INL/MIS-23-73610-Revision-0

Initial results from electron microscopy from AGR 5/6/7 Capsule 1 fuel and

July 2023

Lu Cai

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Initial results from electron microscopy from AGR-5/6/7 Capsule 1 fuel and applications of other microanalysis techniques to irradiated TRISO fuel

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

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Outline

- Characterization of particles from AGR-5/6/7 Capsule 1
  - Background and Motivation
  - X-ray computed tomography on four particles from Compact 1-7-9
  - SEM/EDS of particles from Compacts 1-7-9 and 1-7-4
  - Summary

- Local Thermal Conductivity Measurement on AGR-2 particles

Characterization of Particles from AGR-5/6/7 Capsule 1

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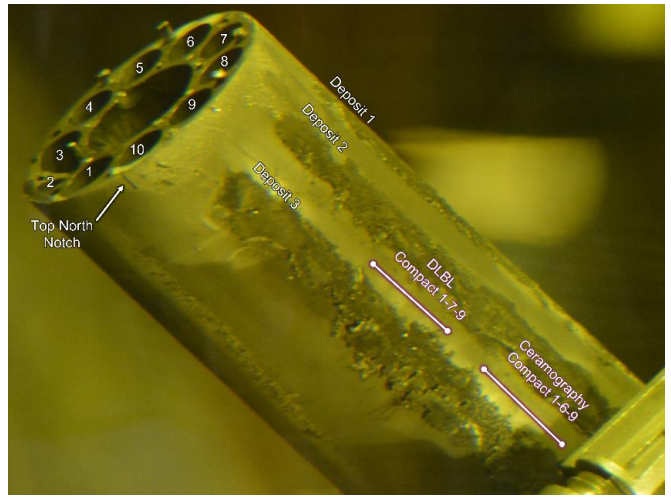
Background and Motivation – Capsule 1

- AGR-5/6/7 Capsule 1 had significant rates of in-pile TRISO fuel particle failure, likely caused by Ni from thermocouples reacting with SiC layers.

- Four randomly-selected particles from Compact 1-7-9 were examined with x-ray tomography for signs of coating degradation and evidence of nickel attack.

- A few particles from Compacts 1-7-9 and 1-7-4 were investigated by scanning electron microscope (SEM) to compare the microstructure and to look for evidence of nickel in the fuel



Ref: INL/RPT-22-66720, “Initial Observations from AGR 5/6/7 Capsule 1.”

Characterization of Particles from AGR-5/6/7 Capsule 1

X-ray Computed Tomography

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Miles T. Cook

Quintin D. Harris

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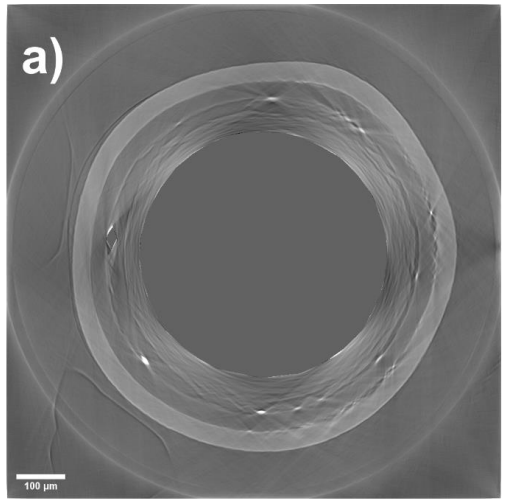
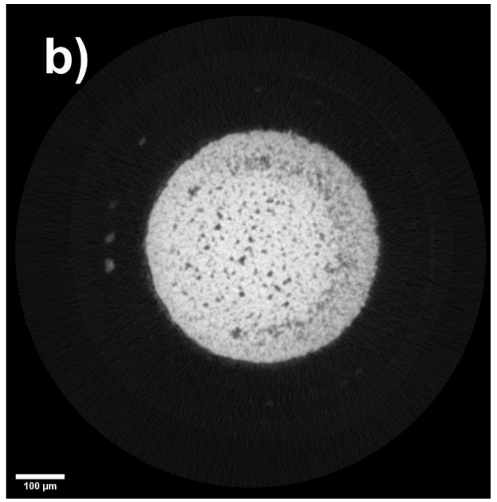
XCT- Particles A, C, and D

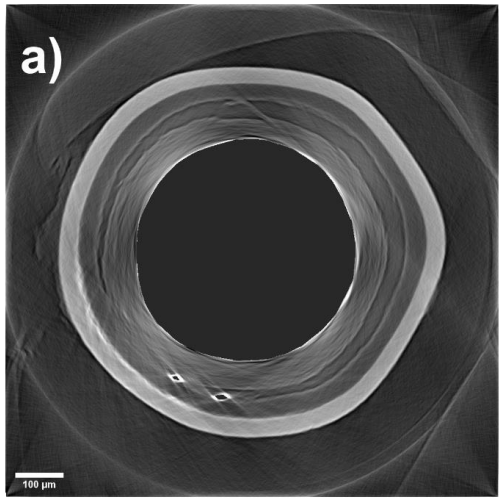
Particle A

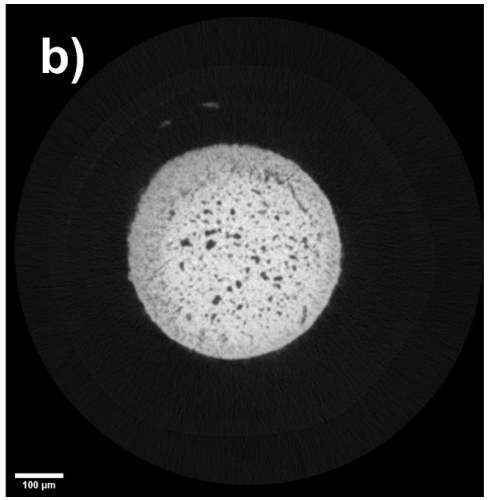




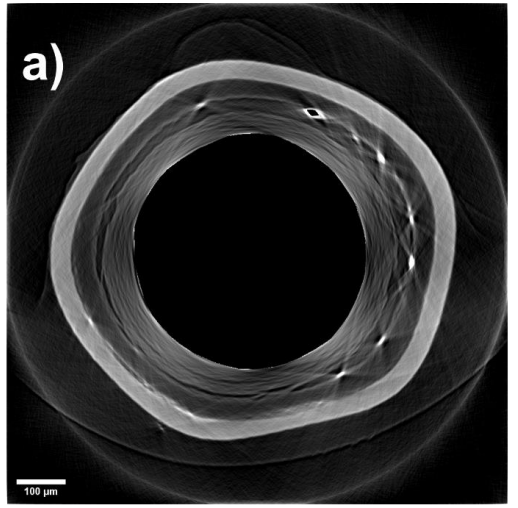
- Low energy (a) and high energy (b) XCT scans through the particle center
- Each of three particles particle has an intact SiC layer.
- The IPyC layers appear well adhered to the SiC layer.
- The IPyC and buffer layers appear to have separated over the majority of the volume of each particle.
- A number of dense features are observed within the TRISO layers.

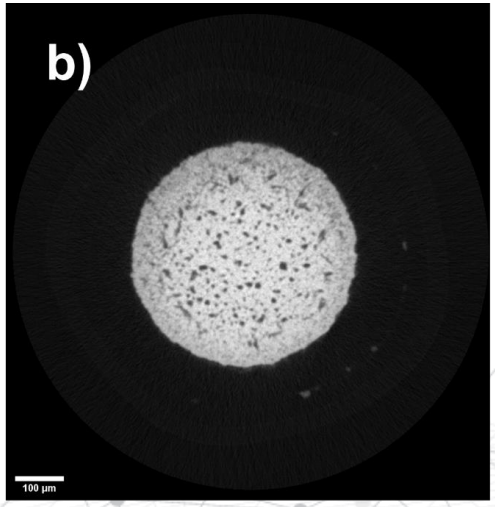
Particle C



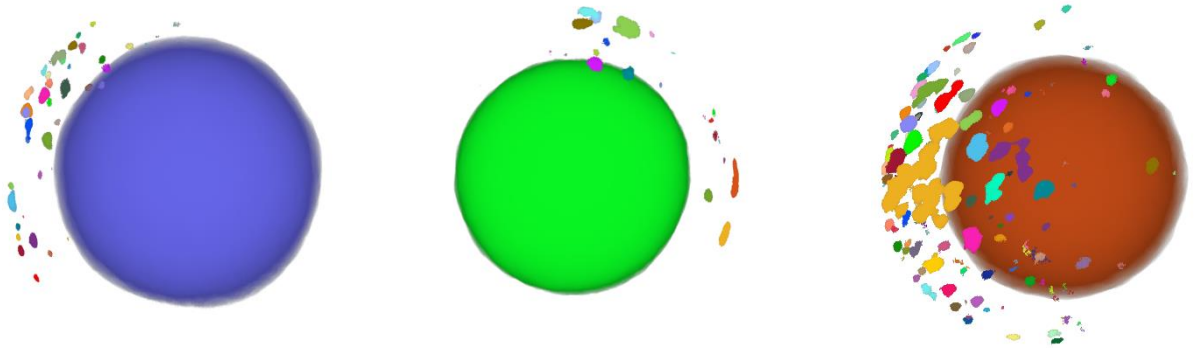


Particle D



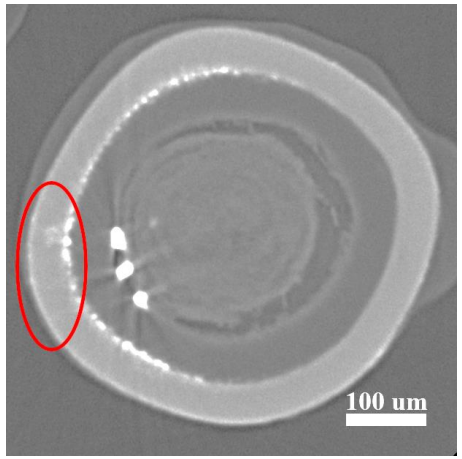


XCT - Particles A, C, and D



3D- renderings of dense features surrounding the kernels of Particles A, C, and D

- The dense features are primarily observed in large clusters along the IPyC side of the IPyC/Buffer interface where the two layers have separated. These features appeared to be nonuniformly distributed within particles.

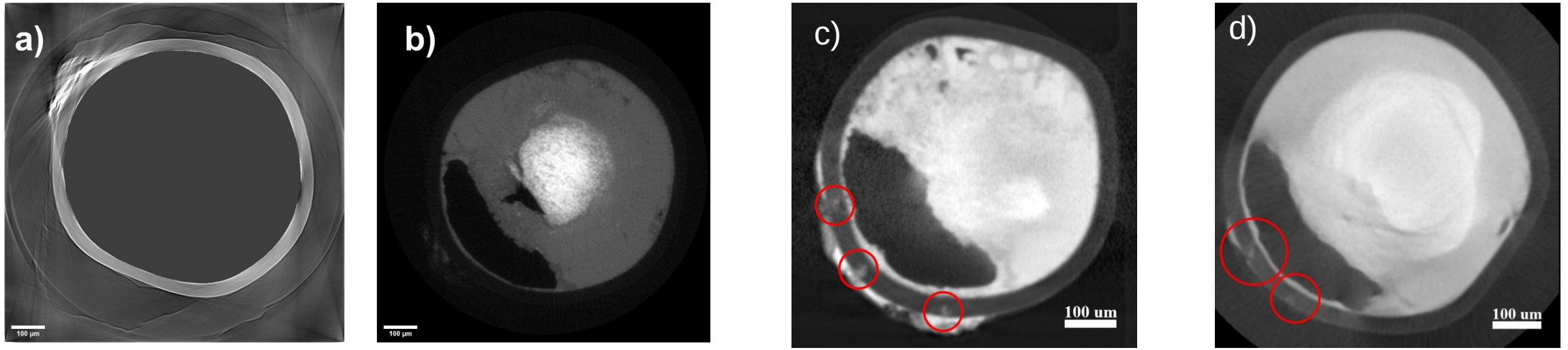


Particle C

- A lot of materials deposited on the SiC/IPyC interface.
- A small quantities of material deposited on the SiC exterior surface (circled in red).

XCT – Particle B

Particle B: no buffer nor IPyC



- Low energy (a) and high energy (b) XCT scans through the particle center, indicating no evidence of buffer nor IPyC. Dense material appeared dispersed throughout the interior of the SiC layer.
- (c) and (d) shows several locations on the SiC exterior surface where the unknown material appears to penetrate into the SiC layer.
- May be an example of a particle originally located near the Compact 1-7-9 surface attacked by nickel.

Characterization of Particles from AGR-5/6/7 Capsule 1

Preliminary SEM/EDS Analysis

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Daniel J. Murray

Fei Teng

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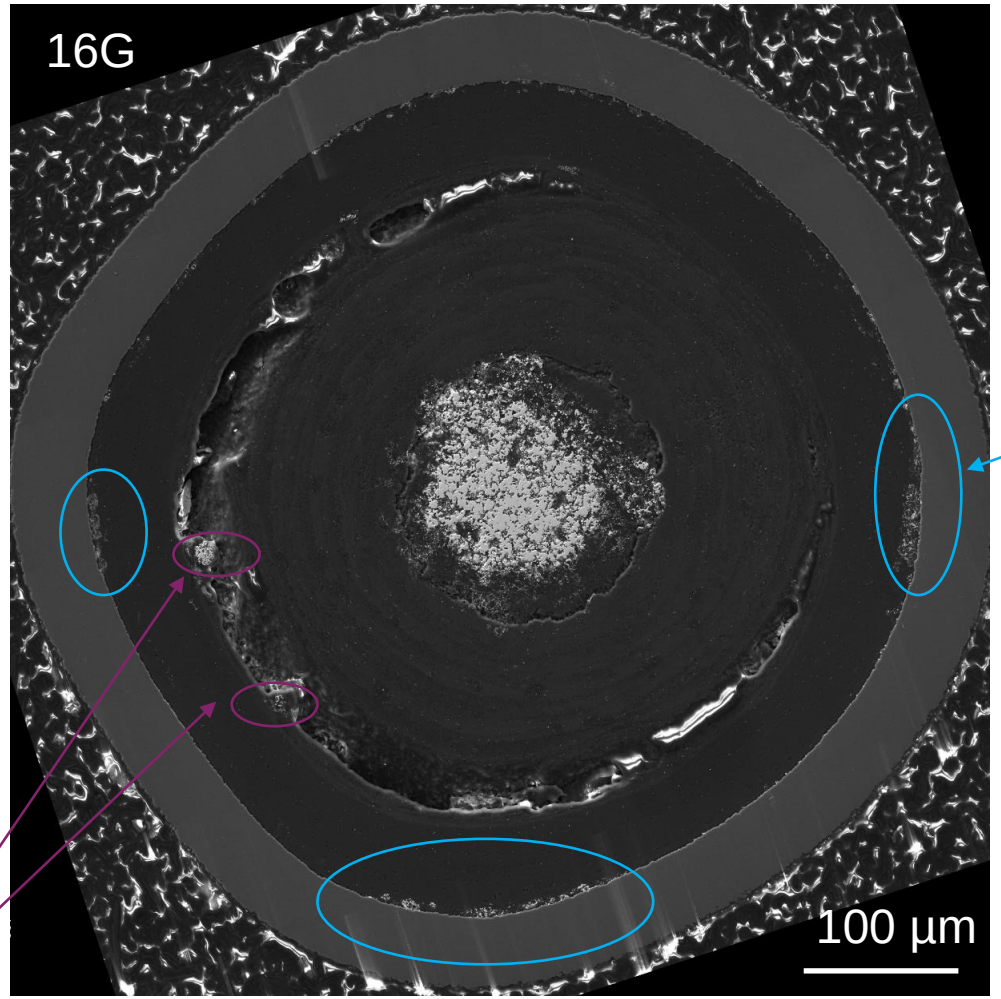
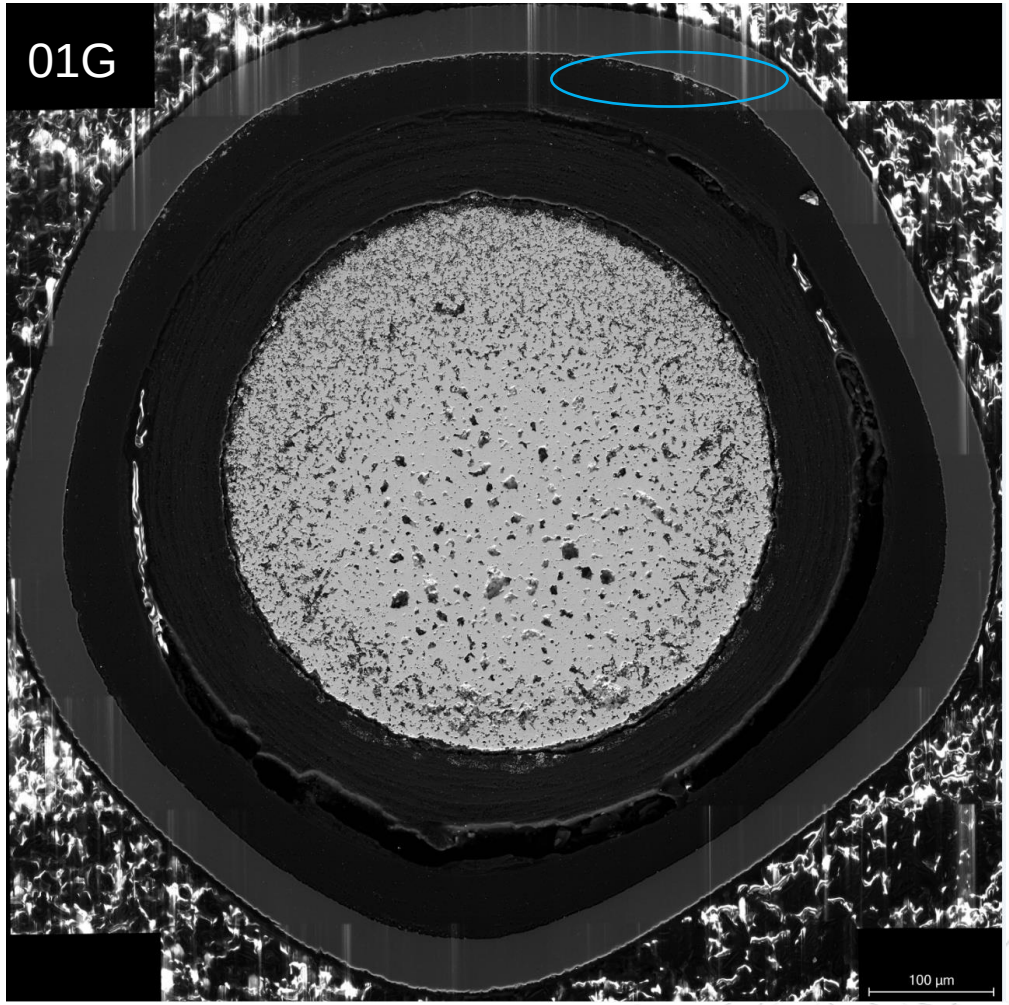
MNT-16G and MNT-01G (Compact 1-7-9)

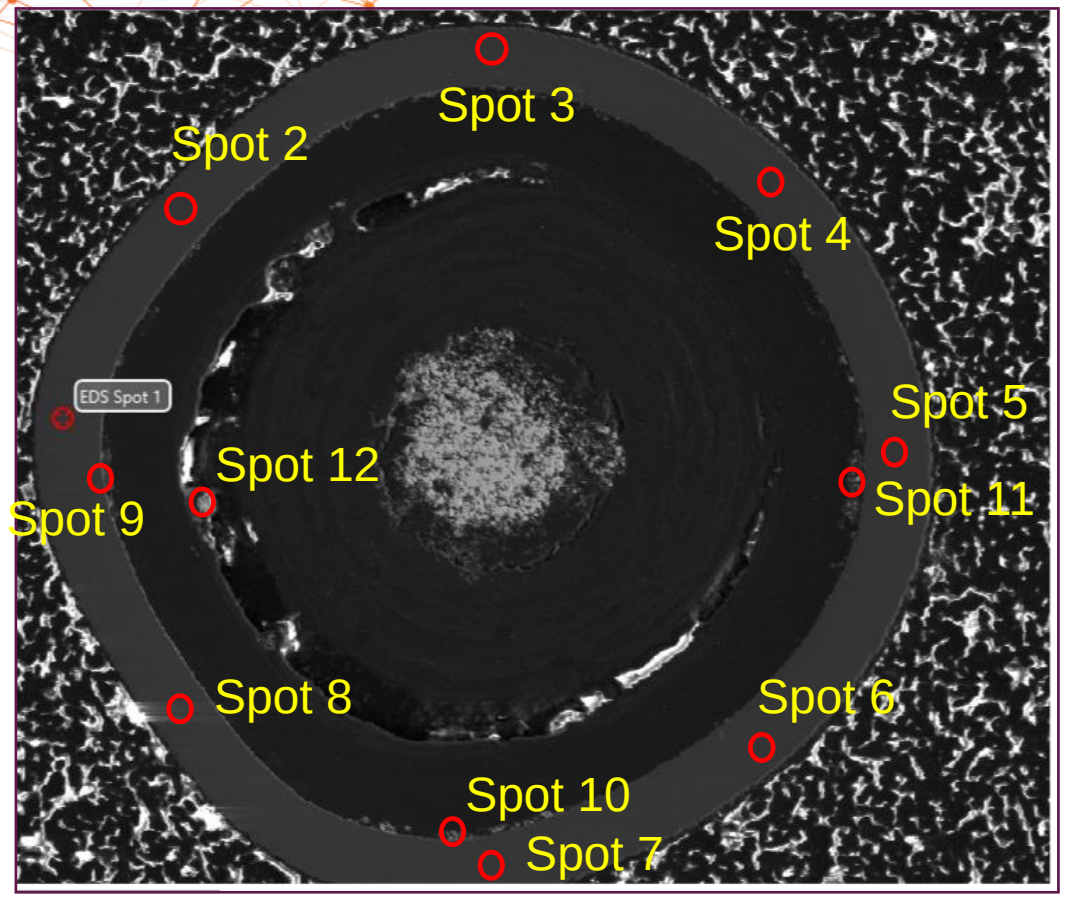


Cluster of high Z materials



High Z materials were also observed at the IPyC side of the IPyC/Buffer interface where the two layers have separated, consistent with the XCT observation.

MNT-16G (Compact 1-7-9)

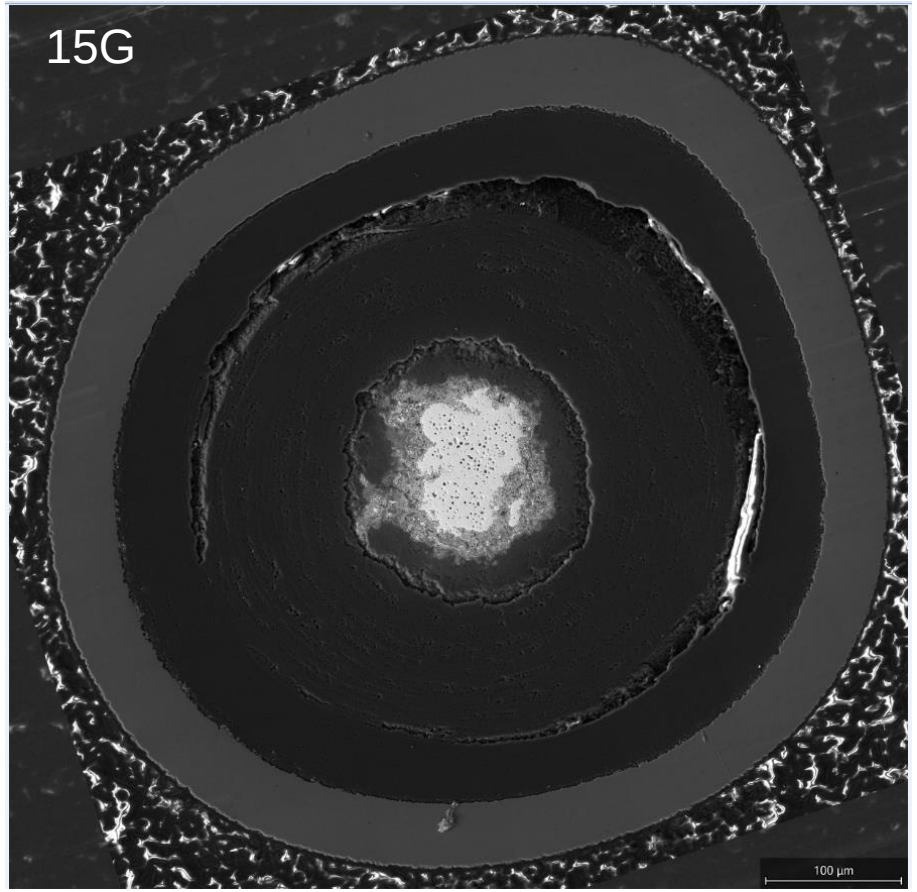


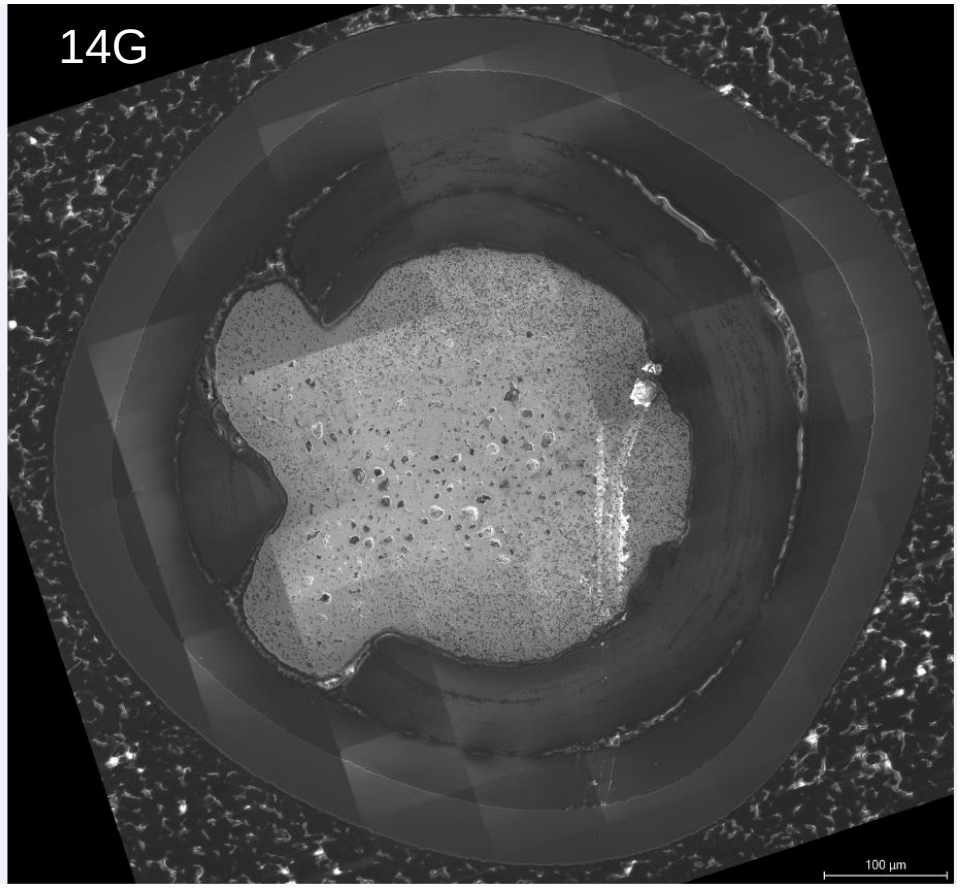
Spot	Location	Ni (wt%)
1	SiC	0.9
2	SiC	N/A
3	SiC	0.3
4	SiC	0.5
5	SiC	N/A
6	SiC	N/A
7	SiC	N/A
8	SiC	N/A
9	SiC/IpyC	2.6
10	SiC/IpyC	7.7
11	SiC/IpyC	12.4
12	IpyC/gap	N/A

Ni is detected in some parts of the SiC layer as well as on the fission product deposits between SiC and IPyC interface.

Note: Ni percentage is semi-quantitative. Si has much strong EDS peaks than C.

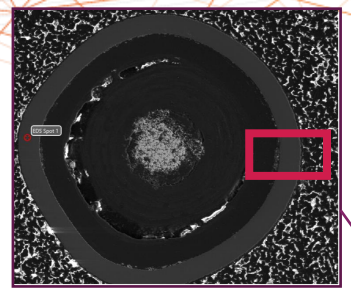
Particles from Compact 1-7-4

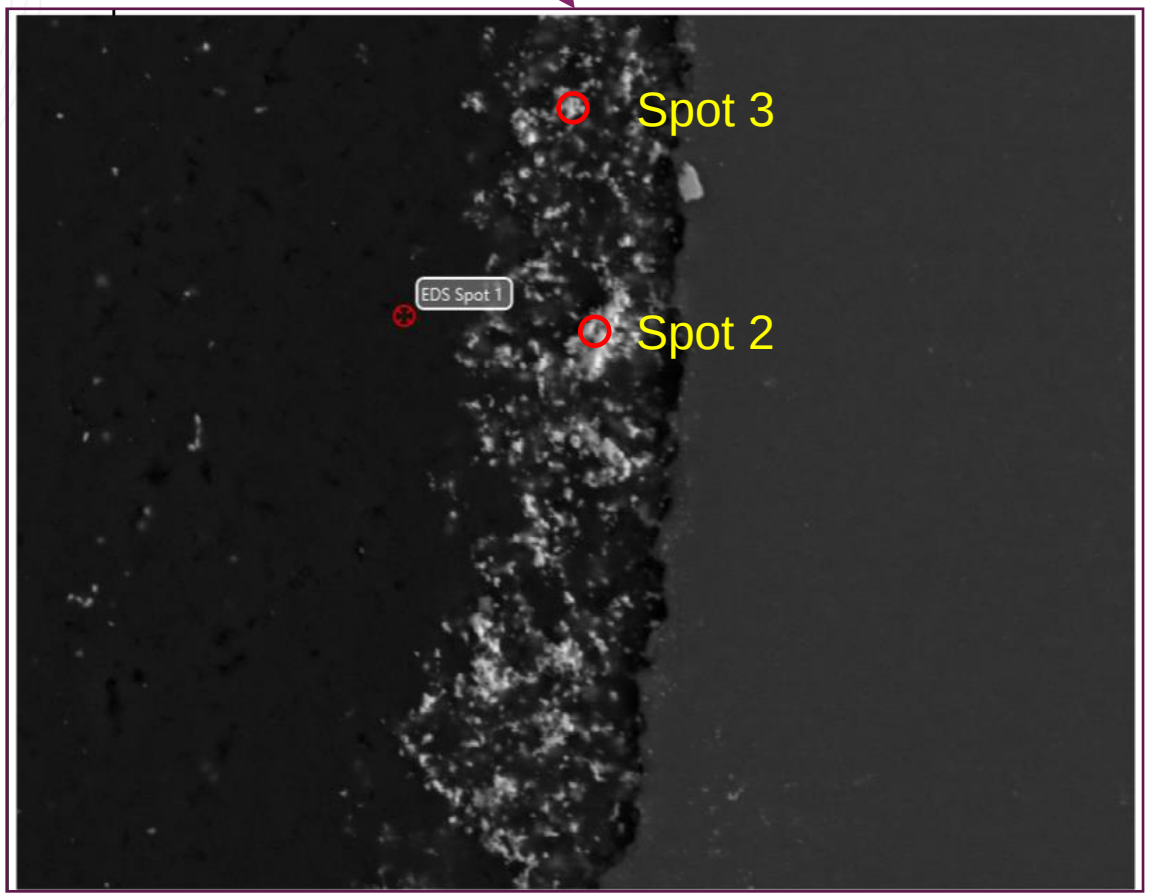


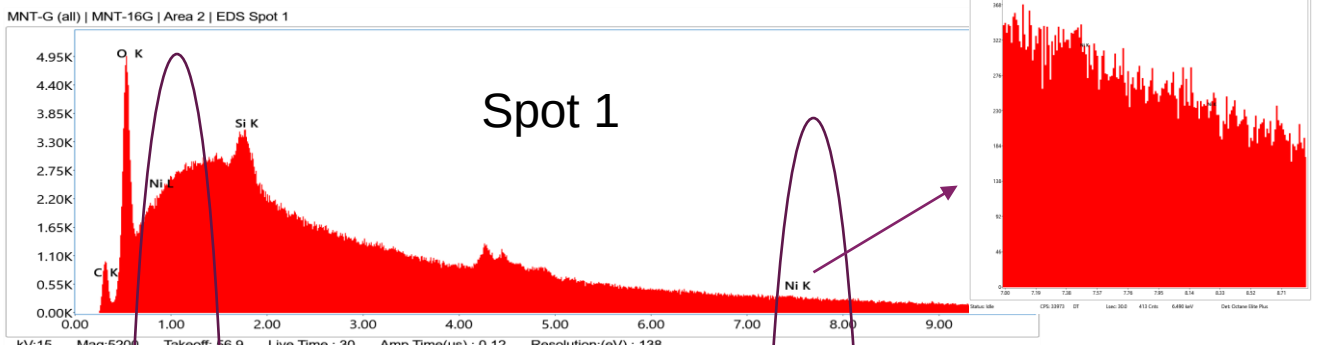


- Visibly less high Z materials deposits at SiC/IPyC
- No detection of Nickel

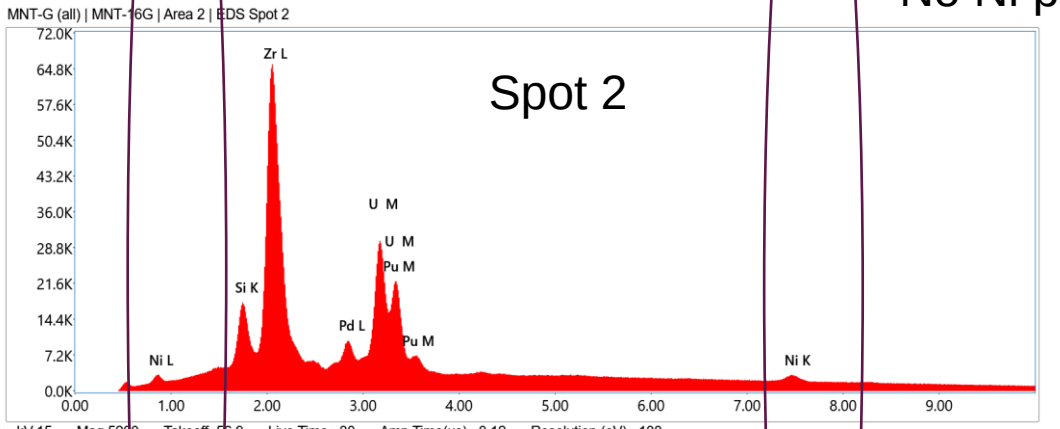
MNT-16G (Compact 1-7-9)

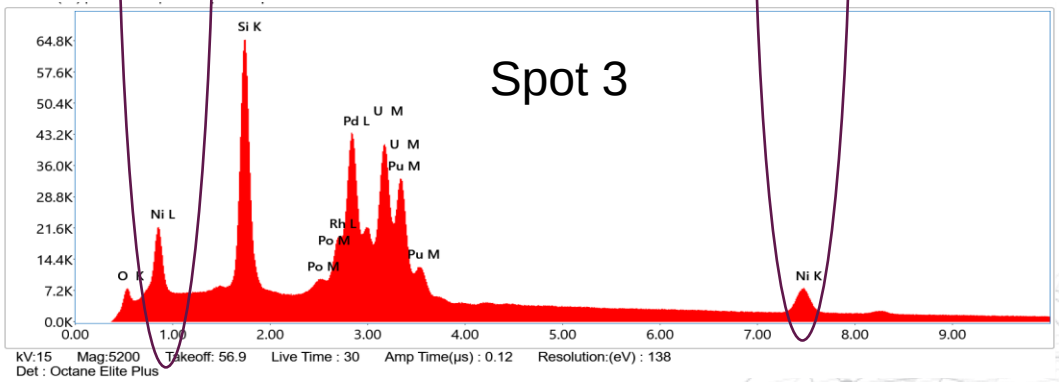




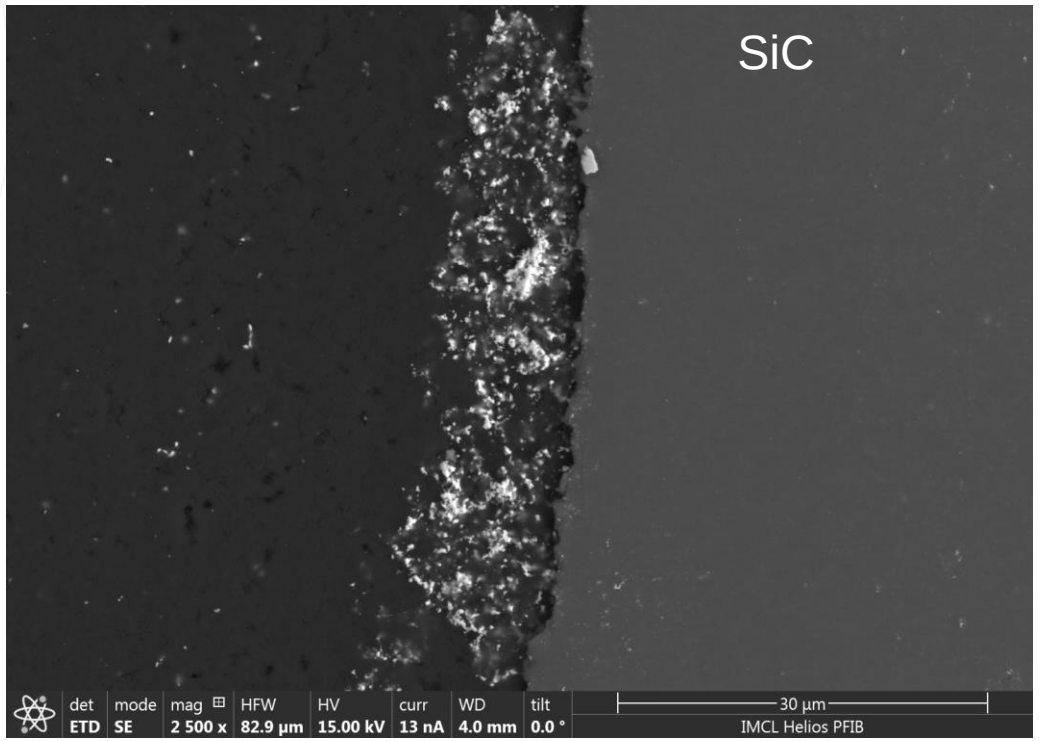


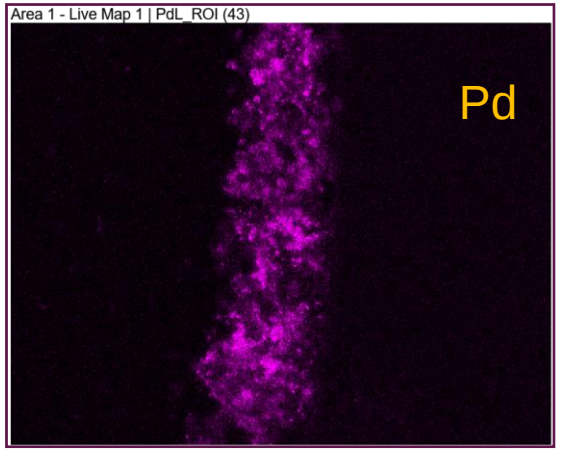
No Ni peak at Spot 1.

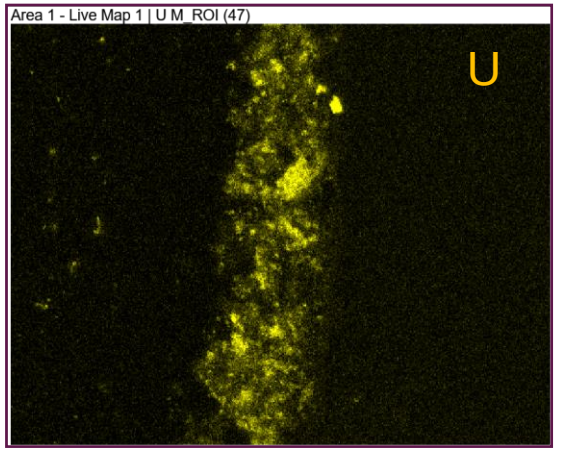


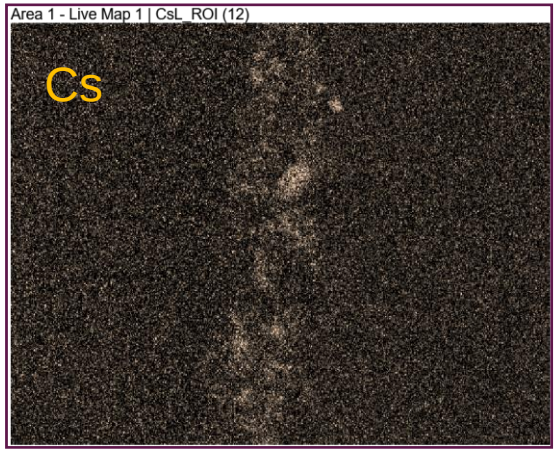


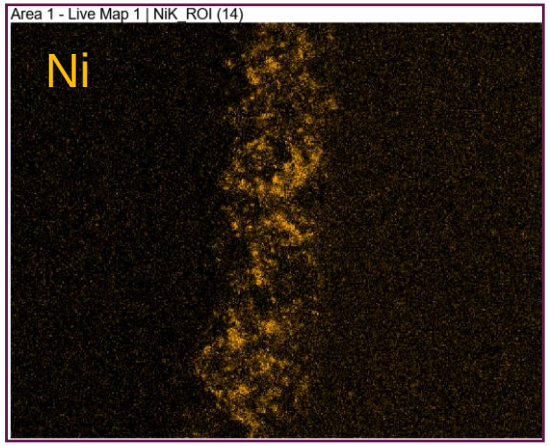
MNT-16G (Compact 1-7-9)







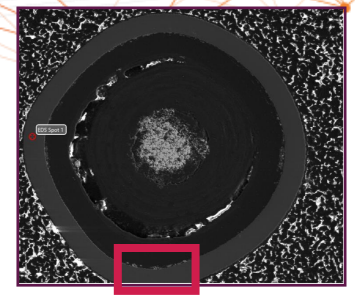


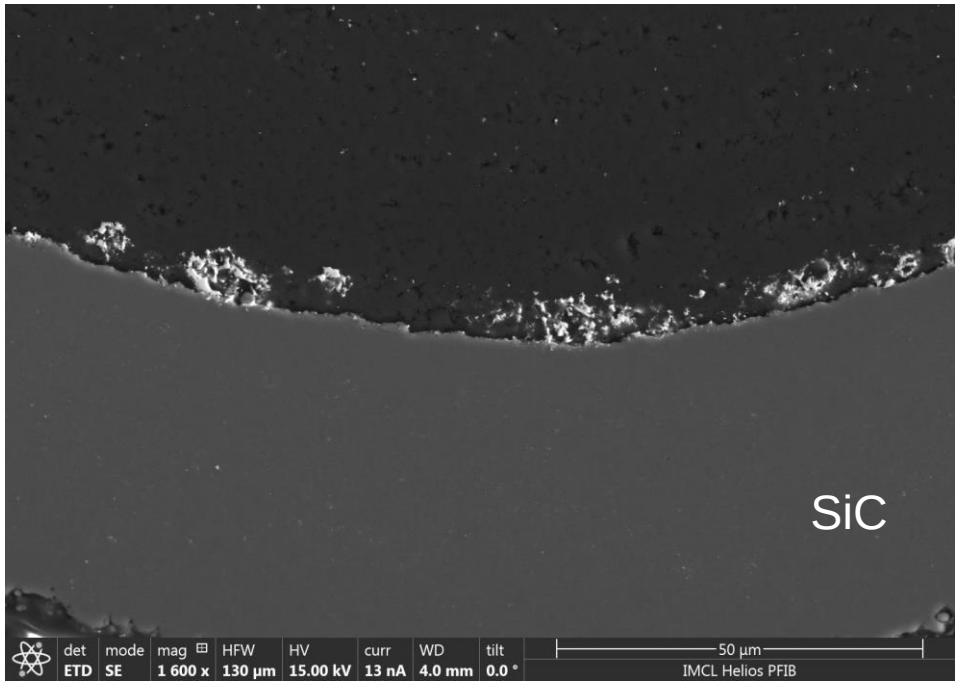


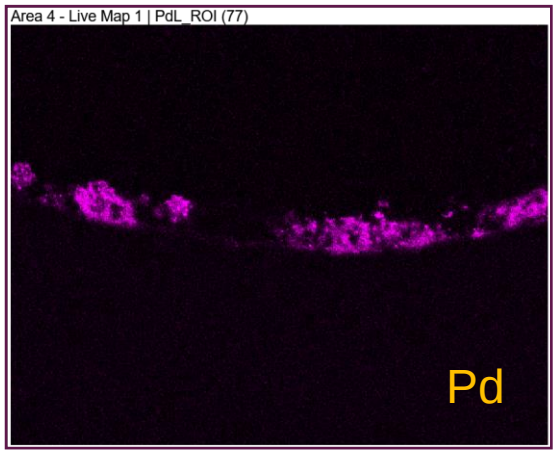
Element	Weight %	Error %
O K	1.7	10.8
Si K	69.5	3.9
Ni K	1.2	6.2
Pd L	5.7	4.8
Ag L	0.4	15.8
Cs L	2.3	4.4
U M	4.1	4.7
Pu M	7.5	4.1

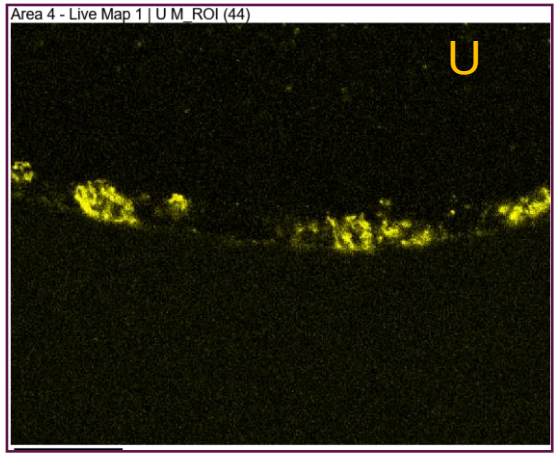
Fission products accumulated at the interface of SiC/IPyC. Ni is detected in these fission products.

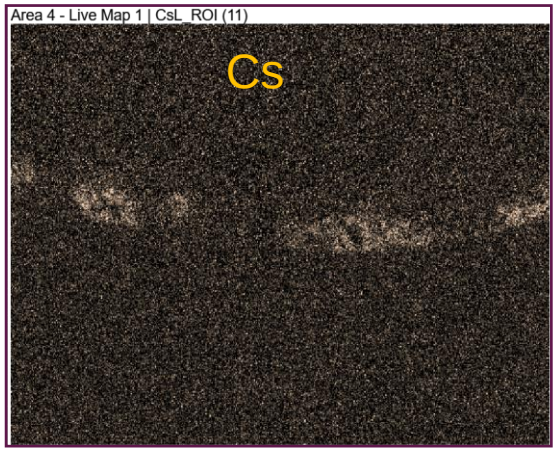
MNT-16G (Compact 1-7-9)

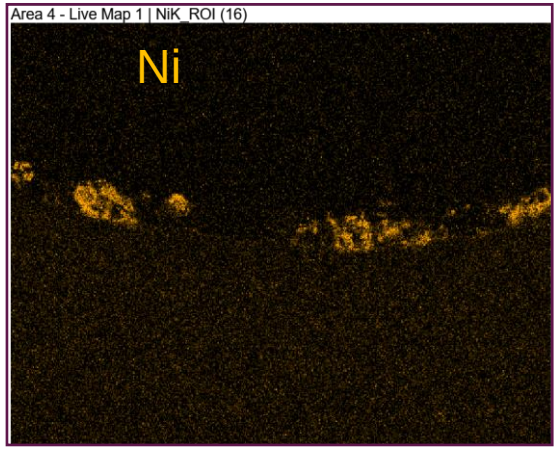






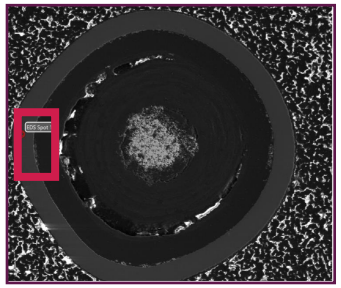


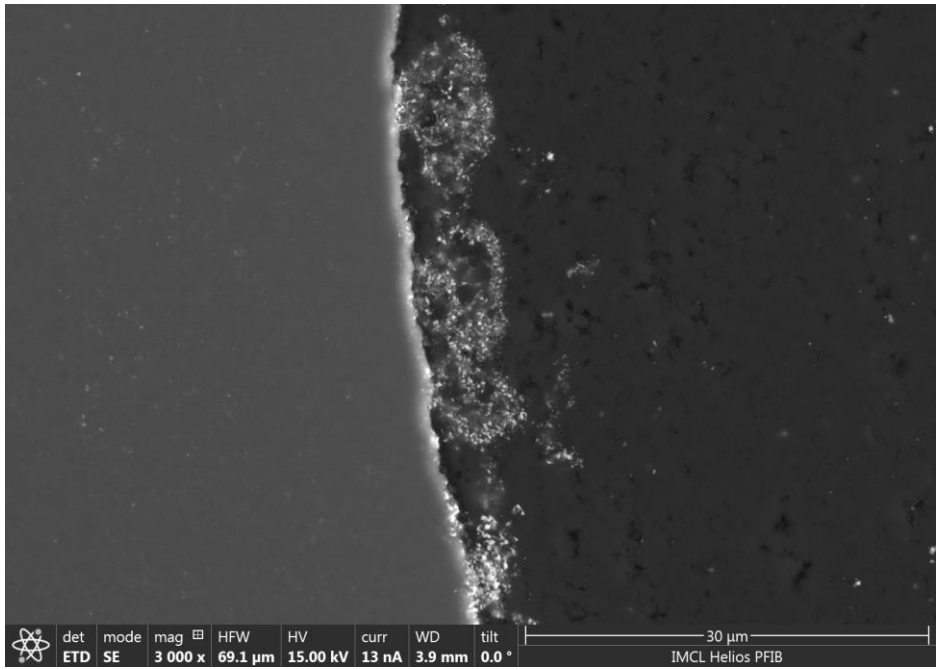


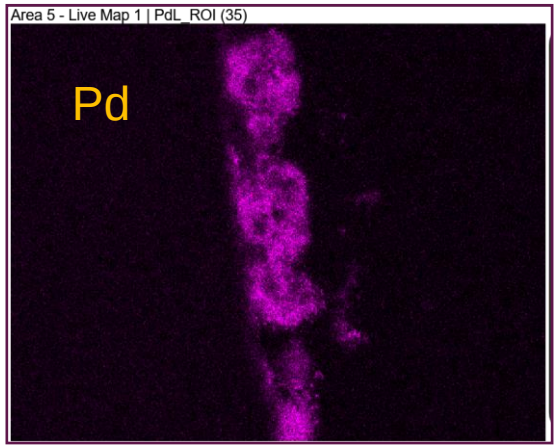


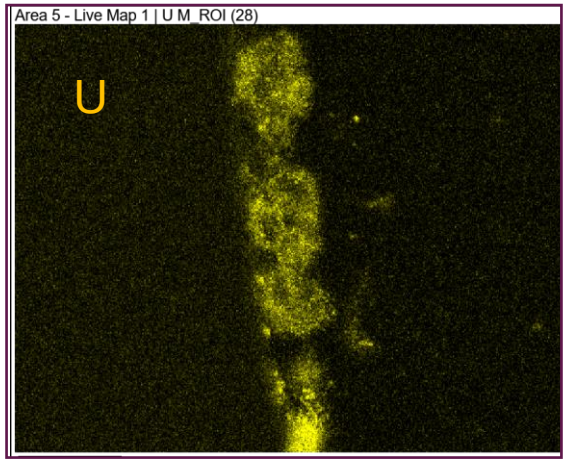
Element	Weight %	Error %
O K	1.9	10.5
Si K	81.9	3.6
Ni K	0.8	9.2
Pd L	3.3	5.1
Cs L	2.1	5.2
Ce L	0.9	11.5
U M	2.9	4.3
Pu M	6.3	4.3

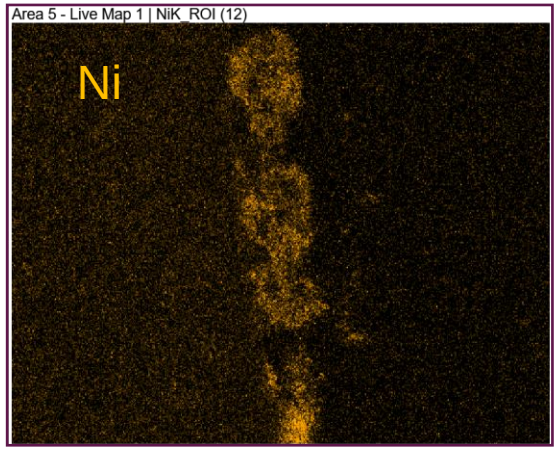
MNT-16G (Compact 1-7-9)







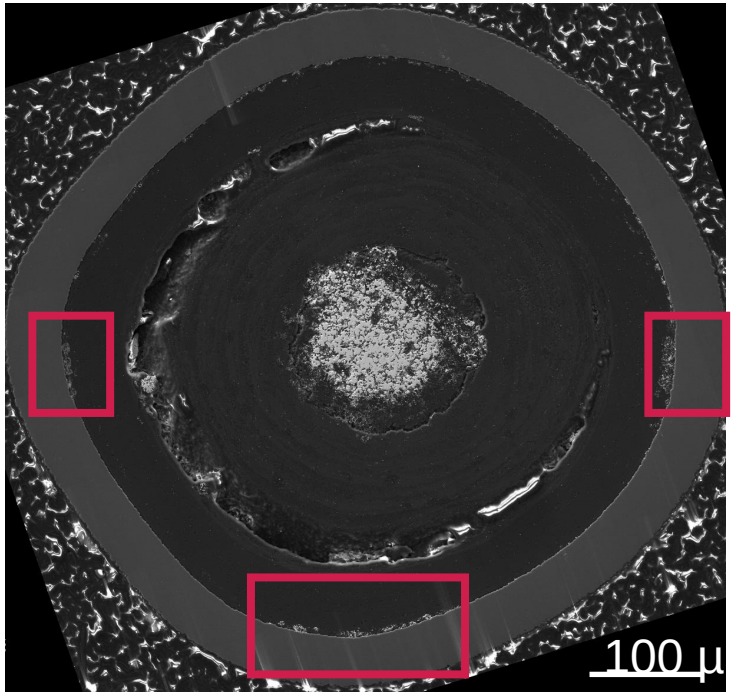




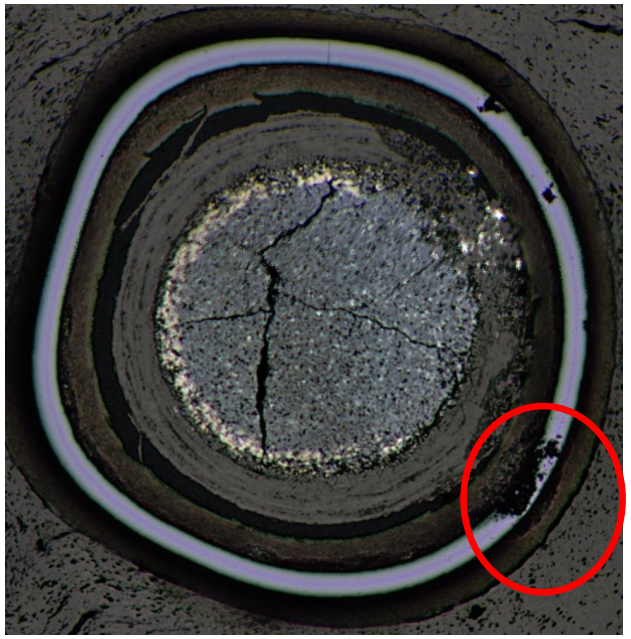
Element	Weight %	Error %
O K	2.6	10.1
Si K	71.9	3.9
Ni K	1.0	5.6
Pd L	4.8	4.8
Ce L	2.5	4.5
U M	3.4	4.4
Pu M	6.5	4.1

Nickel inside the particle

MNT-16G (Compact 1-7-9)



Particle from Compact 1-6-9

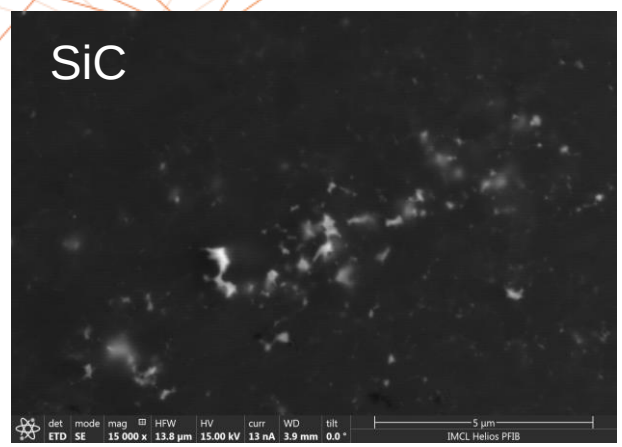


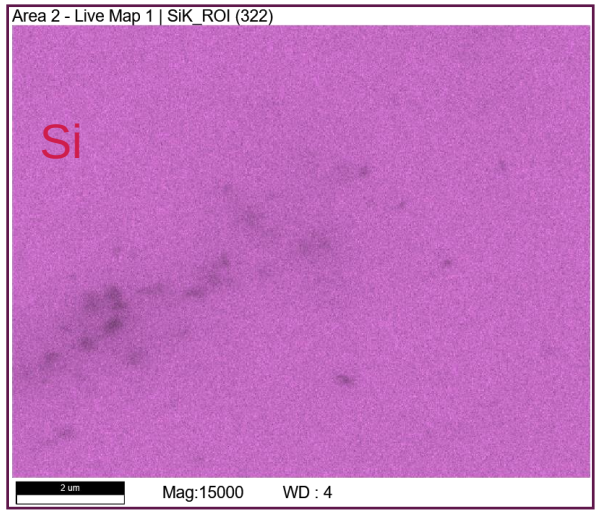
Looks like the nickel attack from inside out ➔Ni penetrated from another plane

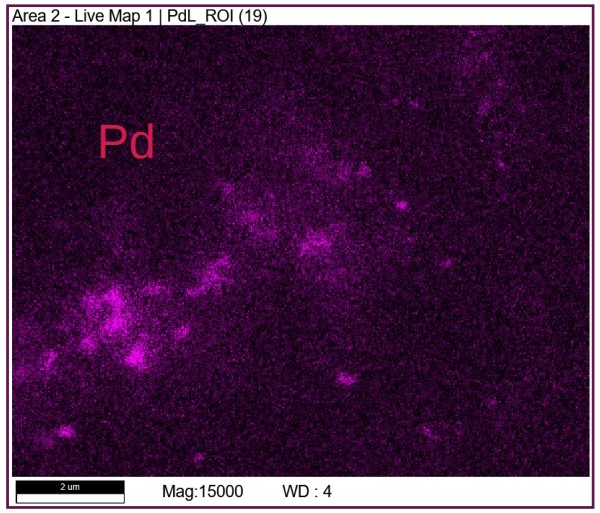
Nickel appears <u>inside</u> the particle with the cluster of fission products.

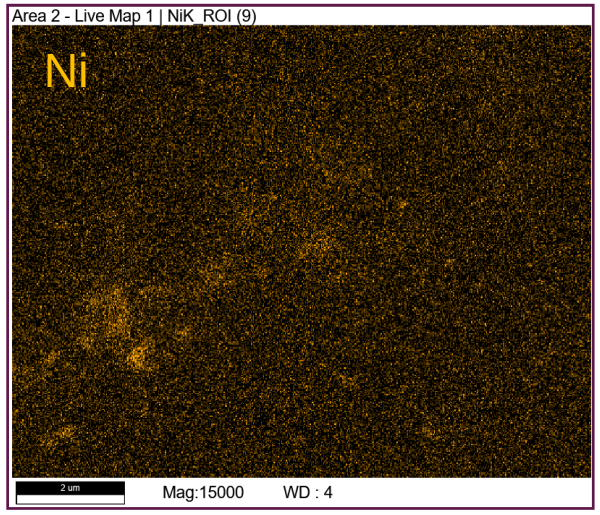
- is Ni penetrated the SiC in some other plane that we can't see here?
- Or is Ni diffusing through the SiC and then accumulating the locations?

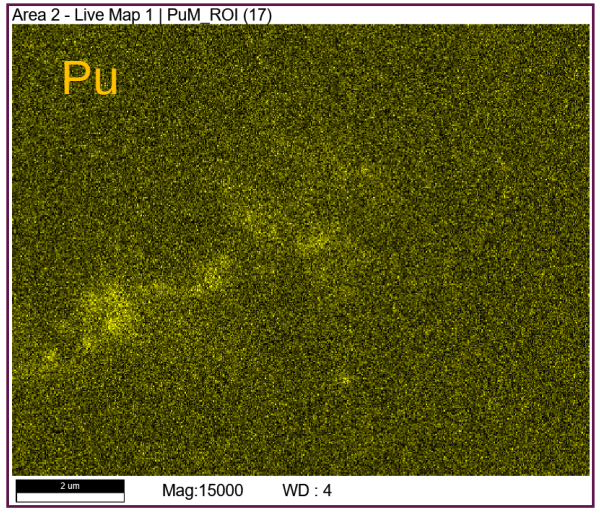
MNT-01G (Compact 1-7-9) – Nickel on SiC











Element	Weight %	Error %
Si K	85.5	3.4
Ni K	0.4	9.9
Pd L	1.0	7.1
Cs L	0.5	10.9
Ce L	0.4	11.6
U M	1.1	6.0
Pu M	7.2	4.0

Fission products with Ni were observed on SiC.

Summary on Capsule 1 Particle Characterization

- SiC degradation was observed in Particle B with XCT. Non-uniform dense features were observed in large clusters along the IPyC side of the IPyC/Buffer interface where the two layers have separated in the other three particles.
- SEM/EDS results confirmed the presence of nickel in the SiC layer co-located with fission products along SiC/IPyC interface. This finding supports the hypothesis that particle failures were caused by the overheated thermocouples.
- We are currently working on characterizing the cross-section of Compact 1-6-9 with SEM EDS to observe evidence of nickel in the fuel matrix.

Thermal Conductivity of AGR-2 Particles

Collaborators:

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Ethan Hisle

Amey Khanolkar

Tsvetoslav Pavlov

Subhashish Meher

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Background and Motivation – Thermal Conductivity

- Measurements of thermophysical properties are used in thermomechanical fuel performance code to model the fuel performance in normal and abnormal conditions. In PARFUME:
  - Thermal conductivity of SiC:

$$k = \frac{17885}{T+273} + 2 \qquad \text{W/(m·K), T in °C}$$

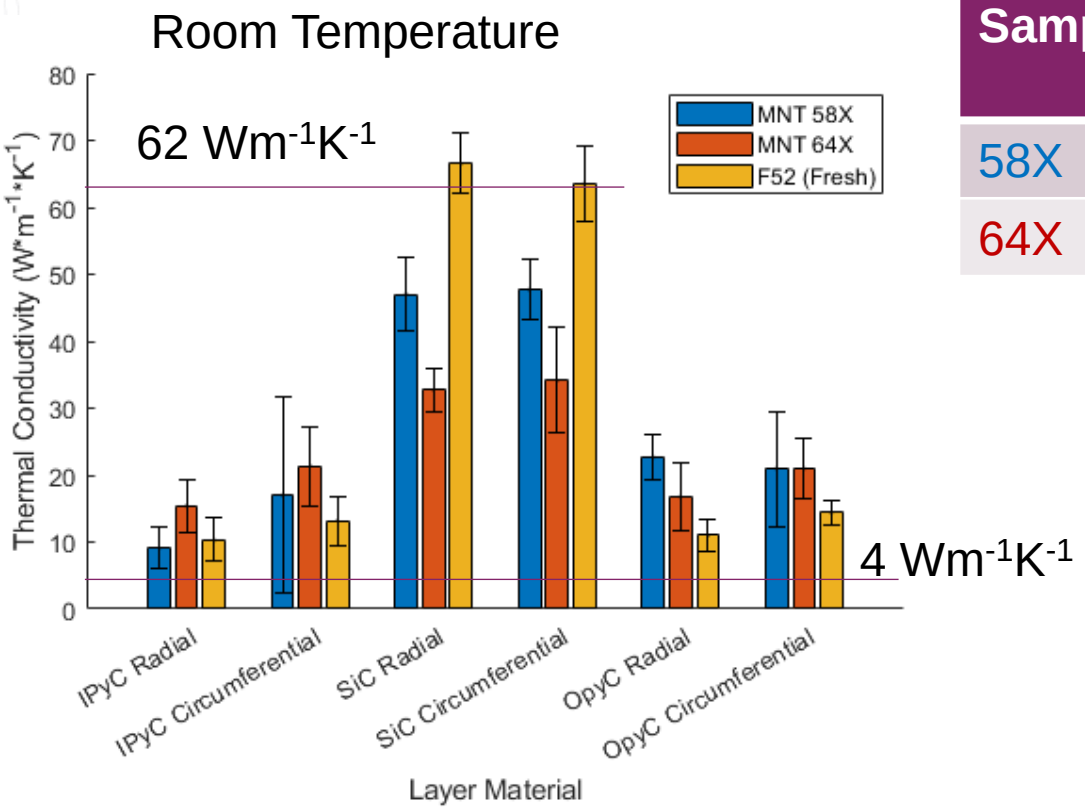
  - Thermal conductivity of IPyC and OPyC: 4 $Wm^{-1}K^{-1}$

- The sensitivity study (INL/EXT-18-44631) shows that variations of the thermal conductivity in each layer have negligible impact on the probability of SiC failure.

- This study uses a thermal conductivity microscope (TCM) to provide microscale measurements of thermal diffusivity and thermal conductivity. The objectives are to evaluate:
  - Accuracy of the values used in the models
  - How irradiation impacts the thermal conductivity

Ref: INL/EXT-08-14497 Rev.1, "PARFUME Theory and Model Basis Report".
INL/EXT-18-44631, "Assessment of Material Properties for TRISO Fuel Particles used in PARFUME".

Results – Thermal Conductivity



Irradiated Sample	Compact (AGR-2)	TAVA temperature (°C)	Burnup (% FIMA)	Fluence (10^{25} n/m^2 E>0.18 MeV)
58X	2-4-3	1216	11.52	3.08
64X	5-1-3	1078	11.09	3.03

- The SiC thermal conductivity from the fresh fuel matches well with that in PARFUME.
- IPyC and OPyC have a 25 to 50% **increase** in thermal conductivity after irradiation. Layers appear to densify which may improve thermal transport.
- SiC thermal conductivity **decreases** *at least* 30 – 50% post irradiation depending on compact which is in line with presence of irradiation induced defects.

Ref: INL/EXT-08-14497 Rev.1, "PARFUME Theory and Model Basis Report".
INL/EXT-18-44631, "Assessment of Material Properties for TRISO Fuel Particles used in PARFUME".

Summary – Thermal Conductivity

- OPyC and IPyC thermal conductivity increases with irradiation.
- SiC thermal conductivity decreases with irradiation.
- The changes in both instances are small relative to the ranges for these properties used in the PARFUME sensitivity study.

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