AGR-2 PIE and Safety Testing

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Advanced Gas Reactor
TRISO Fuels Program Review

Idaho Falls, Idaho
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Outline

- Overview of AGR-2 Post-Irradiation Examination (PIE) at ORNL
  - Compacts under analysis
  - Current status of PIE
- PIE of as-irradiated compacts
  - Update on detection and analysis of UCO-TRISO particles that failed during irradiation
    - Reported in detail at June 2016 annual review
  - General observations on buffer fracture
  - Ag, Eu, and Sr retention in UCO and UO_2 fuel particles
- PIE on safety-tested compacts
  - Summary results
  - CO corrosion of SiC in UO_2 fuel during safety testing
  - First look at SiC failure in AGR-2 UCO — Compact 5-4-1 after 1800°C safety test
  - Bob Morris to present details of fission product release during safety testing
- Summary

Overview of AGR-2 PIE at ORNL

- AGR-2 PIE at ORNL has been in progress for two+ years (May 2015-July 2017).
  - 24 AGR-2 compacts are now at ORNL (including four from PBMR Capsule 4).
- PIE and safety testing have been completed or are in progress on 18 AGR-2 US compacts.
  - 8 as-irradiated compacts
  - 10 safety-tested compacts with post-safety test PIE
- Additional AGR-2 PIE has been performed at INL
  - Precision Gamma Scanner (PGS)
    - of test train
    - of compacts (Ag retention)
    - of graphite holders (mapped distributions of Cs, Eu, Ag)
  - Ceramography
    - four compacts were sectioned and polished
  - Leach-Burn-Leach (LBL) and particle gamma scans
    - one compact

Transferring shielded pigs into ORNL hot cell

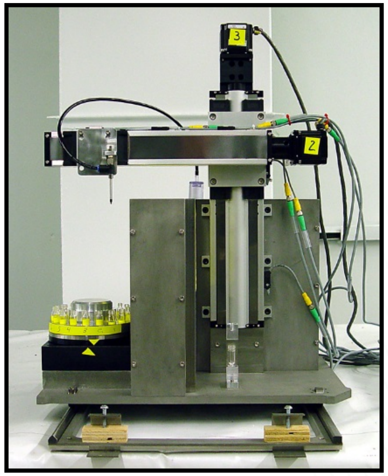
AGR-2 Compact Irradiation Conditions

As-irradiated Compacts

Compact ID	ORNL Fabrication ID	Fuel Type	Average Burnup	Fast Fluence	TAVA Temp.	TAM Temp.
AGR-2 2-2-1	LEU09-OP2-Z126	UCO	12.47% FIMA	3.35×10^{25} n/m^2	1287°C	1353°C
AGR-2 2-2-3	LEU09-OP2-Z092	UCO	10.80% FIMA	2.99×10^{25} n/m^2	1261°C	1335°C
AGR-2 5-2-3	LEU09-OP2-Z062	UCO	10.42% FIMA	3.00×10^{25} n/m^2	1108°C	1184°C
AGR-2 5-3-3	LEU09-OP2-Z040	UCO	10.07% FIMA	2.91×10^{25} n/m^2	1093°C	1172°C
AGR-2 5-4-2	LEU09-OP2-Z059	UCO	12.03% FIMA	3.14×10^{25} n/m^2	1071°C	1168°C
AGR-2 6-2-3	LEU09-OP2-Z104	UCO	8.22% FIMA	2.30×10^{25} n/m^2	1095°C	1157°C
AGR-2 6-3-3	LEU09-OP2-Z085	UCO	7.46% FIMA	2.14×10^{25} n/m^2	1060°C	1134°C
AGR-2 3-3-1	LEU11-OP2-Z106	UO_2	10.46% FIMA	3.49×10^{25} n/m^2	1062°C	1104°C

Time-average, volume-average (TAVA) and time-average maximum (TAM) irradiation temperature from (Hawkes, INL/ECAR-2476)
Fission per initial metal atom (FIMA) from daily depletion calculation (Sterbentz, INL/ECAR-2066)

Soxhlet extractor for leach-burn-leach (LBL)



Irradiated Microsphere Gamma Analyzer (IMGA)

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AGR-2 Compact Irradiation Conditions

Safety-tested Compacts

Compact ID	ORNL Fabrication ID	Fuel Type	Average Burnup	Fast Fluence	TAVA Temp.	TAM Temp.
AGR-2 3-3-2	LEU11-OP2-Z034	UO_2	10.54% FIMA	3.53×10^{25} n/m^2	1062°C	1105°C
AGR-2 3-4-1	LEU11-OP2-Z188	UO_2	10.62% FIMA	3.47×10^{25} n/m^2	1013°C	1085°C
AGR-2 3-4-2	LEU11-OP2-Z150	UO_2	10.69% FIMA	3.50×10^{25} n/m^2	1013°C	1085°C
AGR-2 2-1-2	LEU09-OP2-Z079	UCO	12.62% FIMA	3.25×10^{25} n/m^2	1219°C	1324°C
AGR-2 2-2-2	LEU09-OP2-Z075	UCO	12.55% FIMA	3.39×10^{25} n/m^2	1287°C	1354°C
AGR-2 2-3-1	LEU09-OP2-Z125	UCO	12.63% FIMA	3.42×10^{25} n/m^2	1296°C	1360°C
AGR-2 2-3-2	LEU09-OP2-Z066	UCO	12.68% FIMA	3.46×10^{25} n/m^2	1296°C	1360°C
AGR-2 5-2-2	LEU09-OP2-Z128	UCO	12.34% FIMA	3.39×10^{25} n/m^2	1141°C	1210°C
AGR-2 5-4-1	LEU09-OP2-Z028	UCO	12.05% FIMA	3.12×10^{25} n/m^2	1071°C	1168°C
AGR-2 6-4-2	LEU09-OP2-Z049	UCO	9.26% FIMA	2.21×10^{25} n/m^2	1018°C	1106°C

Time-average, volume-average (TAVA) and time-average maximum (TAM) irradiation temperature from (Hawkes, INL/ECAR-2476)
Fission per initial metal atom (FIMA) from daily depletion calculation (Sterbentz, INL/ECAR-2066)

Core Conduction Cooldown Test Facility (CCCTF)

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Standard ORNL PIE Process

- Receive and inspect compacts
- Perform safety test in 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) the matrix and particles
  - BL matrix from sieving step separately
  - BL 90% of the particles after IMGA survey (save 10% unburned TRISO as an archive)
- Gamma scan particles with Irradiated Microsphere Gamma Analyzer (IMGA)
  - ≤100-second quick survey of all particles to find low-Ce and low-Cs particles
  - 4–6-hour scans to measure particle inventories (^{106}Ru, ^{110m}Ag, ^{125}Sb, ^{134}Cs, ^{137}Cs, ^{144}Ce, ^{154}Eu)
- 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 in x-ray
  - random midplane cross sections of particles with varied inventories
  - scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) to obtain microstructural and elemental information

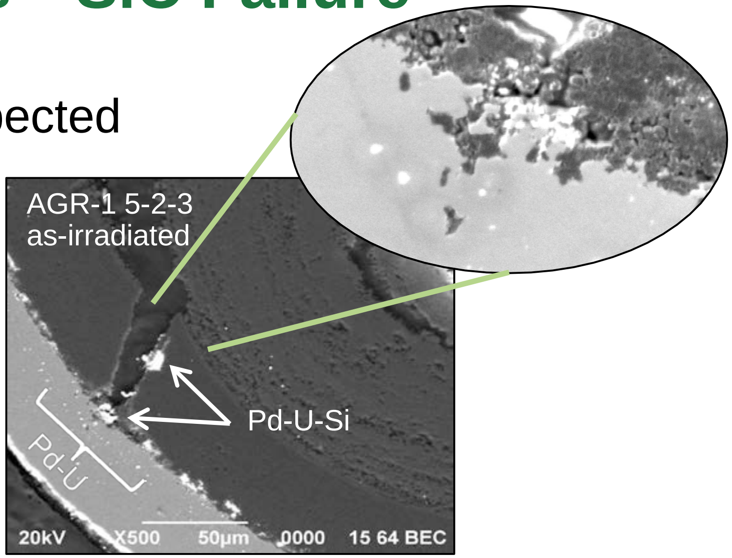
Status of AGR-2 PIE at ORNL

AGR-2 Compact ID	Safety Tested or As-Irradiated	DLBL	IMGA	X-ray	Optical Microscopy	SEM
2-2-3	as-irradiated	complete	complete	complete	complete	complete
5-2-3	as-irradiated	complete	complete	complete	complete	complete
3-3-1	as-irradiated	complete	complete	complete	complete	complete
5-3-3	as-irradiated	complete	complete	complete	complete	complete
6-3-3	as-irradiated	complete	complete	complete	complete	complete
5-4-2	as-irradiated	complete	complete	complete	complete	in progress
2-2-1	as-irradiated	in progress			FY18	FY18
6-2-3	as-irradiated	in progress		FY18	FY18	FY18
3-3-2	1600°C	complete	complete	complete	complete	complete
3-4-2	1600°C	complete	complete	complete	complete	complete
2-2-2	1600°C	complete	complete	complete	complete	complete
5-2-2	1600°C	complete	complete	complete	complete	complete
2-3-1	1600°C	complete	complete	complete	complete	complete
5-4-1	1800°C	complete	complete	complete	complete	complete
2-3-2	1800°C	complete	complete	in progress	in progress	
3-4-1	1700°C	DL complete				FY18
6-4-2	1600°C	complete	in progress			
2-1-2	1800°C	FY18	FY18	FY18	FY18	FY18

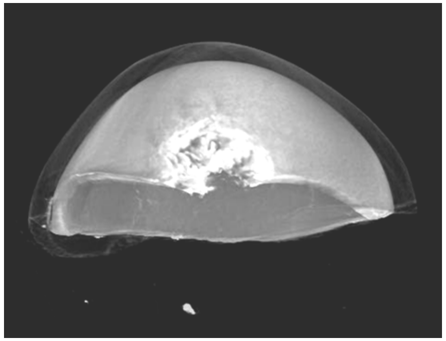
Red = in progress as of 7/18/17 (several safety tests in queue for cup/can leaching by Nuclear Analytical Chemistry)
Gray = FY18 scope

PIE of As-Irradiated AGR-2 Compacts—SiC Failure

- Initial compact PIE focused on compacts with suspected cesium release.
  - During AGR-1 PIE, compacts adjacent to Cs in the graphite sleeves were found to contain failed SiC.
  - Failure related to localized chemical degradation of SiC in particles whose IPyC layer was cracked by buffer shrinkage, exposing SiC to higher concentrations of Pd and U.



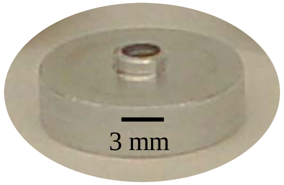
- AGR-2 Compact 2-2-3
  - One defective TRISO and three failed SiC were found by IMGA.
  - SiC corrosion was still localized, but over a much greater volume of the SiC layer.
  - The extensive SiC corrosion appeared to be related to Ni attack.
    - suspect failure of adjacent thermocouple
- AGR-2 Compact 5-2-3
  - Two kernels were leached during deconsolidation/leach.
  - A recovered SiC half-shell had extensive SiC corrosion.
    - Ni not detected in leached half-shell



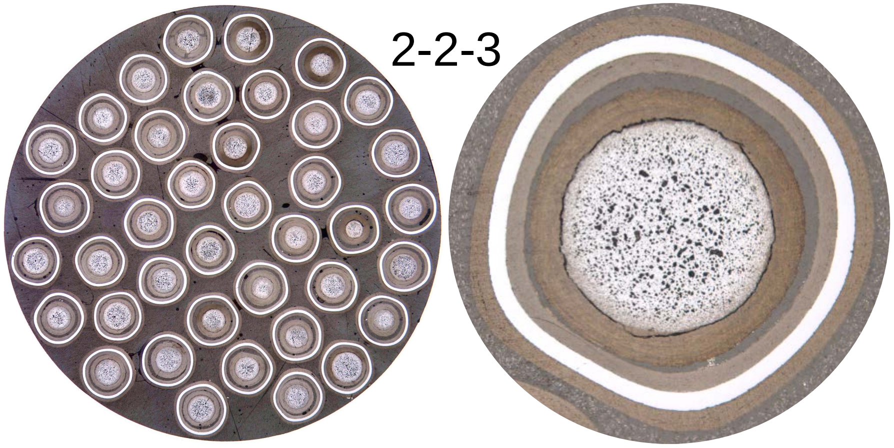
- AGR-2 Compacts 5-3-3 and 6-2-3
  - Cs in adjacent graphite sleeve, but no failed-SiC particles were recovered

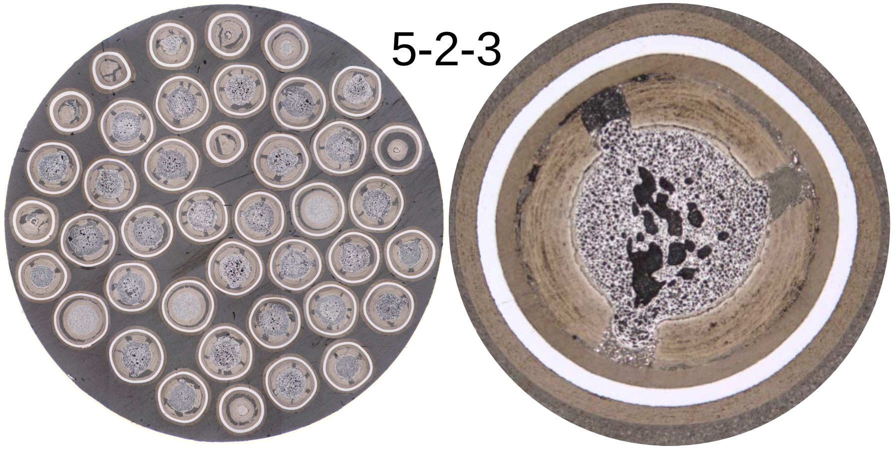
PIE of As-Irradiated AGR-2 Compacts—Buffer Fracture

- Particles polished near midplane using 40-particle Minimet mounts.
- Buffer fracture was sensitive to irradiation temperature.



Compact ID	Fuel Type	Irradiation Temp. (TAVA & TAM)	Average Burnup (FIMA)	Number of Imaged Particles	Number with Visible Buffer Fracture	Fraction with Visible Buffer Fracture
2-2-3	UCO	1261°C & 1353°C	10.8%	74	1	1.4%
5-2-3	UCO	1108°C & 1184°C	10.4%	88	76	86%
5-3-3	UCO	1093°C & 1172°C	10.1%	43	37	86%
6-3-3	UCO	1060°C & 1134°C	7.5%	44	0	0%
3-3-1	UO2	1062°C & 1104°C	10.5%	35	0	0%





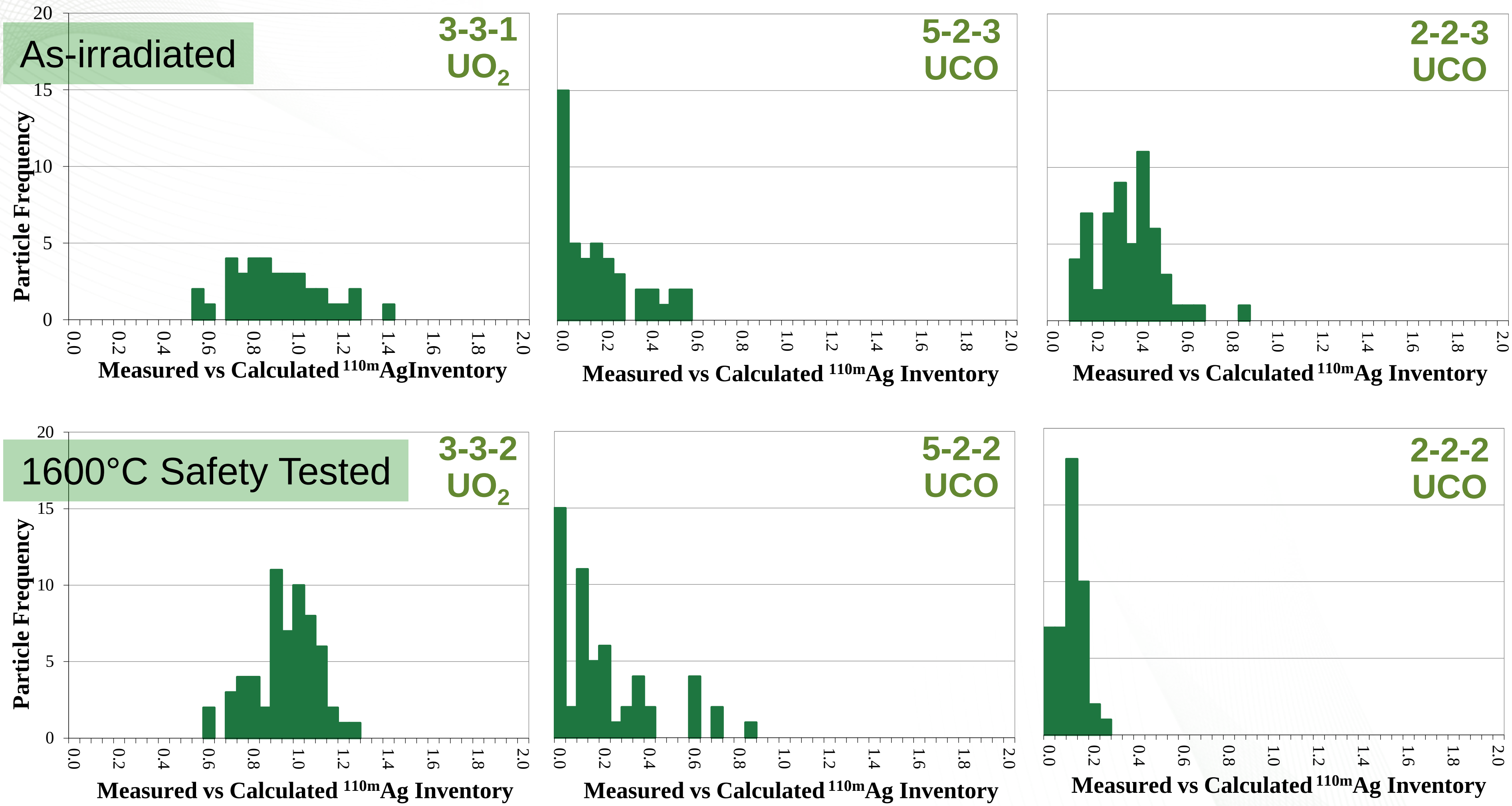
Silver (^{110m}Ag) Release

- PGS data compares well with IMGA+CCCTF+LBL.
- Average exposed ^{110m}Ag detected by LBL in Compact 542 was less than released in twin Compact 541 at 1800°C, indicating release through SiC.

Compact ID	Fuel Type	PGS	IMGA	Safety Test	LBL
5-3-3	UCO	23.3% (-1.0%, +1.9%)	23.3–27.0% (±1.0%)	-	<0.03%
5-2-3	UCO	17.0% (-1.3%, +2.3%)	15.1–17.5% (±0.7%)	-	0.05–0.07%
5-4-2	UCO	50.6% (-1.6%, +4.2%)	56–61% (±2%)	-	0.5–0.5%
5-2-2	UCO	21.8% (-1.1%, +3.0%)	19.8–21.8% (±0.7%)	1.6% (±0.2) at 1600°C	<0.03%
5-4-1	UCO	85.7% (-2.1%, +2.1%)	66.7–67.2% (±1.2%)	17.3% (±2%) at 1800°C	<0.05%
2-2-3	UCO	30.1% (-1.1%, +1.2%)	32.9% (±0.5%)	-	0.06–0.13%
2-2-2	UCO	12.7% (-1.0%, +3.0%)	9.9–10.8% (±0.5%)	0.7% (±0.2%) at 1600°C	<0.2%
2-3-1	UCO	16.0% (-1.1%, +1.7%)	6.0–13.2% (±1.2%)	~2% at 1600°C	<0.5%
3-3-2	UO_2	94.2% (-1.1%, +1.1%)	94.1% (±0.4%)	1.7% (±0.4%) at 1600°C	0.04–0.05%
3-4-2	UO_2	115.6% (-1.1%, +1.1%)	110.8% (±0.5%)	1.1% (±0.3%) at 1600°C	0.5–0.6%
3-3-1	UO_2	94.1% (-1.0%, +1.0%)	90.6% (±1.1%)	-	0.2–0.2%

PGS data from Harp, INL-LTD-15-36599
IMGA data based on 45–60-particle sample

TRISO Particle Silver Retention



UO$_2$ particles retained Ag better than UCO particles, probably due to lower irradiation temperatures (TAVA <1075°C)

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Exposed Eu and Sr in LBL Leachates

- Eu and Sr release through intact SiC was much higher from Compact 2-2-3 than from Compact 5-2-3, due to the higher irradiation temperature.

Equivalent kernel inventory of exposed ^{154}Eu and ^{90}Sr

DLBL Stage	Compact 2-2-3 (1261°C)		Compact 5-2-3 (1108°C)	
	^{154}Eu	^{90}Sr	^{154}Eu	^{90}Sr
Deconsolidation	3.30	4.66	0.65	0.71
1st preburn leach	9.11	13.23	1.48	0.91
2nd preburn leach	2.48	0.71	0.39	0.35
1st postburn leach of matrix ash	25.64	6.88	1.16	0.28
2nd postburn leach of matrix ash	0.87	0.73	0.23	0.04
Subtotal prior to particle burn-leach	41.40	26.19	3.89	2.29
1st postburn leach of particles	8.50	6.46	0.46	0.14
2nd postburn leach of particles	0.10	0.74	0.01	0.006
Subtotal from particle burn-leach	8.60	7.21	0.47	0.14
Total leached	50.00	33.40	4.36	2.43

Some Eu and Sr does not dissolve in nitric until after the burn

Compact 5-2-3 release may have been dominated by 2–3 failed particles

Greater than 10x more Eu and Sr released at higher temperature

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Exposed Eu and Sr in LBL Leachates

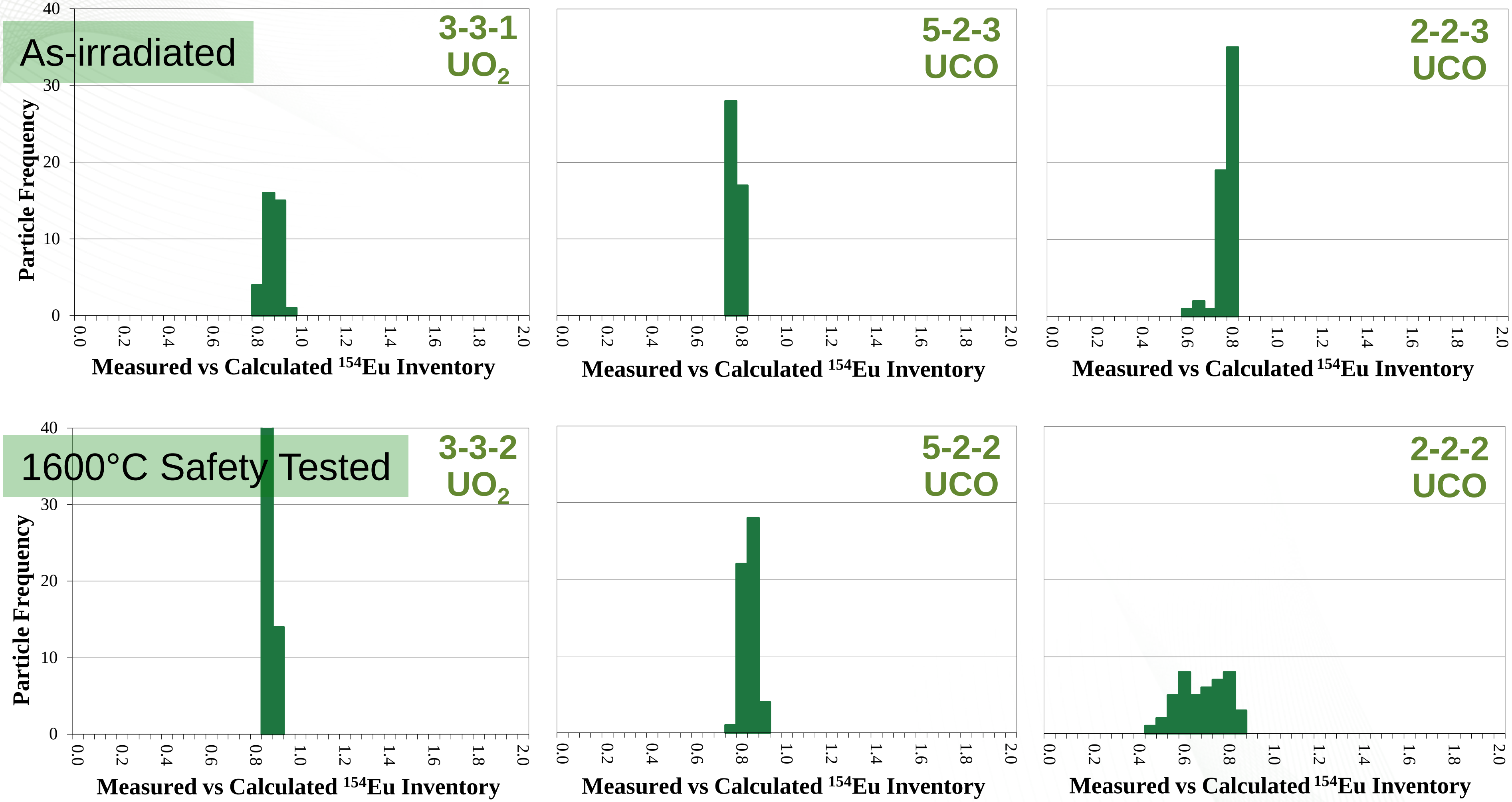
- Eu and Sr release through intact SiC was lower from UO_2 Compact 3-3-1 than from UCO compacts, due to the kernel chemistry.

Equivalent kernel inventory of exposed ^{154}Eu and ^{90}Sr

DLBL Stage	Compact 3-3-1 (1062°C)		Compact 5-3-3 (1093°C)		Compact 6-3-3 (1060°C)	
	^{154}Eu	^{90}Sr	^{154}Eu	^{90}Sr	^{154}Eu	^{90}Sr
Deconsolidation	0.09	0.008	↓	↓	↓	↓
1st preburn leach	0.07	0.003				
2nd preburn leach	0.01	0.002				
1st postburn leach of matrix ash	0.12	0.08	0.84	0.38	2.05	0.03
2nd postburn leach of matrix ash	0.003	0.001	0.02	0.006	0.05	0.003
Subtotal prior to particle burn-leach	0.29	0.09	0.86	0.39	2.10	0.03
1st postburn leach of particles	0.03	0.007	0.08	0.02	0.21	0.01
2nd postburn leach of particles	<0.003	0.001	<0.002	0.002	0.01	0.005
Subtotal from particle burn-leach	0.03	0.008	0.08	0.02	0.22	0.015
Total leached	0.32	0.10	0.94	0.41	2.32	0.05

↑ Moderately less Eu and Sr released from UO_2

TRISO Particle Europium Retention



Some Capsule 2 UCO particles released measurable Eu due to higher irradiation temperatures (TAVA >1250°C)

Measured vs. calculated ^{154}Eu inventory of 0.80–0.85 represents full retention due to overestimation in calculated inventory

AGR-2 Safety Testing

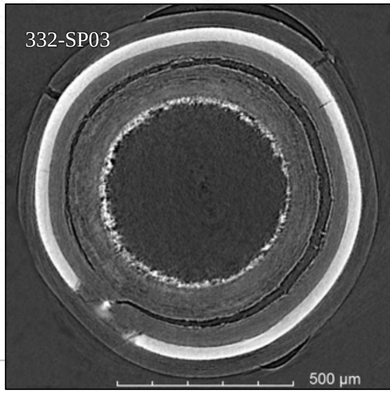
- High Eu and Sr release from Capsule 2 compacts irradiated at higher temperature
- High SiC failure in safety-tested UO_2 compacts due to CO corrosion of SiC.

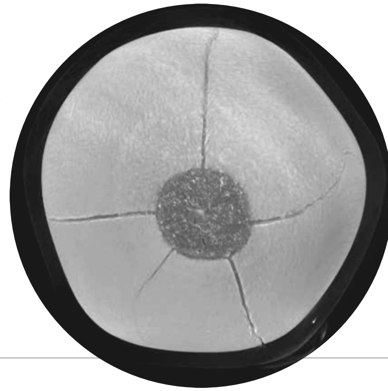
Compact ID	Fuel Type	TAVA Temp.	Average Burnup (FIMA)	Safety Test Temp.	Failed SiC	^{134}Cs Release	^{110m}Ag Release	^{154}Eu Release	^{90}Sr Release
5-2-2	UCO	1141°C	12.3%	1600°C	0	5.9×10^{-6}	1.7×10^{-2}	1.1×10^{-3}	7.9×10^{-4}
5-4-1	UCO	1071°C	12.1%	1800°C	1–2	1.0×10^{-4}	1.7×10^{-1}	6.0×10^{-3}	2.3×10^{-3}
2-2-2	UCO	**1287°C**	12.6%	1600°C	0	2.5×10^{-7}	7.3×10^{-3}	**4.7×10^{-2}**	**4.1×10^{-2}**
2-3-1	UCO	**1296°C**	12.6%	1600°C	0	4.0×10^{-6}	1.8×10^{-2}	**8.8×10^{-2}**	**8.6×10^{-2}**
2-3-2	UCO	**1296°C**	12.7%	1800°C	1–2?	$\sim3\times10^{-4}$	$\sim2\times10^{-2}$	**$\sim2\times10^{-1}$**	TBD
2-1-2	UCO	**1219°C**	12.7%	1800°C	1–2?	$\sim2\times10^{-4}$	$\sim2\times10^{-2}$	**$\sim3\times10^{-2}$**	TBD
6-4-2	UCO	1018°C	9.3%	1600°C	0-1?	6.2×10^{-5}	3.4×10^{-3}	2.7×10^{-4}	8.7×10^{-5}
3-3-2	**UO_2**	1062°C	10.5%	1600°C	**≥6**	**2.1×10^{-3}**	1.7×10^{-2}	3.8×10^{-4}	1.4×10^{-3}
3-4-2	**UO_2**	1013°C	10.7%	1600°C	**≥26**	**9.3×10^{-3}**	1.1×10^{-2}	3.2×10^{-4}	2.7×10^{-3}
3-4-1	**UO_2**	1013°C	10.6%	1700°C	**high**	**$\sim9\times10^{-2}$**	$\sim8\times10^{-2}$	TBD	TBD

Approximate values (~) indicate post-safety test analysis is incomplete.
Compact 3-4-1 safety test was terminated after 162 h at 1700°C due to high release, other tests were run for ~300 h at test temperature

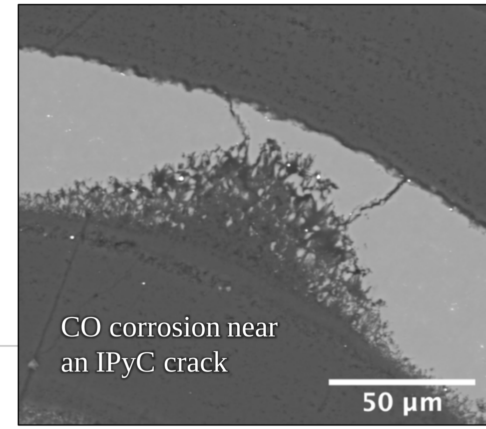
Evidence for CO Corrosion in Failed UO_2 Particles

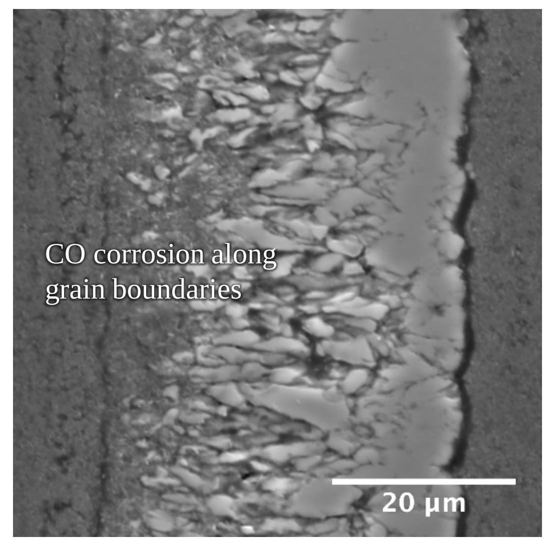
- Large low-density regions found in low-cesium particles after safety testing.



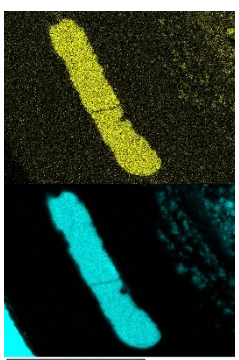


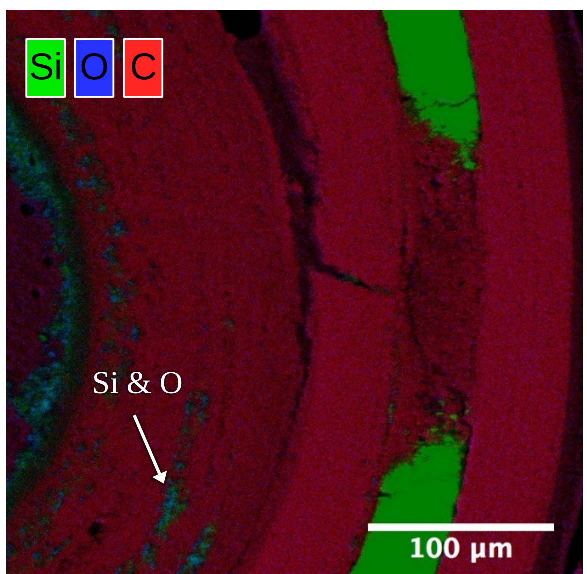
- Grain boundary attack



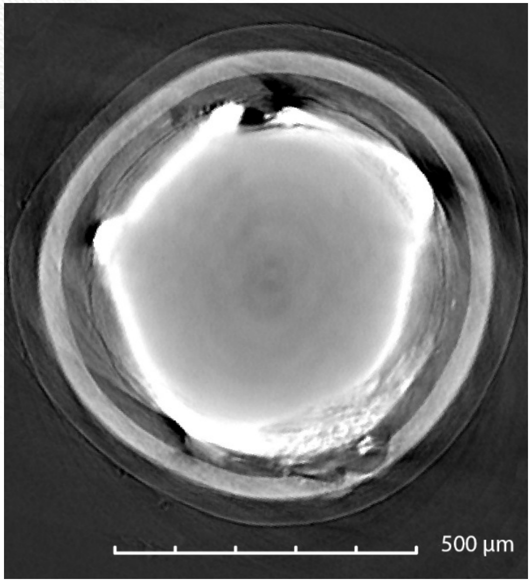


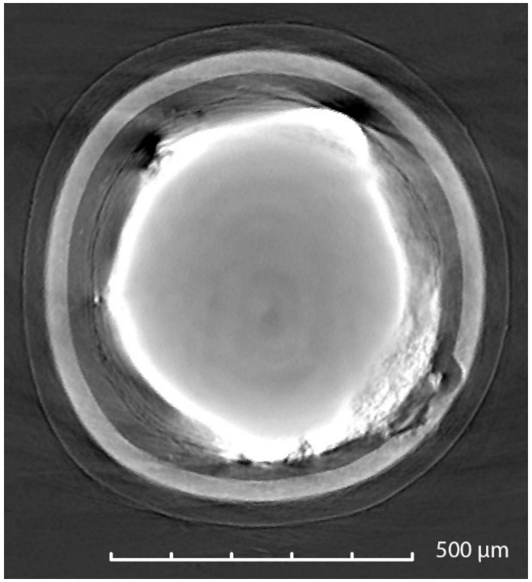
- Si redistribution
- Si-O co-location





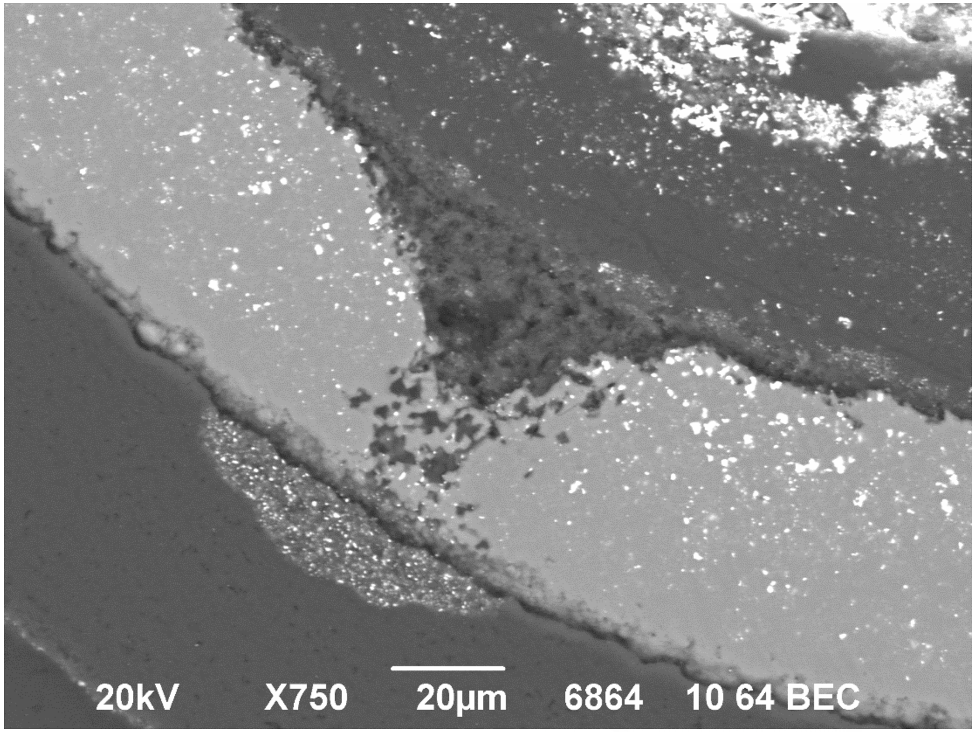
First look at SiC failure in AGR-2 UCO at 1800°C





X-ray Tomographs

Backscattered-electron image



- X-ray showed localized SiC degradation where IPyC was compromised.
- SEM/EDS showed carbon-rich pathway through degraded SiC surrounded by high concentrations of U and fission products.

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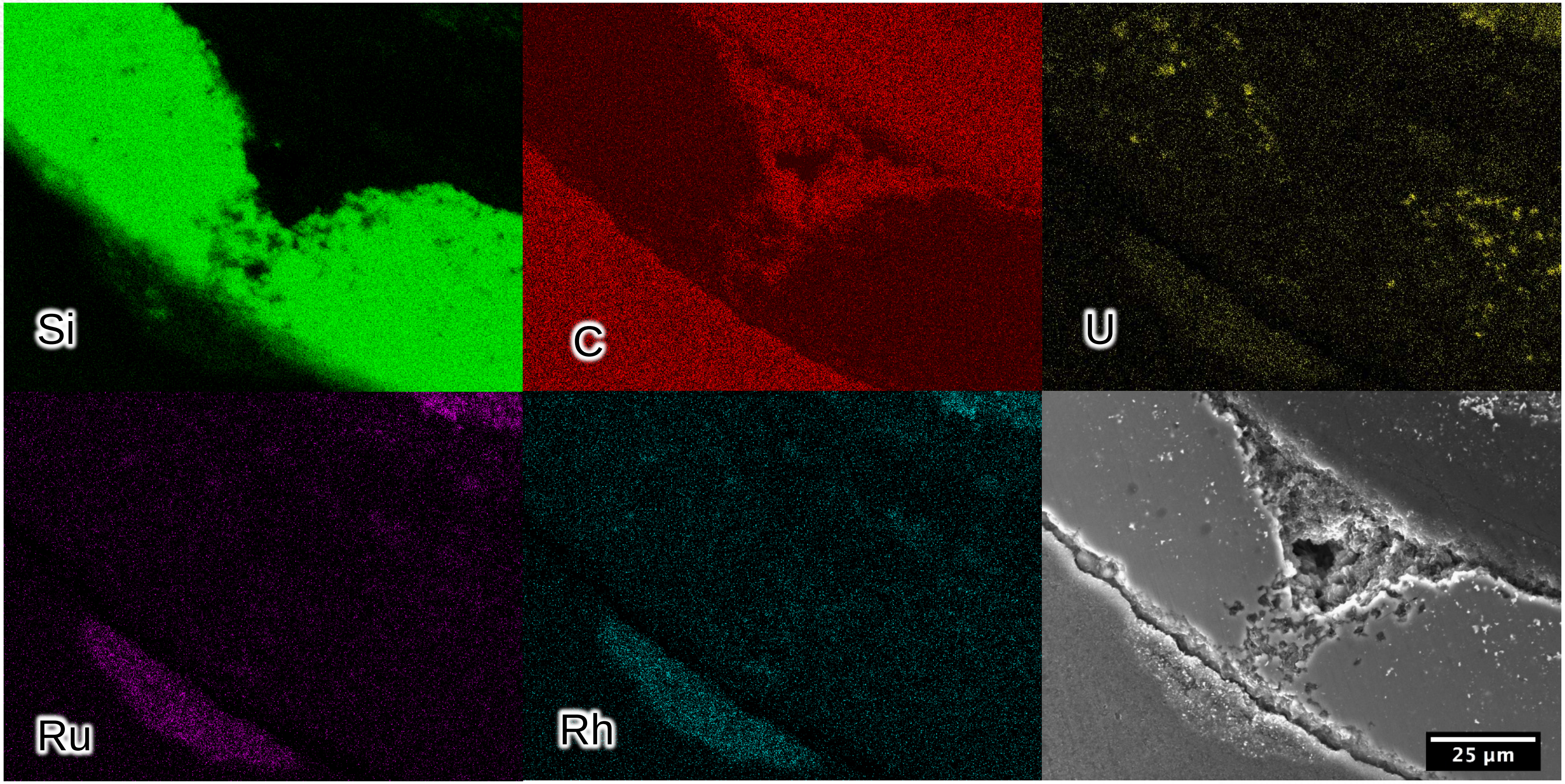
First look at SiC failure in AGR-2 UCO at 1800°C



- Pd presence was very low due to out diffusion at 1800°C (previously observed).
- Ru and Rh were co-located with U and Si in material extending through defect and out into OPyC.

Summary

- PIE is complete on 5 as-irradiated and 6 safety-tested AGR-2 compacts.
- PIE is in progress on 3 as-irradiated and 4 safety-tested AGR-2 compacts.
- AGR-2 UCO fuel
  - ≤1 SiC failure has been observed as a result of 1600°C safety testing to this point.
  - High-temperature Capsule 2 compacts show greatly reduced tendency for buffer fracture, which should reduce SiC failure like that observed in AGR-1 due to reduced probability for IPyC fracture. This may be impacting 1800°C safety test performance, which is showing lower failure fractions compared to AGR-1 fuel.
  - Capsule 5 showed significantly more buffer fracture compared to AGR-1, but this may not directly correlate to the probability for IPyC fracture if buffer/IPyC interface is weaker. Initial indication is that IPyC fracture is also less prevalent in AGR-2 UCO, compared to AGR-1 UCO TRISO.
  - Higher irradiation temperature also resulted in more Eu and Sr release through intact SiC during irradiation, and more Eu and Ag migration out of compact.
  - Gamma scanning with the IMGA indicated that individual Capsule 2 particles released measurable amounts of europium due to higher irradiation temperatures.
  - AGR-2 Compact 2-2-3 as-irradiated particles with SiC failure exhibited much more SiC degradation than AGR-1 fuel (this appears to have been related to Ni attack and not necessarily inherent to the fuel performance).

Summary

- AGR-2 UO_2 fuel
  - Particle failure fractions during 1600°C safety testing have been much higher for AGR-2 UO_2 fuel than AGR-1 or AGR-2 UCO fuel.
  - SiC degradation in some safety-tested particles with failed SiC was extensive and appeared to be localized at IPyC cracks. This was due to higher CO concentrations in UO_2 fuel resulting in CO corrosion.
  - CO corrosion will limit the extent of safety testing UO_2 fuel at margin temperatures (1700°C or 1800°C). One 1700°C test has been completed but had to be terminated early due to high cesium release.
  - Europium was slightly better retained in the AGR-2 UO_2 fuel than in UCO fuel. This is expected based on kernel chemistry, which favors formation of europium carbide in UCO fuel versus more stable europium oxide in UO_2 fuel.
    (Homan et al., Nucl. Tech. 35 (1977) 428–441).
  - Silver was better retained in UO_2 fuel than in UCO, but this is likely a temperature effect, not a function of the kernel chemistry.

Summary

- AGR-1 and AGR-2 PIE is showing that each component of the TRISO particle system is important to overall irradiation performance.
  - Retention of actinides and fission products and limiting CO production by optimization of the kernel chemistry to control oxygen potential is the first line of defense for good fission product retention.
  - Shrinkage of the buffer layer is unavoidable, but it is important that it does not negatively impact the IPyC layer; a weak buffer/IPyC interface can help protect the IPyC.
  - Intact IPyC provides important protection to the SiC from chemical attack, most observed SiC failure has been related to IPyC fracture exposing the SiC to elevated concentrations of fission products.
  - SiC provides structural stability and retention of most fission products but cannot stand alone, especially when fuel is heated to safety test temperatures.
  - Intact OPyC can be the last line of defense to retain gaseous reaction products and maintain stable kernel chemistry in particles that experience SiC failure.

Thank you for your attention

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