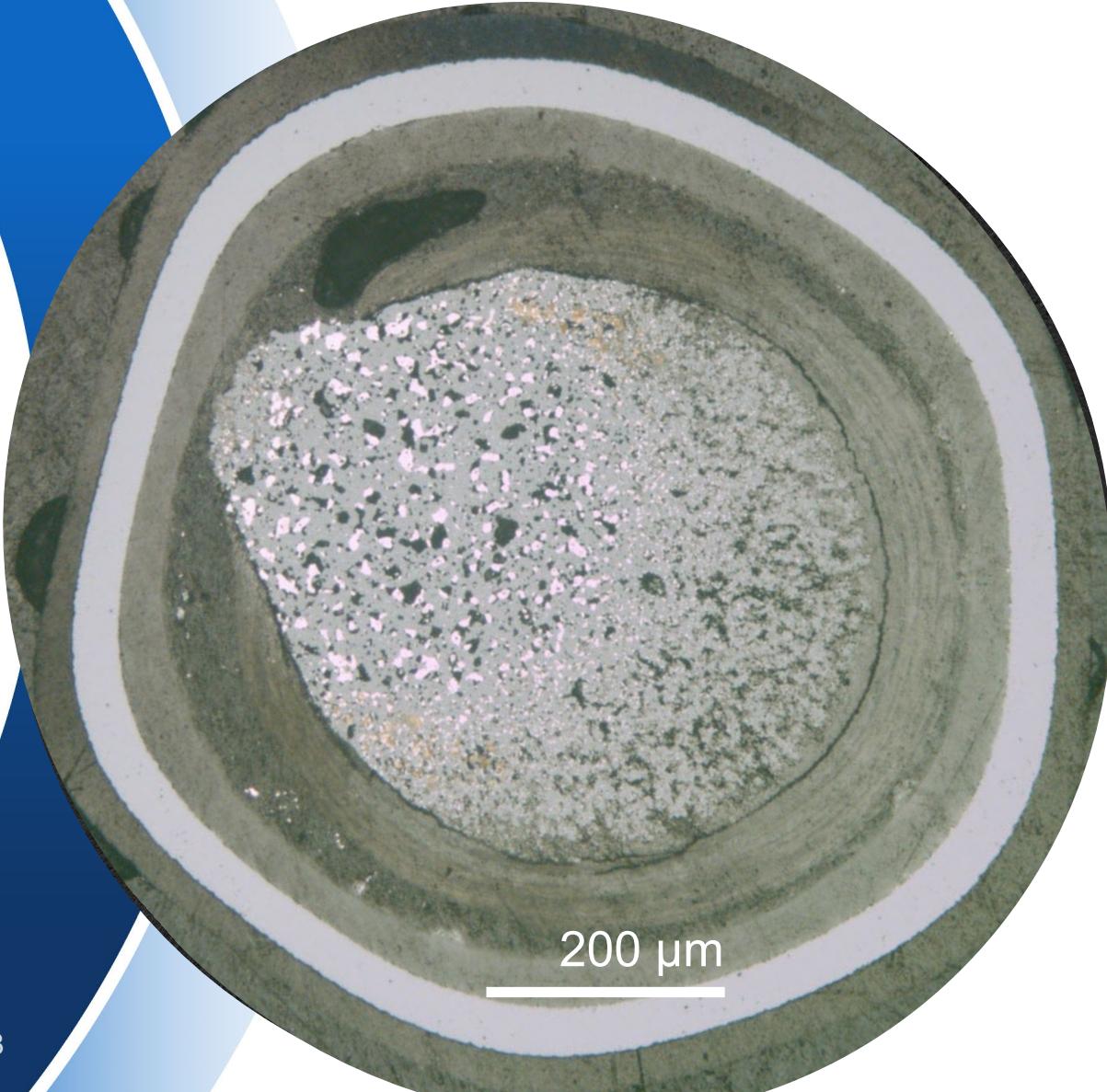
As-Irradiated Compact 3-6-3 Random Samples (14.8% FIMA, 1363°C TAVA Temperature)



- Buffer-IPyC connection point on opposite end of direction of migration

As-Irradiated Compact 3-6-3 RS-08

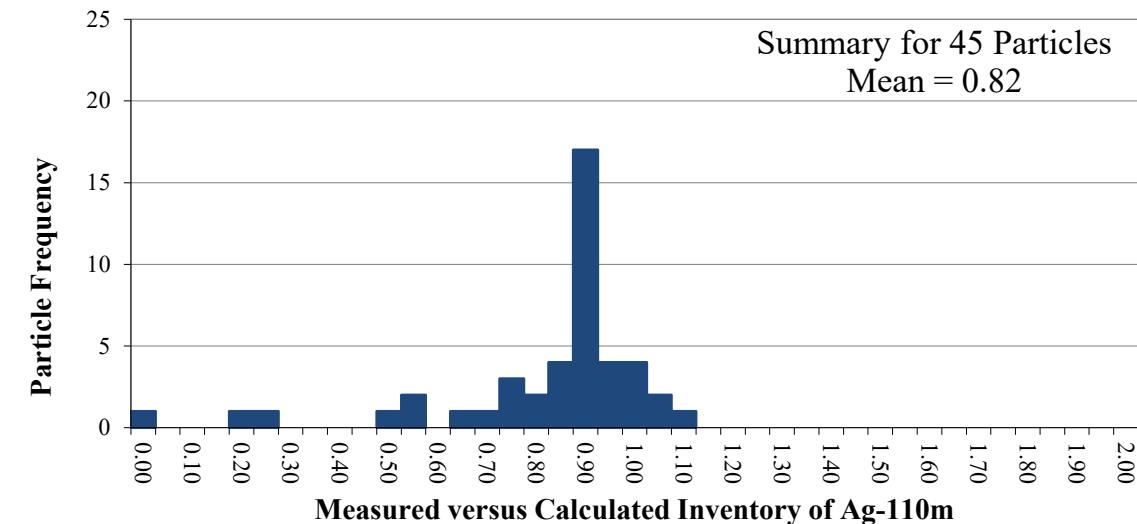
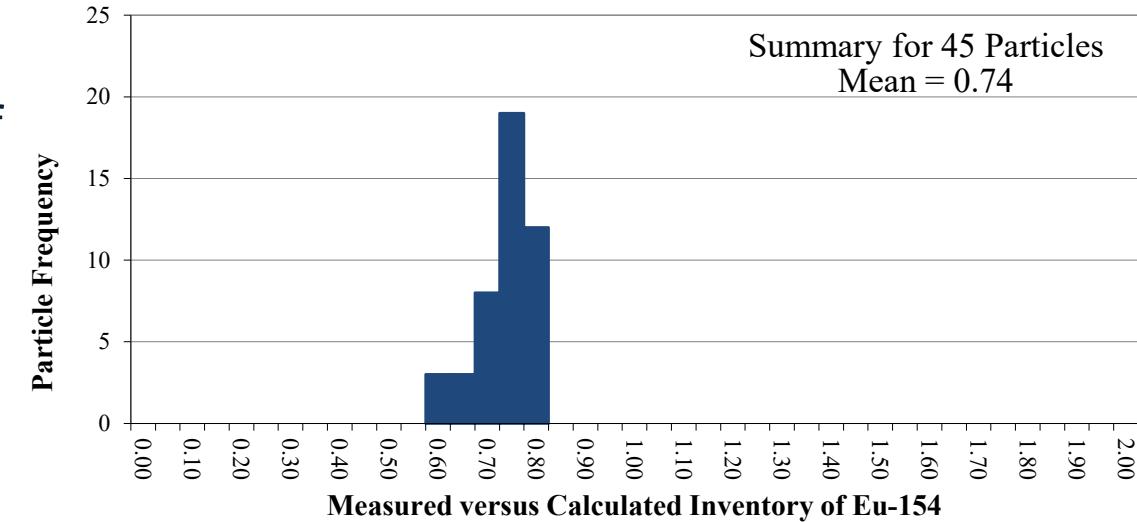
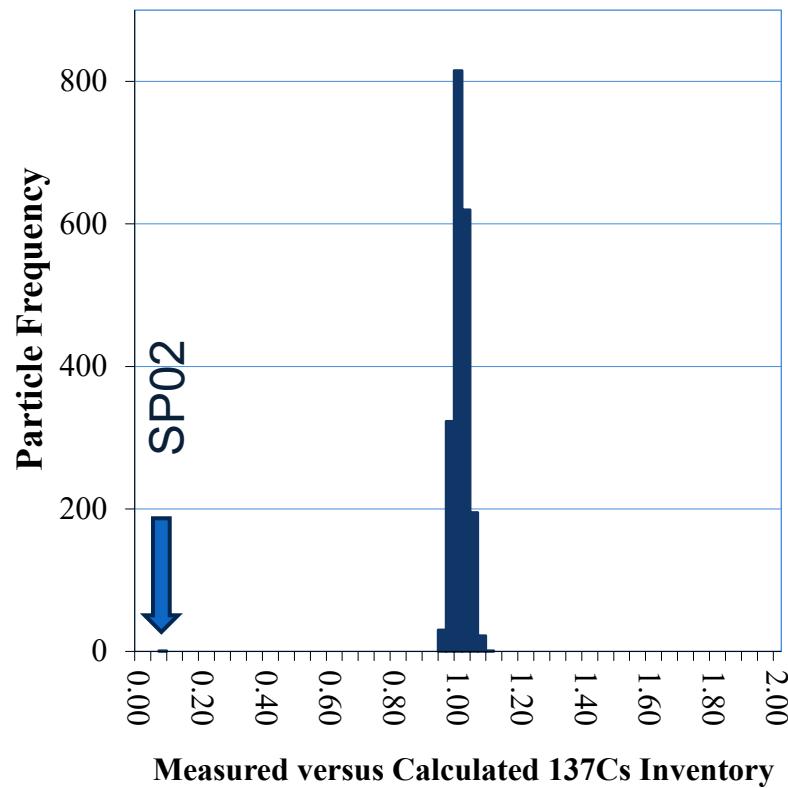
(14.8% FIMA, 1363°C TAVA Temperature)



As-Irradiated Compact 3-6-3

(14.8% FIMA, 1363°C TAVA Temperature)

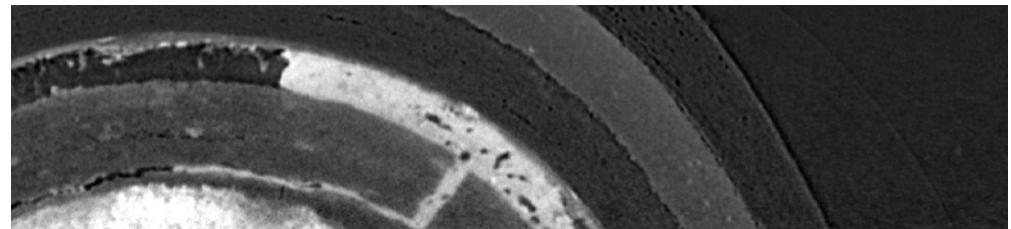
- IMGA data shows acceptable fission product retention despite of kernel migration
- One low- ^{137}Cs particle identified



As-Irradiated Compact 3-6-3 Special Particle

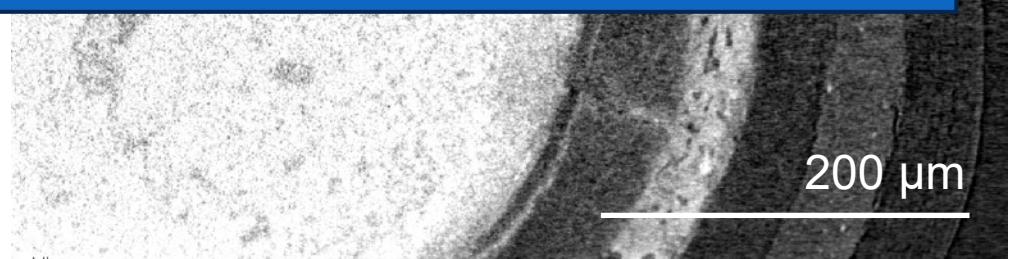
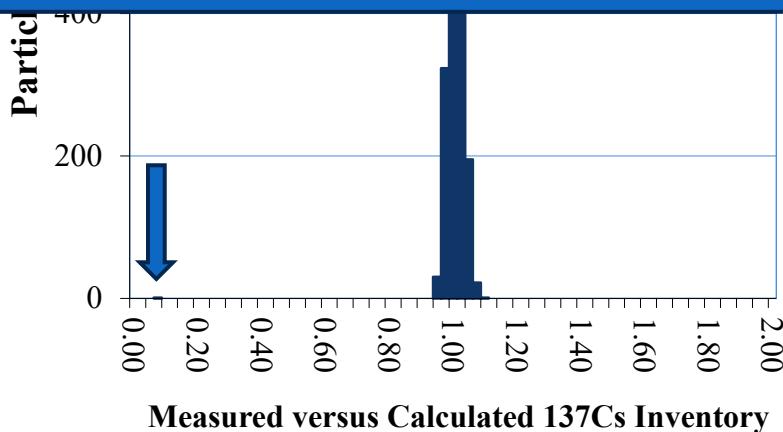
(14.8% FIMA, 1363°C TAVA Temperature)

- Potential IPyC crack and SiC degradation
- More PIE needed to investigate



Key take aways from Compact 3-6-3

- Unclear what mechanism dominates transport of carbon (diffusion vs. CO dissociation and carbon deposition)
- Further tests could elucidate behavior (XCT, SEM, elemental analysis)
- In Compact 3-6-3, migrated kernels do not reach the IPyC, fission product retention is not impacted. Gross failure not observed



XCT image of potential SiC degradation



Summary of ~2 Years of AGR-5/6/7 PIE at ORNL

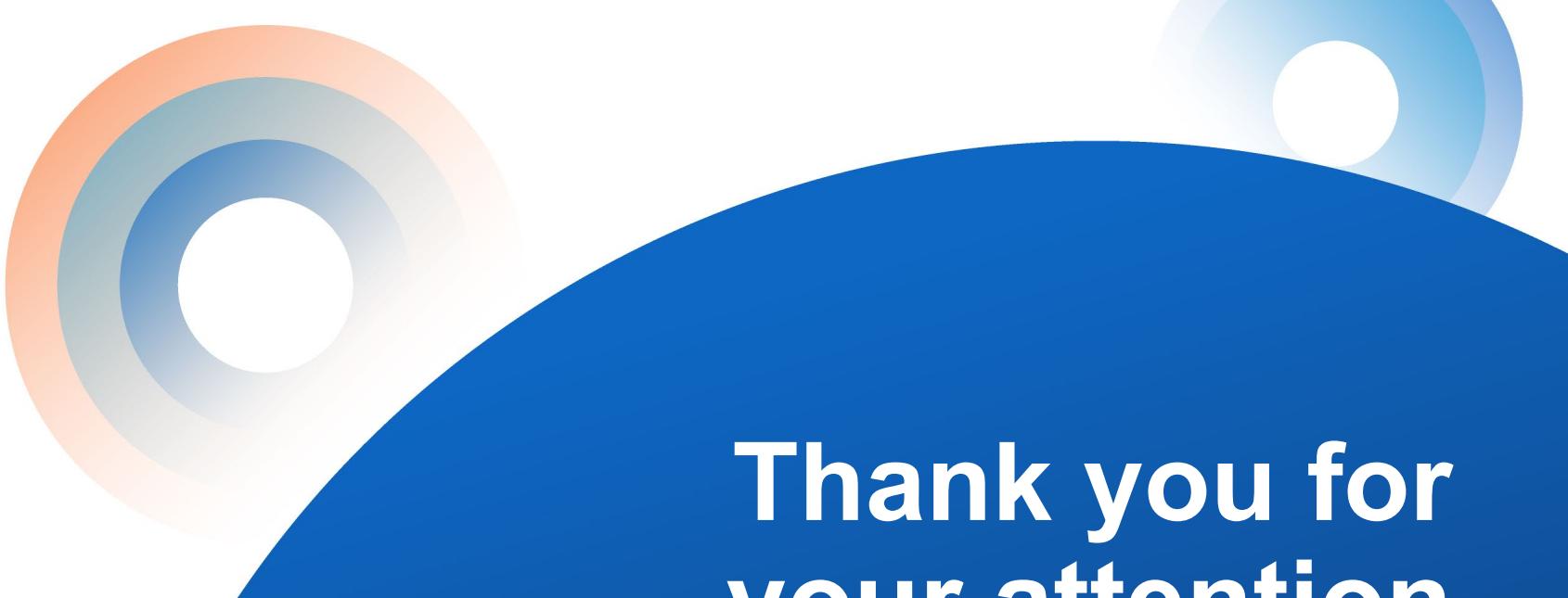
- AGR-5/6/7 PIE at ORNL has been in progress since April 2022.
 - Seven as-irradiated compacts are in various stages of destructive PIE.
 - Twelve compacts have been safety-tested and are in various stages of post-safety test destructive PIE.
- Similarities and some differences compared with AGR-1 and AGR-2 UCO compact safety testing and PIE are noted and will be studied further.
 - We will soon have more results from the later steps in the destructive PIE to help evaluate results from safety testing and DLBL.
 - We need to build a broader database by examining more AGR-5/6/7 compacts to get sufficient statistics to verify trends in behavior before drawing any broad conclusions.





GAS-COOLED REACTOR

ADVANCED REACTOR
TECHNOLOGIES PROGRAM



Thank you for your attention

William F. Cureton

curetonwf@ornl.gov

This work was sponsored by US Department of Energy
Office of Nuclear Energy Advanced Reactors Technologies
Advanced Gas Reactor Fuel Development and Qualification
Program



Extra Slides



FY22Q3–FY24Q3 AGR-5/6/7 Progress at ORNL

363 DLBL Results

		Total Inventory, uCi		ICP-MS Analyses						
		Sample OD		²³⁴ U	²³⁵ U	²³⁶ U	²³⁸ U	²³⁷ Np	²³⁹ Pu	²⁴⁰ Pu
		Post-burn additional matrix ash leach #2								
0	0	Post-burn matrix leach #3								
Max Total of deconsolidation acid and preburn leach #1 and #2				< 2.82E-01	3.05E-01	2.38E-01	2.71E-01	4.30E-01	3.92E-01	6.50E-01
Min Total of deconsolidation acid and preburn leach #1 and #2				2.77E-01	3.05E-01	2.38E-01	2.71E-01	4.30E-01	3.92E-01	6.50E-01
	max preburn compact totals with water rinse			< 2.82E-01	3.05E-01	2.38E-01	2.71E-01	4.30E-01	3.92E-01	6.50E-01
	min preburn compact totals with water rinse			2.77E-01	3.05E-01	2.38E-01	2.71E-01	4.30E-01	3.92E-01	6.50E-01
max Post-burn Particle totals leach #1 and #2				2.75E+00	2.86E+00	2.60E+00	2.69E+00	2.74E+00	2.40E+00	3.04E+00
min Post-burn Particle totals leach #1 and #2				2.75E+00	2.86E+00	2.60E+00	2.69E+00	2.74E+00	2.40E+00	3.04E+00
max Post-burn Matrix totals leach #1 and #2				< 9.10E-02	9.45E-02	7.93E-02	1.81E-01	2.61E-01	2.72E-01	4.70E-01
min Post-burn Matrix totals leach #1 and #2				8.82E-02	9.45E-02	7.93E-02	1.81E-01	2.61E-01	2.72E-01	4.70E-01
max postburn compact totals leach #1 and #2				< 2.84E+00	2.96E+00	2.68E+00	2.87E+00	3.00E+00	2.68E+00	3.51E+00
min postburn compact totals leach #1 and #2				2.84E+00	2.96E+00	2.68E+00	2.87E+00	3.00E+00	2.68E+00	3.51E+00
maximum grand total leach #1 and #2				< 3.12E+00	3.26E+00	2.92E+00	3.14E+00	3.43E+00	3.07E+00	4.16E+00
minimum grand total leach #1 and #2				3.11E+00	3.26E+00	2.92E+00	3.14E+00	3.43E+00	3.07E+00	4.16E+00



Cumulative Safety Test Releases from Nine AGR-5/6/7 Compacts

Compact	⁹⁰ Sr	¹⁰⁴ Pd	^{110m} Ag	¹³⁴ Cs	¹⁵⁴ Eu	⁸⁵ Kr
AGR-5/6/7 2-2-2 (1600°C) (845°C TAVA, 14.0% FIMA)	2.8E-3 (6.3)	1.8E-3 (4.2)	2.6E-2 (59)	8.3E-6 (0.019)	6.5E-3 (15)	
AGR-5/6/7 2-2-4 (1600°C) (856°C TAVA, 14.3% FIMA)	3.0E-3 (6.8)	9.4E-4 (2.1)	2.1E-2 (48)	7.2E-6 (0.016)	6.9E-3 (16)	
AGR-5/6/7 5-5-3 (1600°C) (773°C TAVA, 7.6% FIMA)	8.3E-6 (0.028)	4.8E-4 (1.6)	1.8E-3 (6.2)	1.3E-5 (0.045)	6.6E-5 (0.22)	
AGR-5/6/7 2-3-2 (1800°C) (874°C TAVA, 14.4% FIMA)	7.1E-2 ~(160)	3.1E-2 (70)	~9.6E-3 ~(22)	~1.0E-3 ~(2.3)	>8.8E-2 >(199)	2.4E-4 (0.53)
AGR-5/6/7 3-1-2 (1600°C) (1193°C TAVA, 13.8% FIMA)	1.3E-2 ~(30)	9.1E-4 (2.1)	~5.1E-3 ~(12)	~4.4E-4 ~(1.0)	9.5E-3 (21)	3.4E-5 (0.076)
AGR-5/6/7 5-2-2 (1800°C) (789°C TAVA, 8.8% FIMA)	1.7E-3 (5.8)	3.0E-2 (103)	~3.5E-1 ~(1180)	~4.8E-4 ~(1.6)	4.0E-3 (13)	4.8E-4 (1.6)
AGR-5/6/7 5-6-2 (1600°C) (634°C TAVA, 6.8% FIMA)	6.9E-5 (0.24)	3.2E-4 (1.1)	1.7E-2 56.068	2.0E-4 (0.66)	1.4E-4 (0.48)	5.5E-5 (0.19)
AGR-5/6/7 4-1-2 (1600°C) (774°C TAVA, 13.7% FIMA)	1.4E-3 (3.0)	4.5E-4 ~(1)	2.0E-1 (444)	3.9E-5 (0.085)	2.0E-3 (4.5)	
AGR-5/6/7 2-5-1 (1600°C) (851°C TAVA, 14.8% FIMA)	1.2E-3 (2.6)	3.0E-4 (0.66)	6.3E-2 (137)	9.3E-6 (0.02)	2.7E-3 (5.9)	

Values are presented as compact fraction and particle-equivalent (in parentheses)

Estimated uncertainty in solute analysis is $\pm 10\%$ and less than values for ^{110m}Ag are estimated from first leach.





GAS-COOLED REACTOR

ADVANCED REACTOR TECHNOLOGIES PROGRAM

