Advanced Gas Reactor Fuels Program Review

AGR-1 Advanced Electron Microscopy Progress

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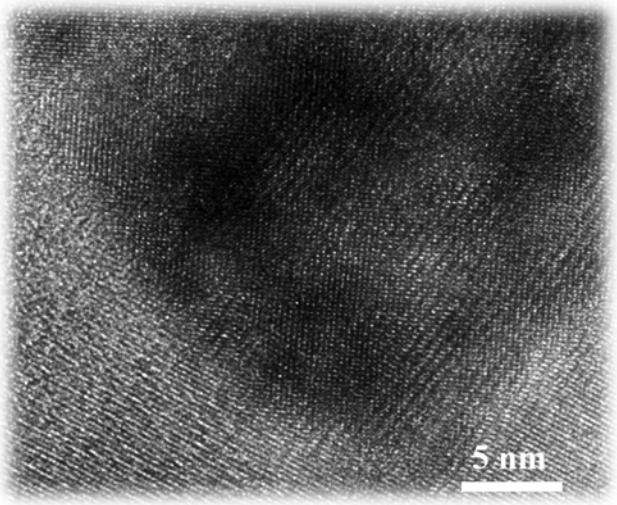
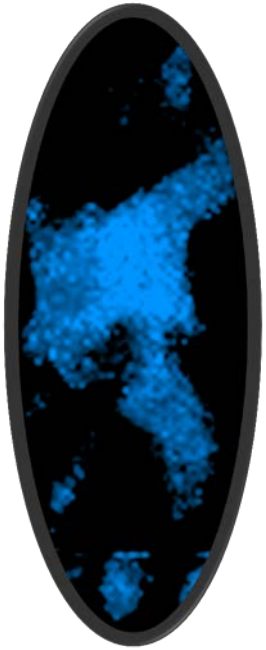
July 15-16, 2015





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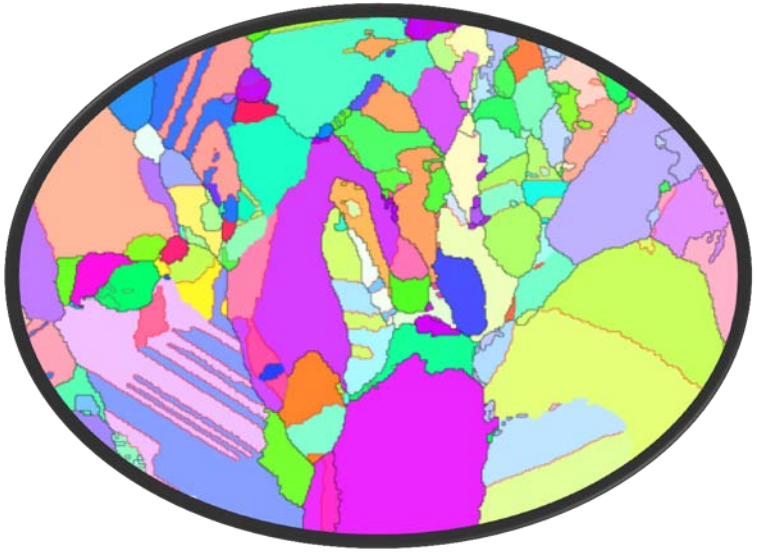
Outline

- Approach and advanced techniques applied
- Progress overview
- Preliminary results
- Technical highlights
- Activities in progress and collaborations
- Publications and conference papers
- Acknowledgements



Approach and Electron Microscopic Techniques

Decontamination in glove box

Electron probe micro-analyzer (EPMA)

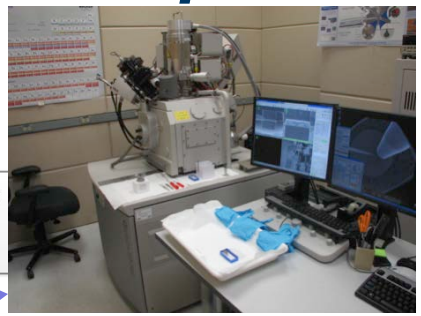
Scanning Electron Microscopy (SEM)

- Main elements of precipitates
- Precipitate distribution
- Identify areas for TEM ★

Electron back scatter diffraction (EBSD)

Focused Ion Beam (FIB)

- Preparation for TEM, STEM, APT, HRTEM
- EBSD, TKD; ASTAR collection



FEI Quanta 3D FEG Dualbeam FIB at EML at MFC

Transmission Electron Microscopy (TEM)

- Micro-Nano sized imaging
- Analytical tools (EDS)
- 2 D imaging

Scanning Transmission Electron Microscopy (STEM) ★

Energy Dispersive Spectroscopy (EDS)
Energy Filtered TEM (EFTEM)
Electron Energy Loss Spectroscopy (EELS)

- Nano sized imaging
- Analytical tools
- 2 D imaging

★ Atom Probe (APT)

- 3D analytical tool

★ High Resolution Transmission Electron Microscopy (HRTEM)

- Atomic structure and imaging
- EELS higher resolution

Transmission Kikuchi Diffraction (TKD)

- Crystallographic information

ASTAR ★

- Crystallographic information

FEI Tecnai G^2 F30 STEM at the Center for Advanced Energy Studies (CAES)

AGR-1 Progress Overview

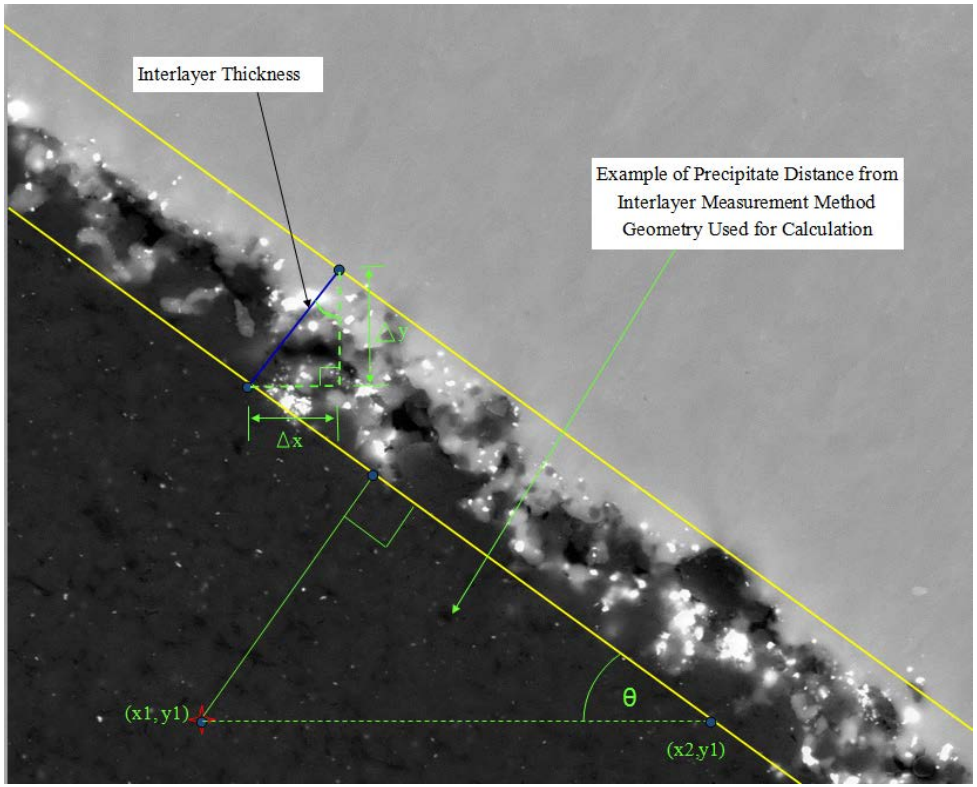
Technique	Compact 6-3-2	Compact 4-1-1	Compact 5-3-1	Compact 1-3-1	Compact 4-3-3	Compact 5-2-3	Un-irradiated	Planned Deliverables (Publications)
Basic Scanning Electron Microscopy (SEM)								**Summary Report 1** Conference Presentation 1 [1]
Basic SEM	FY2012	FY2012	FY2013	FY2013	FY2014	Not planned	Not planned	
SEM Montage	FY2012	FY2013	FY2014 FY2015 (22U)	FY2014 FY2015 (26V)	FY2014	Not planned	Not planned	
Advanced Microscopy Techniques*								**Summary Report 2**
Basic TEM	FY2012/ FY2013	FY2013	Not planned	Not planned	FY2014	FY2014	FY1012/ FY2013	
APT	NSUF 2014 NSUF 2015	Not planned	Not planned	Not planned	Not planned	Not planned	Not planned	(NSUF-RTE report 1) (Conference presentation (2016))
EPMA	Not planned	Not planned	Not planned	Not planned	Not planned	FY2014	FY2012	
STEM	FY2013 NSUF 2014	FY2013/FY2014	FY2014	FY2015 CP 099 complete CP 066 in progress	FY2015 Particle 4 in progress	FY2015	Not planned	**Failure Analysis Report 3 (may be replaced by paper)** Journal paper 1 [2] (Journal paper 2) [3] Journal paper 7 [11] Journal paper 8 [12] Conference paper 1 [4] Conference paper 2 [8]
EELS , EFTEM	(NSUF 2014/2015)	Not planned	Not planned	Not planned	Not planned	Not planned	Not planned	
HRTEM	Not planned	FY2014	Not planned	FY2015	Not planned	Not planned	Not planned	Conference paper 1 [5] Journal paper 4 Journal paper 10 (2016) Conference presentation (2016)
EBSD	FY2013/FY2014	FY2013/FY2014	Not planned	Not planned	Not planned	Not planned	FY2012	
TKD	FY2014	FY2014	Not planned	Not planned	Not planned	Not planned	Not planned	Conference presentation 2 [6]
STEM (UCO kernel)	FY2015 (new)					FY2015 (new)		Journal paper 9 (2016)
PED (ASTAR)	NSUF 2014	Not planned	FY2015	FY2015	Not planned	Not planned	NSUF 2014	(NSUF-RTE report 2) Conference paper 3 [7] Journal paper 5 [9] Journal paper 6 [10]

Preliminary Results: SEM Fission product distribution Montage

Precipitate Distribution and Quantification

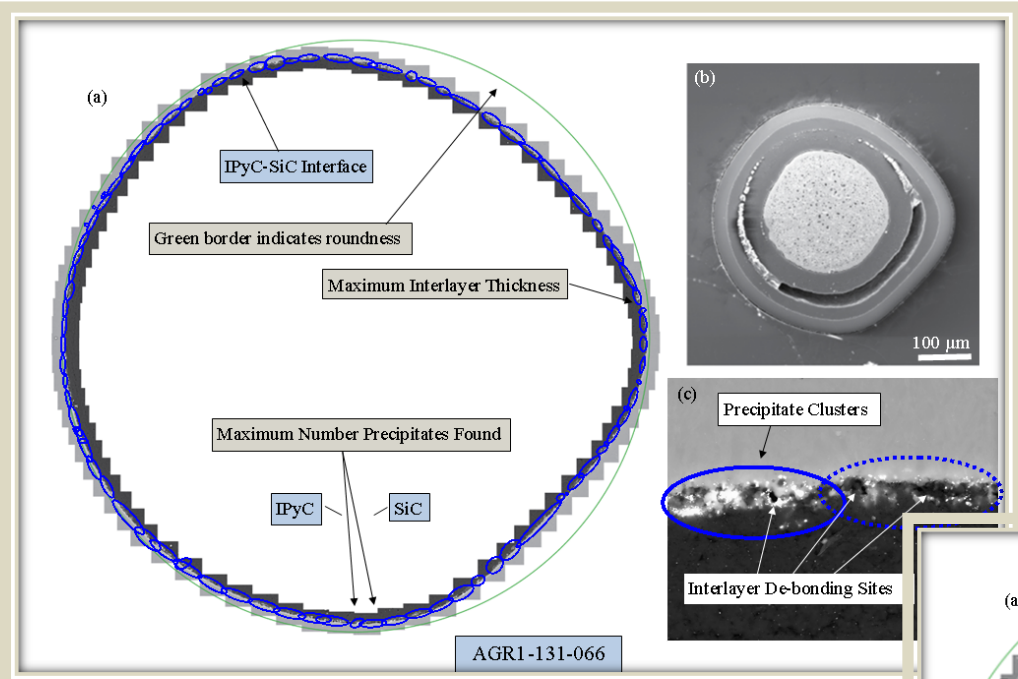
Phased Approach:

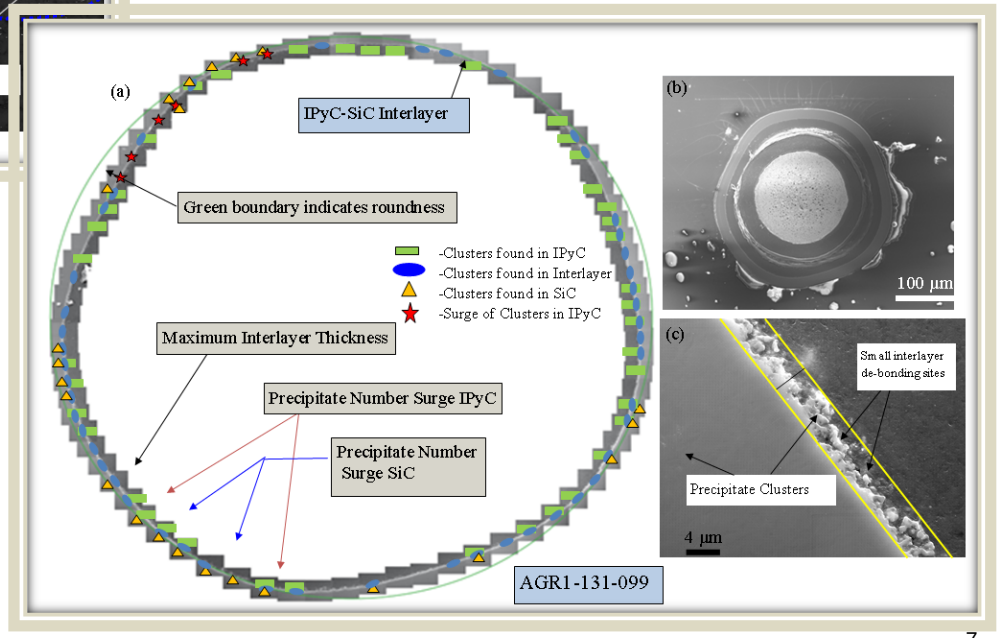
- JEOL 7000 FEG SEM: sequential secondary electron images of the inner-pyrolitic carbon (IPyC)-SiC interface at 4000x magnification
- ~81 micrographs per particle
- Identify Pd precipitates with energy-dispersive x-ray spectroscopy (EDS)
- Integrate in montage Adobe Photoshop
- Identify Pd precipitate clusters
- Measure depth of Pd precipitates in IPyC and SiC
- Measure interlayer thickness



[Ref. Van Rooyen et al., TMS 2015, 144th Annual Meeting, March 15-19, 2015; Walt Disney World Orlando, Florida, USA]

Precipitate Distribution and Quantification (AGR1-131)





[Ref. Van Rooyen et al., TMS 2015, 144th Annual Meeting, March 15-19, 2015; Walt Disney World Orlando, Florida, USA]

Precipitate Distribution and Quantification in SiC

Sample Description								
COMPACT	6-3-2	1-3-1	1-3-1	5-3-1	5-3-1	4-1-1	4-1-1	4-1-1
MOUNT #	48T	26V	30V	20U-1	23U	120T	120T	120T
CP #	34	66	99	38	31	21	29	30
Number of Precipitates								
Average	Not available	72	2	146	183	Not available	43	21
Maximum	Not available	275	197	373	500	Not available	136	62
Minimum	Not available	13	45	44	93	Not available	4	3
Furthest Distance from Interlayer (µm)								
Average	6.7	10.24	13.6	14.3	14.2	Not available	11.46	10.26
Maximum	15.3	17.39	19.95	19.7	21	Not available	18.95	18.25

- Large variation in number of precipitates between particles from same compact for Variant 1 & 3 fuel particles
- AGR1-131-099: Precipitates moved furthest in SiC layer
- AGR1-531-031: Most precipitates in SiC layer

[Ref. Van Rooyen et al., TMS 2015, 144th Annual Meeting, March 15-19, 2015; Walt Disney World Orlando, Florida, USA]

Effect of interlayer thickness

INTERLAYER THICKNESS (µm)								
COMPACT	6-3-2	1-3-1	1-3-1	5-3-1	5-3-1	4-1-1	4-1-1	4-1-1
MOUNT #	48T	26V	30V	20U-1	23U	120T	120T	120T
CP #	34	66	99	38	31	21	29	30
Average	1.85	3.39	2.37	1.91	1.87	3.73	2.20	2.18
Maximum	3.69	5.61	3.84	3.00	2.95	6.58	3.59	3.19
Minimum	0.65	1.88	1.27	1.00	1.08	1.86	1.3	1.23

- Large variation between particles from same compact for Variant 3 fuel particles
- AGR1-411-021 largest interlayer thickness

[Ref. Van Rooyen et al.,TMS 2015, 144th Annual Meeting, March 15-19, 2015; Walt Disney World Orlando, Florida, USA]

SEM Conclusions and next Actions

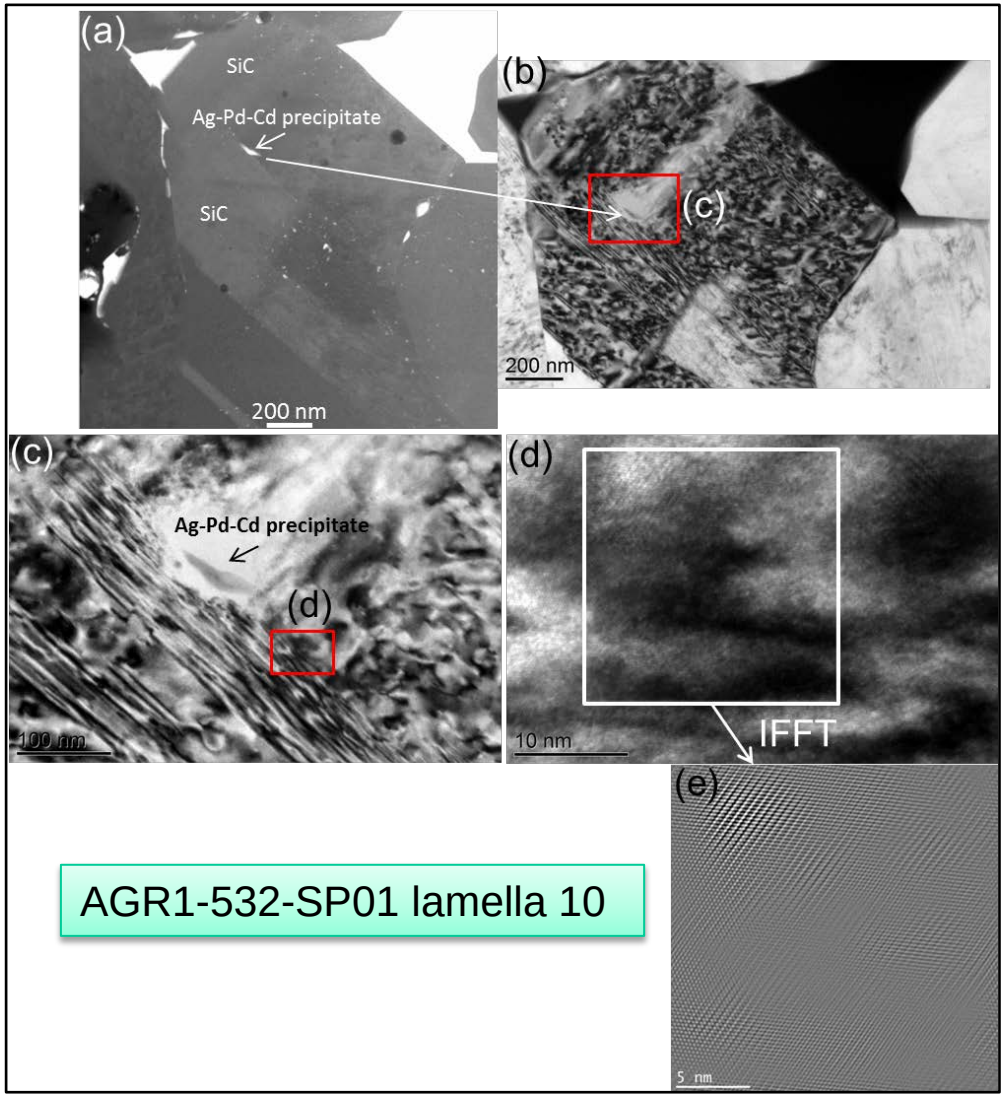
- Successful achievement of developing a simplistic tool for evaluating the precipitate distribution as a comparative tool
- A standardize method for interlayer thickness was developed and demonstrated. (Note that no standard interlayer thickness measurement has been established in the HTR community to date).
- Precipitate montage results summary in SiC layer:
  - Large variation in number of precipitates between particles from same compact for Variant 1 & 3 fuel particles
  - **AGR1-131-099: Furthest movement of precipitates in SiC layer**
  - AGR1-531-03: Most precipitates in SiC layer
- Precipitate montage results summary in IPyC layer:
  - Large variation in number of precipitates between particles from same compact for Variant 3 fuel particles
  - AGR1-411-029: Most precipitates in IPyC layer
  - AGR1-411-029: Furthest movement of precipitates in IPyC layer
- **AGR1-411-021 has the largest interlayer thickness**
- Next actions to include full interpretation to integrate the quantification results with the actual Ag releases of the specific coated particle.

Preliminary Results: STEM

- AGR1-532-SPO1 (Variant 1, 17.4% FIMA, 1059°C TAVA)
- AGR1-131-099 (Variant 3, 15.3% FIMA, 1092°C TAVA)

Identified a Ag-Pd-Cd precipitate at stacking fault inside a SiC grain: AGR1-532-SPO1

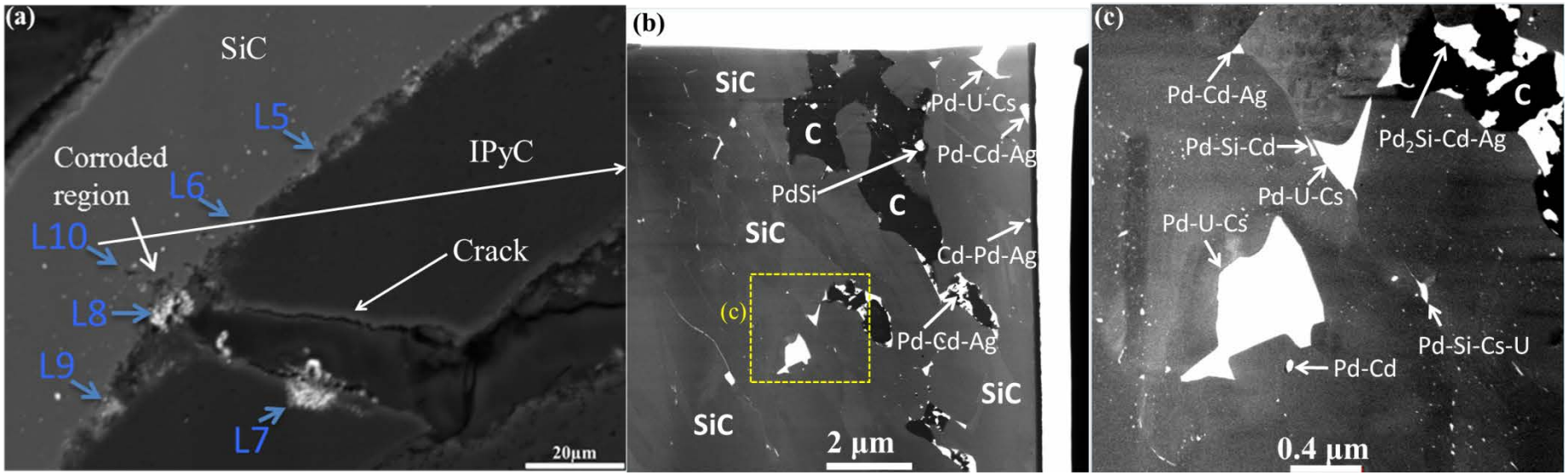
- Ag predominantly at grain boundaries and triple junction
- Previously one finding of Ag inside a SiC grain AGR1-632-35 [3]).
- Precipitate at stacking fault and not at grain boundary (STEM, TEM, STEM EDS and inverse fast fourier transformation (IFFT))



[Ref [3]. Bin Leng, Isabella van Rooyen, Yaqiao Wu, Izabela Szlufarska, Kumar Sridharan, *STEM/EDS Analysis of Fission Products in Irradiated TRISO Coated Particles of the AGR-1 Experiment*, peer review comment resolution in process, to be submitted to Journal of Nuclear Materials]

[Ref this work: Haiming Wen, Isabella J. van Rooyen, John D. Hunn, Tyler J. Gerczak, *"Advanced electron microscopy study of fission products Pd, Ag and Cs in carbon areas in the locally corroded SiC layer in a neutron irradiated TRISO fuel particle"*, to be submitted to Nuclear Engineering and Design or the Journal of Nuclear Materials, July 2015]

Particle AGR1-523-SP01, lamella 10

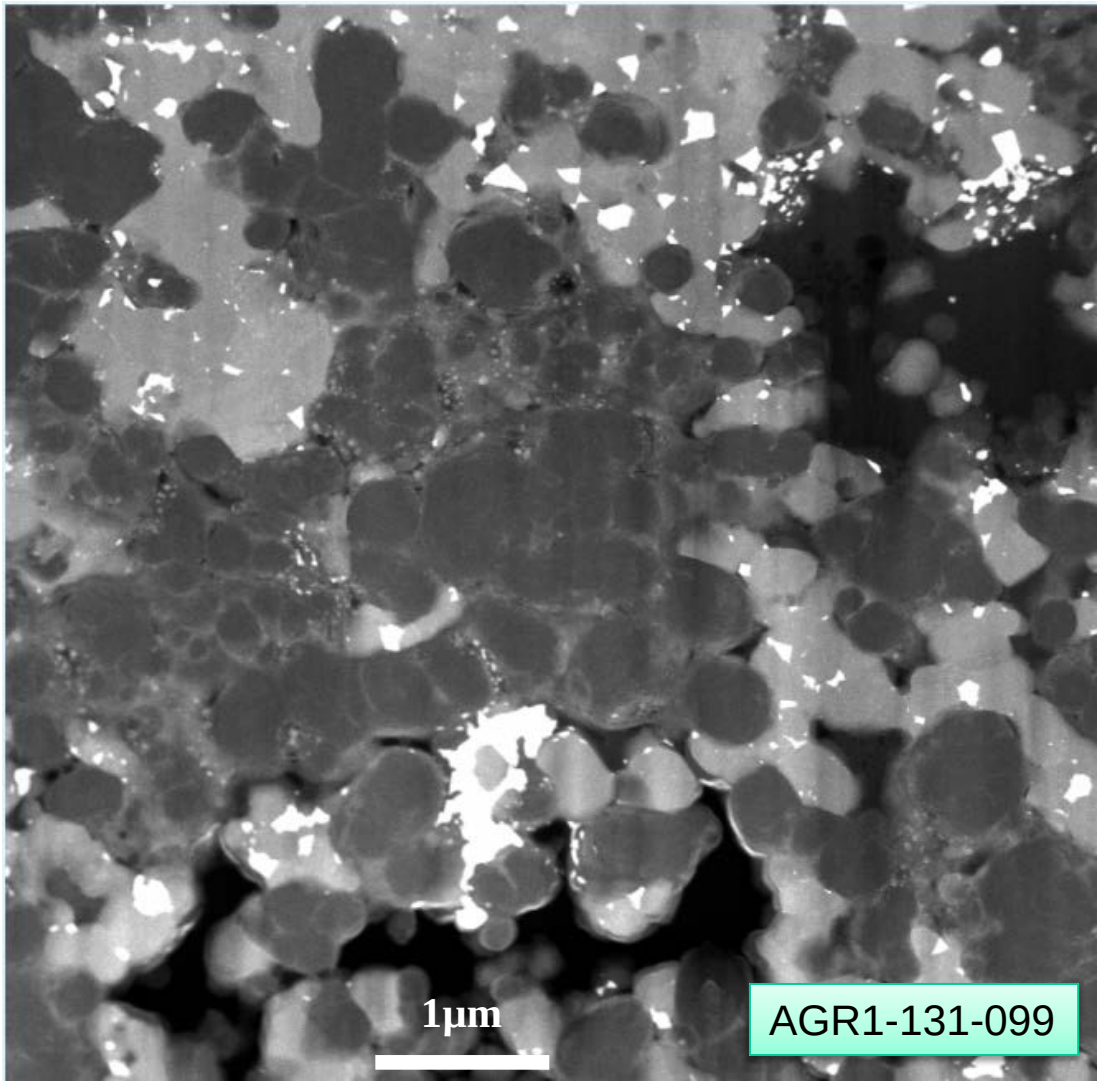


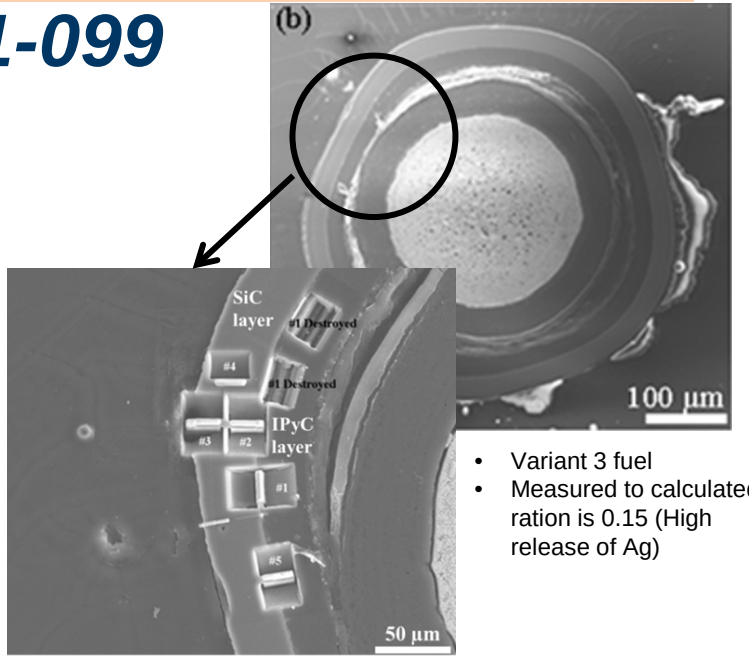
- Almost all the precipitates located in the pure carbon rich areas are identified as Pd_2Si or PdSi, with a relatively small fraction of Pd.
- Cd and/or Ag are frequently observed in palladium silicides and Pd
- Overall, in the specific carbon areas studied in ~14% precipitates contain Ag, and ~20% precipitates contain Cd.

[Ref this work: Haiming Wen, Isabella J. van Rooyen, John D. Hunn, Tyler J. Gerczak, *"Advanced electron microscopy study of fission products Pd, Ag and Cs in carbon areas in the locally corroded SiC layer in a neutron irradiated TRISO fuel particle"*, to be submitted to Nuclear Engineering and Design or the Journal of Nuclear Materials, July 2015]

STEM TRISO Particle AGR1-131-099

IPyC-SiC interface





- Variant 3 fuel
- Measured to calculated ration is 0.15 (High release of Ag)

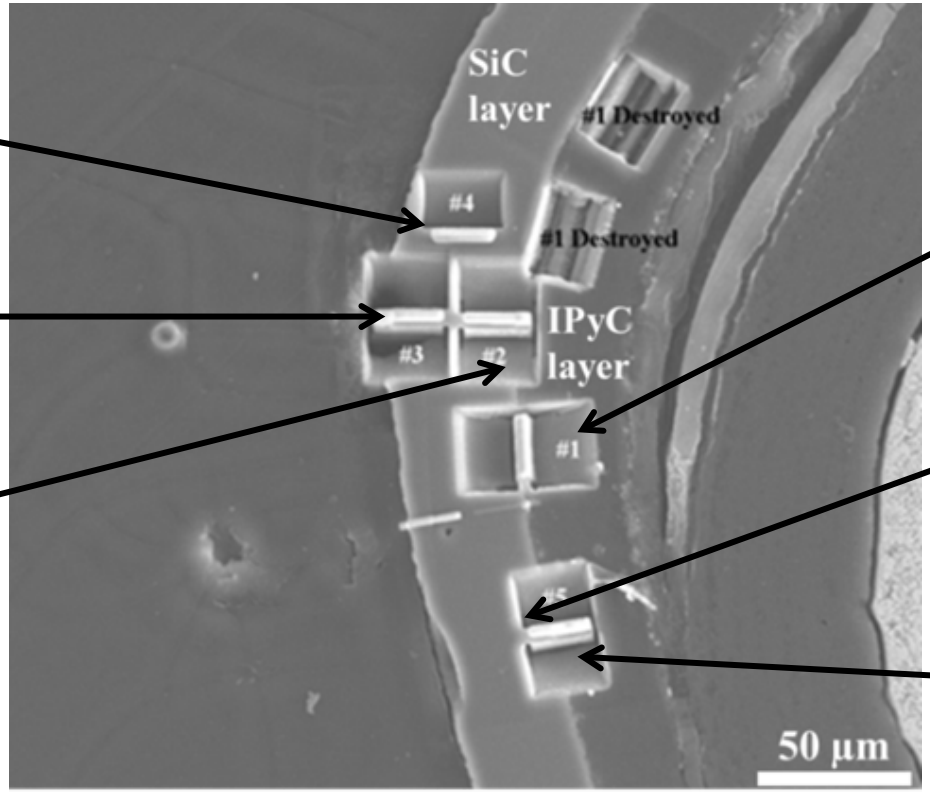
- Relatively low number density of precipitates at IPyC-SiC interface
- Precipitates smaller compared with AGR1-632-034 particle
- Pores at IPyC-SiC interface
- Smaller SiC grain sizes (~200-300 nm)

AGR1-131-099 Precipitate Composition Summary

SiC layer:
Pd-Si-U, Pd-U, Pd.

SiC layer:
Pd-Si-U, Pd-Si, Pd

SiC layer: Cd
Pd-Si-U, Pd-Si, Pd-U, Pd, U, U-Si



Cd
IPyC-SiC interlayer:
Pd-Si-U, Pd-U, U, U-Si

Cd
SiC layer:
Pd-Si-U, Pd-Si, Pd-U, Pd, U.

Cd
PyC layer:
Pd-Si-U, Pd-Si, U-Si, U

- High Ag release of the particle; no Ag-containing precipitates evident in IPyC or SiC layers.
- Decreased number of U-containing precipitates, further away from IPyC-SiC interlayer.
- U-containing precipitates present nearly across the entire SiC layer.
- U was found separately in the SiC layer, whereas in other compacts, U was not found separately.
- Almost all precipitates at IPyC-SiC interface contain U, different from other compacts.

Haiming Wen, Isabella J van Rooyen, Connie Hill, Tammy Trowbridge, Ben Coryell, *Fission product distribution in TRISO coated particles neutron irradiated to 3.22 X 10^{25} n/m^2 fast fluence at 1092°C*, ASME Power and Energy 2015, June 28 – July 2, 2015, San Diego, California.

Preliminary Results: Crystallographic Information (PED) ASTAR

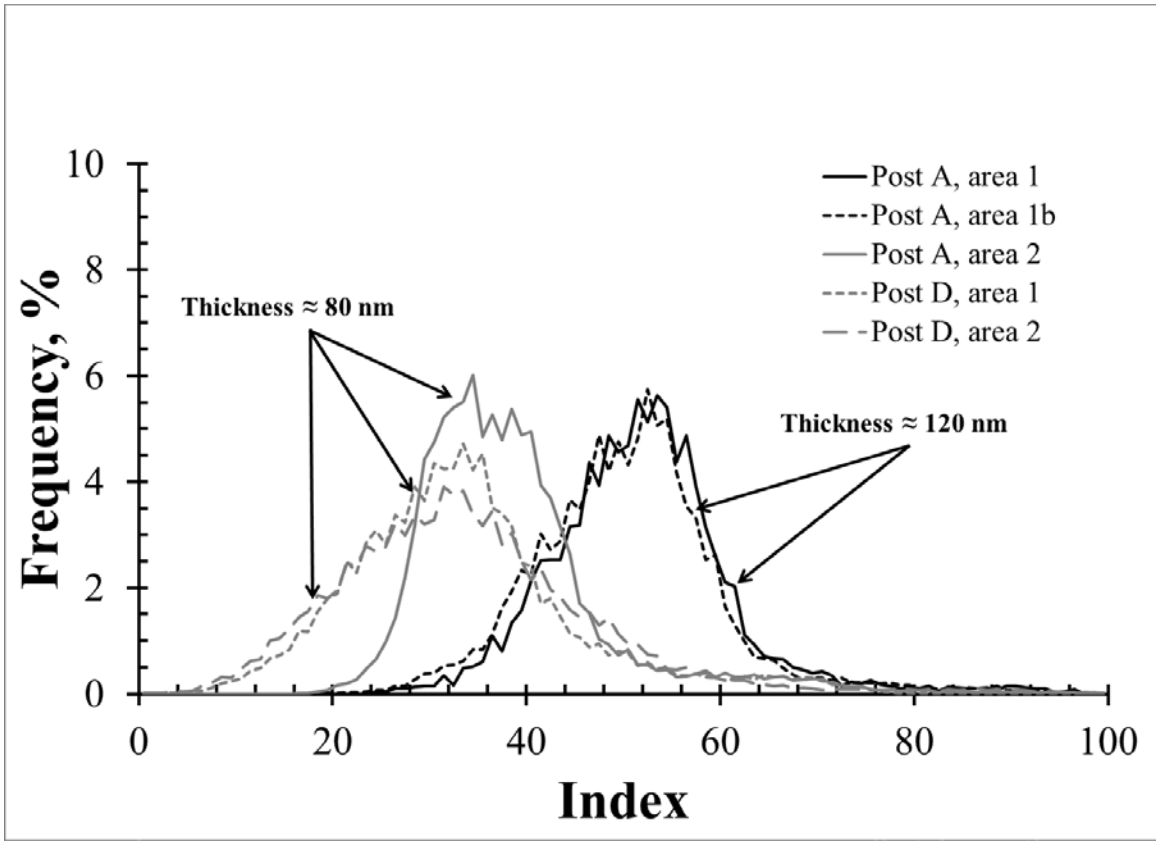
NSUF RTE13-460

Expected Outcomes for Precession Electron Diffraction (PED) measurements

- Implement emerging technique to map crystallographic orientation in the TEM for irradiated materials
- Determine the influence on sample thickness on the quality of crystallographic orientation data
- Crystallographic orientation data from PED will be used to determine grain boundary misorientation of grain boundaries with and without fission products.
  - Determine whether fission products are associated with “special” grain boundary misorientation (CSL boundaries, high angle or low angle)
  - Determine which grain boundary types will not support fission product migration
- Provide information on the fission product transport mechanism for subsequent modeling of fuel behavior during irradiation

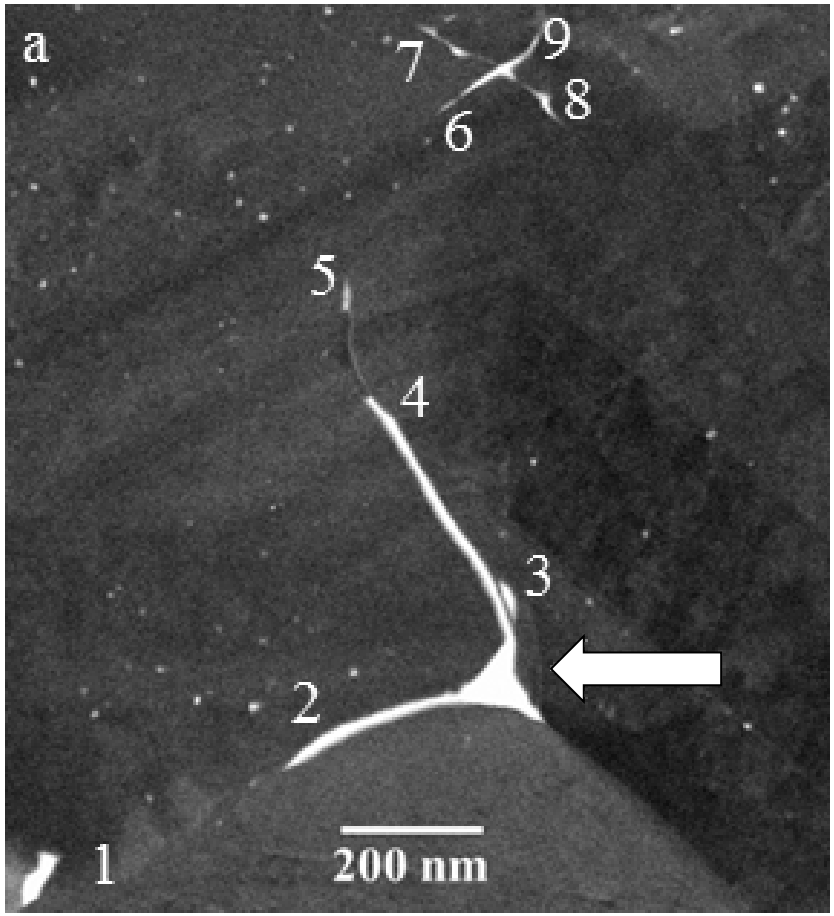
Effect of FIB-lamella thickness: PED measurements (Unirradiated SiC)

- Quality of orientation assignment by PED (ASTAR) indicated by "Index" parameter – larger value indicates high quality
- Thicker samples produce higher average values of the index parameter – thicker samples produce higher quality orientation data
- At some point, overlapping grains will degrade data – grain size dependent

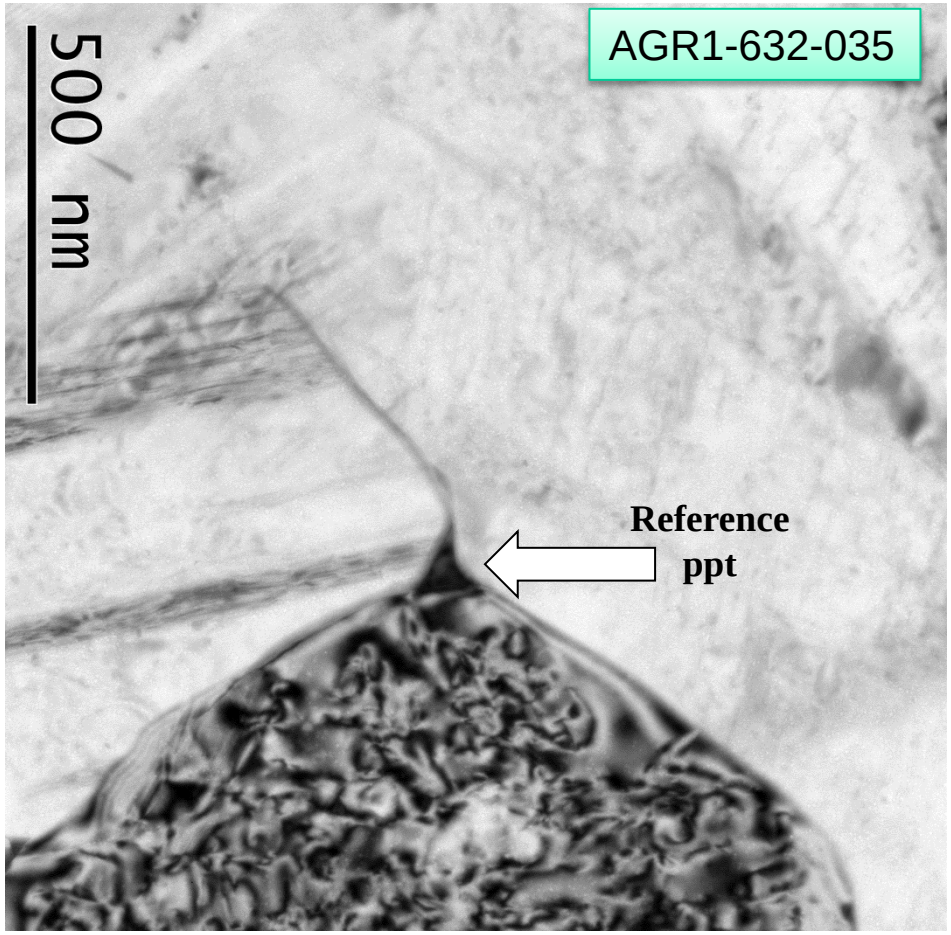


- [I. J. van Rooyen, J. Youngsman, T. M. Lillo, Y. Q. Wu, D. Goran, M. E. Lee, W. E. Goosen5, J. H. Neethling, T. L. Trowbridge and J. W. Madden, Methods for identification of crystallographic parameters of irradiated SiC to understand fission product transport, The 3rd Workshop on HTGR SiC Material Properties, Sept. 30-Oct 1, Jeju Island, South Korea]
- [T. Lillo, I.J. van Rooyen, "Precession Electron Diffraction for SiC Grain Boundary Characterization in unirradiated TRISO Fuel", to be submitted to Nuclear Engineering and Design or the Journal of Nuclear Materials, June/July 2015]
- [Isabella J van Rooyen, Yaqiao Wu, Tom Lillo, John Youngsman and Jan H Neethling, Approach and Micro-analysis techniques applied to study fission product transport mechanisms in neutron irradiated SiC layers, IMRC XXIII, Cancun, Mexico, August 17-21, 2014]

Iradiated SiC PED Experimental Results



STEM - HAADF



Bright Field TEM

[T. Lillo, I.J. van Rooyen, "Influence of SiC Grain Boundary Character on Fission Product Transport in TRISO Fuel" Draft in progress, to be submitted to the Journal of Nuclear Materials, July 2015]

Grain Orientation Map from PED data

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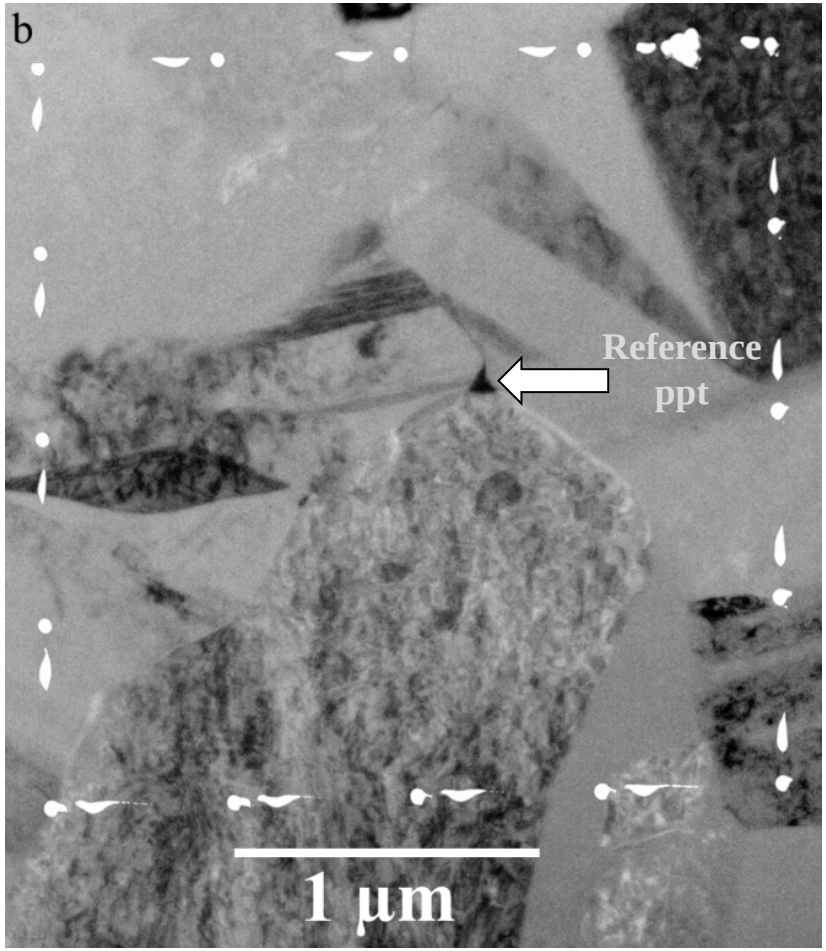
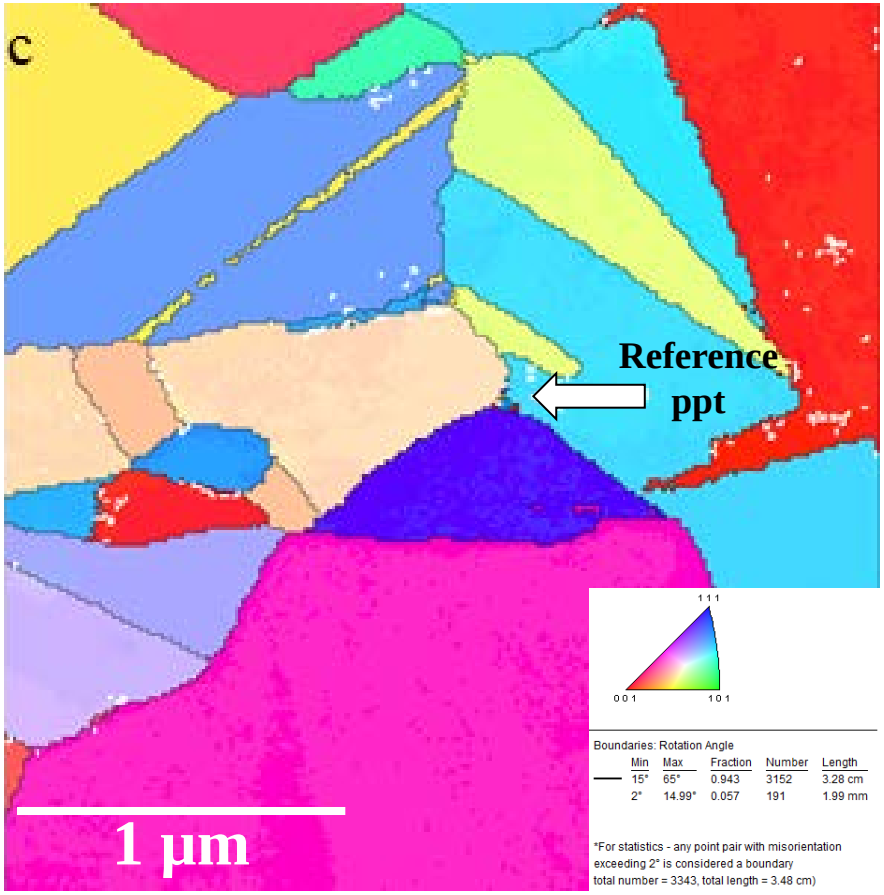


TEM with scan area



Orientation image:

High angle grain boundaries – black
Low angle grain boundaries - white

[T. Lillo, I.J. van Rooyen, "Influence of SiC Grain Boundary Character on Fission Product Transport in TRISO Fuel" Draft in progress, to be submitted to the Journal of Nuclear Materials, July 2015]

Summary PED measurements: Grain boundary Misorientation Angle

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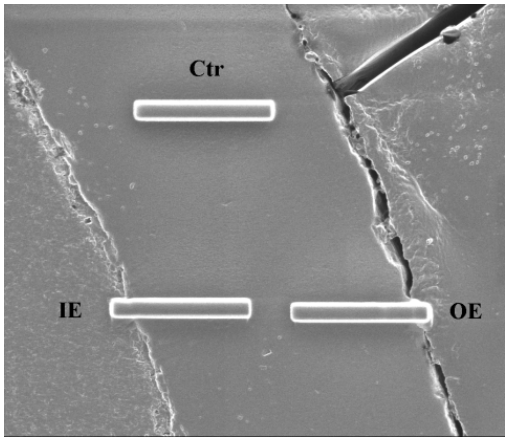
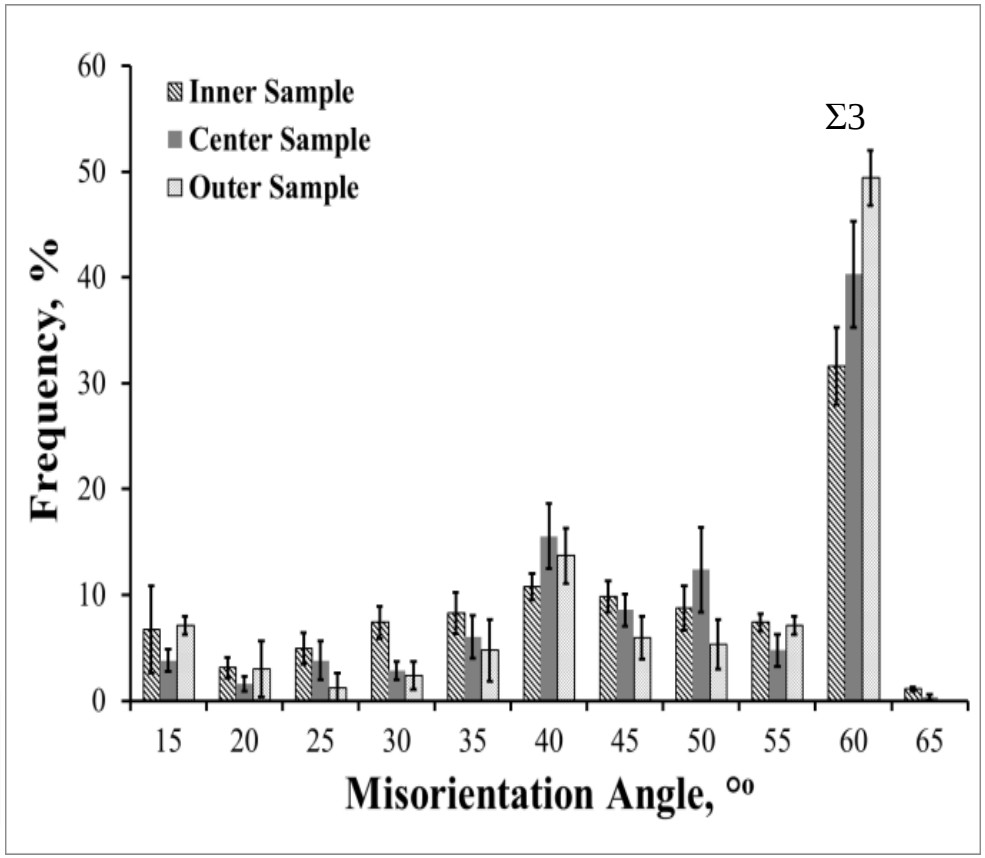


- Total number of grain boundaries analyzed > 900
- Only 11.1% of boundaries analyzed contain fission products



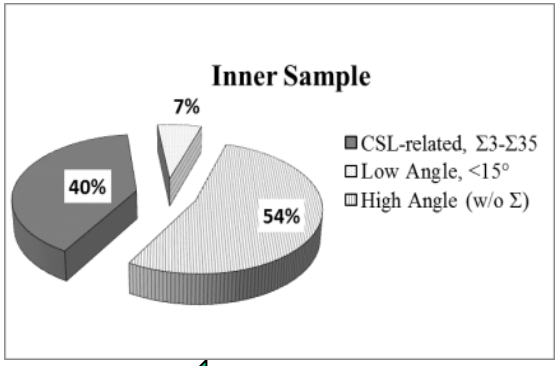
- Majority of grain boundaries: Twin boundaries, with a 60° misorientation angle; smaller peak in distribution occurs around 40°.
- The only difference among Ctr, IE, OE: frequency of twin boundaries.
  - significantly fewer twins in the IE sample where growth the SiC layer began, compared to the OE sample

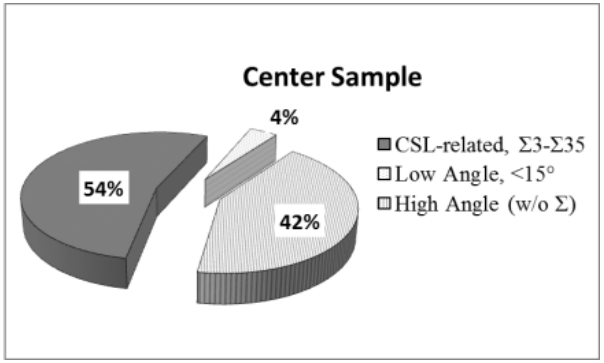
[T. Lillo, I.J. van Rooyen, "Influence of SiC Grain Boundary Character on Fission Product Transport in TRISO Fuel" Draft in progress, to be submitted to the Journal of Nuclear Materials, July 2015]

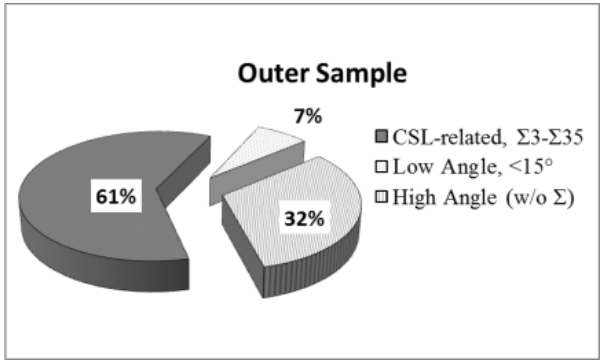
Summary of All PED measurements: Grain boundary type

AGR1-632-035

CSL-related grain boundary fraction







Fraction of random, high angle grain boundaries

Fraction of low angle grain boundaries is relatively constant across the thickness (< 10% of grain boundaries)

[T. Lillo, I.J. van Rooyen, "Influence of SiC Grain Boundary Character on Fission Product Transport in TRISO Fuel" Draft in progress, to be submitted to the Journal of Nuclear Materials, July 2015]

Summary of All PED measurements: Fission product precipitates on the various boundary types

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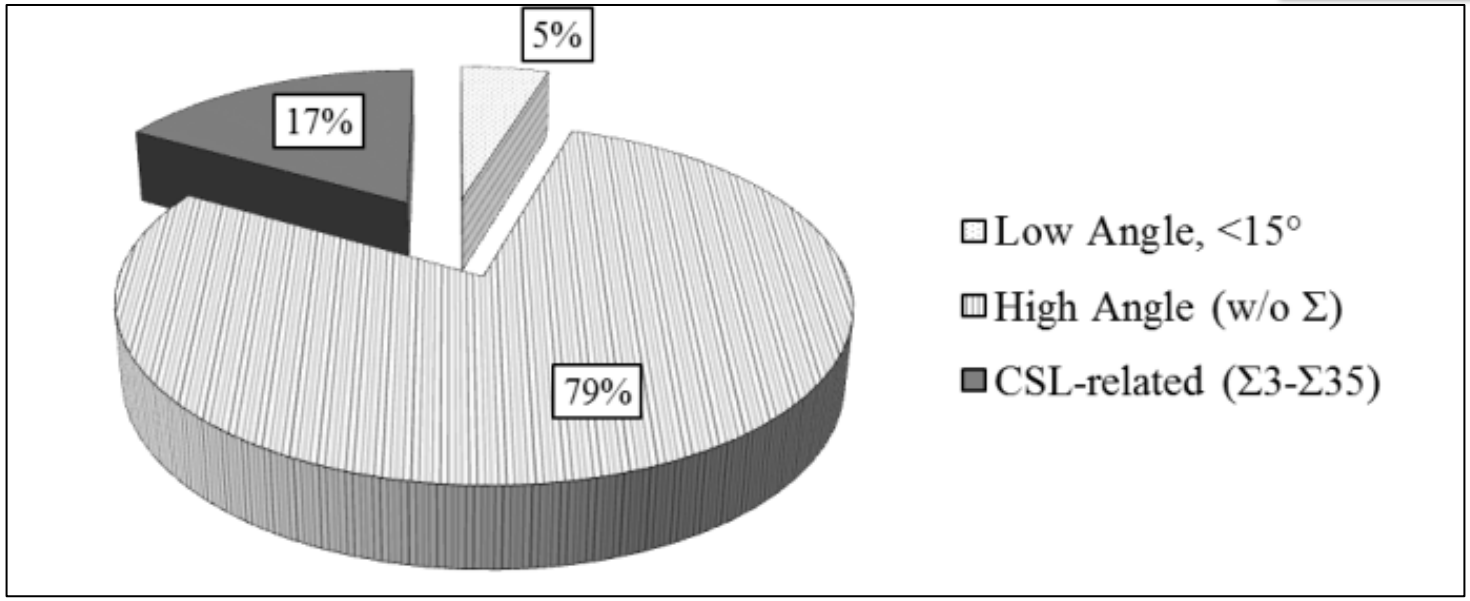

Fission products favor precipitation on random, high angle grain boundaries but can precipitate out on low angle and CSL-related grain boundaries to a limited degree.

[T. Lillo, I.J. van Rooyen, "Influence of SiC Grain Boundary Character on Fission Product Transport in TRISO Fuel" Draft in progress, to be submitted to the Journal of Nuclear Materials, July 2015]

Distributions of Fission Products by Grain Boundary Type.

[T. Lillo, I.J. van Rooyen, "Influence of SiC Grain Boundary Character on Fission Product Transport in TRISO Fuel" Draft in progress, to be submitted to the Journal of Nuclear Materials, July 2015]

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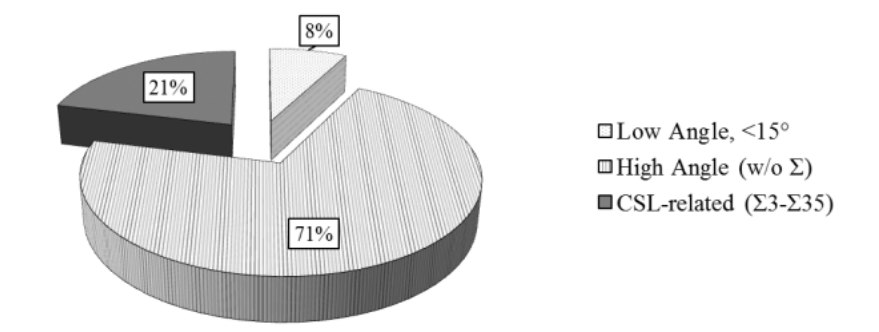
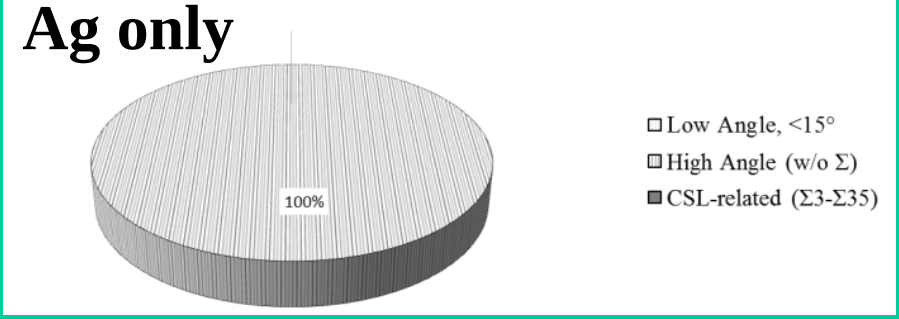
All three grain boundaries types

Pd only



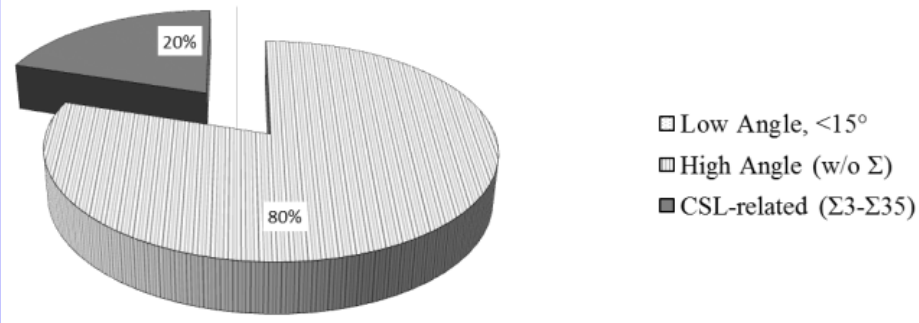
Random, high angle boundaries, never low angle or CSL related boundaries

Ag only

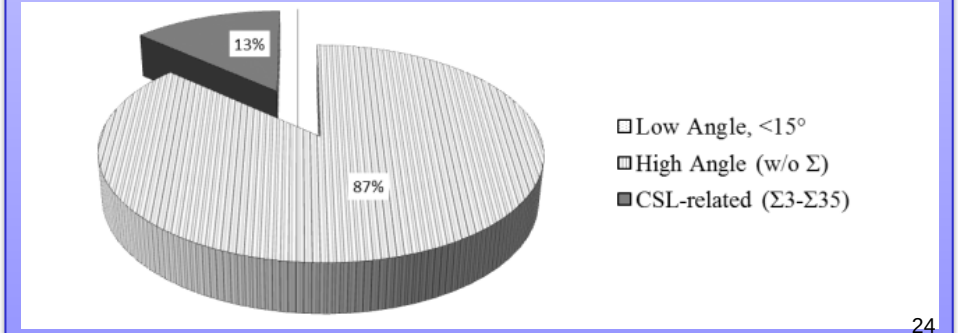


No low angle boundaries

Pd+Ag

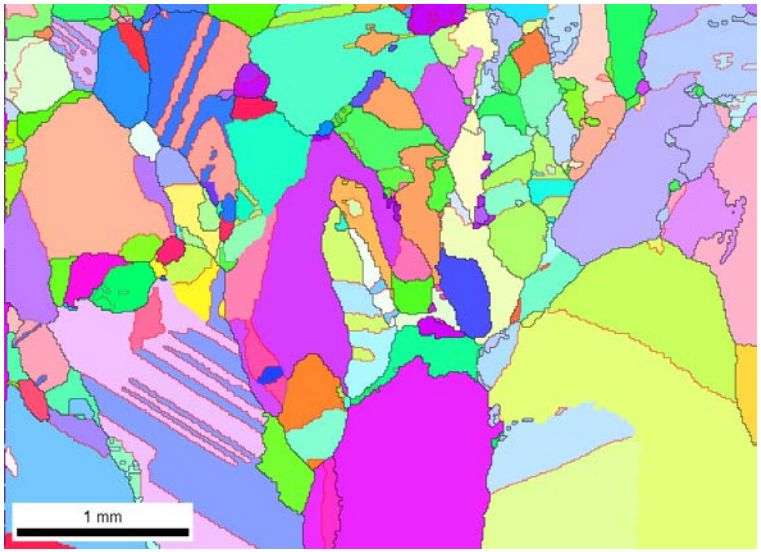


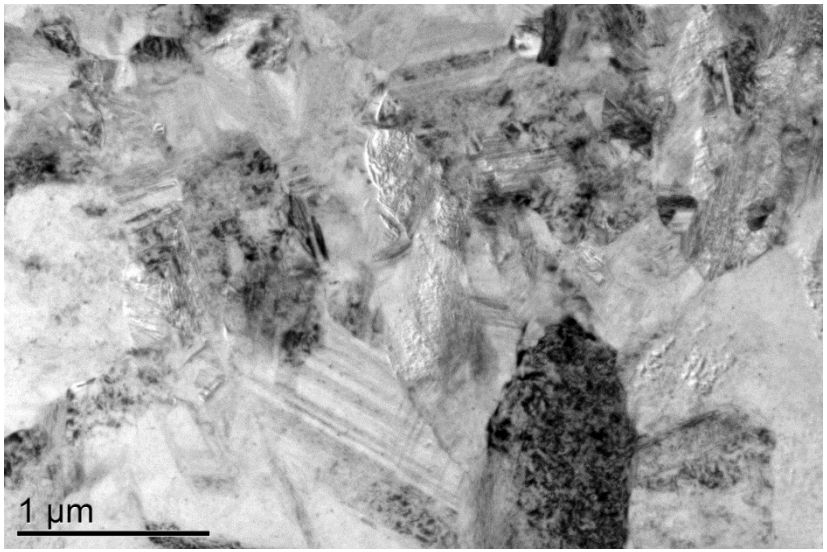
Pd+U



Continuing work under AGR

AGR1-131-099





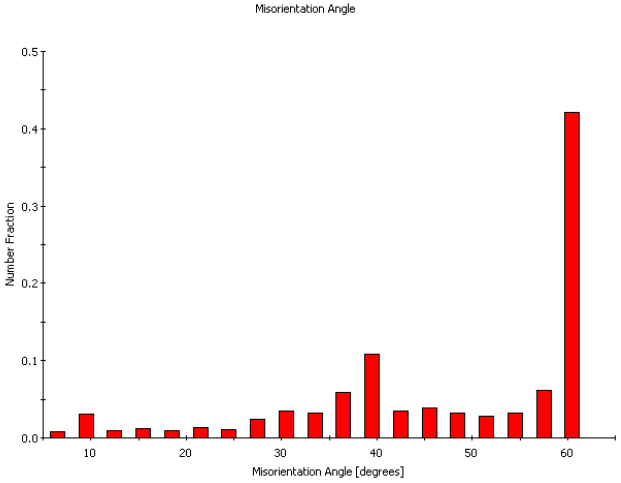
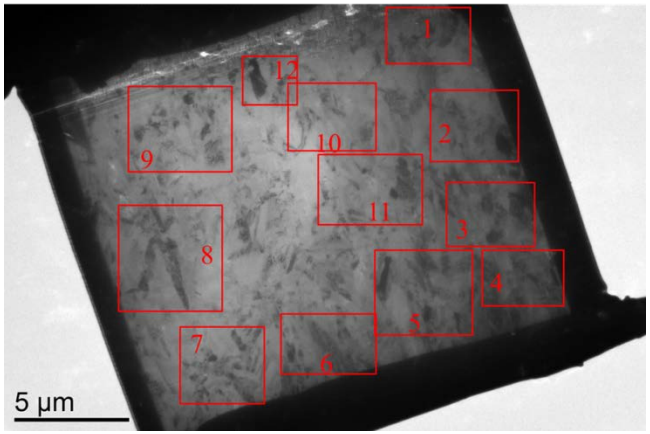


Chart: Misorientation Angle

Angle [degrees]	Number Fraction
6.5	0.00847126
9.5	0.0303613
12.5	0.00886279
15.5	0.011817
18.5	0.00872041
21.5	0.0132764
24.5	0.0107136
27.5	0.0240612
30.5	0.0353088
33.5	0.0321054
36.5	0.0589429
39.5	0.10792
42.5	0.03538
45.5	0.0391173
48.5	0.0316782
51.5	0.0276206
54.5	0.0324969
57.5	0.0613632
60.5	0.421783
63.5	0
Average Number	47.272



- Grain orientation mapping performed in TEM using ASTAR based on precession electron diffraction
- Grain boundary character
- Analysis and interpretation in progress

- ~95% high-angle GBs; ~40% $\sum 3$ twin boundaries
- Results on 12 areas similar to each other

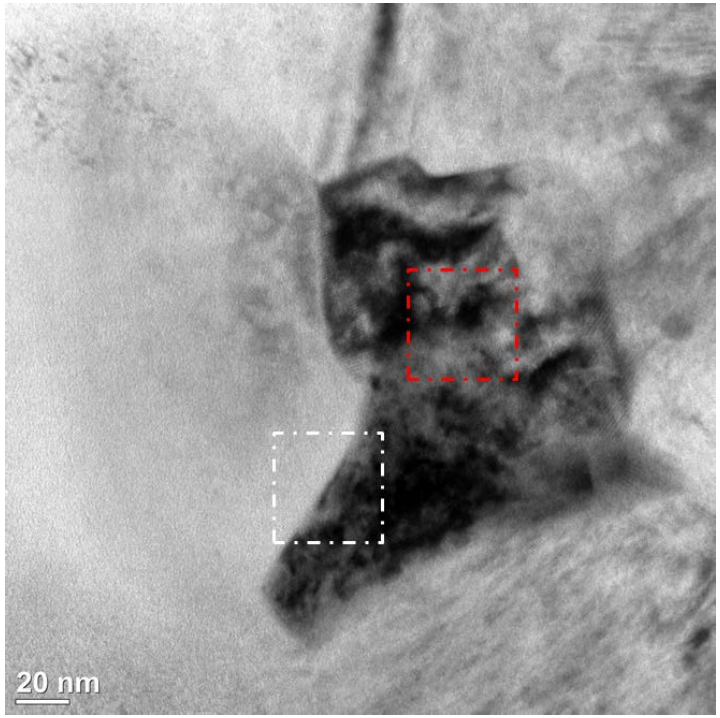
Haiming Wen: INL Post-Doctoral Graduate Researcher

Preliminary Results: HRTEM

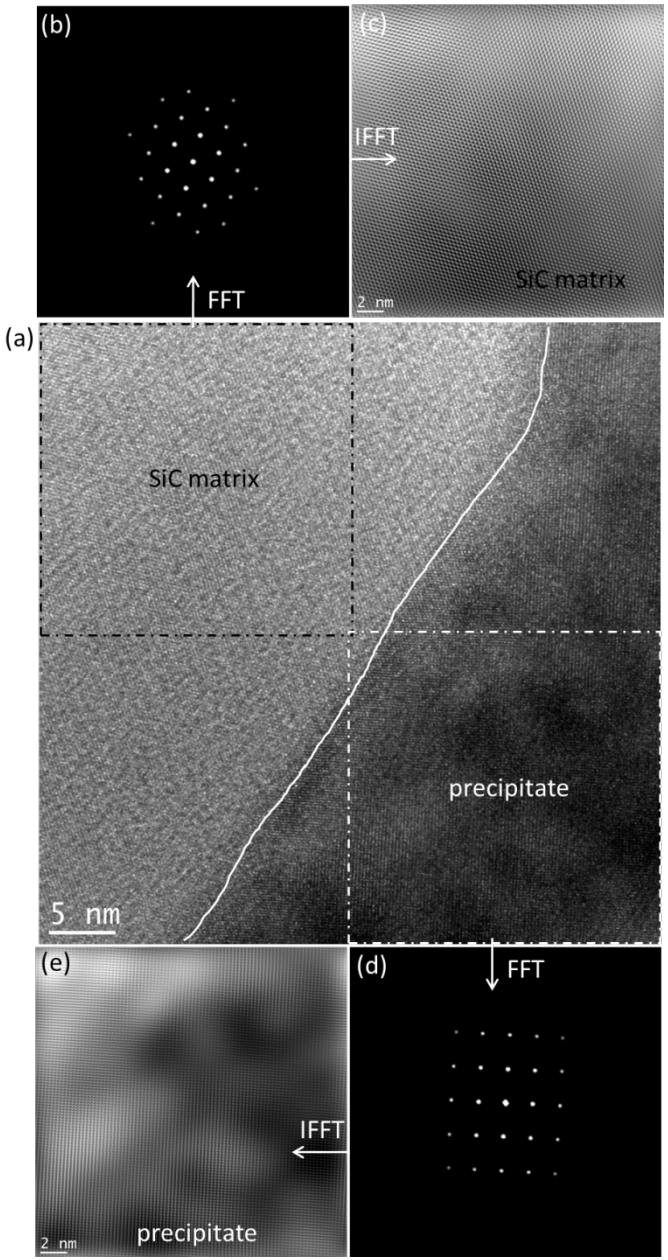
Preliminary HRTEM initiated in May 2015 at LANL (Terry Holesinger) to establish a US based facility in lieu of the successful research conducted in 2013 at the Nelson Mandela Metropolitan University (NMMU) electron microscopy facility.

- LANL FEI Titan 80-300TM with 0.07nm and 0.20nm TEM and STEM resolution respectively (NMMU JEOL ARM 200F TEM which is equipped by two CEOS spherical aberration correctors).
- Work focused on phase information of “larger” precipitates (chemical composition & structure)
- Limits on the spectral scan data. Data was influenced by a persistent drop out of intensity in the collected spectra, possibly a hardware or software issue.

Interface of Precipitate at SiC grain



AGR1-131-Compact 1-3-1-99, Lamella 1

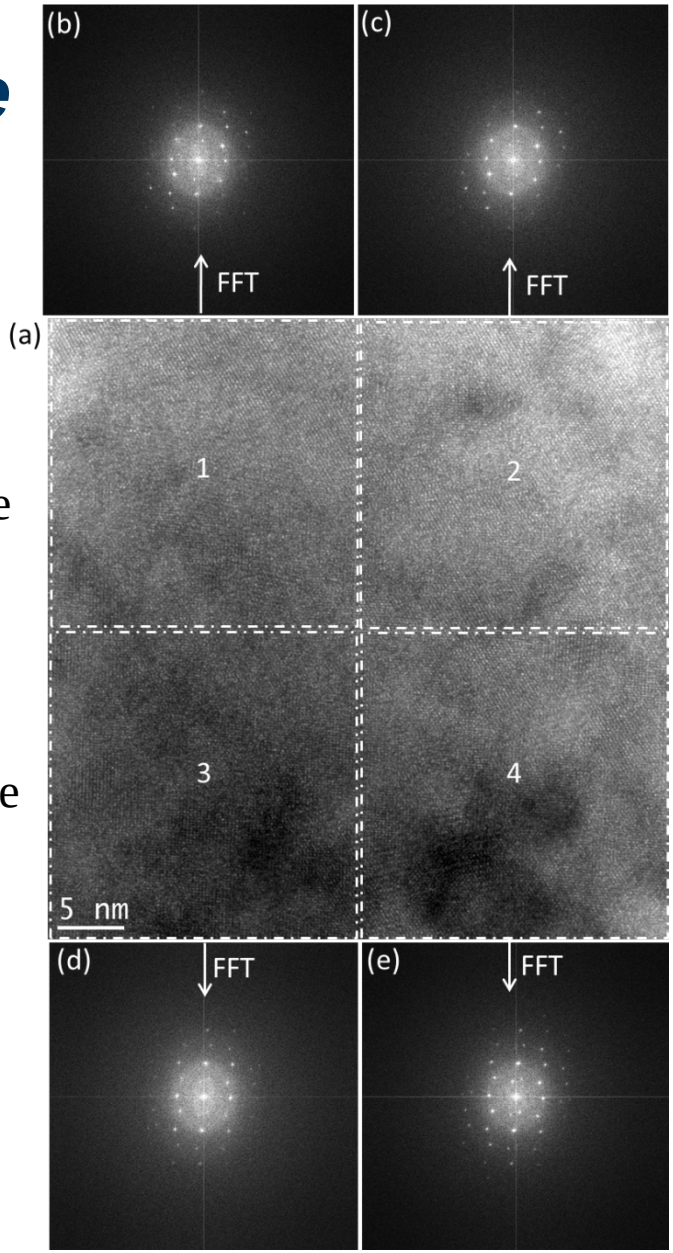


Single phase precipitate

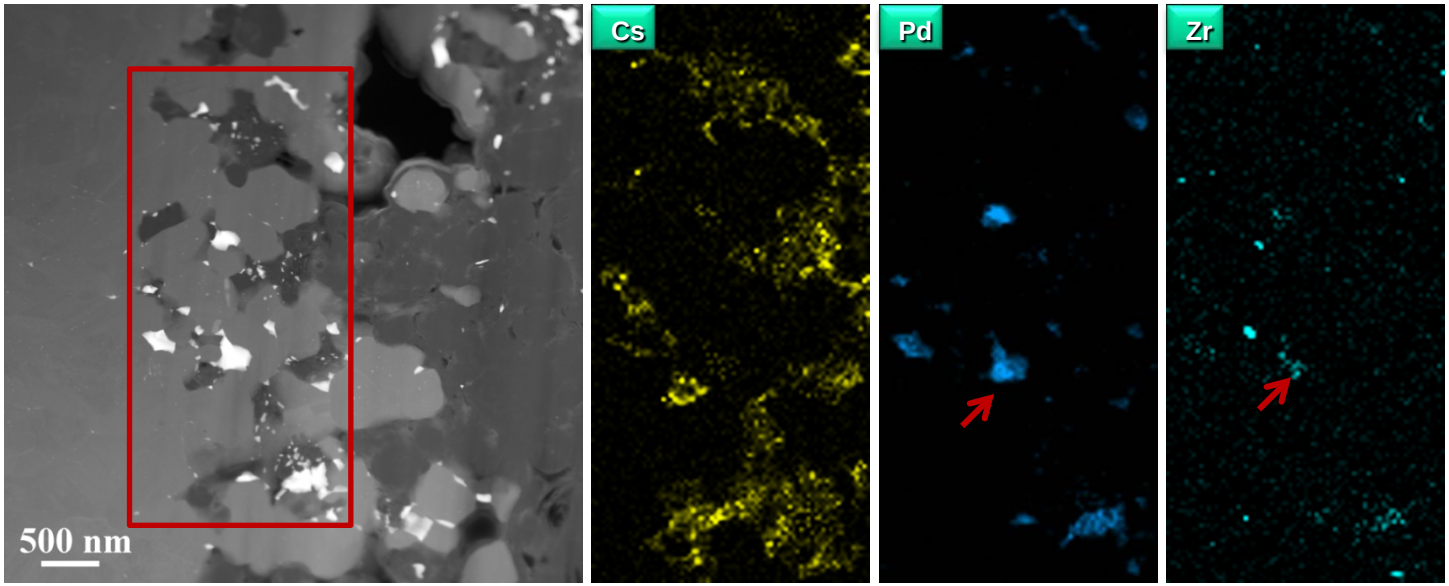
AGR1-131-Compact 1-3-1-99, Lamella 1

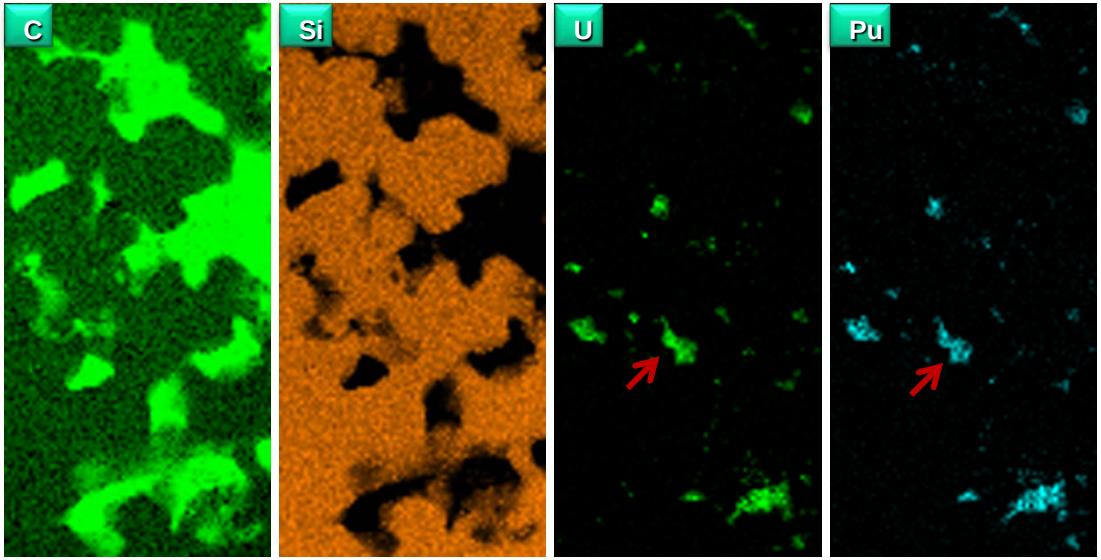
- Top part of the precipitate has slight misorientation (with a few degrees) with the bottom part of the precipitate; however, the crystal structure is identical.

- The small misorientation may be due to a difference in dislocation density between the top and bottom parts of the precipitate.



Multi phase(??) precipitate in SiC

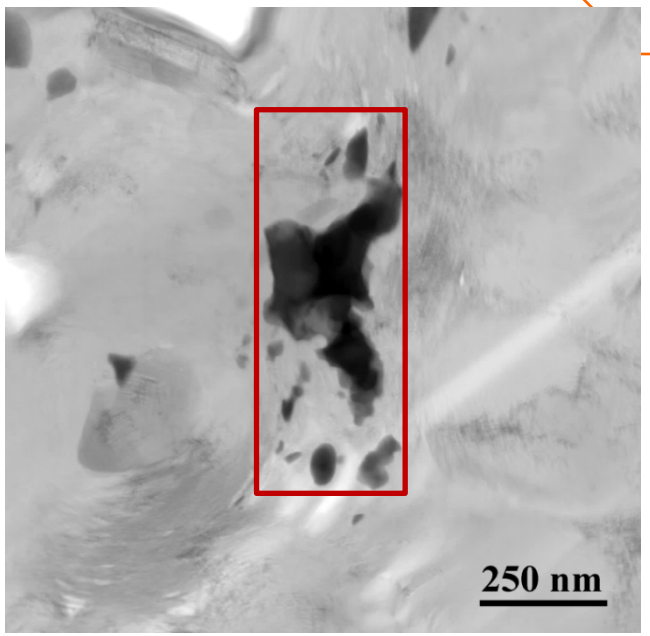




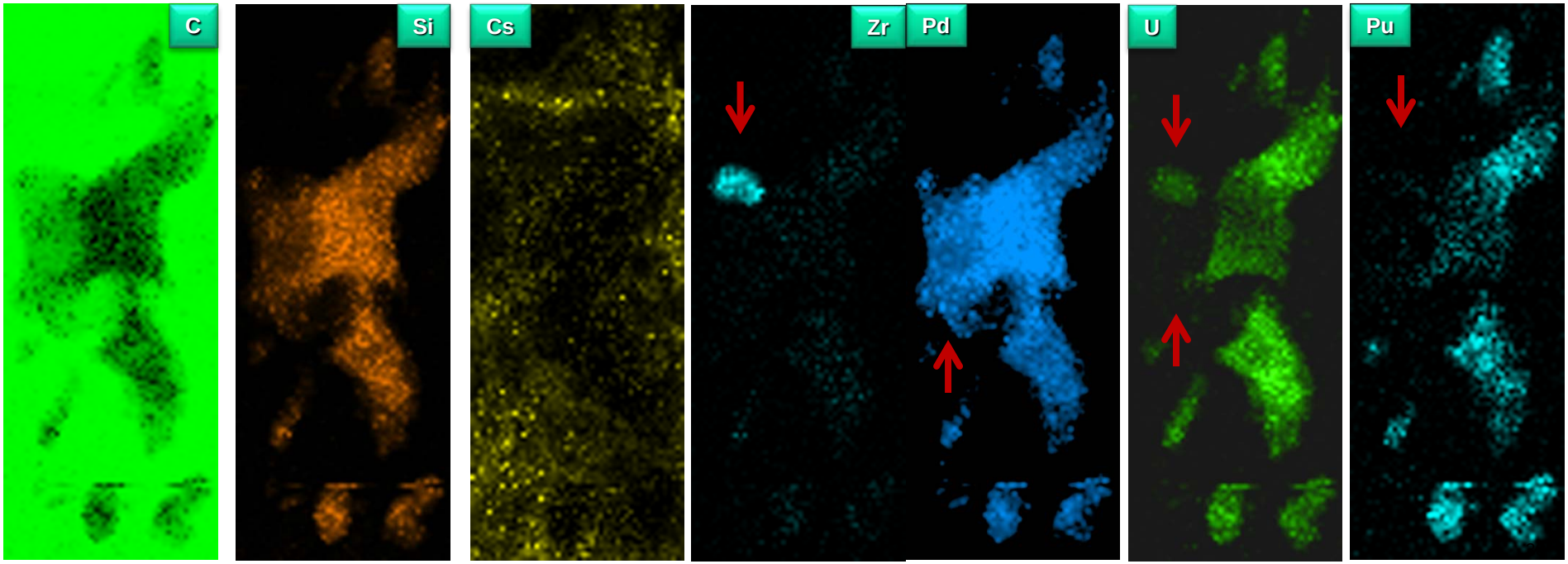
AGR1-131-Compact 1-3-1-99

- Possible multi phase precipitates, needs confirmation with SAD
- Demonstration of Cs pile-up at SiC

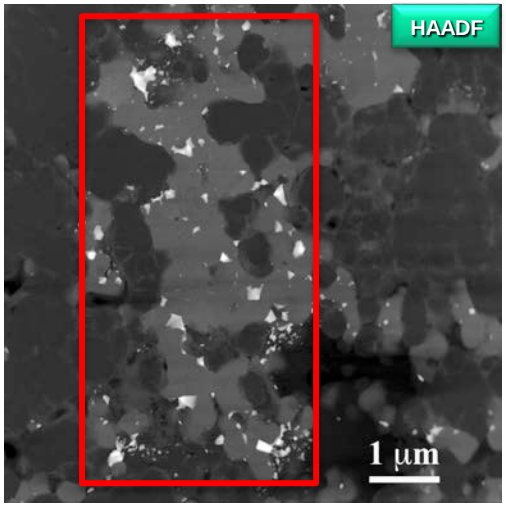
Multi-phased precipitate in IPyC



AGR1-131-Compact 1-3-1-99

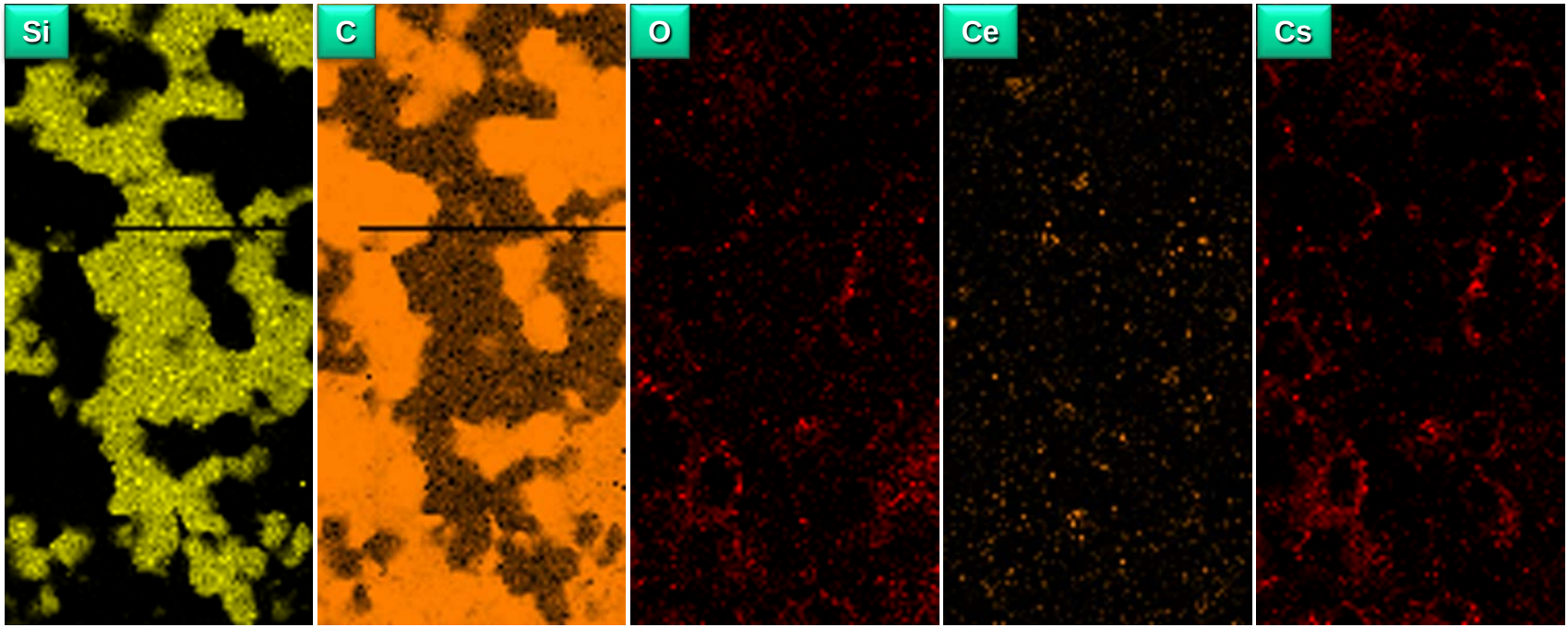


Ce & Cs accumulate at SiC-IPyC interface (coincide to Oxygen)

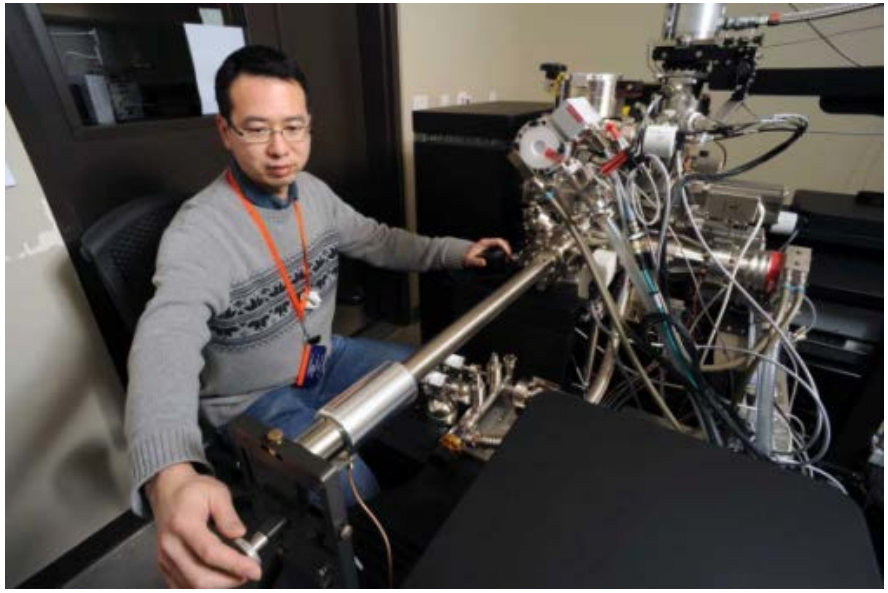


- Caution is warranted as the dwell time for each spot was just 4 seconds.
- Qualitatively, the Cs appears to be more abundant than Ce

AGR1-131-Compact 1-3-1-99



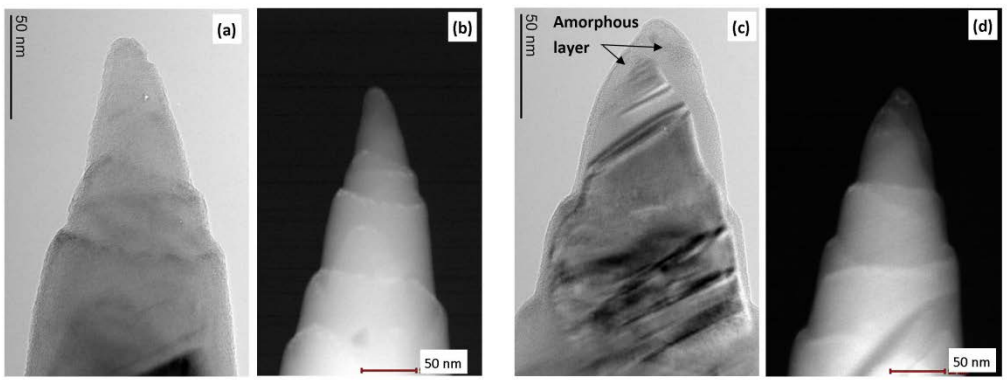
Preliminary Results: APT



(Cameca LEAP 4000XHR at the Center for Advanced Energy Studies (CAES))

(This work is a collaborative effort with Boise State University (Prof Yaqiao Wu) and is partially funded by NSUF-RTE funds).

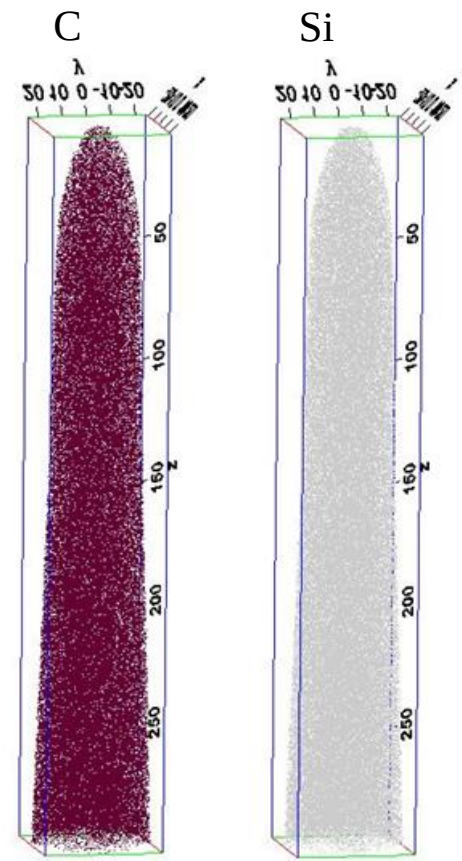
Optimization with unirradiated SiC



Run #	R33_04253
# ions	14 M
Mode	laser
Temperature (setting, K)	90
Laser energy (pJ)	90
Pulse frequency (kHz)	200
Detection Rate (%)	0.1

Run #	R33_04254
# ions	1.7 M
Mode	laser
Temperature (setting, K)	90
Laser energy (pJ)	90
Pulse frequency (kHz)	200
Detection Rate (%)	0.1

Two large datasets up to 300 nm in depth, which contained up to 15 million atoms, were acquired



- The most successful measurements were obtained at a set of parameters of a temperature of 90 K and laser energy of 90 – 100 pJ. Laser parameters for the earlier FY2014 APT work on irradiated SiC consisted generally at lower temperature, i.e., 90 K for current vs. 40 – 80 K for previous.

- APT on irradiated SiC planned for August 2015

Technical Highlights FY2015

- Identification of **Ag in the SiC grain** in two different compacts (Baseline and Variant 3)
- Fission product precipitates can consist of multi- or single phases
- Identification of grain boundary characteristics using PED at nanoscale
  - Fission product preference grain characteristics obtained (Baseline fuel)
  - First application of PED in conjunction with fission products in irradiated fuels
- INL Award for Outstanding Scientific Paper for 2014 was presented on February 27, 2015 to Isabella van Rooyen and Thomas Lillo for “Identification of Silver and Palladium in Irradiated TRISO Coated Particles,” published in Journal of Nuclear Materials 446 (2014) pp. 178-186.
- Good Publication Record (Sept 2015):
  - 6 Conferences papers/presentations
  - 3 INL reports
  - 8 Journal papers
- Collaboration with LANL (HRTEM), ORNL (particle with IPyC layer breach), BSU (APT, PED)
- Establish & implement SEM fission product distribution comparative tool

Acknowledgements and Contributions

- Scott Ploger and Jason Harp: Mount and decontamination preparation for electron microscopy examination and micro-analysis
- Jim Madden: FIB-STEM and APT sample preparation
- Tammy Trowbridge: SEM images
- Connie Hill (Idaho State University, INL intern): SEM montage and interlayer thickness
- Bin Lin (University of Wisconsin-Madison): STEM & APT(NSUF)
- Tom Lillo: STEM, PED (NSUF)
- Haiming Wen: STEM, HRTEM, PED
- Yaqiao Wu (Boise State University): PED, APT, STEM, FTEM and EELS microscopy
- Terry Holesinger (LANL): HRTEM
- John Hunn, Tyler Gerczak (ORNL): IPyC breached particle colloboration
- Isabella van Rooyen: VHTR fuels electron microscopy and micro-analysis lead

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- [1] I. J. van Rooyen, C. Hill, T. Trowbridge, *Fission product distribution patterns as a comparative characterization tool for TRISO fuel performance*, TMS2015, 144th Annual meeting, March 15-19, 2015; Walt Disney World Orlando, Florida, USA.
- [2] T Lillo, IJ van Rooyen, *Distribution of Pd, U and Ag in the SiC Layer of Irradiated TRISO Fuel,* Journal of Nuclear Materials (2015), pp. 97-106, DOI information: 10.1016/j.jnucmat.2015.02.010.
- [3] Bin Leng, Isabella van Rooyen, Yaqiao Wu, Izabela Szlufarska, Kumar Sridharan, *STEM/EDS Analysis of Fission Products in Irradiated TRISO Coated Particles of the AGR-1 Experiment*, peer review comment resolution in process, to be submitted to Journal of Nuclear Materials.
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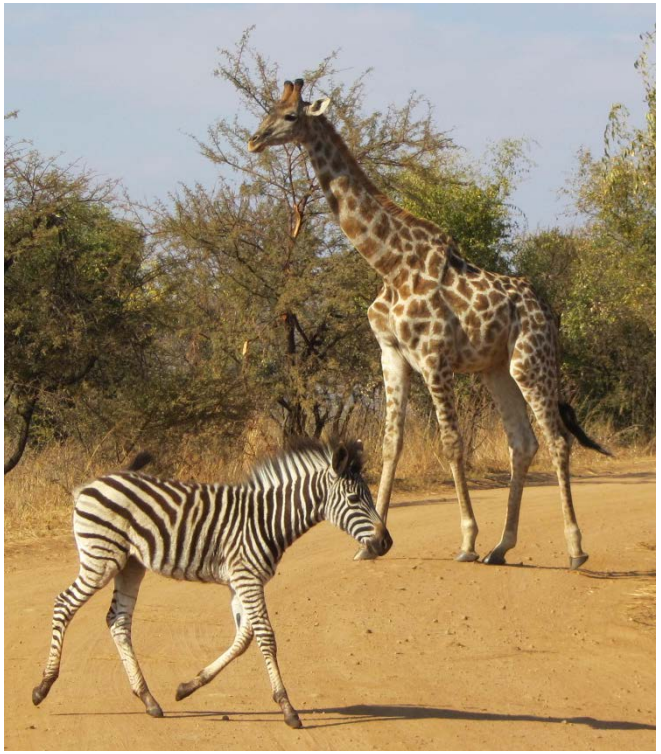
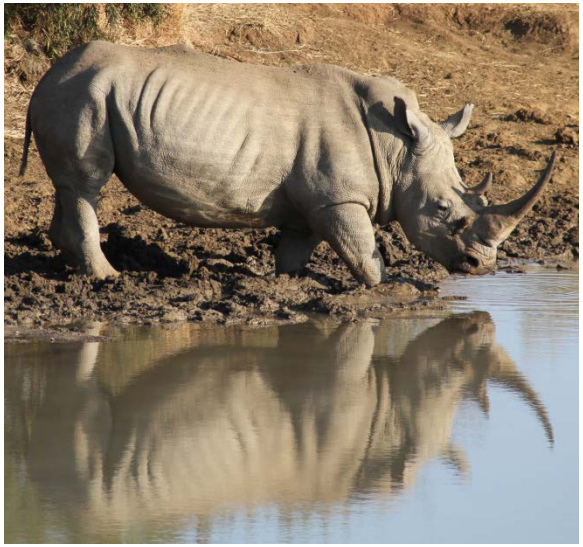
- [6] I. J. van Rooyen, J. Youngsman, T. M. Lillo, Y. Q. Wu, D. Goran, M. E. Lee, W. E. Goosen[5], J. H. Neethling, T. L. Trowbridge and J. W. Madden, *Methods for identification of crystallographic parameters of irradiated SiC to understand fission product transport*, The 3rd Workshop on HTGR SiC Material Properties, Sept. 30-Oct 1, Jeju Island, South Korea
- [7] Thomas M. Lillo, Isabella J. van Rooyen, Yaqiao Wu, "*Grain Boundary Character and Fission Product Precipitation in SiC"*, ANS 2015, June 7-11, 2015, San Antonio, TX.
- [8] Haiming Wen, Isabella J. van Rooyen, John D. Hunn, Tyler J. Gerczak, Charles A. Baldwin, Fred C. Montgomery, *Advanced electron microscopy study of fission product distribution in the failed SiC layer of a neutron irradiated TRISO coated particle*, Microscopy and Microanalysis 2015 (M&M2015), August 2-7, 2015, Portland.
- [9] T. Lillo, I.J. van Rooyen, *"Precession Electron Diffraction for SiC Grain Boundary Characterization in unirradiated TRISO Fuel"*, to be submitted to Nuclear Engineering and Design or the Journal of Nuclear Materials, July 2015.
- [10] T. Lillo, I.J. van Rooyen, *"Influence of SiC Grain Boundary Character on Fission Product Transport in TRISO Fuel"*, Draft in progress, to be submitted to the Journal of Nuclear Materials, July 2015.
- [11] Haiming Wen, Isabella J. van Rooyen, John D. Hunn, Tyler J. Gerczak, *"Advanced electron microscopy study of fission products Pd, Ag and Cs in carbon areas in the locally corroded SiC layer in a neutron irradiated TRISO fuel particle"*, to be submitted to Nuclear Engineering and Design or the Journal of Nuclear Materials, July 2015.
- [12] Haiming Wen, Isabella J. van Rooyen, John D. Hunn, Tyler J. Gerczak, *"Fission products palladium, silver and cesium distribution in the locally corroded SiC layer of a neutron irradiated TRISO fuel particle"*, to be submitted to Nuclear Engineering and Design or the Journal of Nuclear Materials, August 2015.

What is next?? The road to integrate all the results





Questions?

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Technique Acronyms

Acronyms	Description
APT	Atom Probe Tomography
EDS	Energy Dispersive Spectroscopy
EBSD	Electron Back Scattered Diffraction
EELS	Electron Energy Loss Spectroscopy
EFTEM	Energy Filtered TEM
FIB	Focused Ion Beam
HRTEM	High Resolution Transmission Electron Microscopy
SAD	Selected Area Diffraction
SEM	Scanning Electron Microscope
STEM	Scanning Transmission Electron Microscopy
TEM	Transmission Electron Microscope
t-EBSD	Transmission-EBSD
TKD	Transmission Kikuchi Diffraction
WDS	Wavelength Dispersive Spectroscopy

Irradiation Parameters

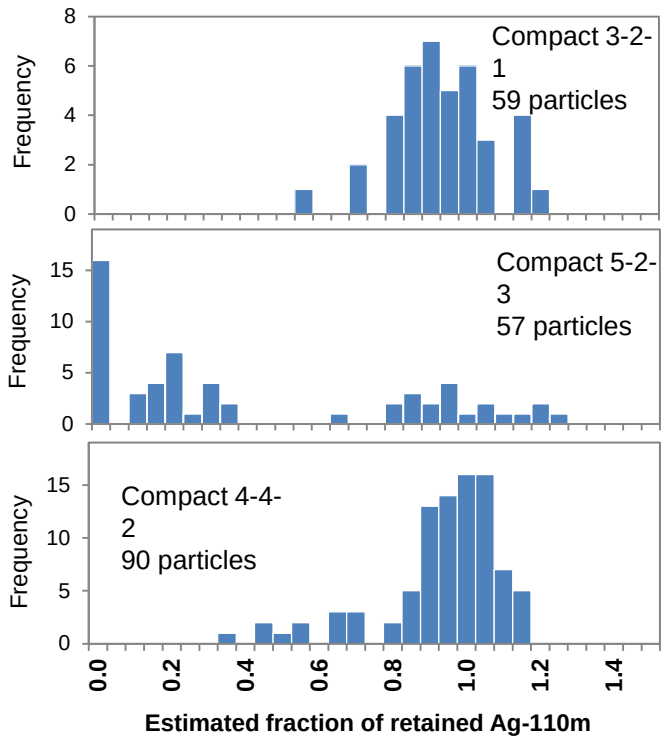
Compact	Fuel Type[a]	TAVA[b] Temp. (°C)	TA Max[c] Temp. (°C)	Burnup (% FIMA)	Fast fluence (10^{25} n/m^2)
6-3-2	B	1070	1144	11.4	2.55
6-1-1	B	1111	1197	15.3	2.43
3-2-1	B	1051	1143	19.1	4.21
5-2-1	V1	1057	1140	17.4	3.71
5-2-3	V1	1059	1141	17.4	3.77
5-3-1	V1	1040	1122	16.7	3.60
4-4-2	V3	1024	1139	16.6	3.59
4-1-1	V3	1072	1182	19.4	4.13
1-3-1	V3	1092	1166	15.3	3.22

[a] B=Baseline; V1=Variant 1, V2= Variant 2, V3=Variant 3

[b] Time average, volume average temperature

[c] Time average maximum temperature

Silver Retention of Individual Particles



Average retained fraction of release ^{110m}Ag in the particle sample

Compact	Retained ^{110m}Ag fraction
6-3-2	0.36 – 0.51
6-1-1	0.39 – 0.45
3-2-1	0.92
5-2-3	0.43 – 0.45
5-2-1	0.30 – 0.45
5-3-1	0.48 – 0.57
4-4-2	0.93
4-1-1	1.02
1-3-1	0.12 – 0.28

- Particles within a single compact could often exhibit a large range of silver retention, from complete release to complete retention

- Possible causes:
  - Temperature variations
  - SiC microstructure variations
  - Kernel and coating morphology
- Detailed microanalysis of particles exhibiting high and low silver release is in progress to compare microstructure

Precipitate Distribution and Quantification in IPyC

Sample Description								
COMPACT	6-3-2	1-3-1	1-3-1	5-3-1	5-3-1	4-1-1	4-1-1	4-1-1
MOUNT #	48T	26V	30V	20U-1	23U	120T	120T	120T
CP #	34	66	99	38	31	21	29	30
Number of Precipitates								
Average	Not available	259	610	222	187	Not available	1848	383
Maximum	Not available	463	1618	600	389	Not available	5317	912
Minimum	Not available	45	48	60	69	Not available	23	56
Furthest Distance from Interlayer (μm)								
Average	2.9	12.73	13.51	14.8	14.3	Not available	14.38	16.18
Maximum	15.3	18.37	17.91	21.5	19.7	Not available	21.79	23.68

- Large variation in number of precipitates between particles from same compact for Variant 3 fuel particles
- AGR1-411-029: Most precipitates in IPyC
- AGR1-411-029: Precipitates moves furthest in IPyC

[Ref. Van Rooyen et al.,TMS 2015, 144th Annual Meeting, March 15-19, 2015; Walt Disney World Orlando, Florida, USA]