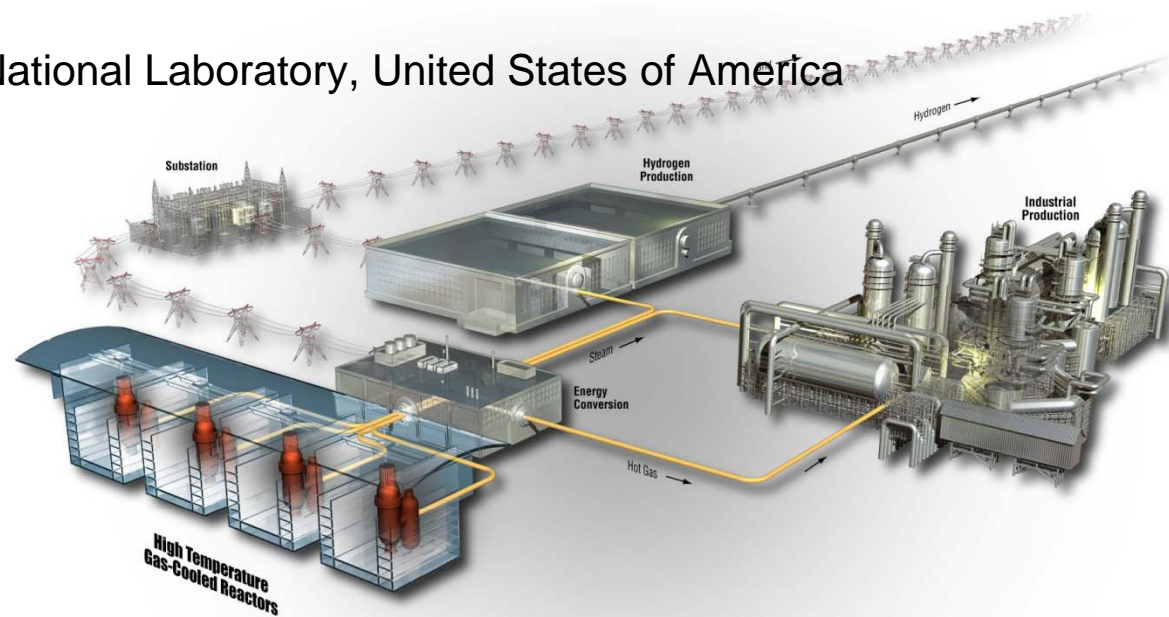


# ***Preliminary findings: Microscopic examination and fission product precipitate identification in irradiated TRISO CPs from the AGR-1 Experiment.***

**I.J. van Rooyen<sup>a</sup>, D. Janney<sup>a</sup>, P. Demkowicz<sup>a</sup>, B. Miller<sup>a</sup>,  
J. Riesterer<sup>a</sup>**

<sup>a</sup> Idaho National Laboratory, United States of America

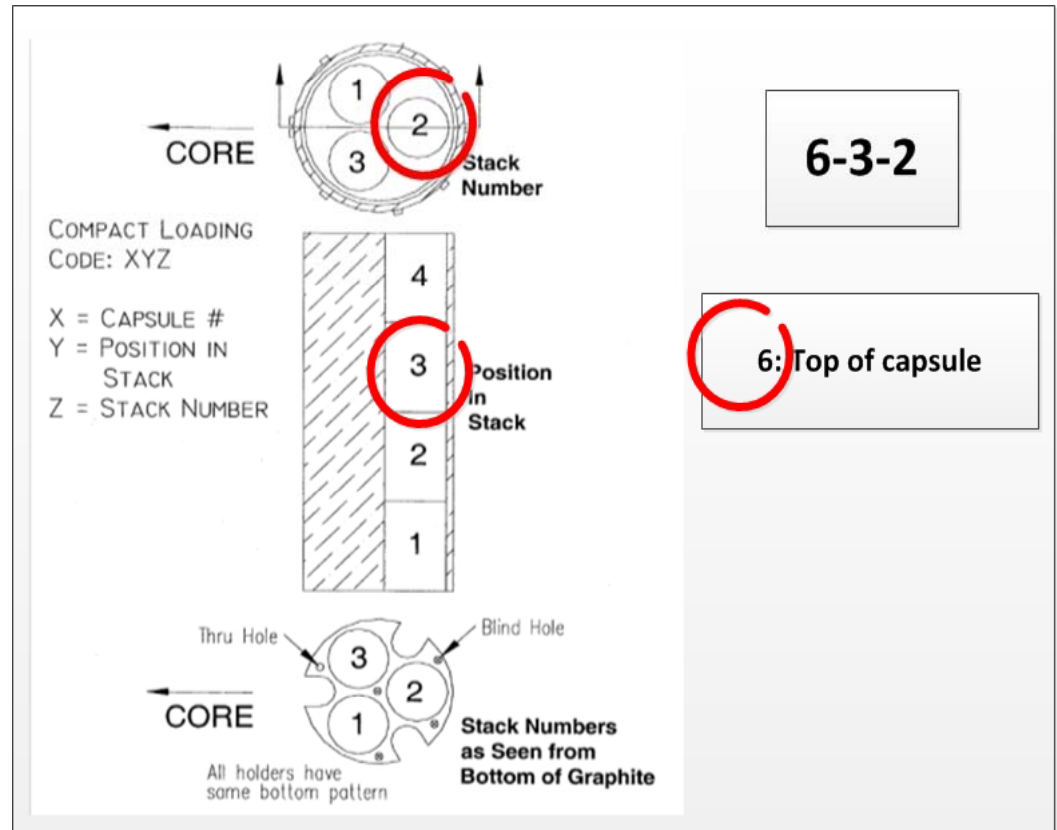


# ***Agenda***

- Introduction
- Objectives of electron microscopy examination
- Sample identification
- Precipitate distribution and quantification
- Precipitate identification
- Conclusion
- Acknowledgements

# Introduction

- AGR-1 irradiation test:  
December 2006 - November 2009
- Four different fuel types
  - variation in deposition parameters for IPyC or SiC
  - UCO kernels, ~ 350  $\mu\text{m}$  with a U-235 enrichment ~19.7%.
- This work is “Baseline” fuel type:
  - Historic German fuel coating process conditions chosen as starting point for baseline fuel.

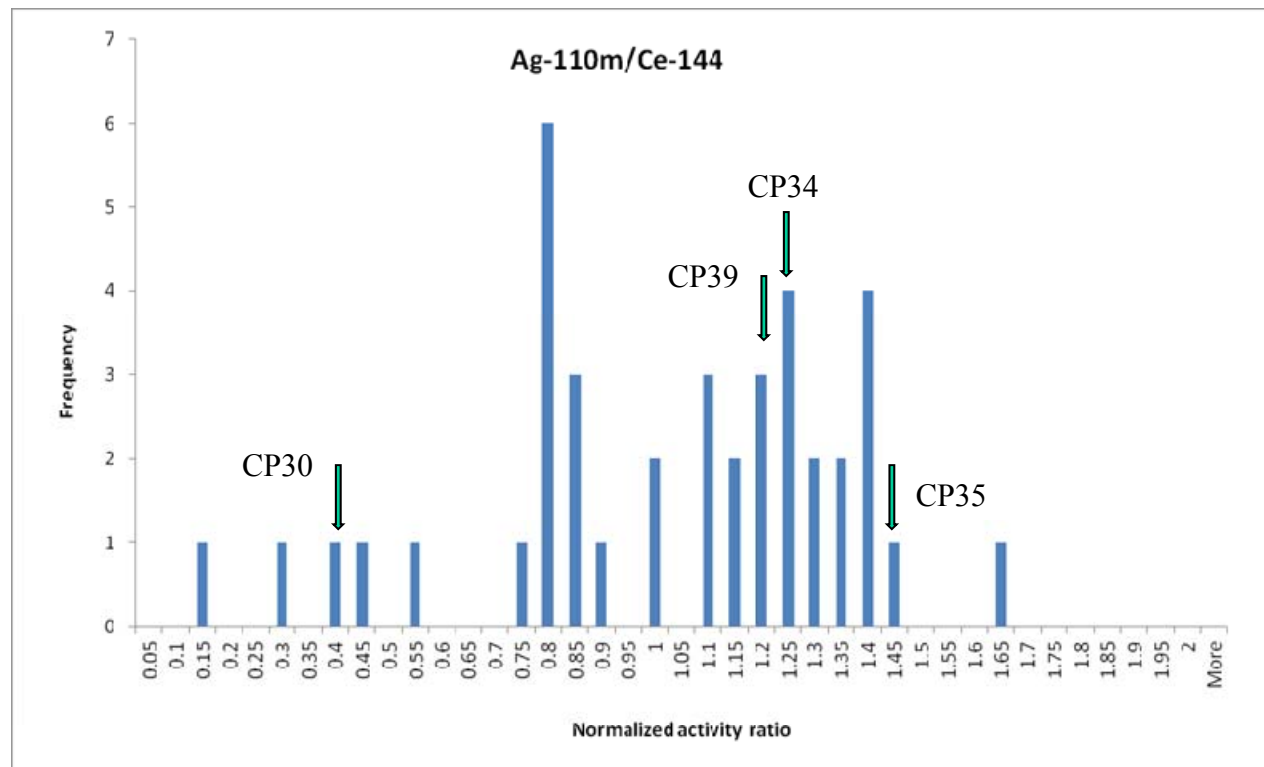


## ***Objectives of Electron Microscopic Examination***

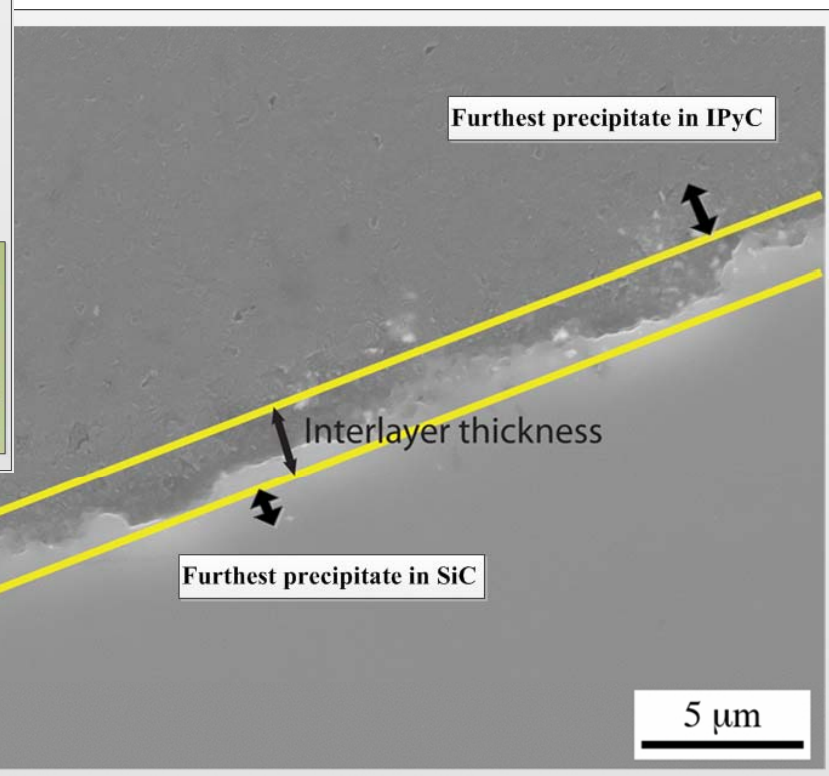
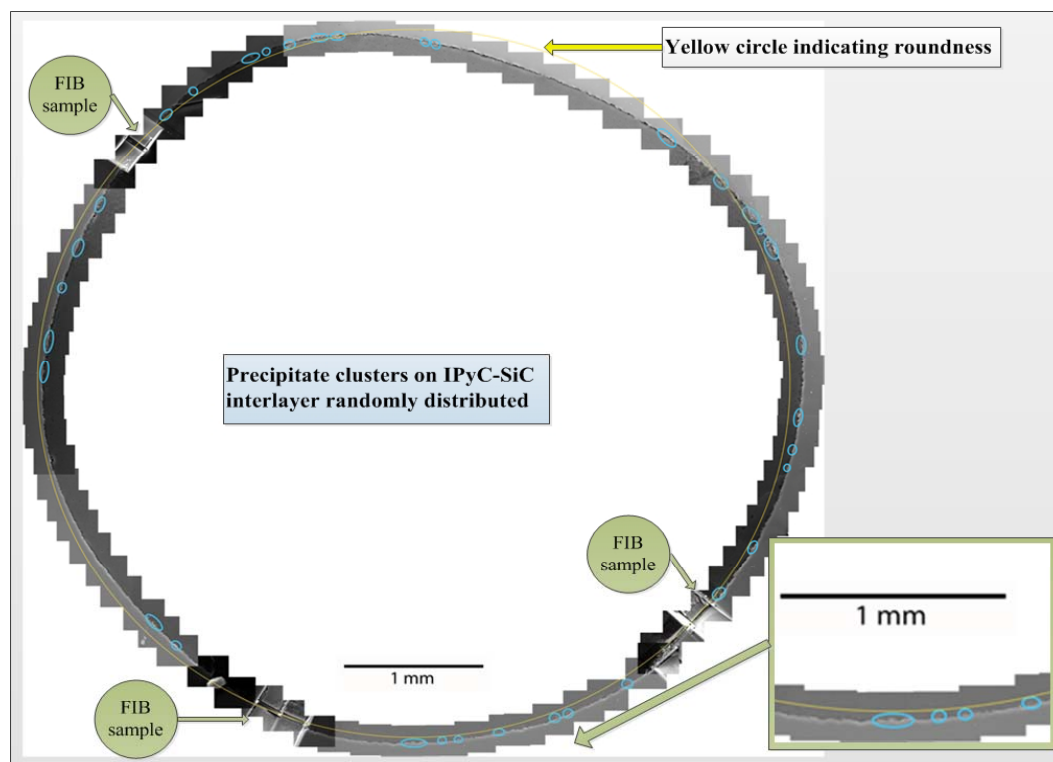
- Characterize Microstructure:
  - Establish irradiation effects
  - Fuel kernel porosity
  - Layer degradation/corrosion
  - Fission product precipitation
  - Migration of fission products in SiC layer
  - Interlayer bonding integrity
  - SiC Grain size, texture & grain boundary alignment

## Sample identification

- Based on IMGA Ag-110m inventory from 40 particles

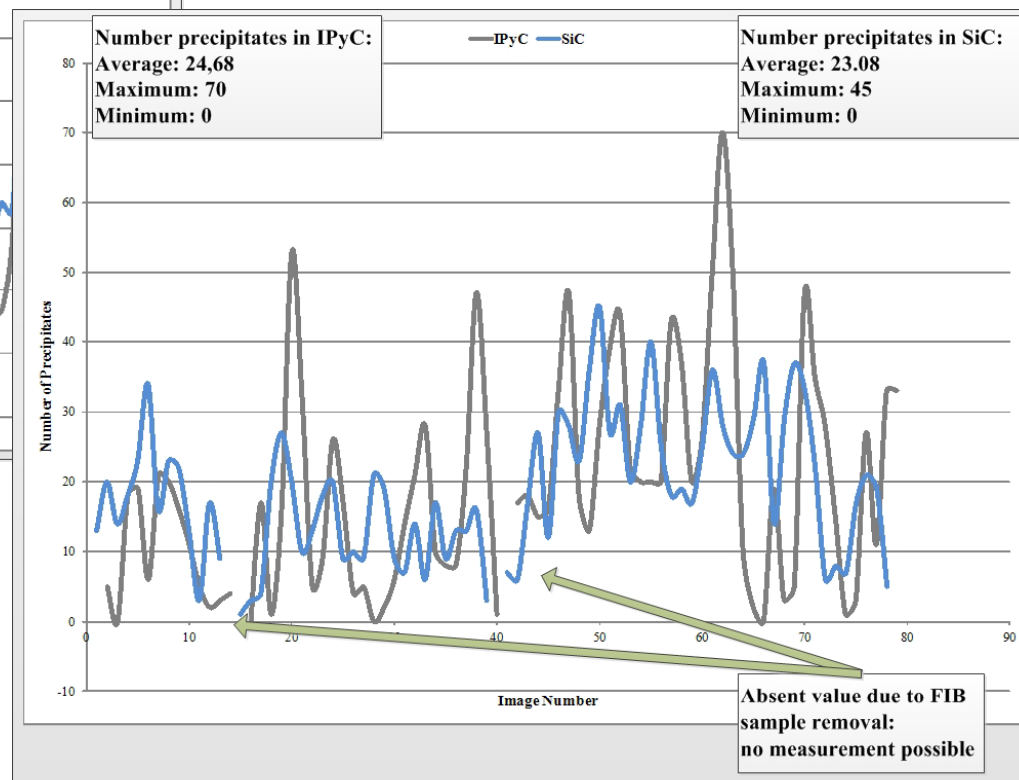
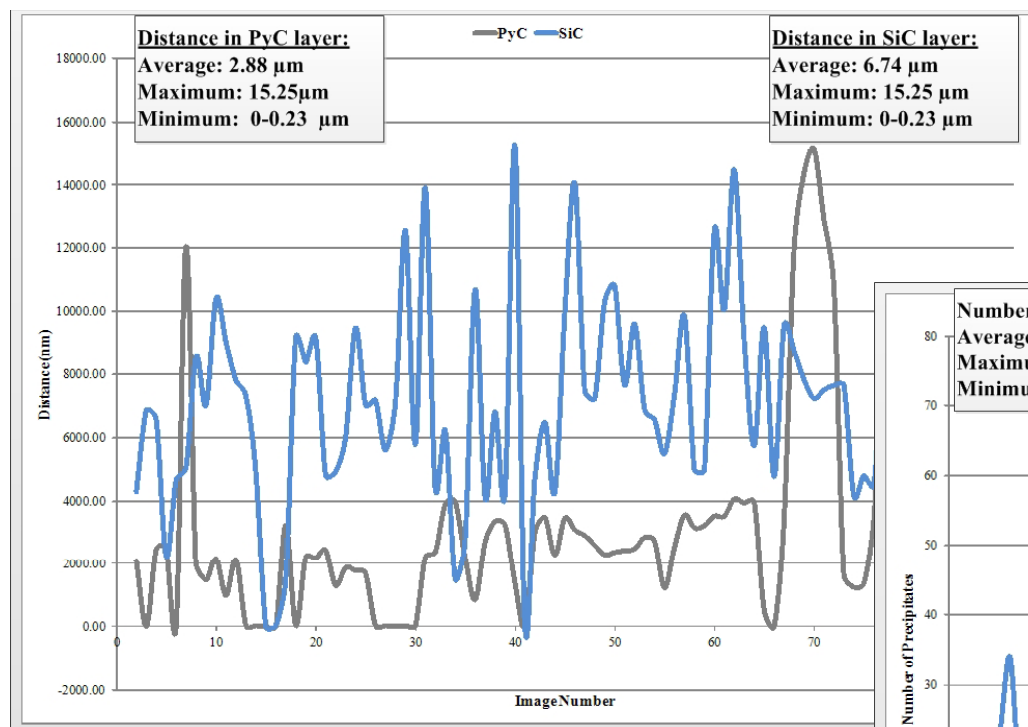


# Precipitate distribution and quantification



CP34 SEM montage: Precipitate clusters randomly distributed

# Precipitate distribution and quantification

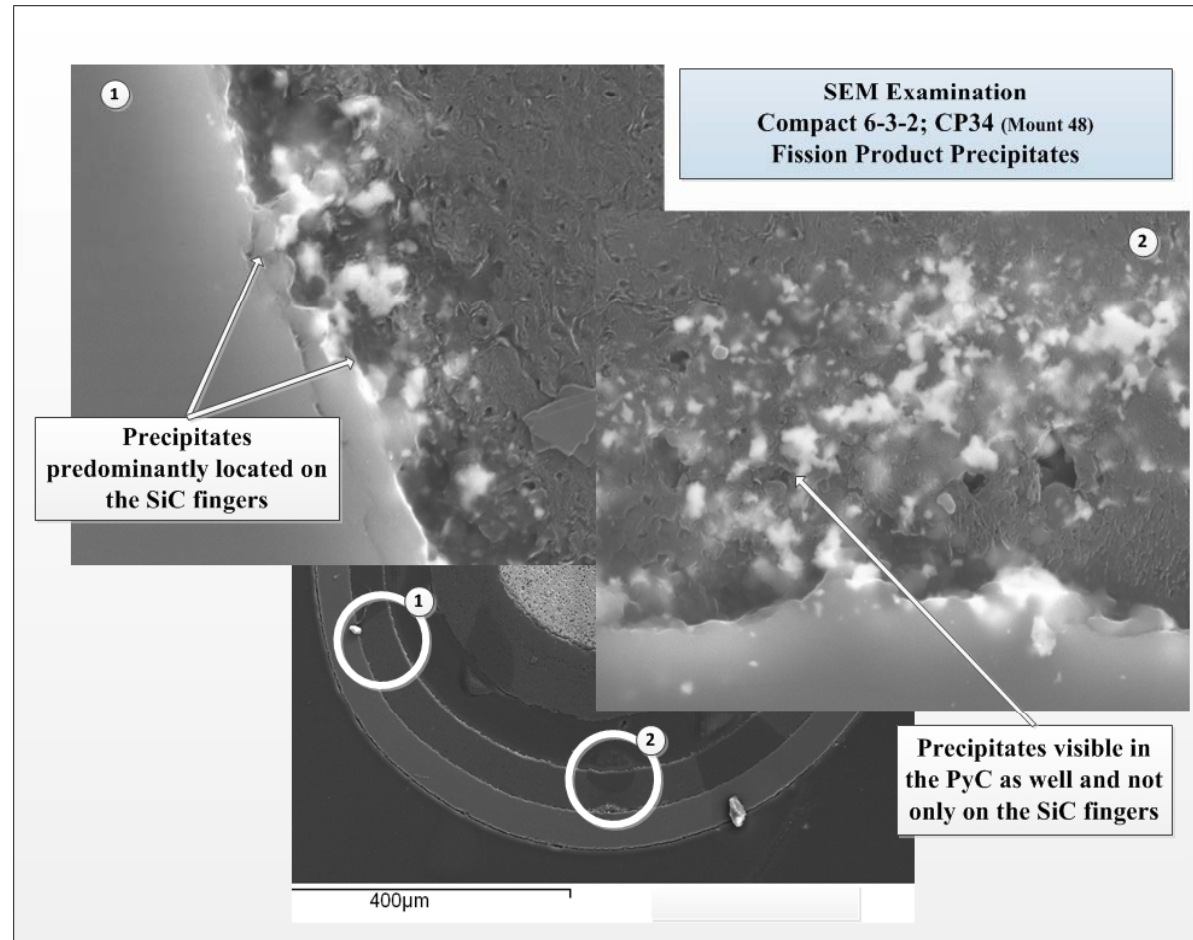


CP34 SEM montage: Precipitate clusters randomly distributed



## ***Precipitate distribution and quantification***

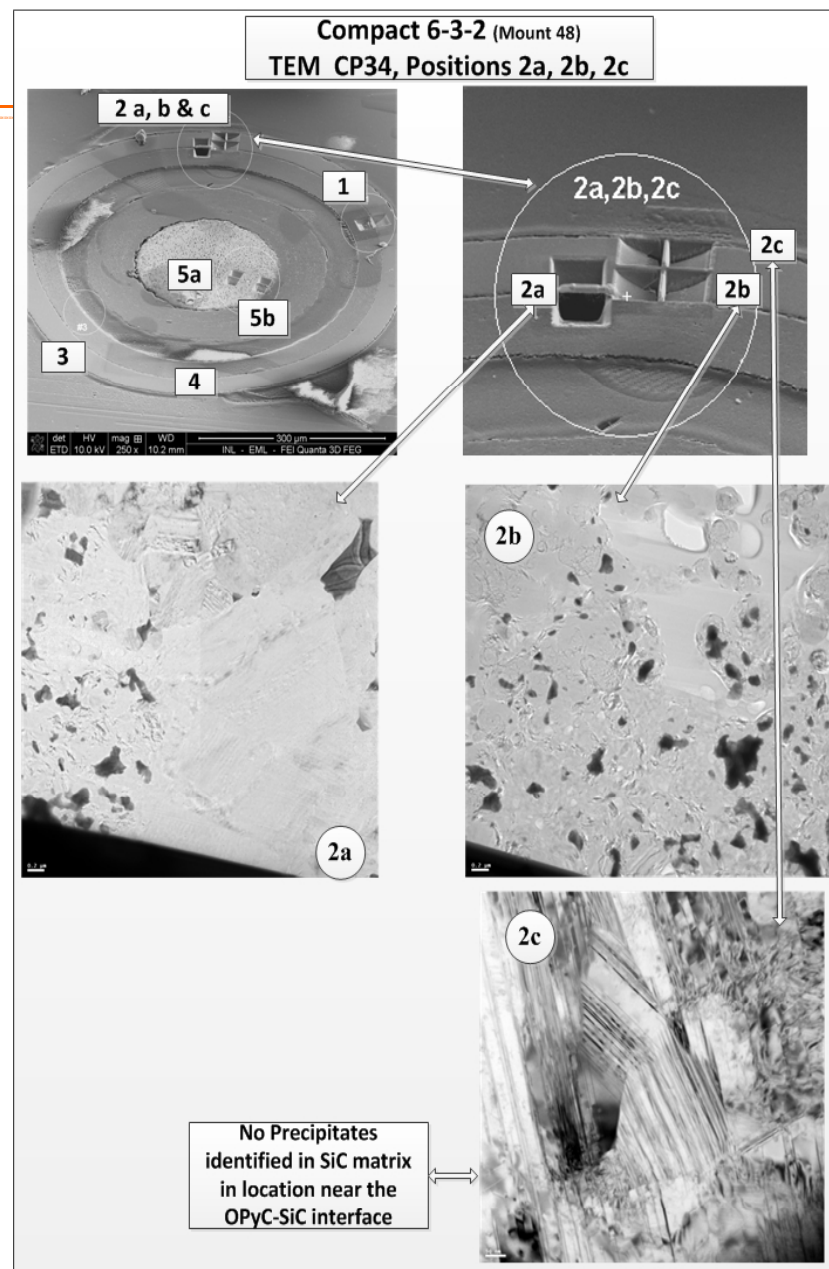
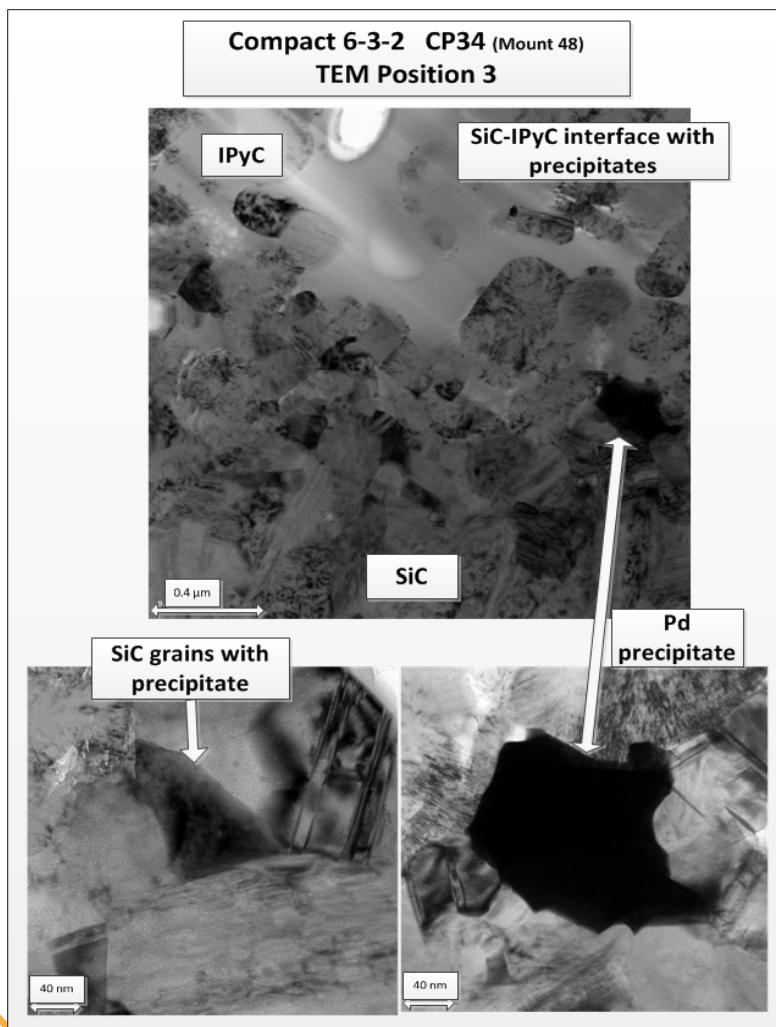
- Pd-rich precipitates located on:
  - SiC fingers,
  - SiC and IPyC in region of SiC-IPyC interlayer
  - Depth of 2-4  $\mu\text{m}$  in SiC





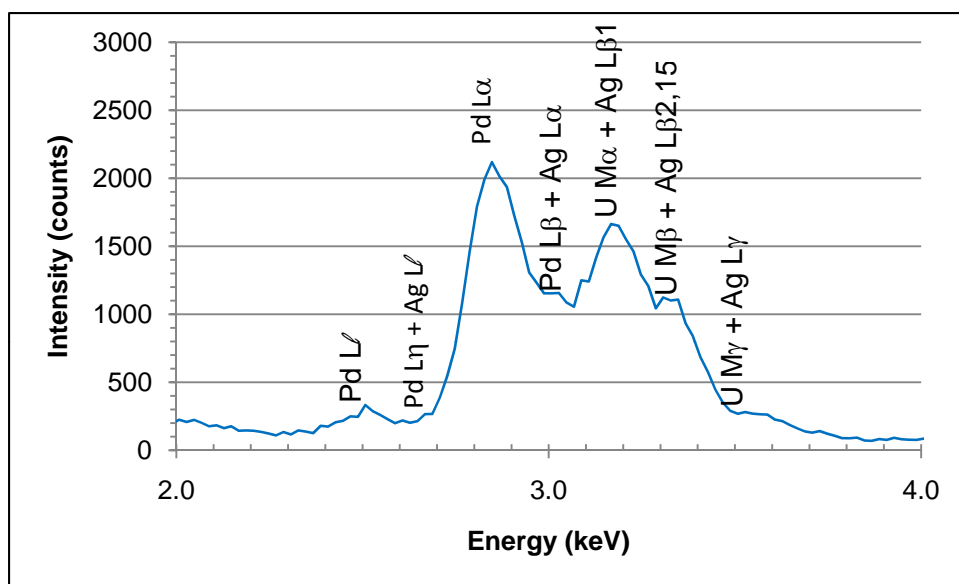
# Precipitate identification

- TEM



## Precipitate identification

- TEM- EDS



All Ag peaks within the standard energy range of current TEM-EDS detector (0-20 keV) are overlapped by peaks from U or Pd, making it impossible to identify Ag qualitatively

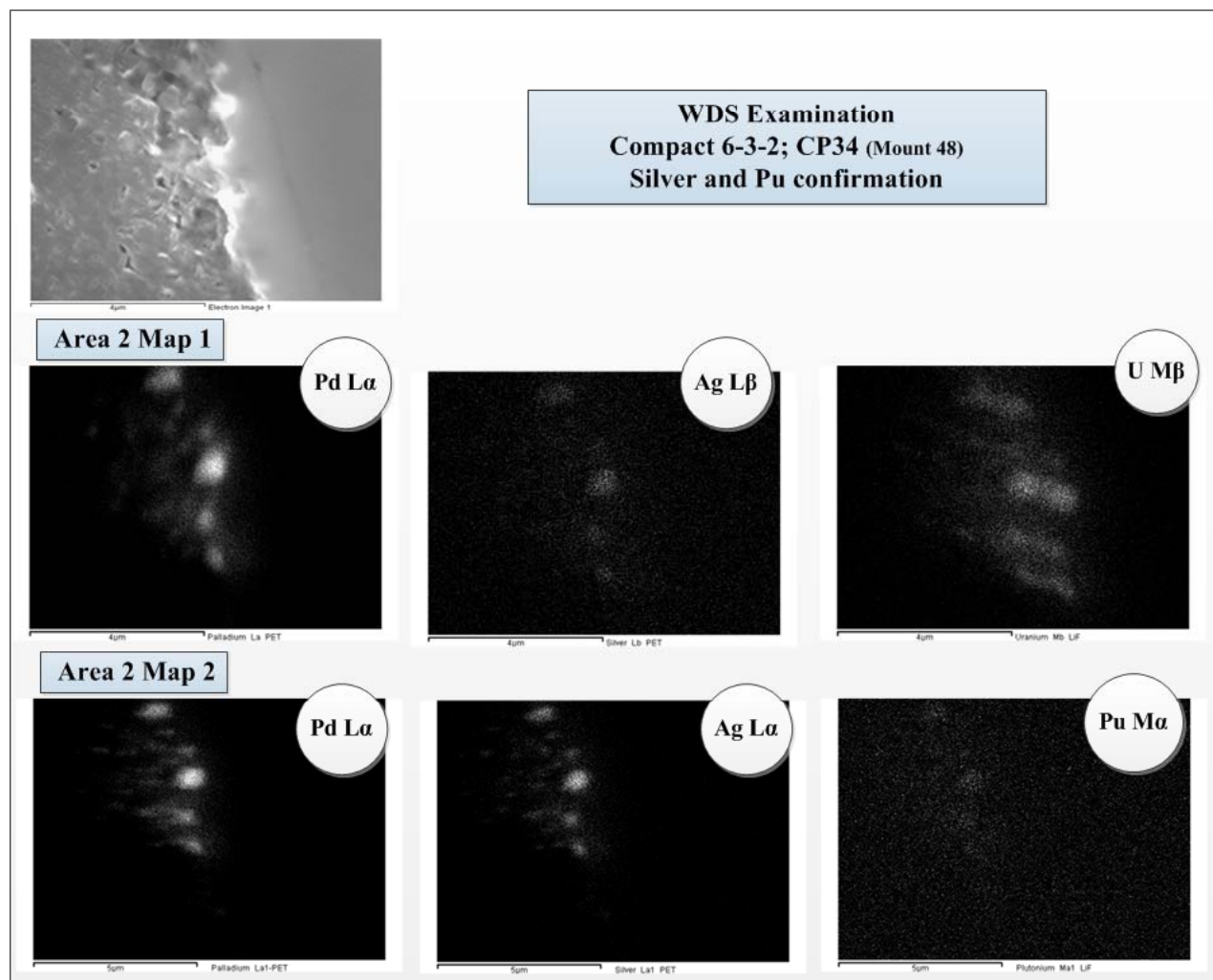
If the EDX detector is configured for an expanded energy range (e.g., 0-40 keV), the Ag K $\alpha$ 1 peak can be used to qualitatively confirm the presence of Ag. This peak may overlap the lower-intensity Ag K $\alpha$ 2 peak (at 21.993 keV), but does not overlap significant peaks from other elements believed to be present in the sample (C, Si, U, Pd, Zr, and Pu).

X-ray	Energy (keV)	Relative intensity	Overlapped by
Ag L $\gamma$	2.633	4	Pd L $\eta$
Ag L $\alpha$	2.984	111	Pd L $\beta$
Ag L $\beta$ 1	3.150	56	U M $\alpha$
Ag L $\beta$ 2, 15	3.347	13	U M $\beta$
Ag L $\gamma$ 1	3.519	6	U M $\gamma$
Ag K $\alpha$ 1	22.166	100	(None)

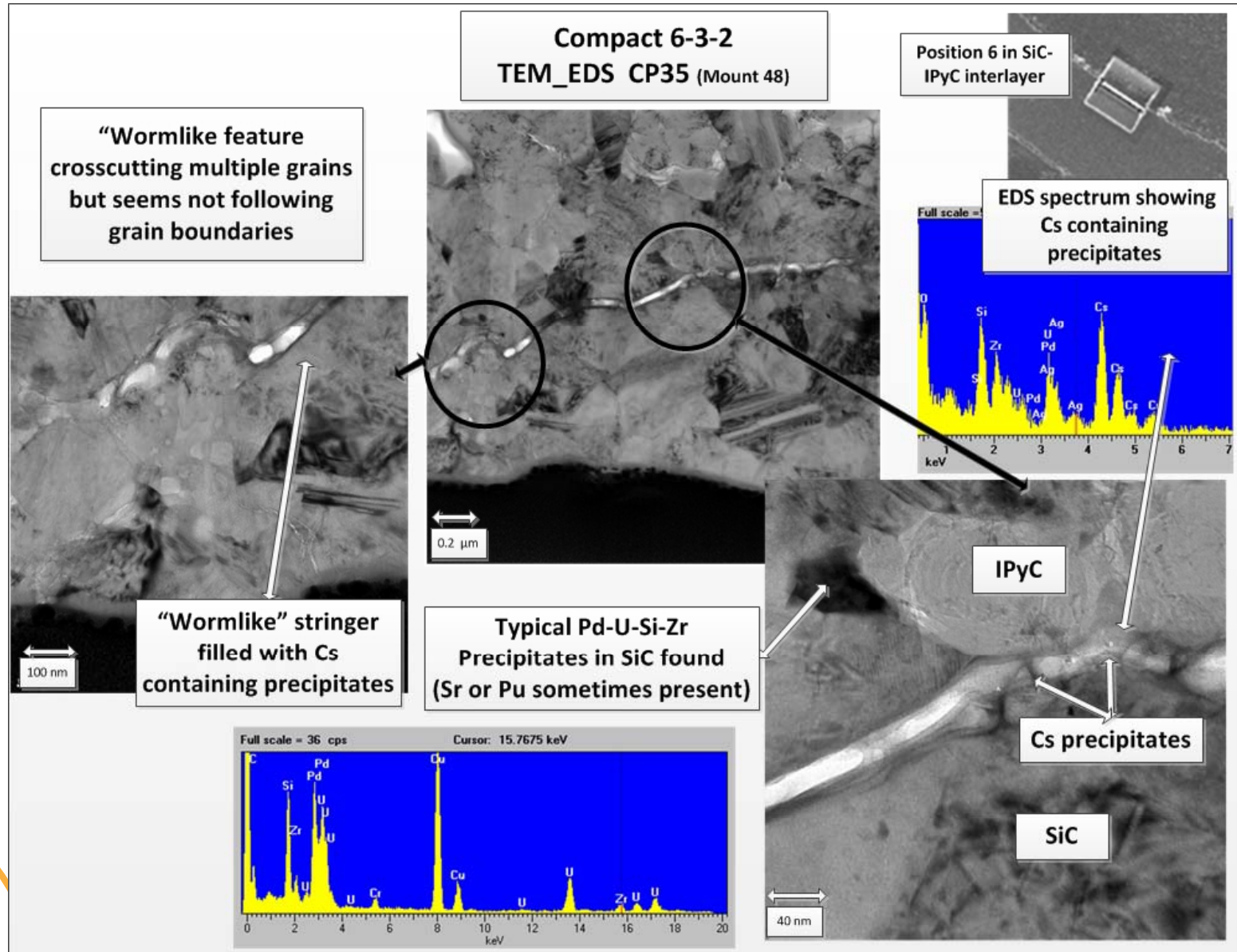
# Precipitate identification: Silver confirmation

## SEM- WDS

- Ag L $\alpha$  & Pd L $\beta$  X-rays (2.984 and 2.990 keV) are similar
- Ag L $\beta$  X-ray is more suitable for WDS maps because it is separated by 78 eV from the closest Pd X-ray (Pd L $\beta$ 3, at 3.072 keV) and by 96 eV from the closest U X-ray (U M $\alpha$ 2, at 3.1596 keV).



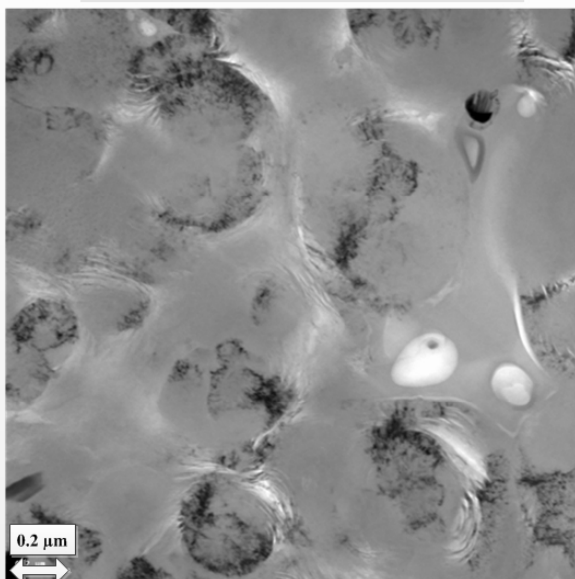
# Precipitate identification



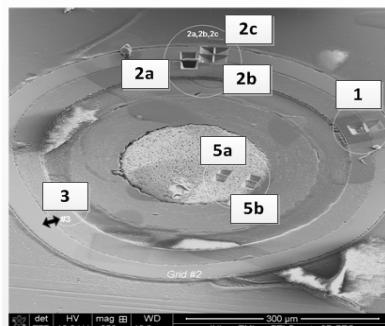


# SiC-IPyC microstructure

Compact 6-3-2 CP34 (Mount 48)  
Representative IPyC Microstructure  
TEM Position 1a

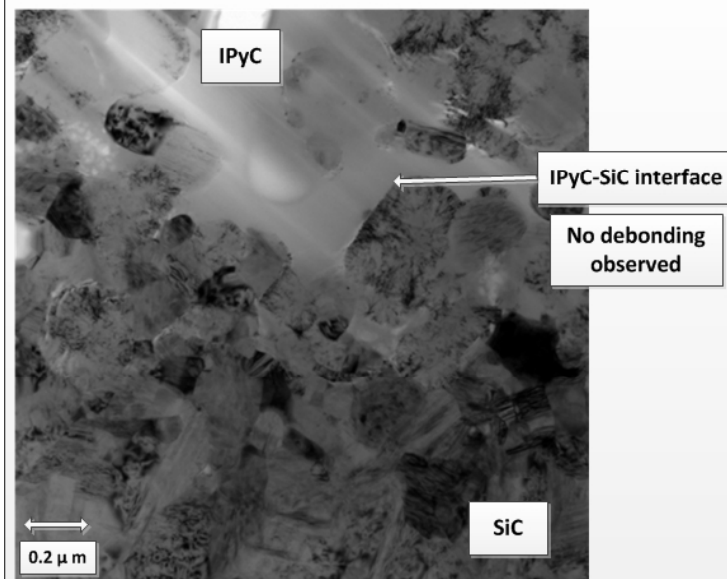


No significant change  
observed

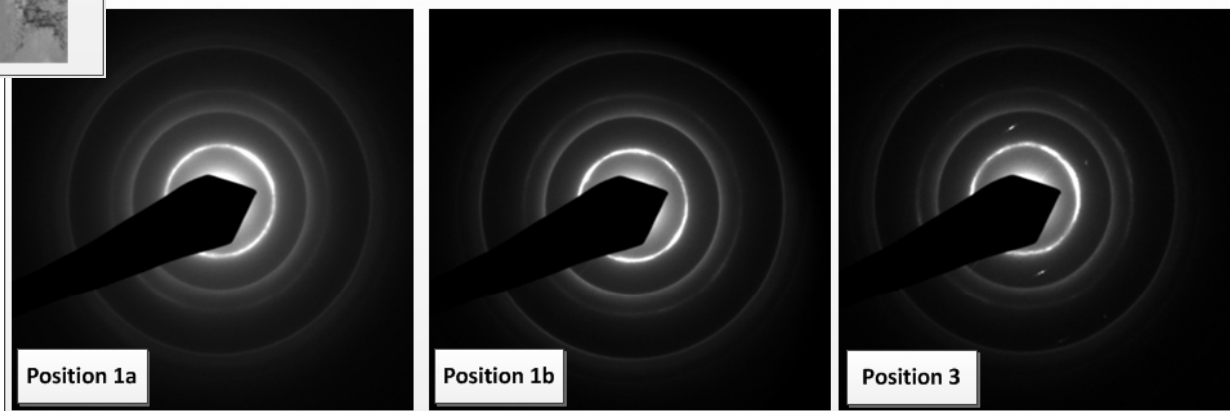


No debonding observed  
in TEM samples  
evaluated (CP34, CP35)

Compact 6-3-2 CP34 (Mount 48)  
IPyC-SiC interface TEM Position 3



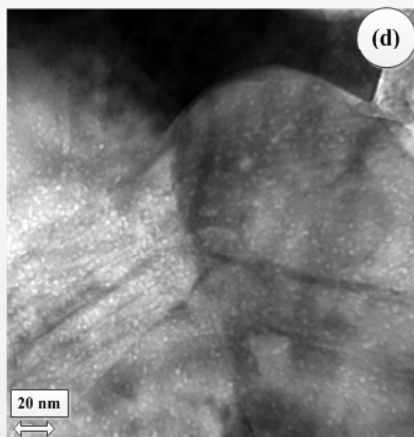
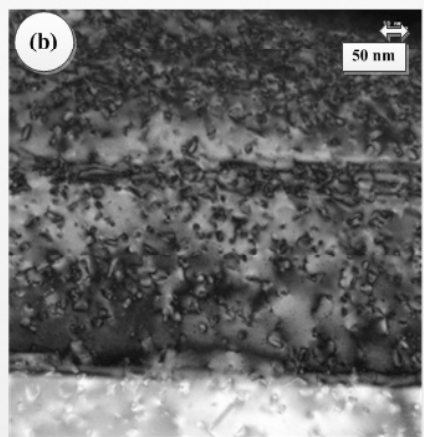
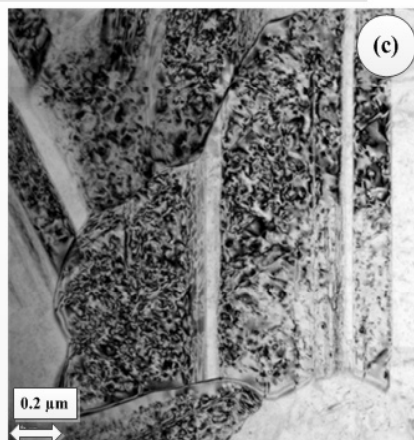
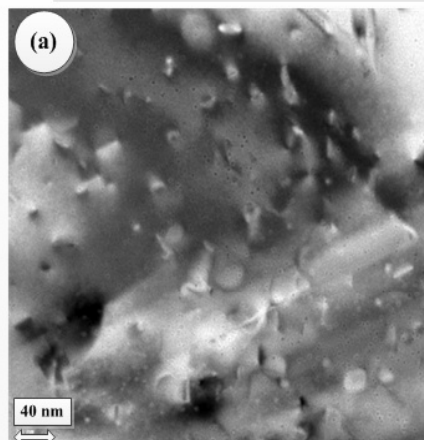
Compact 6-3-2 CP34 (Mount 48)  
IPyC TEM Positions 1 & 3



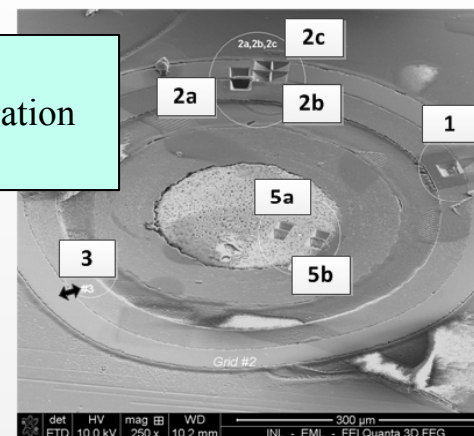
# SiC microstructure

Compact 6-3-2 CP34 (Mount 48)

Irradiation effects in SiC Microstructure TEM position 2c & 3



Typical irradiated microstructures: investigation in process

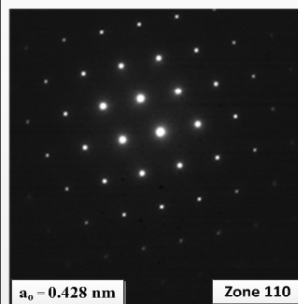


Note sample 4 as planned was not prepared, but additional samples in position 2 were prepared.

Compact 6-3-2 CP34 (Mount 48)  
TEM SiC Phase identification

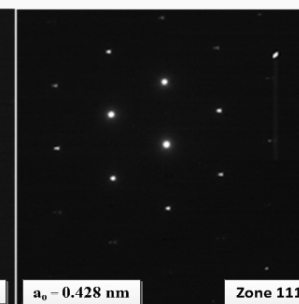
Position 2c  
(original SAD micrograph 2)

3C-SiC



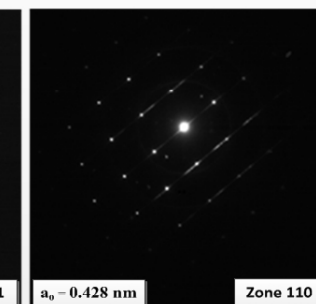
Position 3  
(original SAD micrograph 6)

3C-SiC



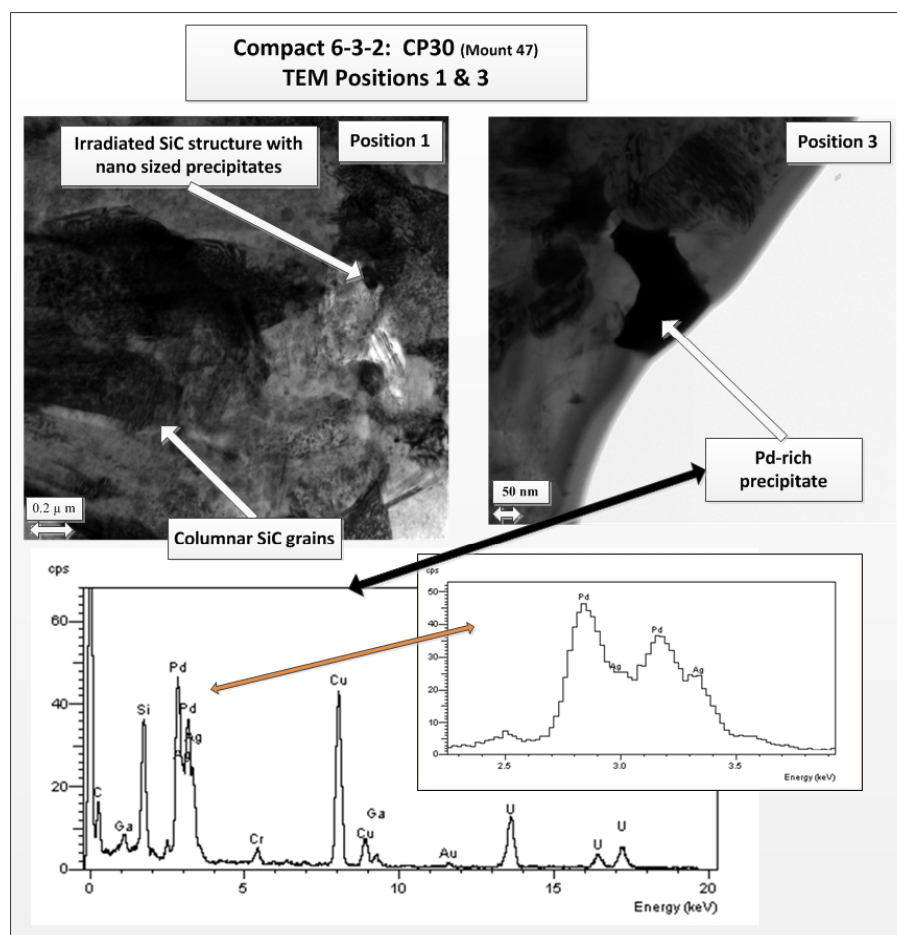
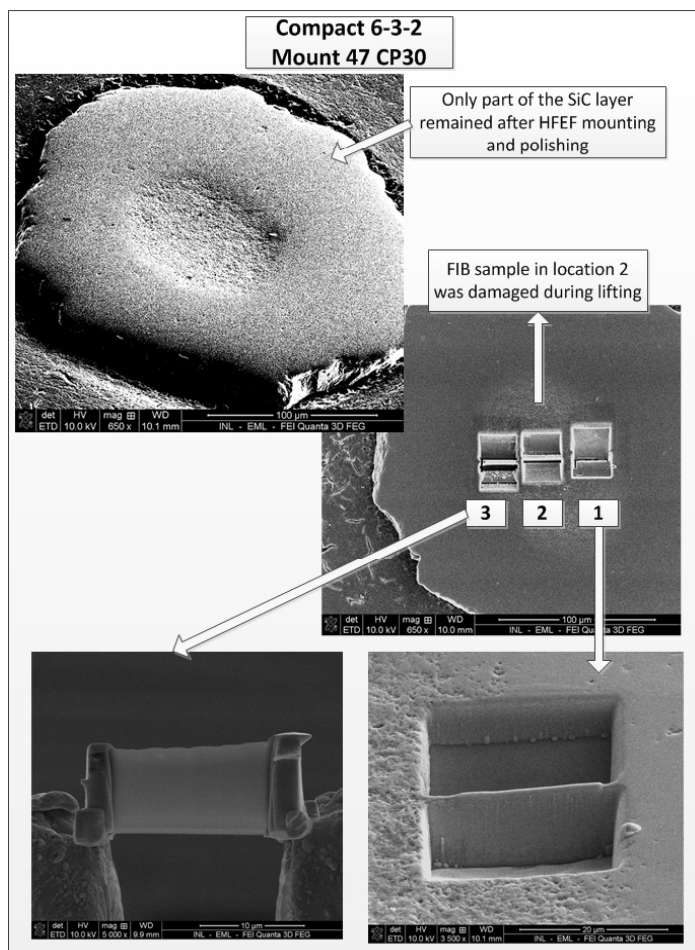
Position 3  
(original SAD micrograph 20)

3C-SiC



No evidence of any phase transformation (CP34, CP35)

# SiC microstructure: Low 110m Ag (CP30)





## ***Conclusions: Preliminary***

- Pd-rich precipitates randomly distributed in SiC-IPyC interlayer and present on both the SiC fingers and IPyC
- Ag identified in Pd-rich precipitates
- No significant difference in SiC microstructure observed between low and high 110m Ag particles----still under investigation
- No SiC phase transformation due to irradiation observed
- Pd-rich precipitates identified but detail will be provided at HTR2012 conference

## ***Acknowledgements***

- Jim Madden: FIB sample preparation
- Scott Ploger & Jason Harp : Mount and decontamination preparation for electron microscopy examination