



# Micro- and Nano-techniques for the Study of Fission Product Precipitation in SiC Layer

June 2021

*Changing the World's Energy Future*

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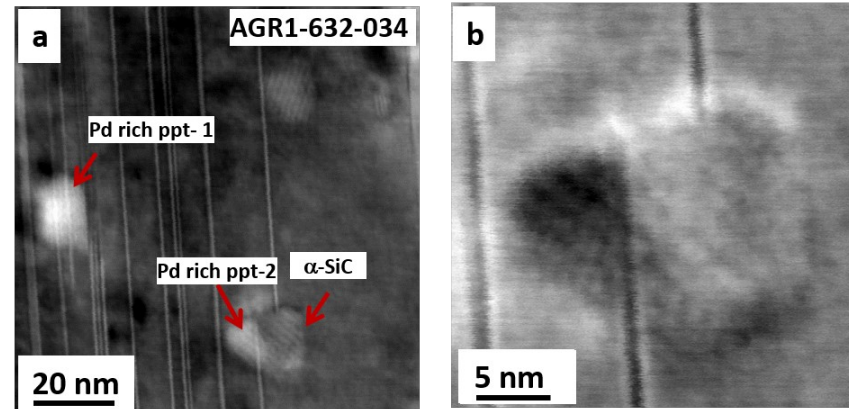
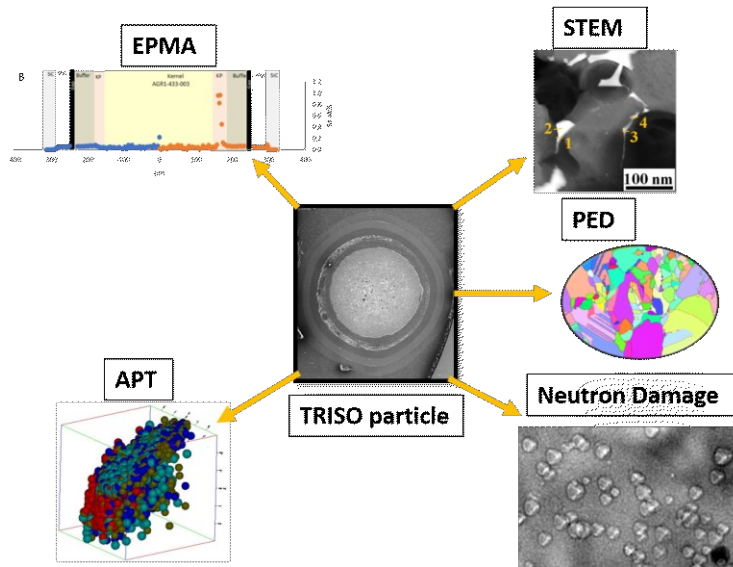
**June 2021**

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**Prepared for the  
U.S. Department of Energy  
Under DOE Idaho Operations Office  
Contract DE-AC07-05ID14517**

## Micro-and Nano-Characterization Techniques



June 2, 2021

Subhashish Meher

Idaho National Laboratory, USA

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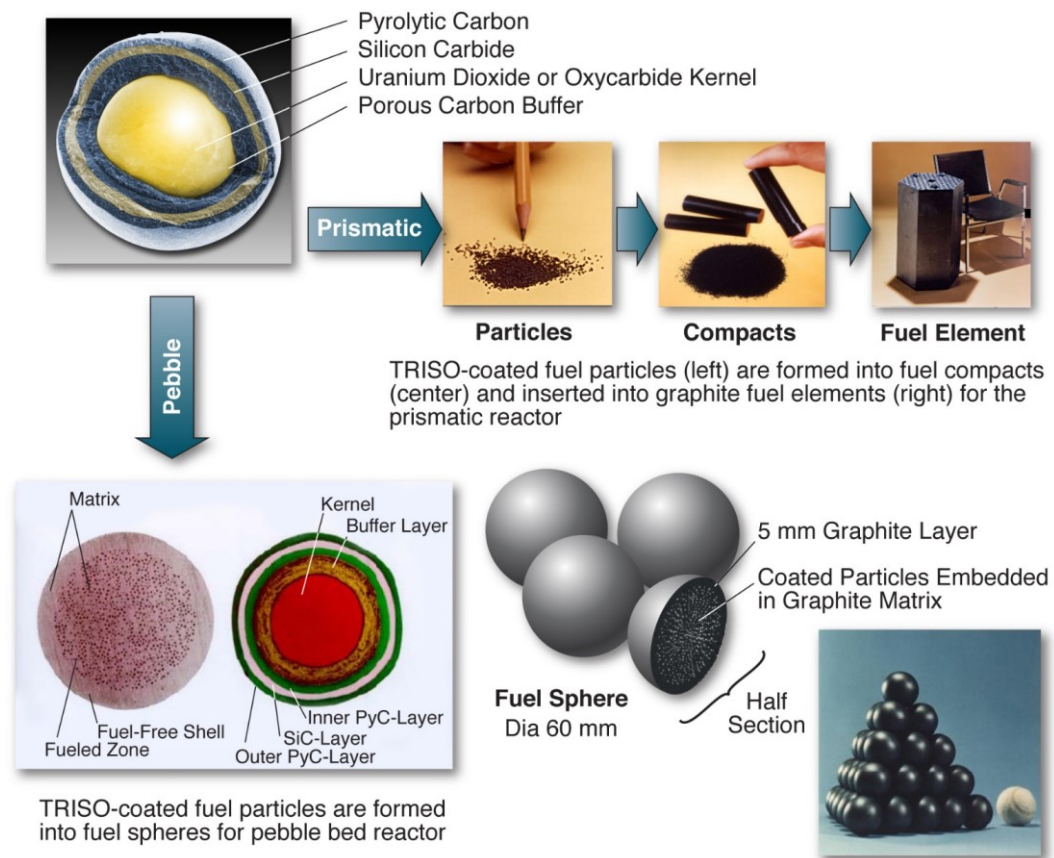
Isabella J. van Rooyen, S. Meher, Karen Wright, Thomas Lillo  
Idaho National Laboratory, USA

INL/CON-20-58872

HTR-2021, June 2-5, 2021, Virtual Conference

# Introduction

- Several advanced reactor designs incorporate tristructural isotropic (TRISO) fuel particles to achieve high coolant temperatures and increased thermodynamic efficiency
- TRISO coatings are deposited on the fuel kernel by chemical vapor deposition (CVD)
- Pyrocarbon and SiC layers retain fission gases. SiC layer is primary “pressure vessel” and retains condensable fission products (e.g. cesium, europium, strontium, etc.)



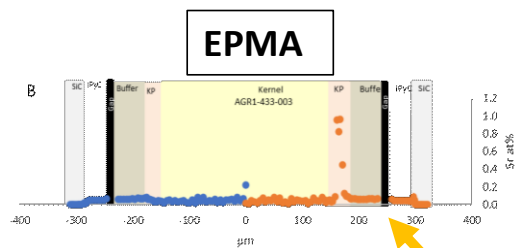
[T. Allen et al., Materials Today, 13 (2010) 14-23]

08-GA50711-01-R1

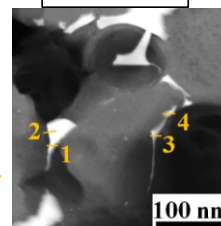
# Introduction

## Micro-and Nano-Characterization Techniques

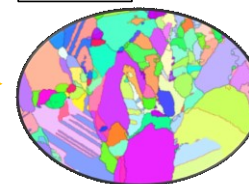
Fission Product  
Identification & Location



STEM



PED

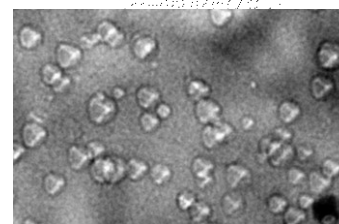


Characterize effects of Radiation upon:

- ❖ kernel porosity
- ❖ layer debonding,
- ❖ fission product precipitation,
- ❖ microstructure
- ❖ Determine microstructural differences between particles exhibited high and low releases of Ag-110m

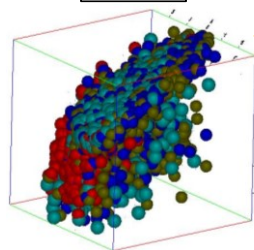
TRISO particle

Neutron Damage

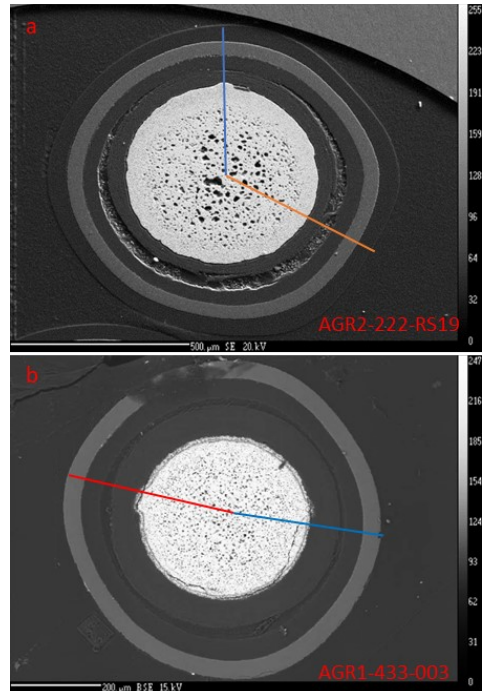


Fission Product Transport  
Mechanism

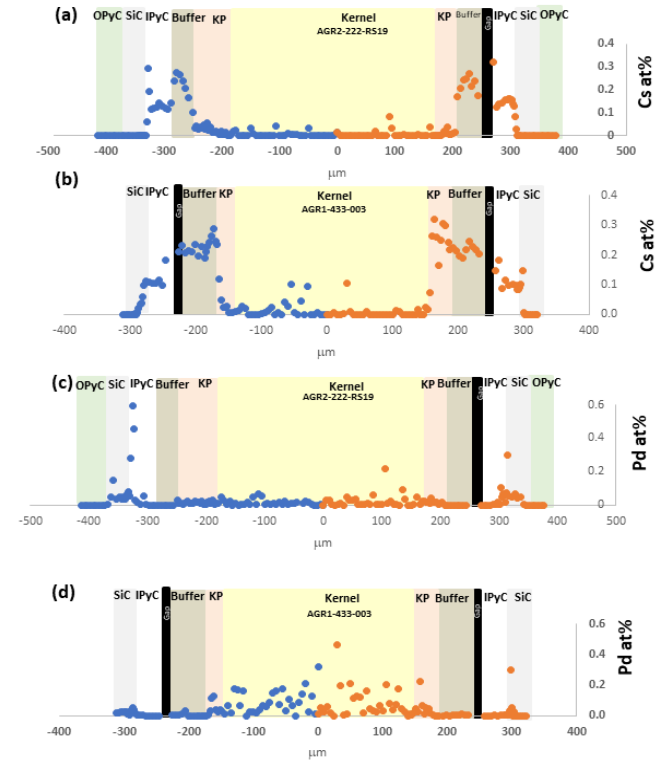
APT



# Electron Probe Microanalyses of AGR-1 and AGR-2 Safety-Tested Particles



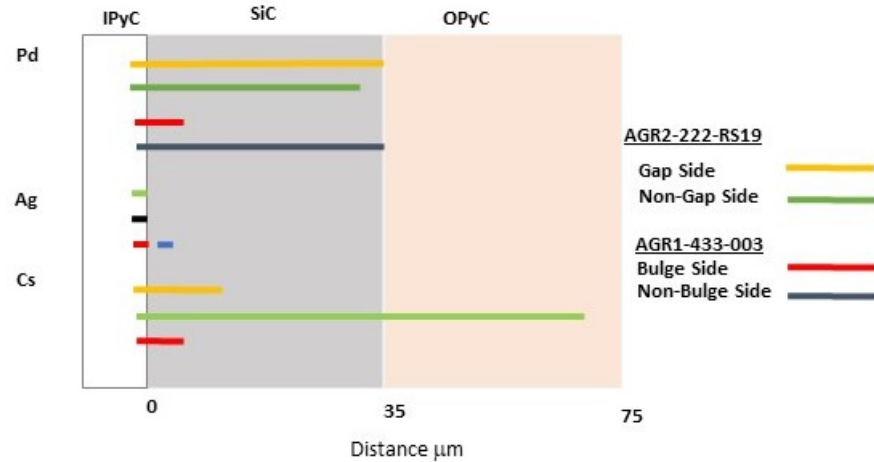
SEM micrographs showing the profile projections from particles (a) AGR2-222\_RS06 and (b) AGR1-433-003, with orange lines at the gap side and blue lines at the non-gap side.



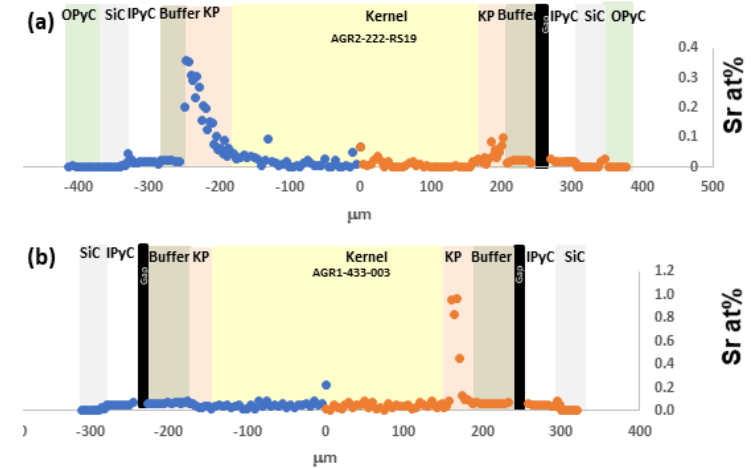
Cs profile across the two radii in particles AGR2-222-RS019 and AGR1-433-003 (a-b); Pd profile across the two radii in particles AGR2-222-RS019 and AGR1-433-003 (c-d). Orange symbols represent a radius with an aberration such as a kernel bulge or a large gap between the buffer and IPyC, while blue symbols represent a radius with fewer imperfections.



# Electron Probe Microanalyses of AGR-1 and AGR-2 Safety-Tested Particles



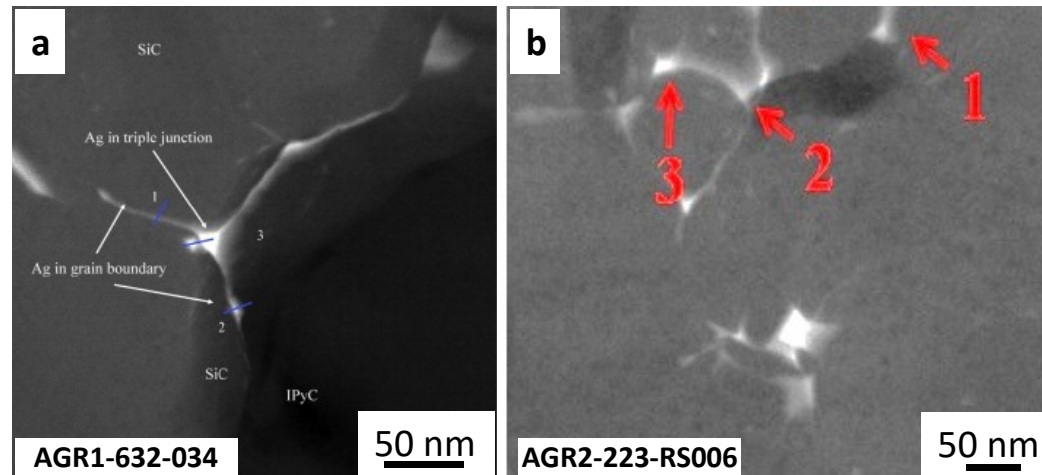
Penetration depth of Pd, Ag, and Cs into the outer layers of both particles. Note that fission product quantity in the OPyC was not measured in particle



Sr profile across the two radii in particles AGR2-222-RS019 and AGR1-433-003. Orange symbols represent a radius with an aberration such as a kernel bulge or a large gap between the buffer and IPyC, while blue symbols represent radii with fewer imperfections.



# Identification of Ag in Grain Boundaries and Triple Points

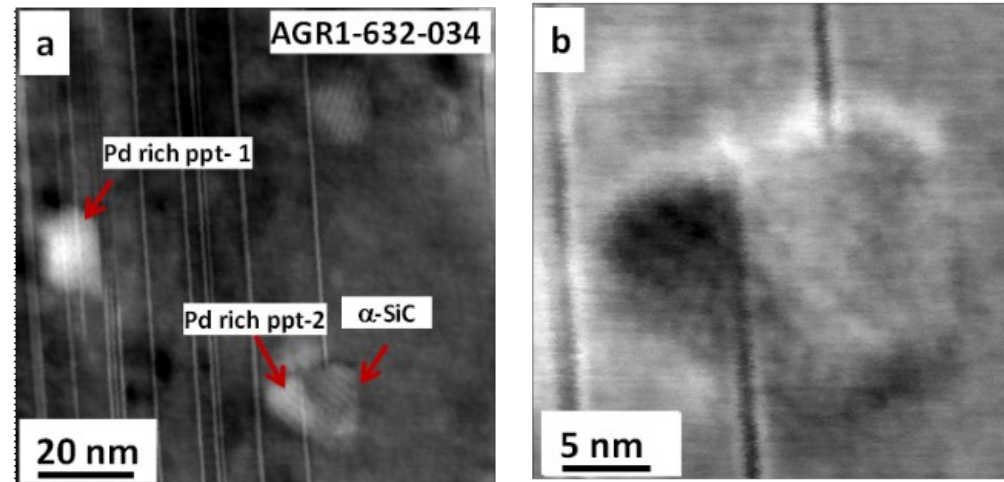


STEM images identify Ag in SiC grain boundaries and triple junctions in (a) AGR-1 [4] and (b) AGR-2 TRISO particle.

Qualitative spot EDS compositions (at.%) from the locations highlighted in Figure 5 (b) taken from particle AGR2-223-RS006.

Point	Si	C	Pd	Ag	Cd	U
1	55.9	41.1	0.46	2.45	0.01	0
2	54.8	40.8	0	3.56	0.75	0.05
3	52.7	41.4	0.17	5.08	0.67	0

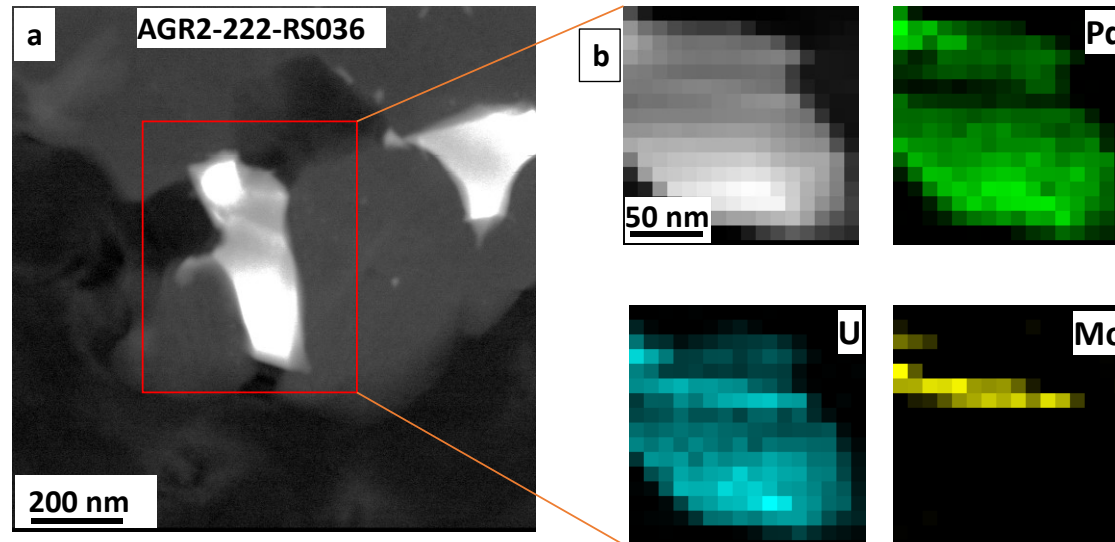
# Identification of Pd both Inter- and Intragranularly in SiC



STEM images show intragranular transport of Pd in the SiC layer of TRISO particles, occurring via a dual-step transformation process.

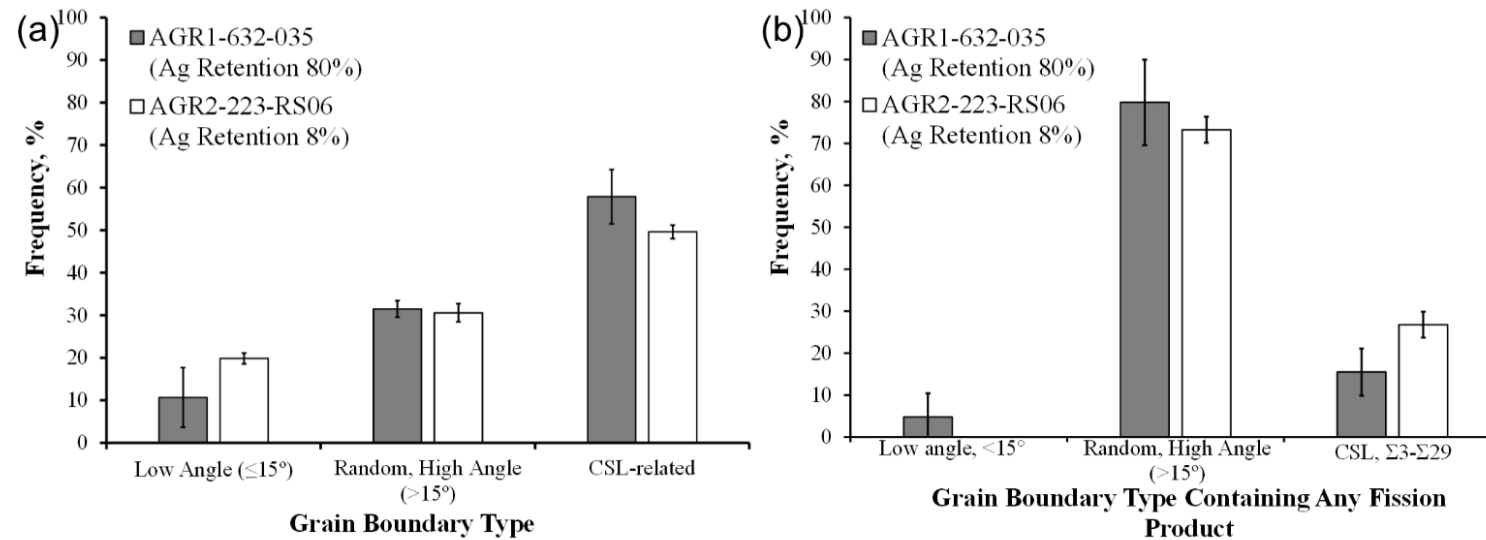
	Si	C	Pd	Ag	Cd	U
Pd-rich ppt-1	66.9	29.4	0.7	0	0.2	0.1
Pd-rich ppt-2	66.5	29.4	0.8	0	0	0
$\alpha$ -SiC	62.1	37.0	0.2	0	0	0

# Qualitative Chemical Composition of Fission Product Precipitates Using EDS



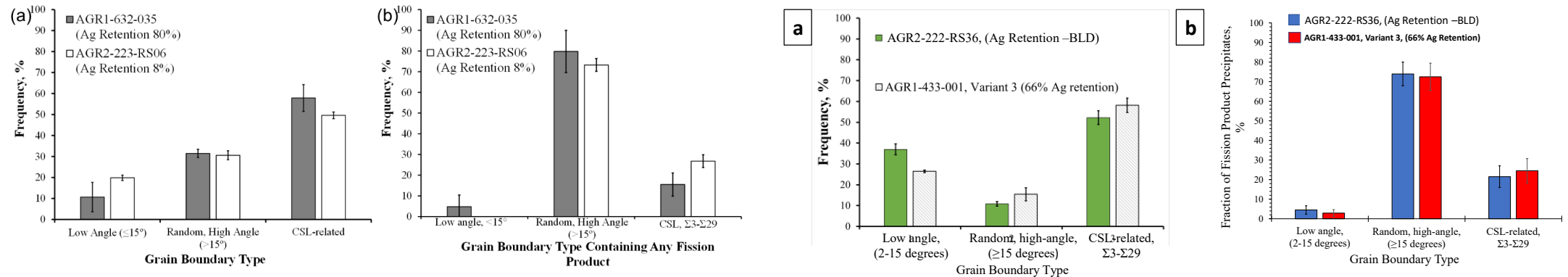
STEM image showing grain boundary fission product precipitates, and (b) associated fission-product spatial maps showing the different chemical segregations within the same large precipitate in the SiC layer of TRISO particle AGR2-222-RS036.

# *SiC Grain Boundary Nature and Their Association with Fission Products Using PED*



Comparison of SiC grain boundary types in AGR-1/AGR-2 irradiated TRISO particles, showing the effect of fabrication methods on grain boundaries and fission product transport in particles with near-identical burnup levels.

# SiC Grain Boundary Nature and Their Association with Fission Products Using PED



Comparison of SiC grain boundary types in AGR-1/AGR-2 irradiated TRISO particles, showing the effect of fabrication methods on grain boundaries and fission product transport in particles with near-identical burnup levels.

Fig. 10: Comparison of SiC grain boundary types in AGR-1/AGR-2 irradiated and safety-tested TRISO particles.

# Conclusions

- EPMA results also suggest that Ag penetration is not detected beyond the SiC-IPyC interface in both AGR-1 and AGR-2 particles investigated.
- TEM results, Ag is identified at intergranular sites within the SiC layer in both the AGR-1 and AGR-2 particles. The Ag is usually accompanied by Cd and a small amount of Pd.
- The fission product Pd can transport inter- and intragranularly in the SiC layer. However, intragranular transport of Pd is accomplished through a dual-step nucleation process requiring polymorphic transformation of  $\beta$ -SiC into  $\alpha$ -SiC.
- Compositional segregation within precipitates in the SiC layer demonstrates that Mo and Pd are segregated within the same precipitate.

# Acknowledgements

- This work was sponsored by the United States (U.S.) Department of Energy, Office of Nuclear Energy, under U.S. Department of Energy Idaho Operations Office Contract DE-AC07-05ID14517, as part of the Advanced Reactor Demonstration Program. The U.S. Government retains and the publisher, by accepting the article for publication, acknowledges that the U.S. Government retains a nonexclusive, paid-up, irrevocable, world-wide license to publish or reproduce the published form of this manuscript, or allow others to do so, for U.S. Government purposes.
- The authors would like to acknowledge the efforts of:
  - Staff at the Materials and Fuels Complex at Idaho National Laboratory.
  - TEM work was performed at the Center for Advanced Energy Studies-Microscopy and Characterization Suite.





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