July 25, 2023

John D. Hunn
ORNL TRISO Team Lead

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

DOE ART Gas-Cooled Reactor (GCR) Review Meeting
Virtual Meeting
July 25 – 27, 2023

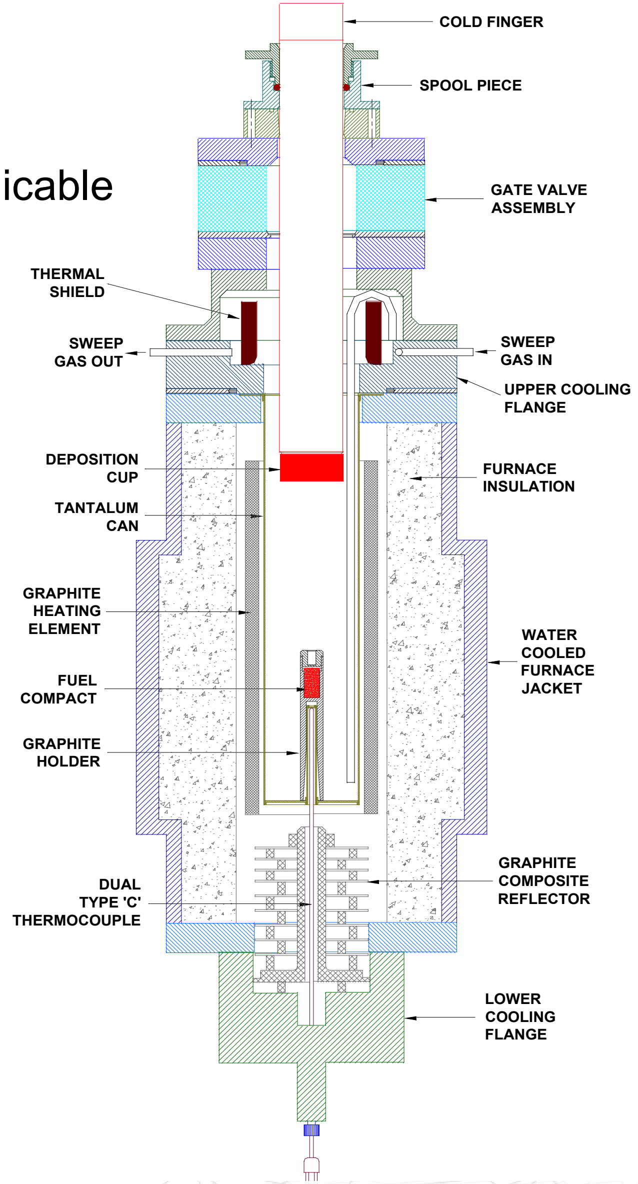
ART
Advanced Reactor Technologies

ORNL Coauthors and Contributors

- Will Cureton — Safety testing in the Core Conduction Cooldown Test Facility (CCCTF)
- Fred Montgomery —Deconsolidation Leach-Burn-Leach (DLBL) and burnup analysis
- Tyler Gerczak — Electron microscopy and Furnace for Irradiated TRISO Testing (FITT)
- Darren Skitt — Irradiated Microsphere Gamma Analyzer (IMGA), materialography, particle heating in FITT
- Grant Helmreich — X-ray Computed Tomography (XCT)
- Jesse Werden — Electron microscopy
- Katherine Montoya — Electron microscopy, analysis of oxidation of FITT specimens
- Zach Burns — Support FITT operation
- 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

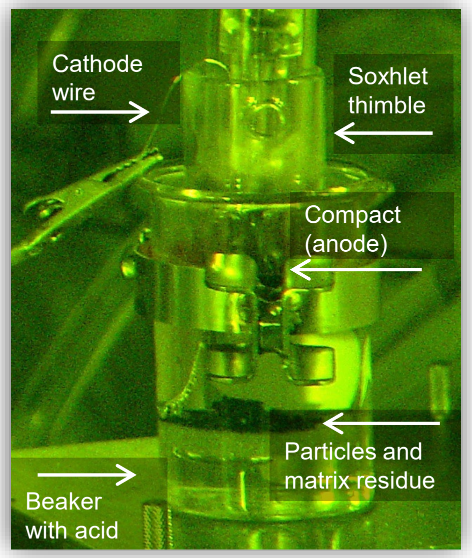
- Perform safety test in ORNL Core Conduction Cooldown Test Facility (CCCTF) if applicable
  - modernized version of the same in cell furnace used for TRISO fuel tests predating AGR
  - cylindrical fuel compact is placed in graphite holder
  - compact in holder positioned on thermocouple finger
  - copper-plated 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, ^{110m}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



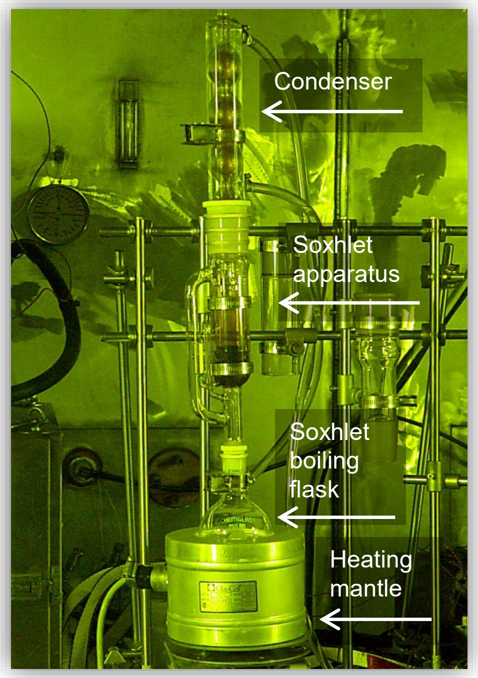
Will Cureton
Safety Testing in the CCCTF

Standard ORNL PIE Process

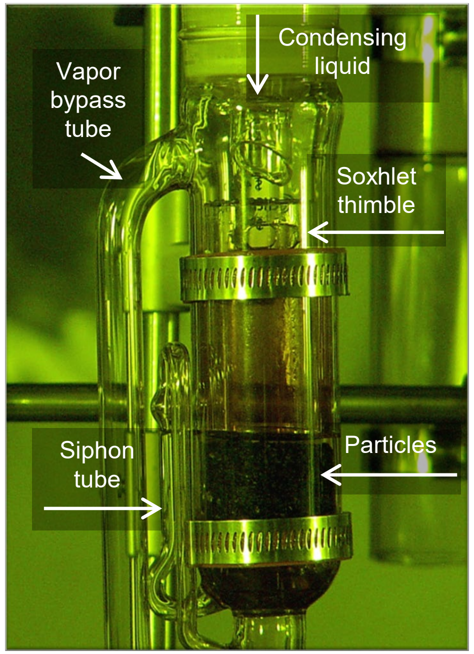
- Perform safety test in ORNL Core Conduction Cooldown Test Facility (CCCTF) if applicable
- Deconsolidate and leach (DL) compact



Deconsolidation Rig



Soxhlet extractor for LBL

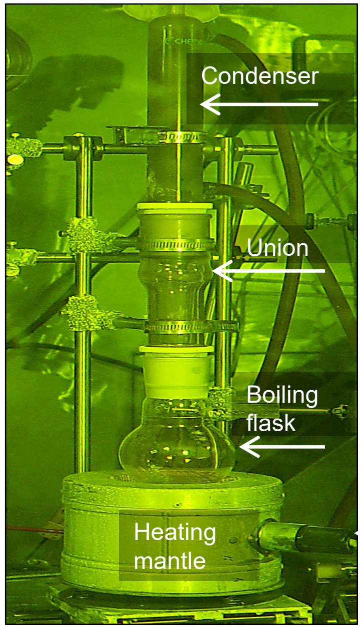


Particles leached in Soxhlet extractor

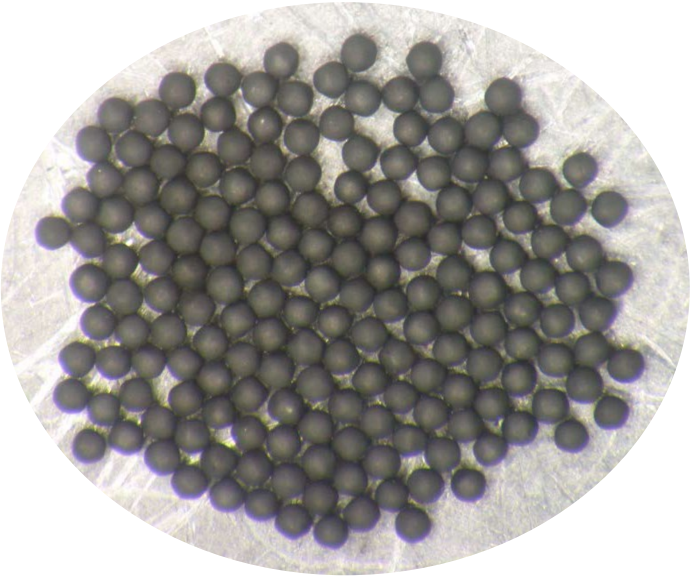
Fred Montgomery
Deconsolidation Leach-Burn-Leach and Burnup Analysis

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

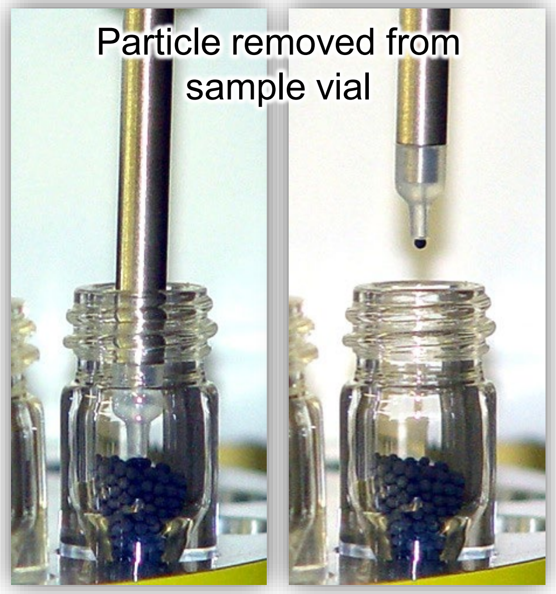


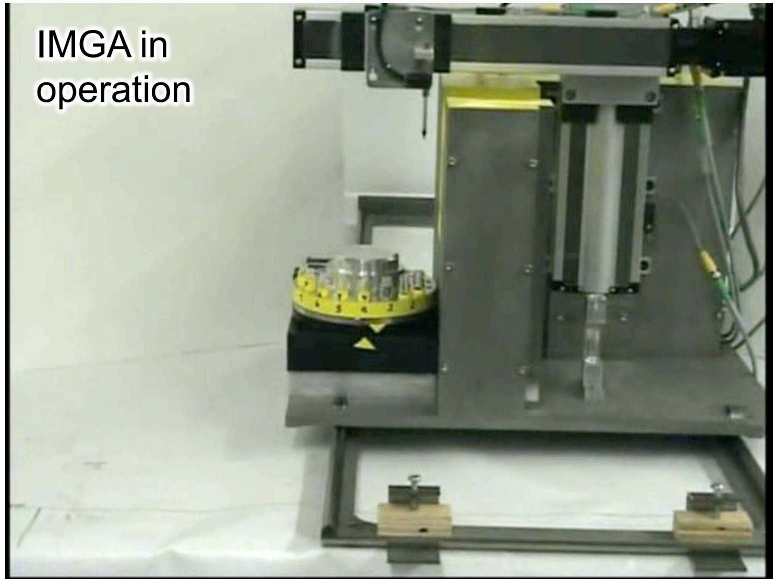
Clean TRISO particles

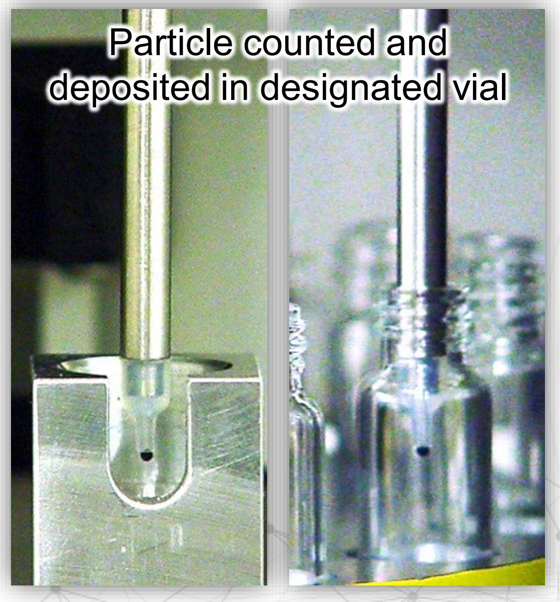
Fred Montgomery
Deconsolidation Leach-Burn-Leach and Burnup Analysis

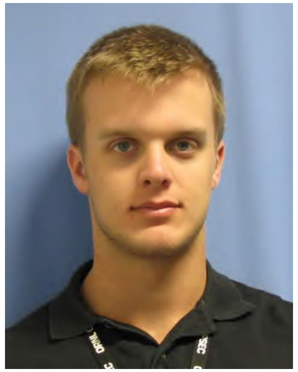
Standard ORNL PIE Process

- 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, ^{110m}Ag, ^{125}Sb, ^{134}Cs, ^{137}Cs, ^{144}Ce, ^{154}Eu)





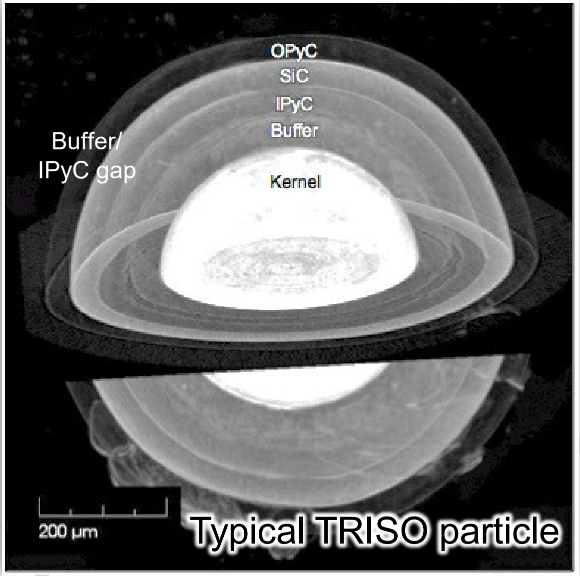


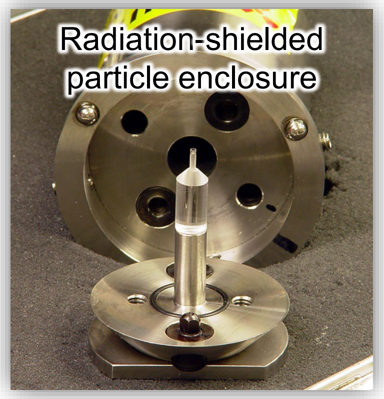


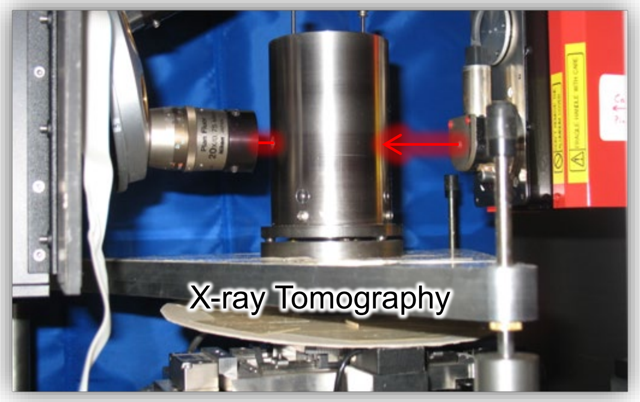
Darren Skitt
IMGA; Particle Mounting, Cross Sectioning, and Optical Microscopy

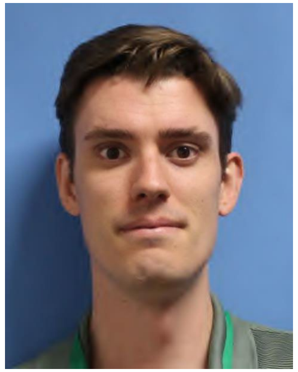
Standard ORNL PIE Process

- 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, ^{110m}Ag, ^{125}Sb, ^{134}Cs, ^{137}Cs, ^{144}Ce, ^{154}Eu)
  - 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)









Grant Helmreich—XCT

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, ^{110m}Ag, ^{125}Sb, ^{134}Cs, ^{137}Cs, ^{144}Ce, ^{154}Eu)
  - Burn-leach 90% of the particles after IMGA survey, saving 10% unburned TRISO as an archive
- Analyze select particles with non-destructive 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)
- 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

Tyler Gerczak, Jesse Werden, and Katherine Montoya
SEM, EDS, and FIB-SEM

FY22Q3–FY23Q3 AGR-5/6/7 Progress at ORNL

- Shipments: First 4 compacts received 3/23/2022; 16 compacts have been shipped to date.

AGR-2 Compact	Average Burnup	Fast Fluence	TAVA Temperature	Safety Test			DLBL	IMGA	XCT	Optical Microscopy	SEM & EDS
				Temperature	Furnace Run	Chemistry					
2-2-1	14.03% FIMA	4.72×10^{25} n/m^2	845°C	As-Irradiated			✓	✓	✓	✓	✓
1-5-9	9.29% FIMA	3.30×10^{25} n/m^2	1070°C	As-Irradiated			✓	✓	✓	✓	
4-1-3	14.06% FIMA	5.01×10^{25} n/m^2	786°C	1600°C FACS test, compact sent to ORNL			DL ✓	✓	✓	in progress	
3-6-3	14.77% FIMA	5.47×10^{25} n/m^2	1363°C	As-Irradiated			DL ✓	FY24	FY24	FY24	FY24
2-2-2	14.02% FIMA	4.72×10^{25} n/m^2	845°C	1600°C	✓	✓	✓	✓	✓	✓	✓
2-2-4	14.33% FIMA	4.94×10^{25} n/m^2	856°C	1600°C	✓	✓	✓	✓	✓	✓	in progress
2-3-2	14.36% FIMA	4.85×10^{25} n/m^2	874°C	1800°C	✓	in progress	DL ✓	✓	in progress		
5-5-3	7.64% FIMA	2.13×10^{25} n/m^2	773°C	1600°C	✓	✓	FY24	FY24	FY24	FY24	FY24
3-1-2	13.76% FIMA	5.48×10^{25} n/m^2	1193°C	1600°C	✓	in progress	DL ✓	in progress	FY24	FY24	FY24
5-2-2	8.82% FIMA	2.99×10^{25} n/m^2	789°C	1800°C	✓		FY24	FY24	FY24	FY24	FY24
5-6-2	6.75% FIMA	1.67×10^{25} n/m^2	634°C	1600°C	in progress		FY24	FY24	FY24	FY24	FY24
4-1-2	13.73% FIMA	4.78×10^{25} n/m^2	774°C	1600°C			FY24	FY24	FY24	FY24	FY24

FIMA = fissions per initial metal (U) atom   TAVA = time-average volume-average   ✓ = completed   some work planned for FY24 may be pulled forward if time and funding allow

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

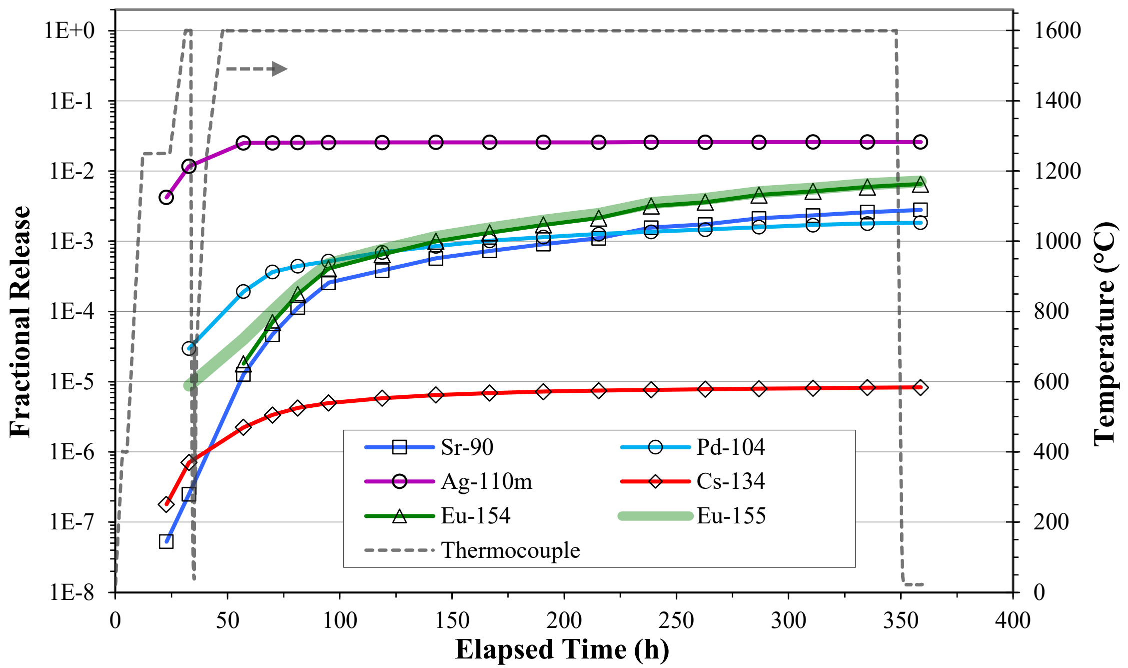
- Low Cs indicates no failed particles.
- Safety test releases include contribution from matrix inventory at end of irradiation and particle releases.
- Matrix inventory affected by irradiation temperature and nuclide time at temperature.

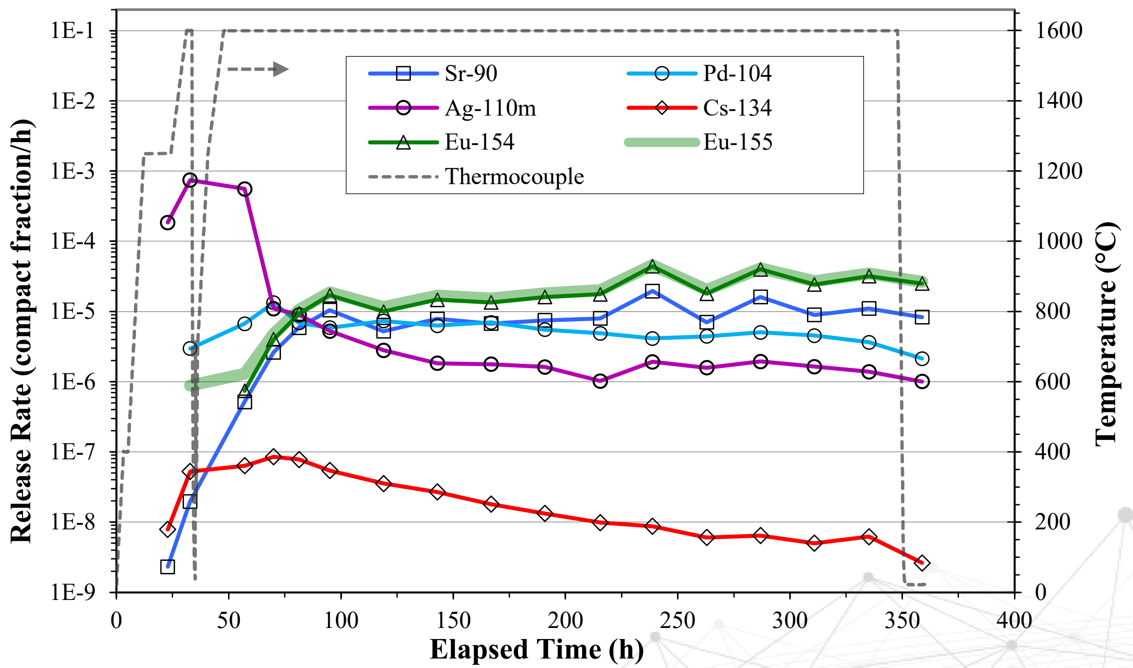
Compact	^{90}Sr	^{104}Pd	^{110m}Ag	^{134}Cs	^{154}Eu
AGR-5/6/7 2-2-2 (1600°C) (845°C TAVA, 14.0% FIMA)	2.8E-3	1.8E-3	2.6E-2	8.3E-6	6.5E-3
	(6.3)	(4.2)	(59)	(0.019)	(15)
AGR-5/6/7 2-2-4 (1600°C) (856°C TAVA, 14.3% FIMA)	3.0E-3	9.4E-4	2.1E-2	7.2E-6	6.9E-3
	(6.8)	(2.1)	(48)	(0.016)	(16)
AGR-5/6/7 5-5-3 (1600°C) (773°C TAVA, 7.6% FIMA)	8.3E-6	4.8E-4	1.8E-3	1.3E-5	6.6E-5
	(0.028)	(1.6)	(6.2)	(0.045)	(0.22)
AGR-5/6/7 2-3-2 (1800°C) (874°C TAVA, 14.4% FIMA)	TBD	TBD	~9.6E-3	~1.0E-3	>8.8E-2
	TBD	TBD	~(22)	~(2.3)	>(199)
AGR-5/6/7 3-1-2 (1600°C) (1193°C TAVA, 13.8% FIMA)	TBD	TBD	~5.1E-3	~4.4E-4	TBD
	TBD	TBD	~(12)	~(1.0)	TBD
AGR-5/6/7 5-2-2 (1800°C) (789°C TAVA, 8.8% FIMA)	TBD	TBD	~3.5E-1	~4.8E-4	TBD
	TBD	TBD	~(1180)	~(1.6)	TBD

Values are presented as compact fraction and particle-equivalent (in parentheses)
Estimated uncertainty in solute analysis is ±10% and less than values for ^{110m}Ag are estimated from first leach.
Some analyses are in progress: TBD = data not yet available; values with preliminary efficiencies presented as approximate or greater than

1600°C Safety Test of Compact 2-2-2
(14.0% FIMA, 845°C TAVA Temperature)

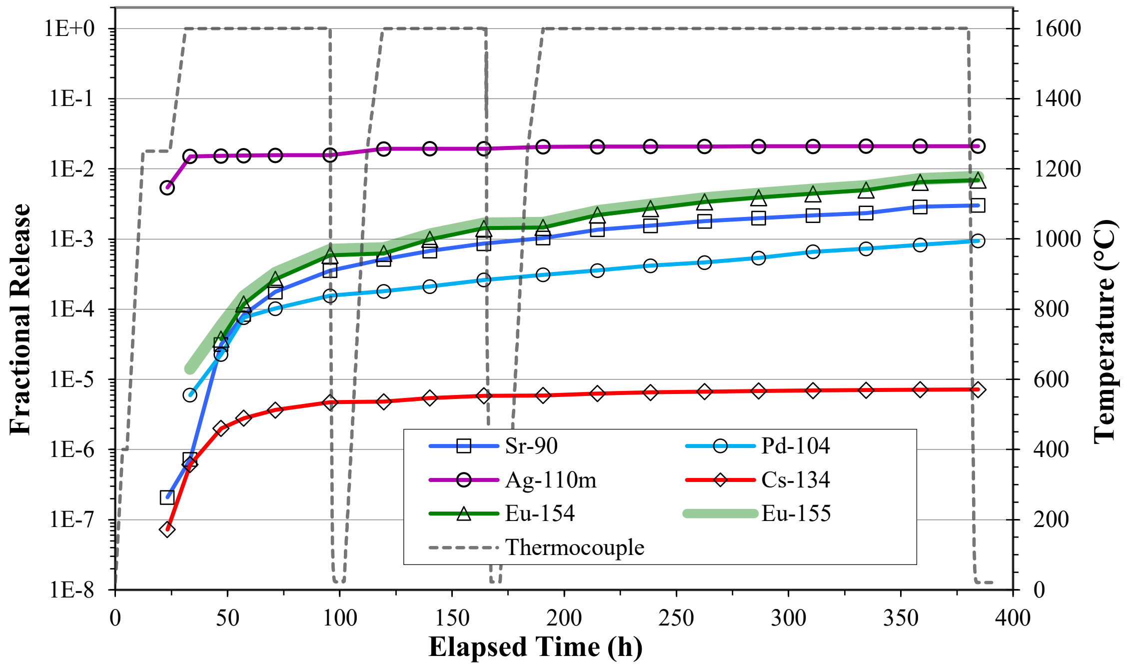
- Unplanned interruption after 2.5 h @ 1600°C because of a power outage.
- No indication of TRISO failure — ^{85}Kr below MDA of 5.6E-7 (<0.0013 particle equivalents).
- No indication of SiC failure — ^{134}Cs release <0.019 particle equivalents.
- ^{110m}Ag release dominated by early release of matrix inventory.
- ^{154}Eu, ^{90}Sr, and ^{104}Pd had typical rate trends related to slow transport to cups and some diffusive release from particles.

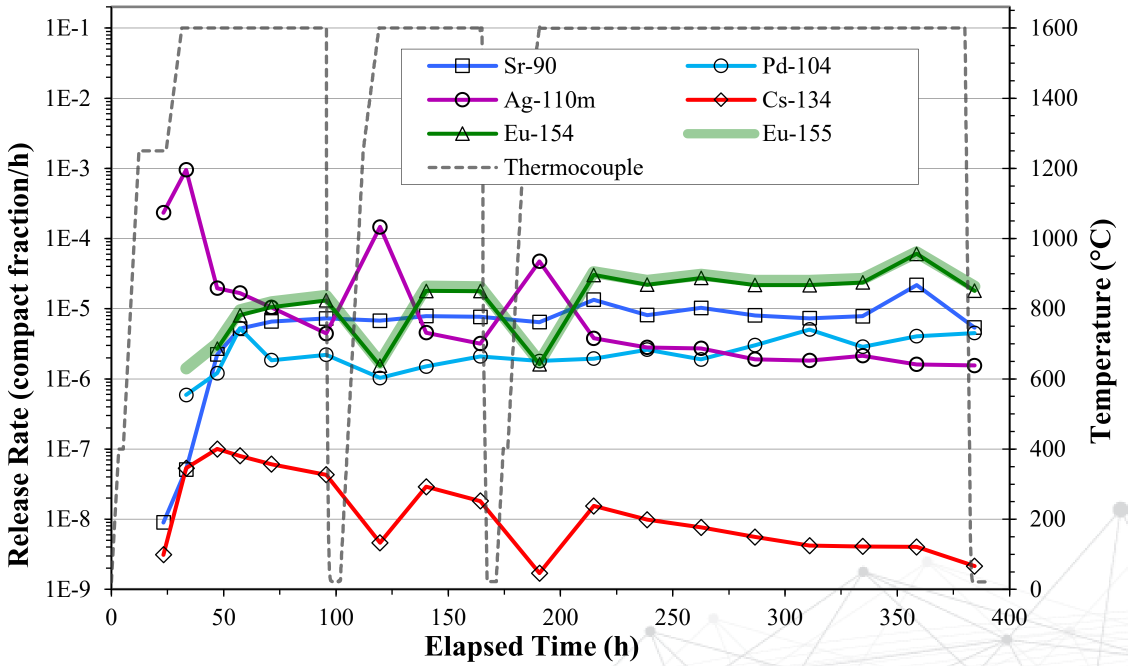




1600°C Safety Test of Compact 2-2-4
(14.3% FIMA, 856°C TAVA Temperature)

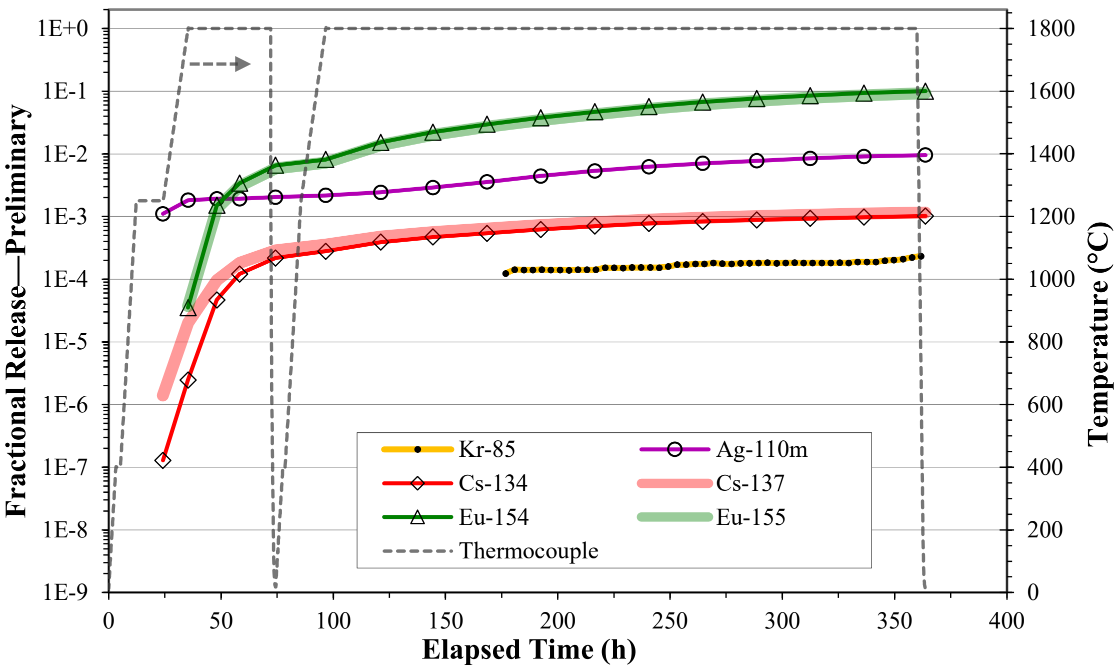
- Two unplanned shutdowns mid-test due to controller bugs.
- No indication of TRISO failure — ^{85}Kr below MDA of 5.5E-7 (<0.0013 particle equivalents).
- No indication of SiC failure — ^{134}Cs release of <0.016 particle equivalents.
- ^{110m}Ag, ^{154}Eu, ^{90}Sr, and ^{104}Pd similar to Compact 2-2-2.

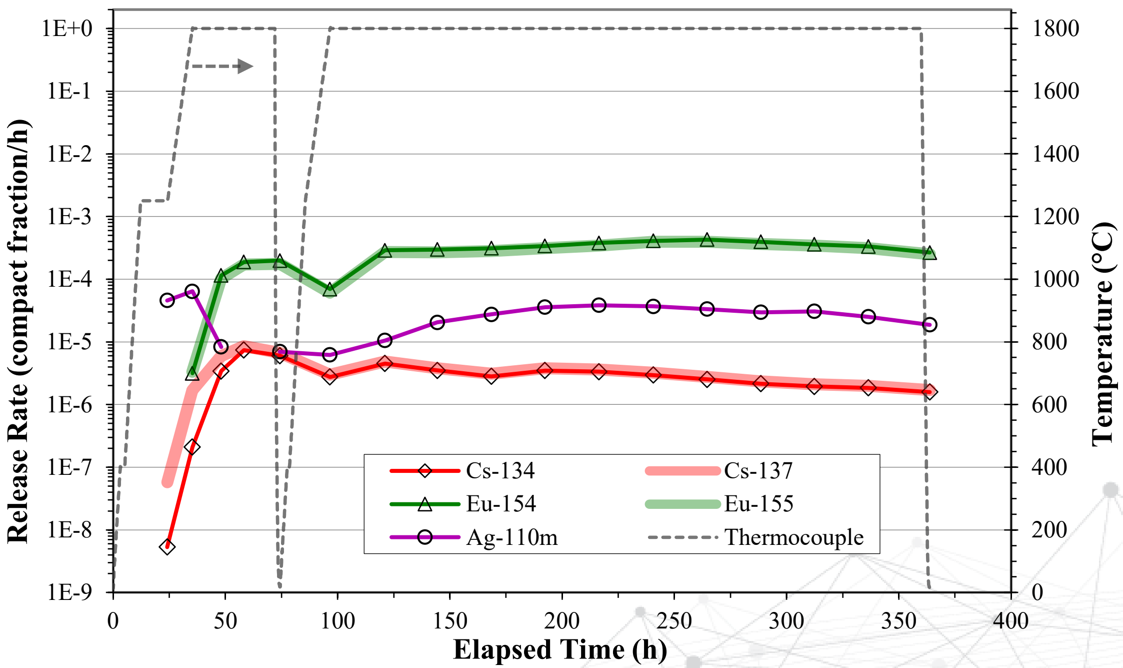




1800°C Safety Test of Compact 2-3-2
(14.4% FIMA, 874°C TAVA Temperature)

- Computer data acquisition card failure required pause in heating test.
- ^{85}Kr accumulation of 2.4E-4 (0.53 particle equivalents) indicative of one particles with degraded or defective TRISO.
- ^{134}Cs release of roughly 2-1/2 particle equivalents indicates particles with degraded or defective SiC.
- ^{154}Eu had typical rate trend with total release of ~10% or more.
- Initially low ^{110m}Ag release was probably related to a low inventory outside the SiC at the end of the safety test.

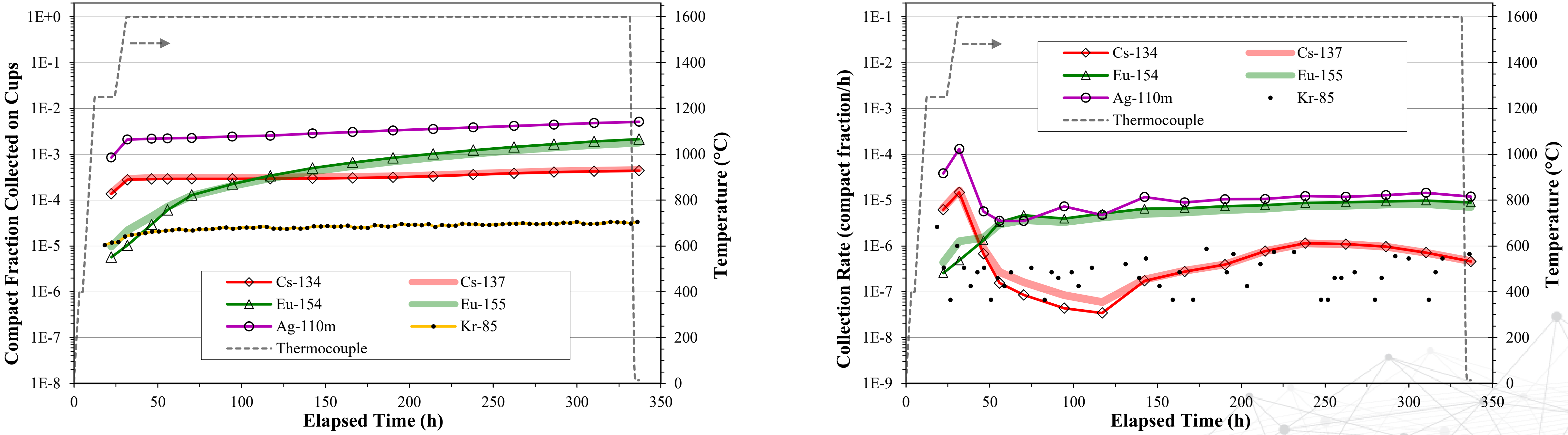




Preliminary: no efficiency correction for inventory on Ta internals

1600°C Safety Test of Compact 3-1-2
(13.8% FIMA, 1193°C TAVA Temperature)

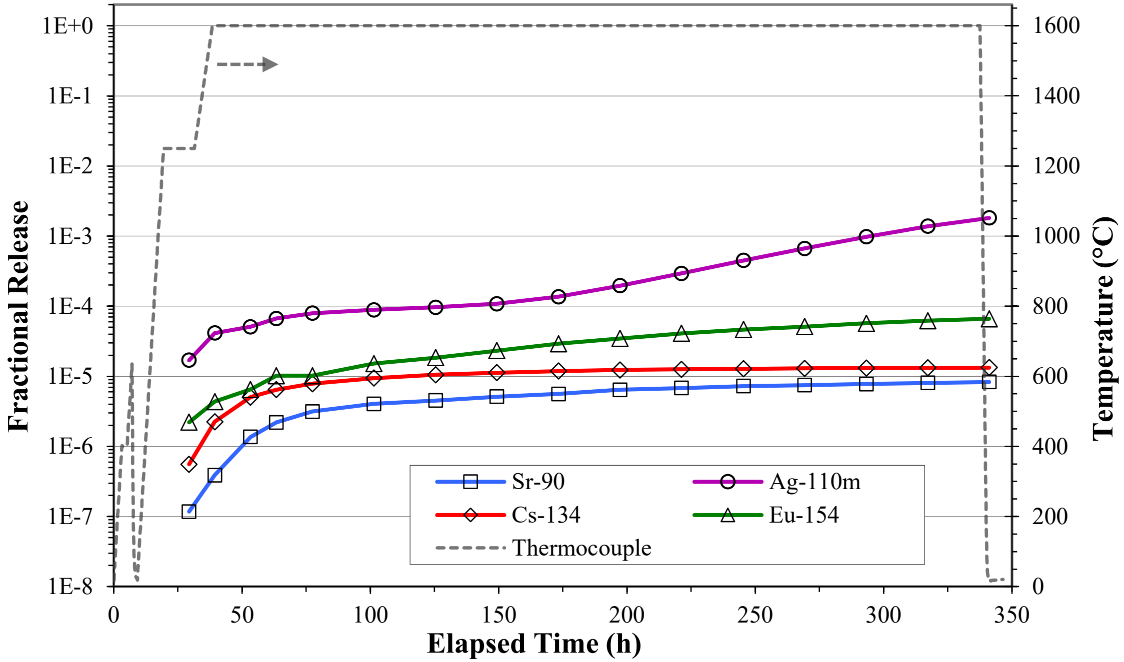
- ^{85}Kr accumulation of 3.4E-5 (0.076 particle equivalents) is higher than normal but low for failed TRISO.
- ^{134}Cs release in the neighborhood of one particle equivalent indicates one or two particles with degraded or defective SiC.
- ^{154}Eu had typical rate trend with total release not yet determined but likely similar to Compact 2-2-2 and Compact 2-2-4 safety tests.
- ^{110m}Ag showed initial release from matrix of roughly 5 particle equivalents plus a steady release throughout test of roughly 5 more that cannot be accounted for by failed or defective particles and is usually not resolvable at 1600°C.

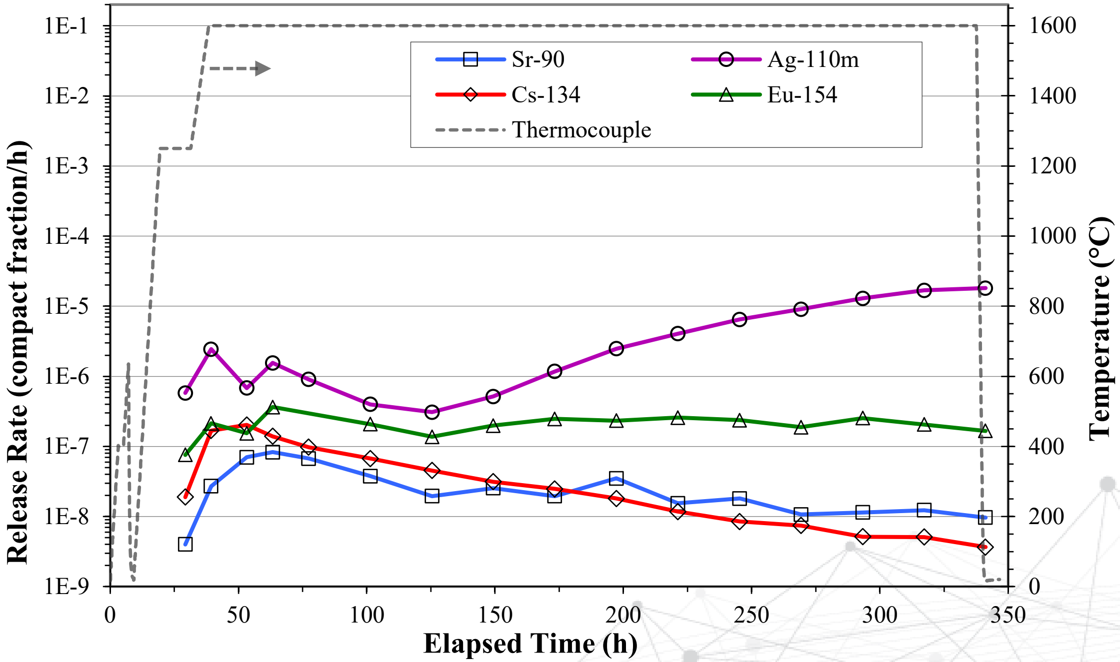


Preliminary: no efficiency correction

1600°C Safety Test of Compact 5-5-3
(7.6% FIMA, 773°C TAVA Temperature)

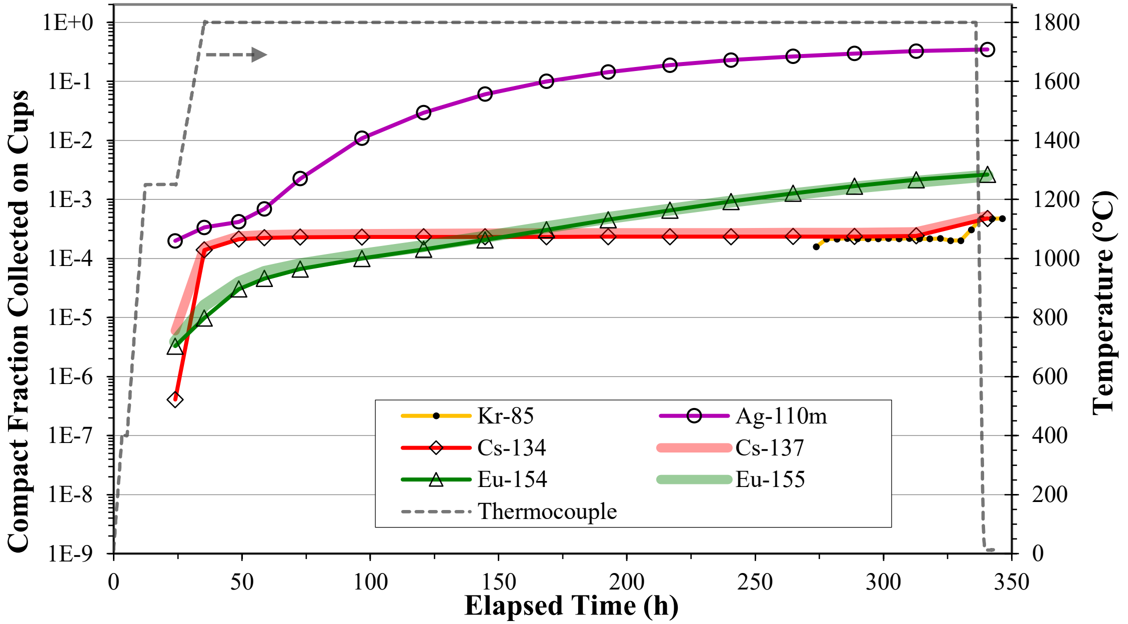
- Inconsequential shutdown during initial heat up due to cooling water interruption.
- No indication of TRISO failure — ^{85}Kr below MDA of 6.6E-7 (<0.0023 particle equivalents).
- No indication of SiC failure — measured ^{134}Cs was <0.045 particle equivalents.
- ^{154}Eu and ^{90}Sr had typical rate trends with low total releases consistent with low-burnup.
- ^{110m}Ag release rate increased with time after ~100 h at 1600°C due to diffusion through SiC. This is normally unresolvable during 1600°C safety tests because total ^{110m}Ag release is usually dominated by ^{110m}Ag in the matrix at the start of test.

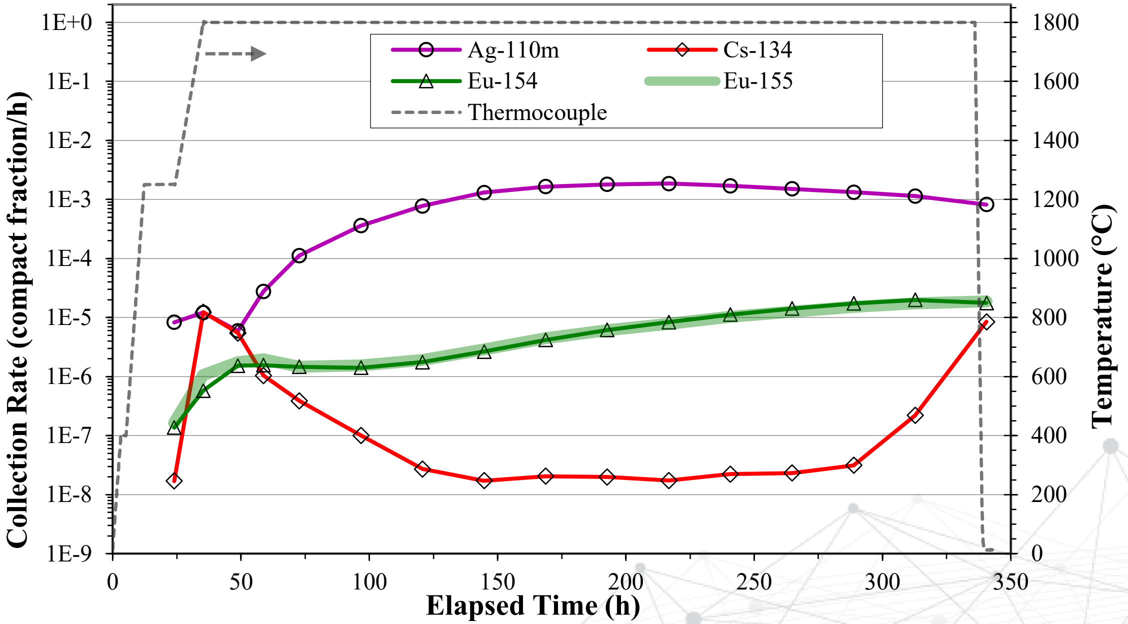




1800°C Safety Test of Compact 5-2-2
(8.8% FIMA, 789°C TAVA Temperature)

- ^{85}Kr accumulation of 4.8E-4 (1.1 particle equivalents) indicative of degraded or defective TRISO.
- Initial ^{134}Cs release in the neighborhood 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.
- Initial ^{110m}Ag and ^{154}Eu release significantly lower than AGR-5/6/7 Compact 2-3-2 at 1800°C is consistent with lower burnup.
- Cumulative ^{110m}Ag release of 30% 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.

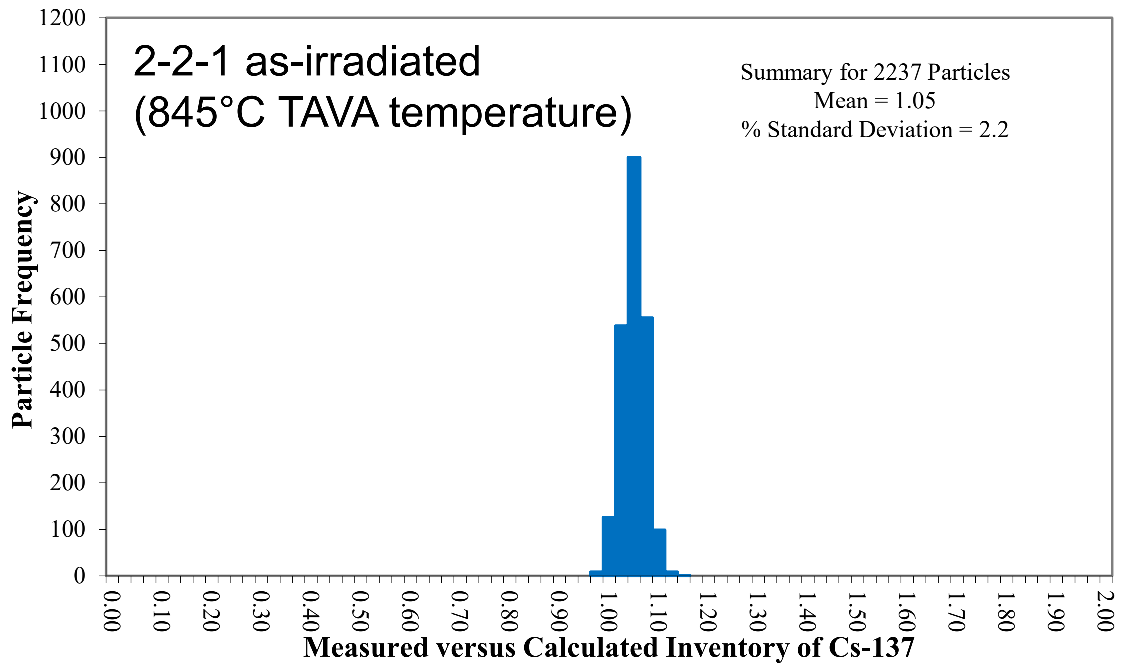


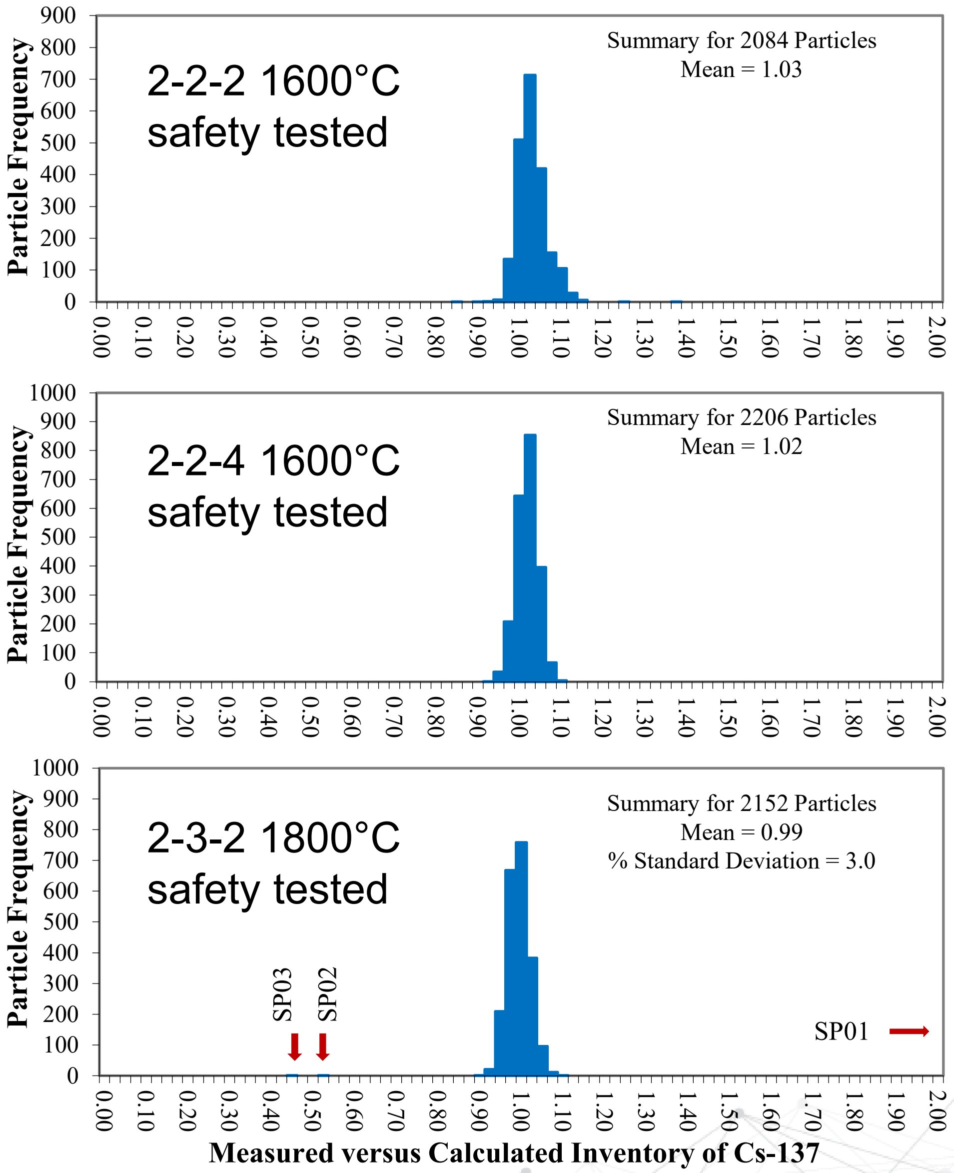


Preliminary: no efficiency correction for inventory on Ta internals

Capsule 2 IMGA Results—^{137}Cs

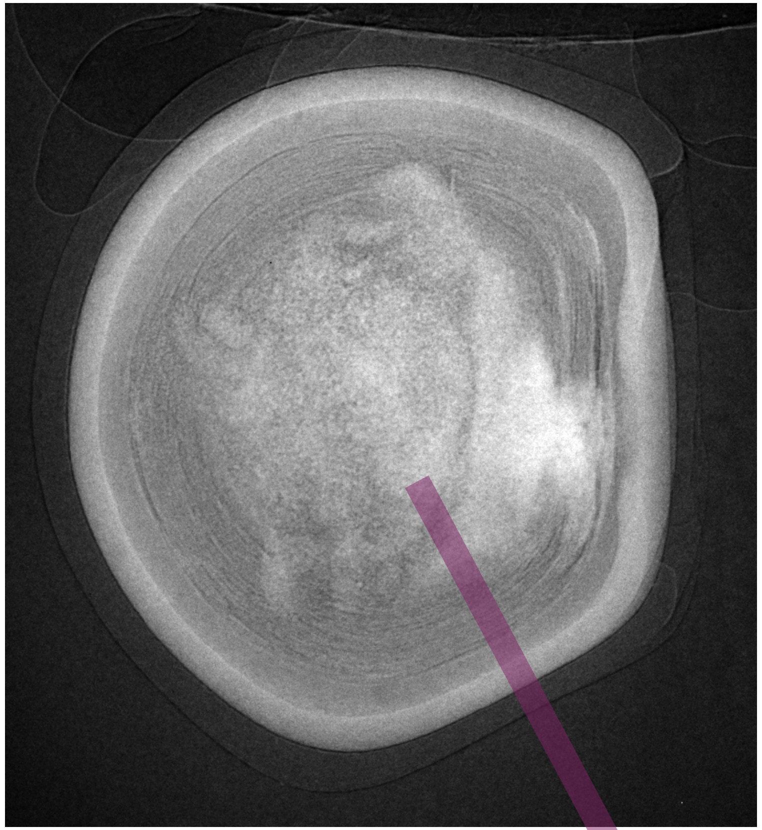
- No significant Cs release from as-irradiated Compact 2-2-1 particles
- No significant Cs release from 1600°C safety tested particles
- One low ^{144}Ce (SP01) and two low ^{137}Cs particles from 1800°C safety test of Compact 2-3-2 (queued for analysis)

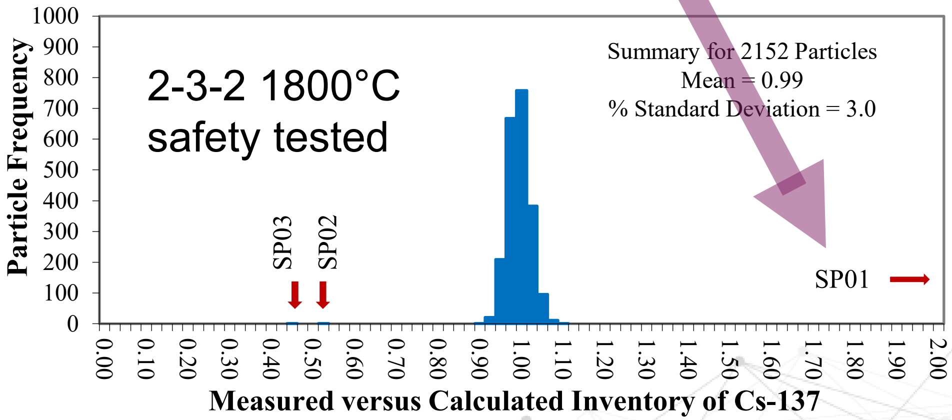




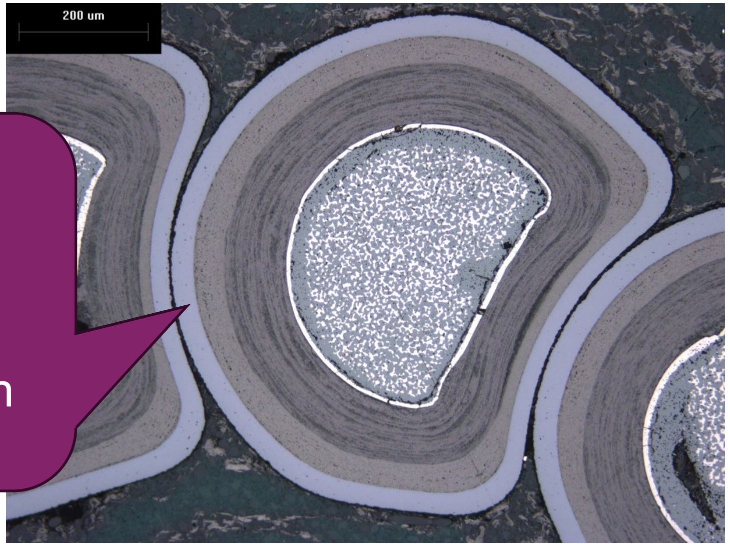
Compact 2-2-3 Particle SP01 had an As-Fabricated Defect

- No significant Cs release from as-irradiated Compact 2-2-1 particles
- No significant Cs release from 1600°C safety tested particles
- One low ^{144}Ce (SP01) and two low ^{137}Cs particles from 1800°C safety test of Compact 2-3-2 (queued for analysis)
- Defective, dimpled coating related to a faceted kernel.



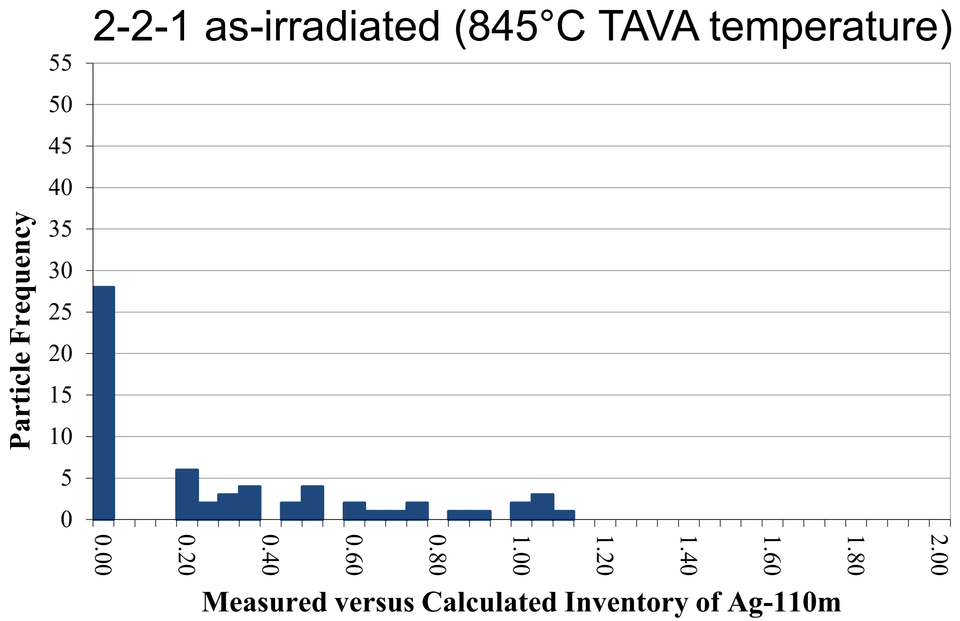


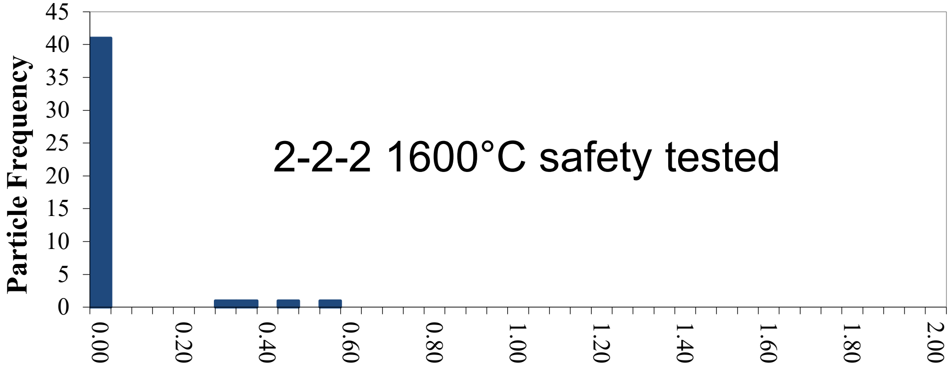
Similar structure observed in AGR-5/6/7 development batches due to kernel fissuring and fragmentation

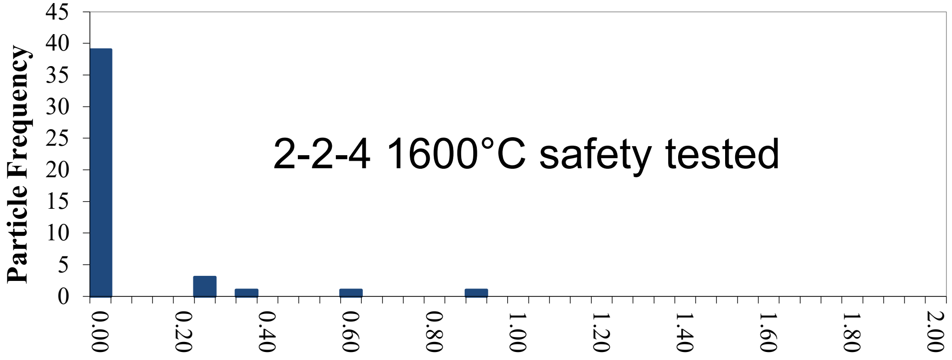


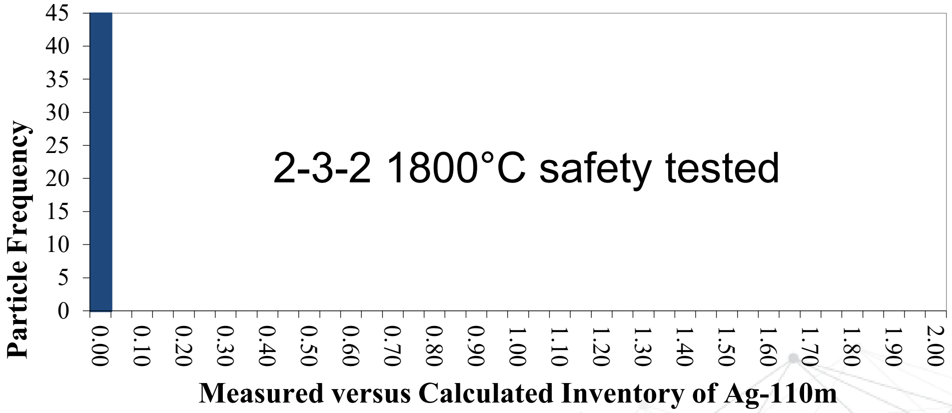
Capsule 2 IMGA Results—^{110m}Ag

- Relatively low silver retention in as-irradiated Compact 2-2-1 particles
- Particles apparently losing silver at 1600°C
- No measurable (<5%) silver retention after 1800°C



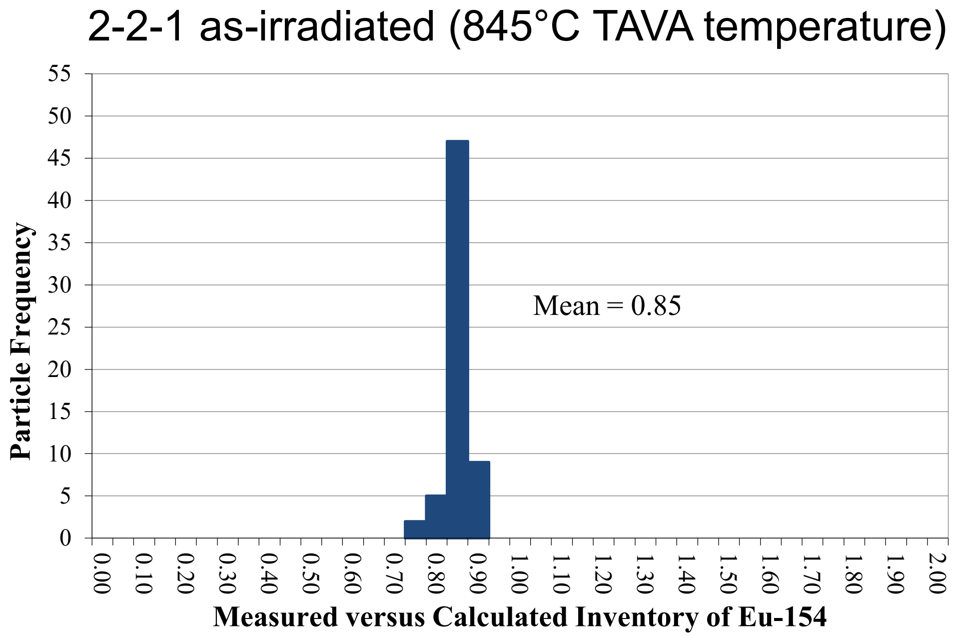


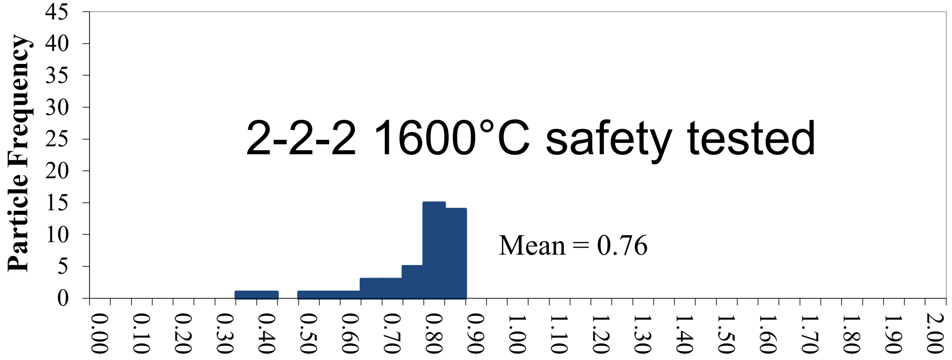


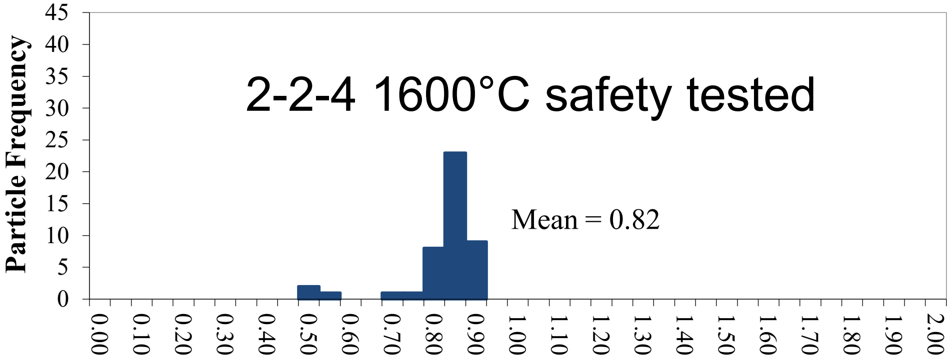


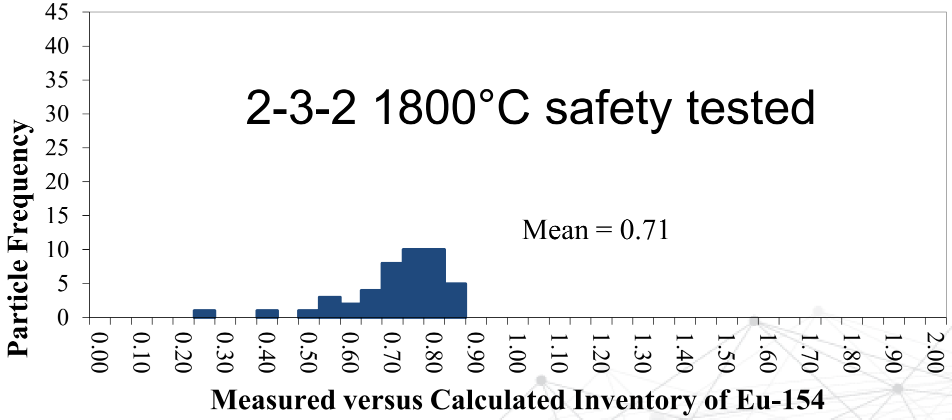
Capsule 2 IMGA Results—^{154}Eu

- High europium retention in as-irradiated Compact 2-2-1 particles
- Some particles losing significant ^{154}Eu at 1600°C
- General reduction in ^{154}Eu retention at 1800°C





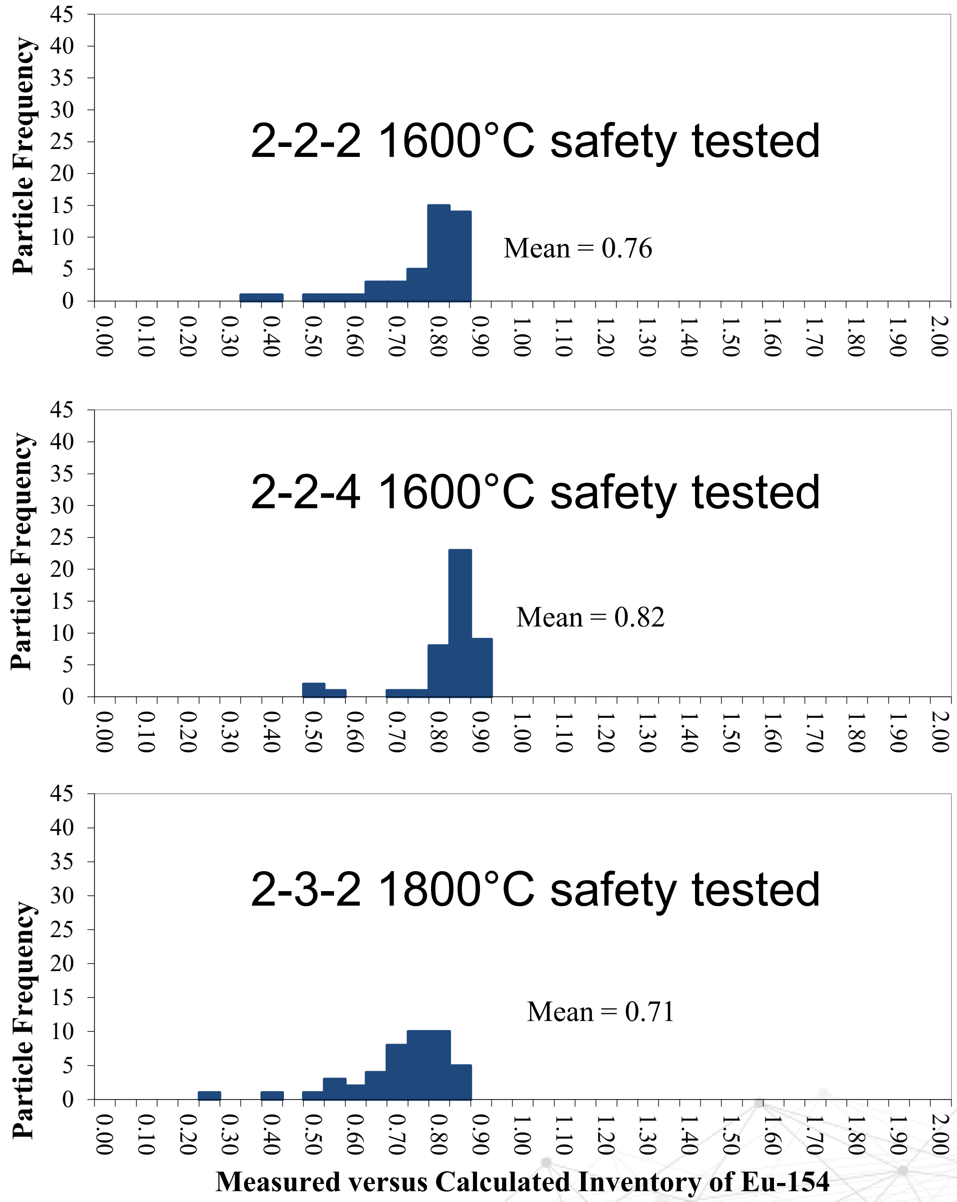




Capsule 2 IMGA Results—^{154}Eu

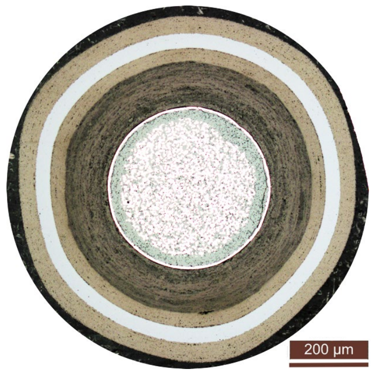
- High europium retention in as-irradiated Compact 2-2-1 particles
- Some particles losing significant ^{154}Eu at 1600°C
- General reduction in ^{154}Eu retention at 1800°C
- DLBL data show more ^{154}Eu in compact matrix after 1600°C safety tests of Compacts 2-2-2 and 2-2-4 compared with similar as-irradiated Compact 2-2-1, which experienced similar burnup and TAVA temperature. Same trends observed for several other nuclides indicate measurable diffusive release at 1600°C.

Compact	^{90}Sr	^{144}Ce	^{154}Eu	^{239}Pu
2-2-1 as-irradiated	6.0E-3	1.3E-3	1.2E-2	2.0E-4
	(14)	(2.9)	(27)	(0.44)
2-2-2 1600°C ST	5.4E-2	6.9E-3	8.1E-2	1.4E-3
	(121)	(16)	(183)	(3.2)
2-2-4 1600°C ST	2.1E-2	9.5E-3	3.2E-2	2.7E-3
	(48)	(22)	(72)	(6.1)
DLBL totals presented as compact fraction and particle-equivalents				

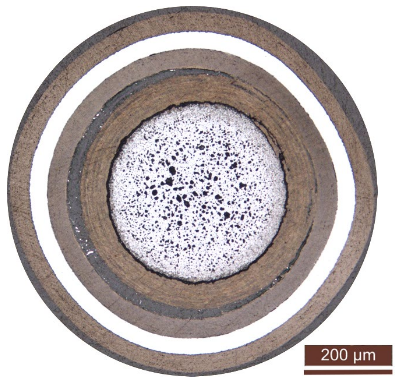


Buffer Shrinkage, Fracture, and Detachment from IPyC

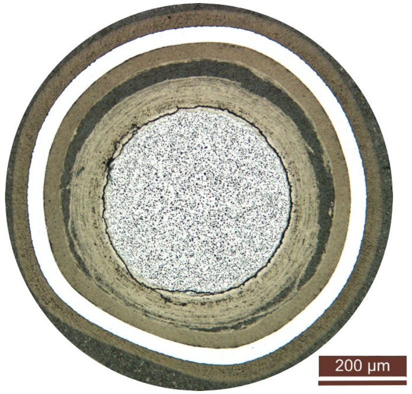
- The low-density buffer layer undergoes significant densification and shrinkage during irradiation that can cause fracture of the layer and detachment from the inner pyrolytic carbon (IPyC) layer.



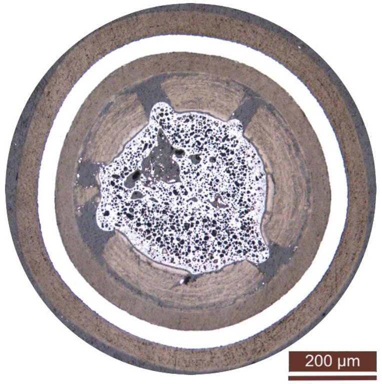
Unirradiated AGR-2 UCO-TRISO



AGR-2 Compact 2-2-3



AGR-2 Compact 6-2-3



AGR-2 Compact 5-2-3

Table 23. AGR-2 UCO compacts with quantified buffer fracture frequency used to

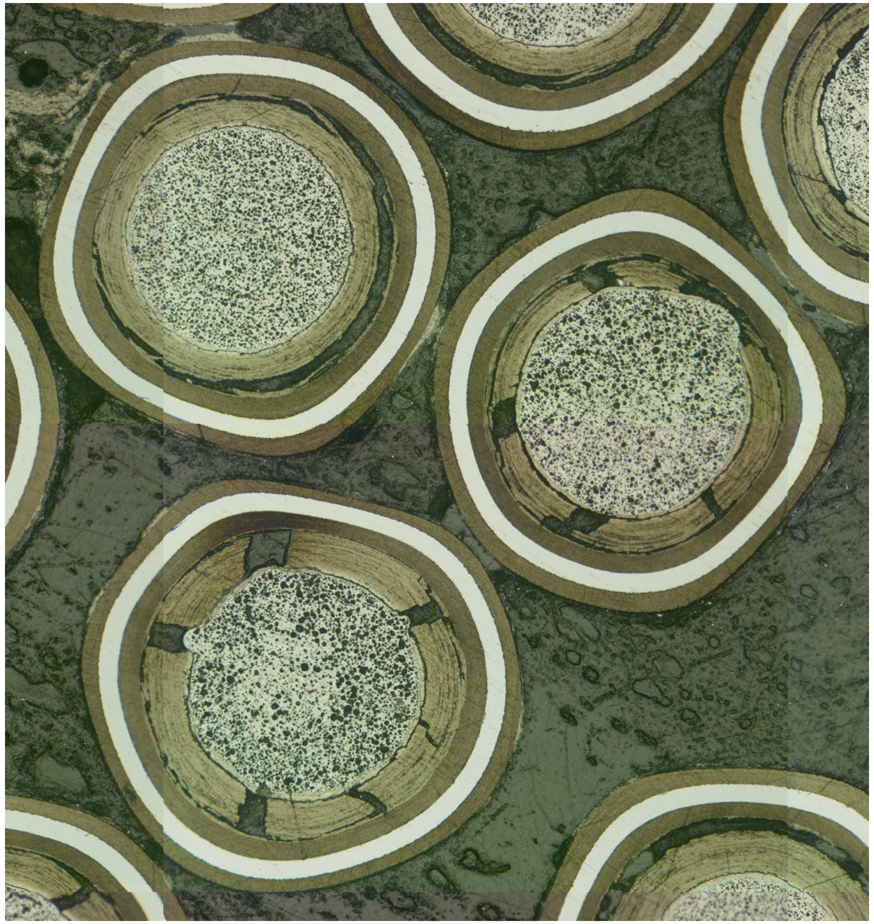
Compact [a]	Temperature (°C) [b]		Burnup [c] (% FIMA)	Fast Fluence [c] (10^{25} n/m^2)	Buffer Fracture
	TAVA	TA_{max}			
2-1-3	1194	1305	10.95	2.88	37 of 198 (18.7%)
2-2-1	1287	1353	12.47	3.35	0 of 44 (0.0%)
2-2-3	1261	1335	10.8	3.00	1 of 74 (1.4%)
2-3-1 (1600°C)	1296	1360	12.63	3.42	0 of 54 (0.0%)
2-3-2 (1800°C)	1296	1360	12.62	3.46	1 of 37 (2.7%)
2-4-3	1216	1324	11.52	3.08	3 of 159 (1.9%)
5-1-3	1078	1177	11.09	3.03	70 of 181 (38.7%)
5-2-3	1108	1184	10.4	3.00	76 of 88 (86.4%)
5-3-3	1093	1172	10.1	2.90	37 of 43 (86.0%)
5-4-2	1071	1168	12.03	3.14	27 of 35 (77.1%)
6-2-3	1095	1157	8.22	2.30	7 of 44 (15.9%)
6-3-3	1060	1134	7.50	2.10	0 of 44 (0.0%)
6-4-2 (1600°C)	1018	1106	9.26	2.21	13 of 48 (27.1%)

from AGR-2 Final Report INL/EXT-21-64279

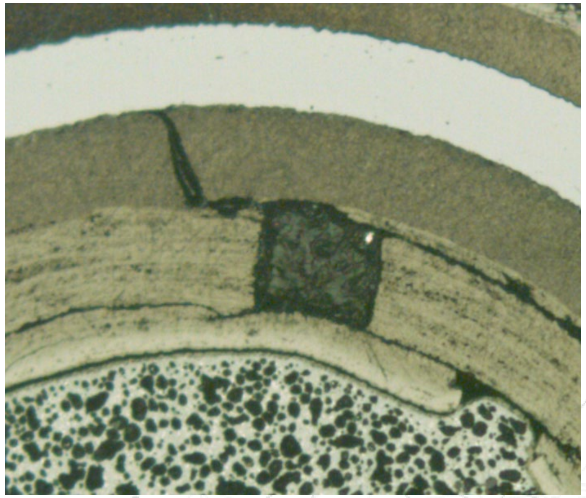
As-Irradiated Compact 2-2-1 Random Sample

As-Irradiated Compact 2-2-1 Random Sample

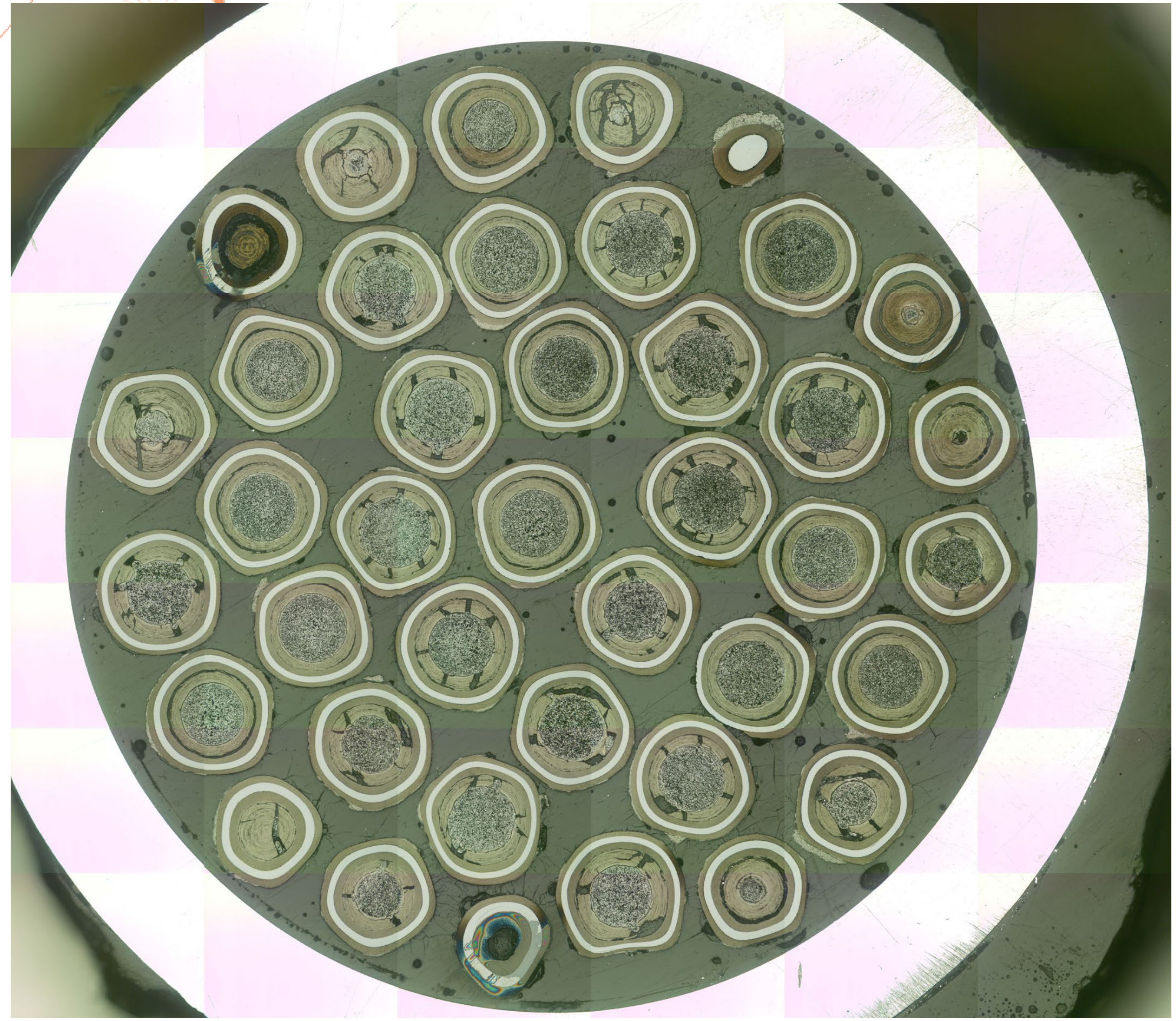
40 out of 76 with Buffer fracture



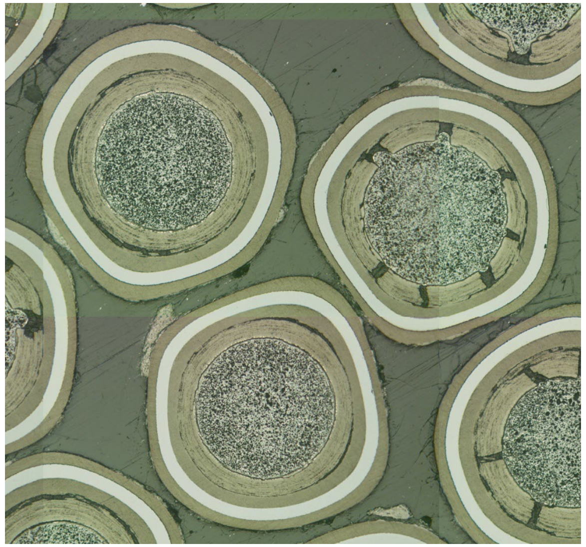
5 out of 76 with IPyC fracture



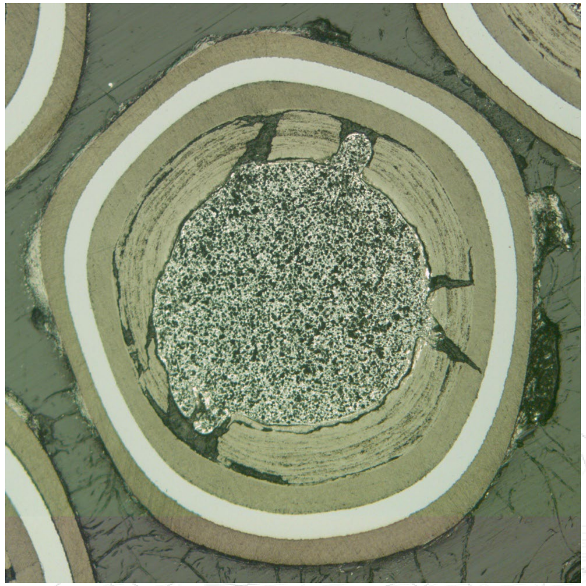
1600°C Safety-Tested Compact 2-2-2 Random Sample



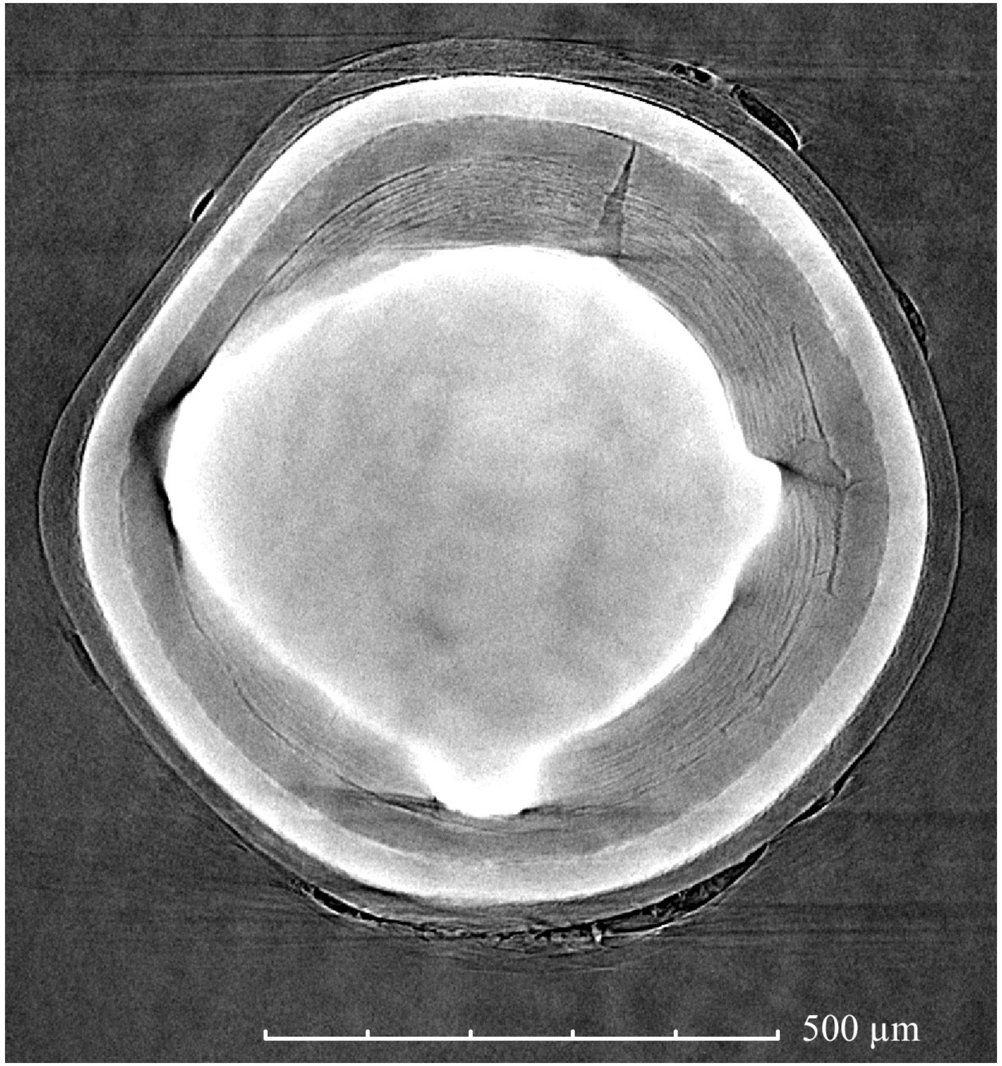
21 out of 33 with Buffer fracture



1 IPyC fracture



1600°C Safety-Tested Compact 2-2-4 Random XCT Sample



Randomly-selected particle from Compact 2-2-4

Arrowhead fracture from buffer to IPyC
with some separation at IPyC/SiC interface

Summary of First Five Quarters of AGR-5/6/7 PIE at ORNL

- AGR-5/6/7 PIE at ORNL has been in progress since April 2022.
  - Three as-irradiated compacts are in various stages of destructive PIE.
  - Seven 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 have already been 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 also 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.

Thank you for your attention

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