Exceptional service in the national interest

Sandia National Laboratories

IPF Z Orientation Map

SEM Pole Piece
e- beam
specimen
EBSD CCD
Conventional TKD Detector Geometry

SEM Pole Piece
e- beam
specimen
EBSD CCD
Horizontal TKD Detector Geometry

e- beam
specimen
TEM Pole Piece
Fast Camera
Phosphor screen
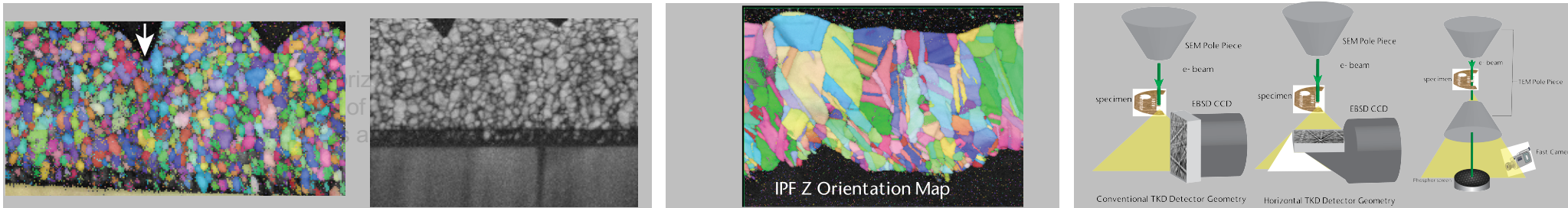
Crystallographic Orientation Image Mapping with Multiple Detector Configurations at 30 – 300 kV

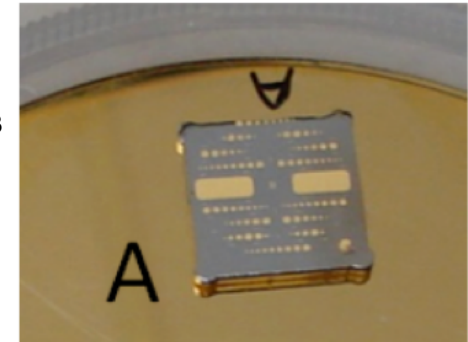Joshua D. Sugar[1], Joseph T. McKeown[2], Daniel C. Bufford[3], and Joseph R. Michael[3]

[1]Sandia National Laboratories, Livermore, CA; [2]Lawrence Livermore National Laboratory, Livermore, CA; [3]Sandia National Laboratories, Albuquerque, NM

U.S. DEPARTMENT OF ENERGY
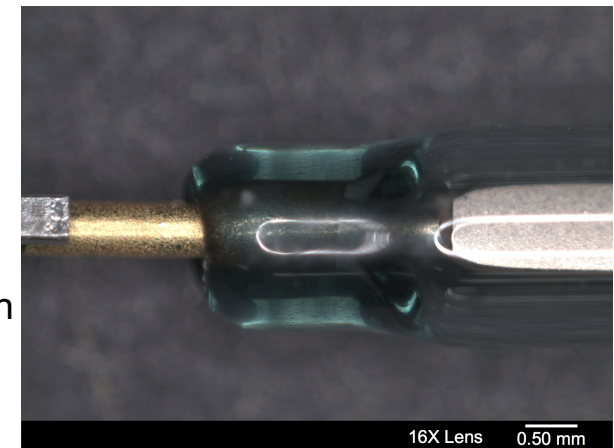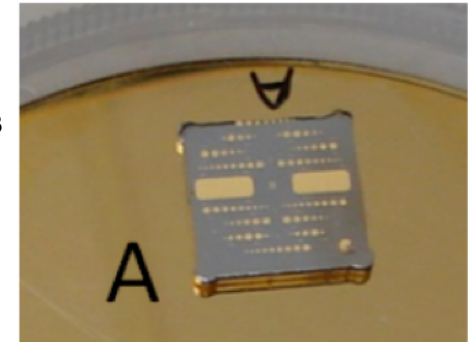National Nuclear Security Administration

Microstructural Analysis of Long-Life Electrical Contacts

- Sandia is interested in understanding the aging of long-lived electrical contacts for a variety of materials and purposes
  - How does the grain structure and chemistry evolve from initial processing to end of life?
- The chemical and morphological of these contacts can be characterized with electron microscopy
- We can use TEM and SEM-based techniques to characterize microstructural properties
  - Composition: EDS/EELS
  - Grain structure: Imaging
  - Crystallographic Orientation/Texture: EBSD/TKD/PED

Array of Au pads on Bi_2Te_3

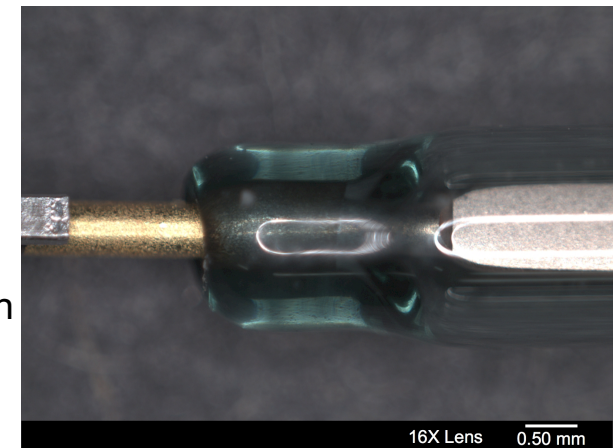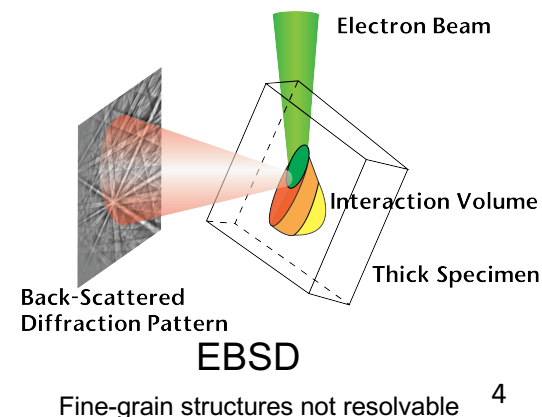Au-plated kovar pin on electrical switch

16X Lens   0.50 mm

2

Microstructural Analysis of Long-Life Electrical Contacts

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Array of Au pads on Bi_2Te_3

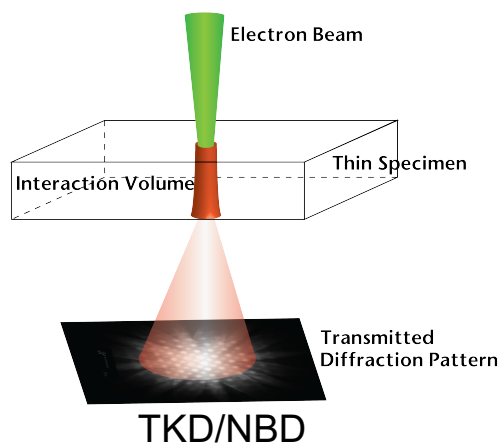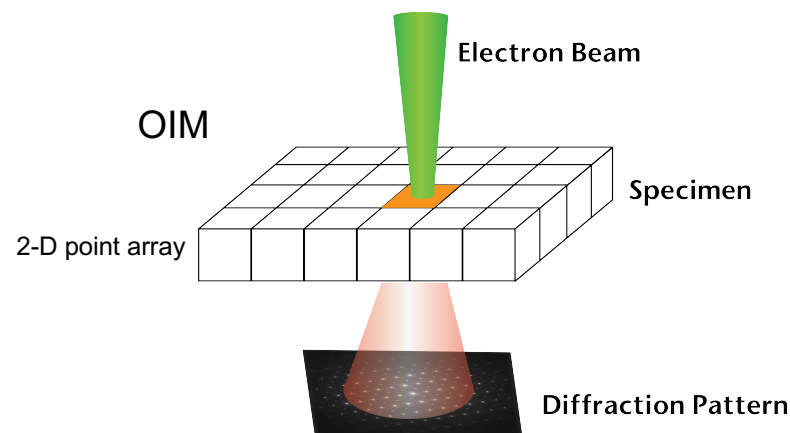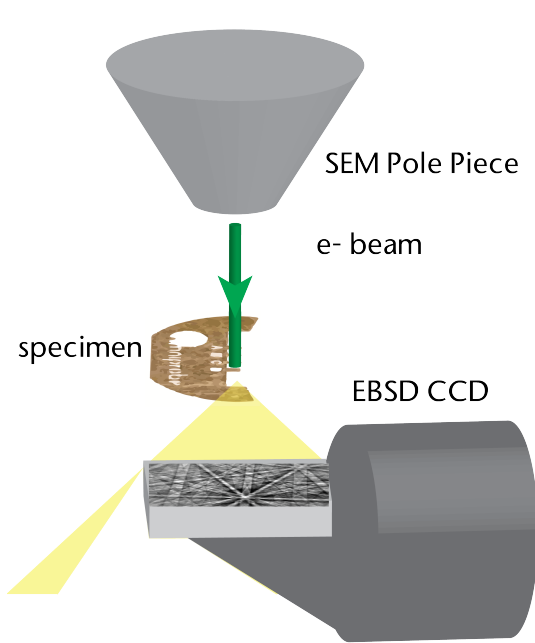Au-plated kovar pin on electrical switch

16X Lens   0.50 mm

TEM-like Resolution in SEM with Thin Samples

- Orientation Image Mapping (OIM) is done by collecting a diffraction pattern at a 2-D array of points
    - Kikuchi patterns can be collected in the SEM @ max 30 kV
        - Backscattered for bulk samples: EBSD resolution limited by interaction volume
        - Transmission for thin samples: TKD
    - Nanobeam diffraction patterns (spots) collected in a TEM @ max ~300 kV

- Can the same scale features be mapped at 30 kV and 300 kV? Do they map the same thing?

OIM

Electron Beam

Specimen

2-D point array

Diffraction Pattern

Electron Beam

Interaction Volume

Thin Specimen

Transmitted Diffraction Pattern

TKD/NBD

Electron Beam

Interaction Volume

Thick Specimen

Back-Scattered Diffraction Pattern

EBSD

Fine-grain structures not resolvable

4

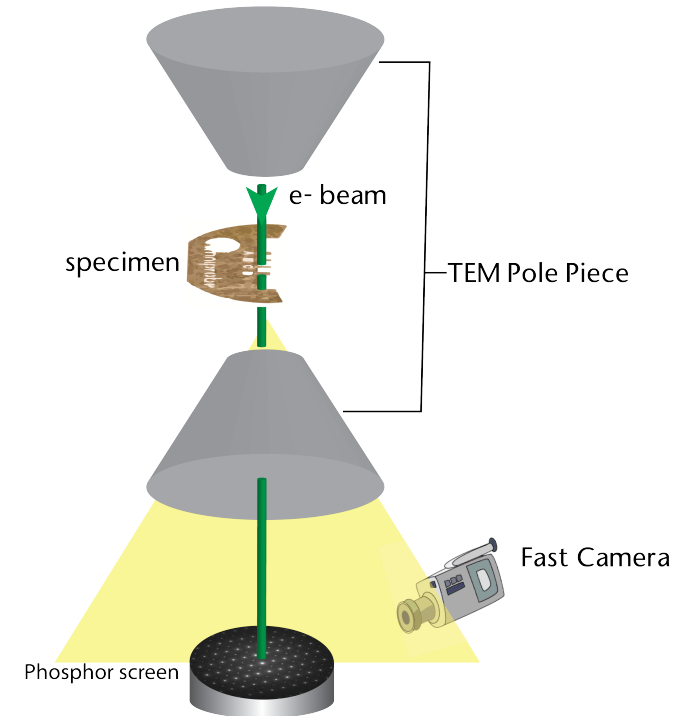# Comparison of 3 Different Experimental Arrangements

Horizontal TKD Detector Geometry

Zeiss Supra 55 VP @ 30 kV
Bruker eFlash HR EBSD
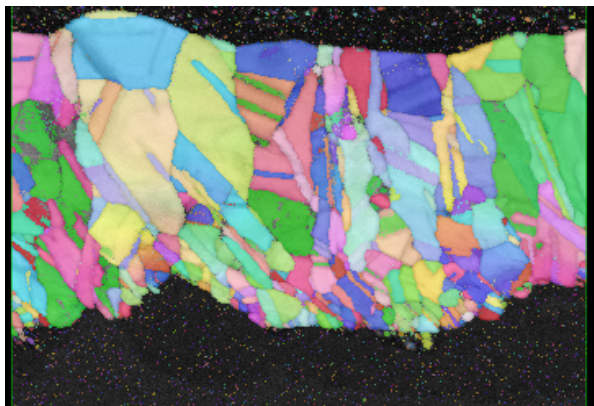Optimus flat on-axis attachment
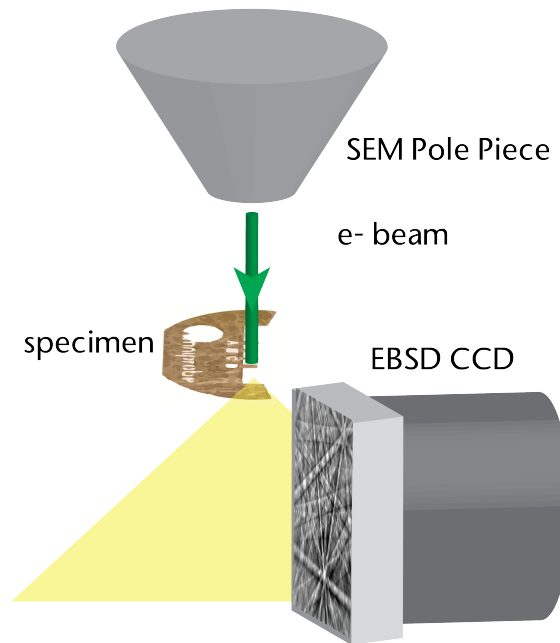
Conventional TKD Detector Geometry

FEI Helios Nanolab 660 @ 30 kV
Oxford Nordlys Max 2 EBSD
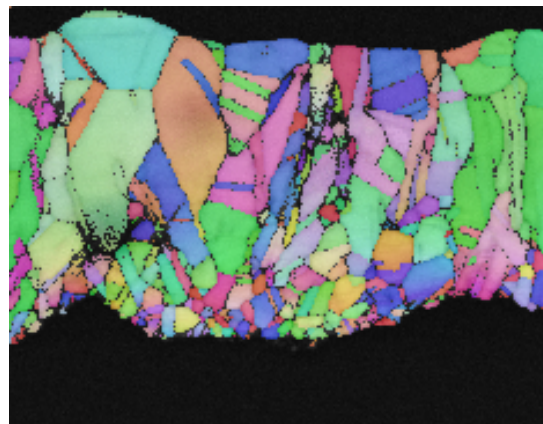
Philips CM 300 @ 300 kV
Nanomegas Astar

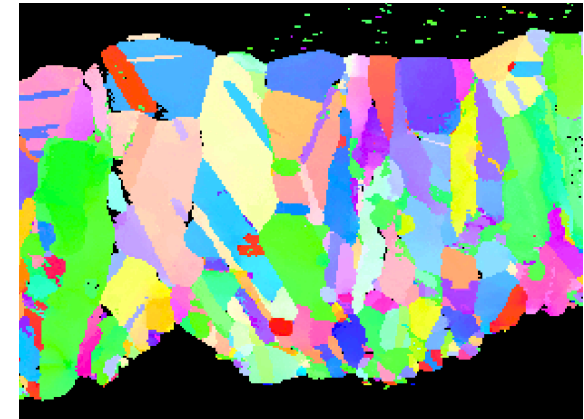# Same Results for Pulsed Plated Au Grains > ~10 nm
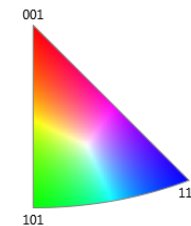

200 nm

4 nm/pixel
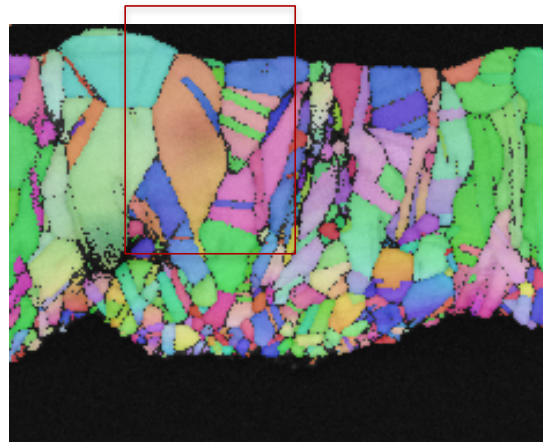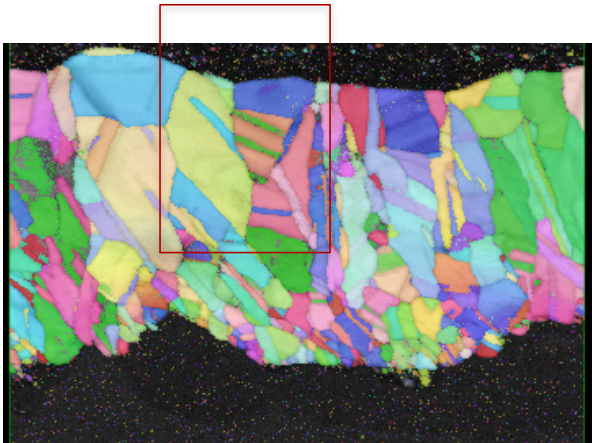
Horizontal

30 kV

200 nm

5 nm/pixel

Vertical

200 nm

300 kV

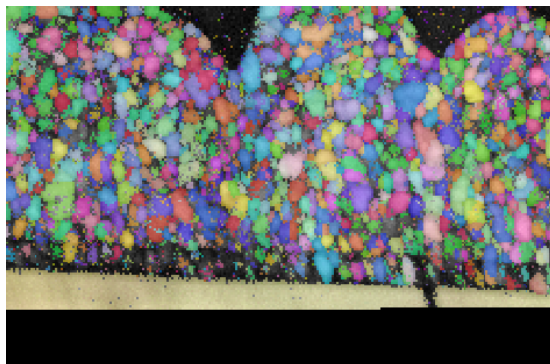# Same Results for Pulsed Plated Au Grains > ~10 nm

IPF Z Maps
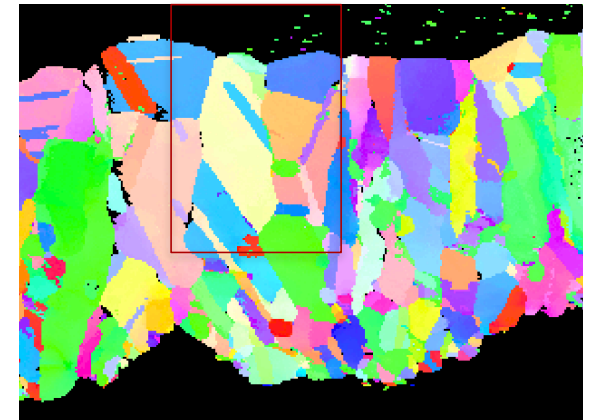
- Some distortion from drift
- Ability to detect twins is not the same
- For the most part the result is the same
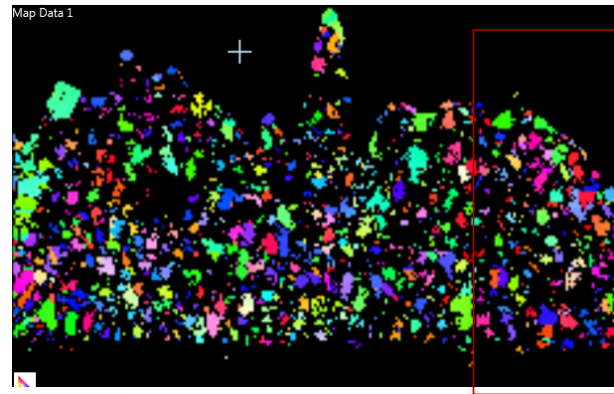- 30 kV results are more similar than the 300 kV results

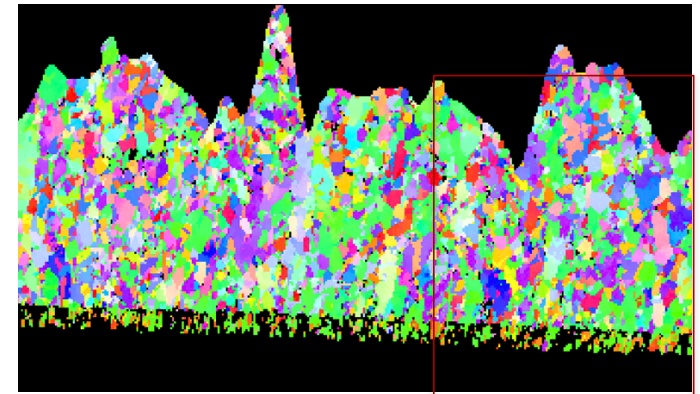# Fine-Grained Structure (DC-plated) Data Show Significant Differences

200 nm

200 nm

200 nm

4 nm/pixel

5 nm/pixel

300 kV

Horizontal
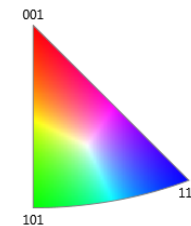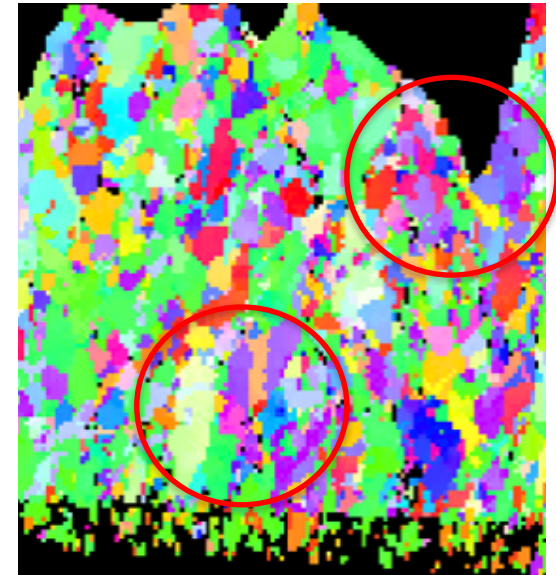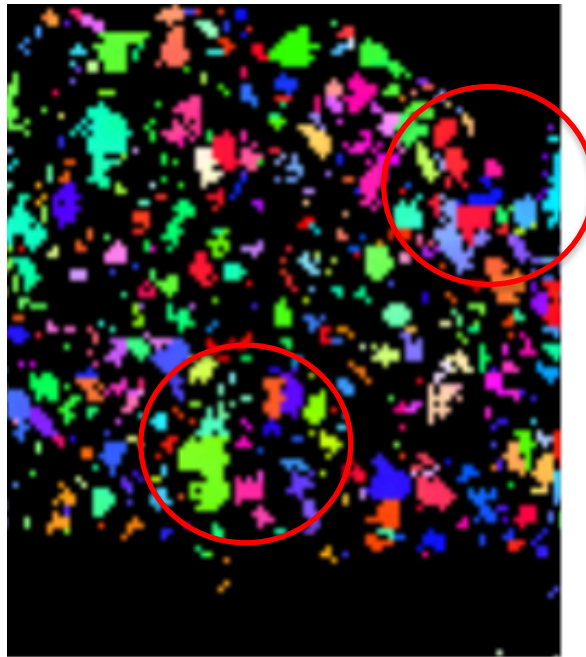
Vertical

30 kV

Fine-Grained Structure (DC-plated) Data Show Significant Differences

- Some regions are similar
- Vertical TKD detector has many missed pixels
- TEM data shows more 101 (green) than horizontal TKD

Why are these different?

What Causes the Observed Differences in These Techniques?

- Drift
  - Stage stability is different

- Electron Optics Mode
  - Final electrostatic vs. electromagnetic lens for high resolution

- Contamination
  - Different effective thickness of contaminant

- Electron Energy
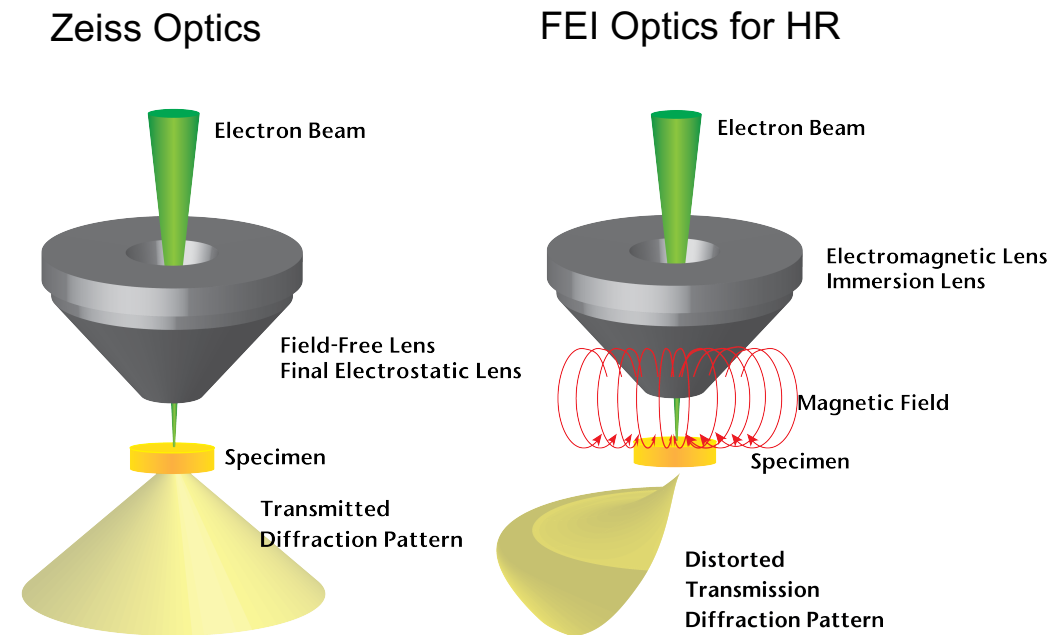 - Electron mean free path difference

What Causes the Observed Differences in These Techniques?

- Drift
  - Stretching or compressing of grains NOT orientation
  - Depending on beam current, detector gain, binning, etc. acquisition times can range from 1 ms to 100s of ms
  - Ample time for drift
  - Drift correction routines at this pixel dwell time cause jittery images
- Electron Optics Mode
- Contamination
- Electron Energy

1 μm   Drift Correction On

1 μm   Drift Correction Off
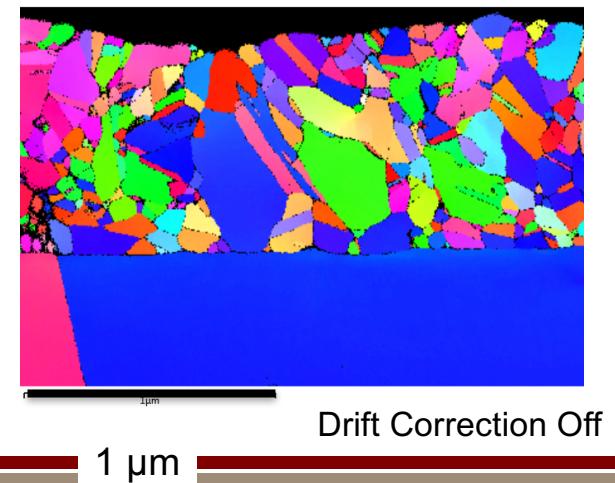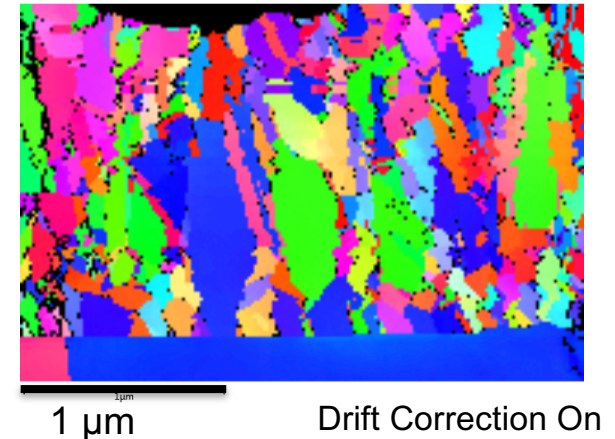
What Causes the Observed Differences in These Techniques?

- Drift
- Electron Optics Mode for TKD
  - Smallest probes obtainable in field-free mode and immersion mode are different
  - FEI uses immersion for high resolution Zeiss does not
  - Can we get fine-scale maps in FEI system?
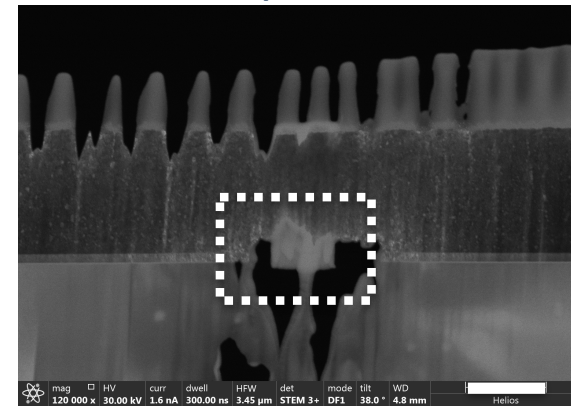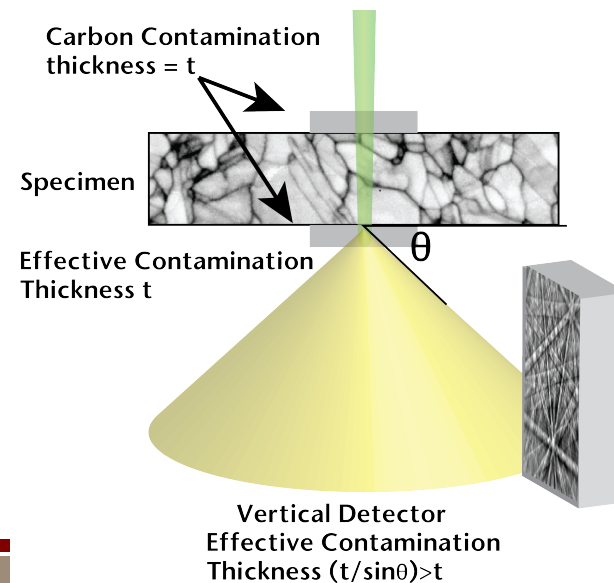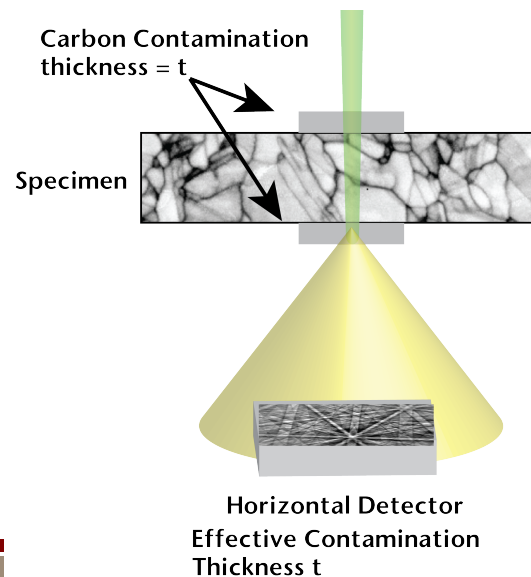- Contamination
- Electron Energy

Zeiss Optics

Electron Beam

Field-Free Lens
Final Electrostatic Lens

Specimen

Transmitted
Diffraction Pattern

FEI Optics for HR

Electron Beam

Electromagnetic Lens
Immersion Lens

Magnetic Field

Specimen

Distorted
Transmission
Diffraction Pattern

Does this matter?

What Causes the Observed Differences in These Techniques?

- Drift
- Electron Optics Mode
- Contamination
  - Build up of amorphous C will scatter the diffracted electrons
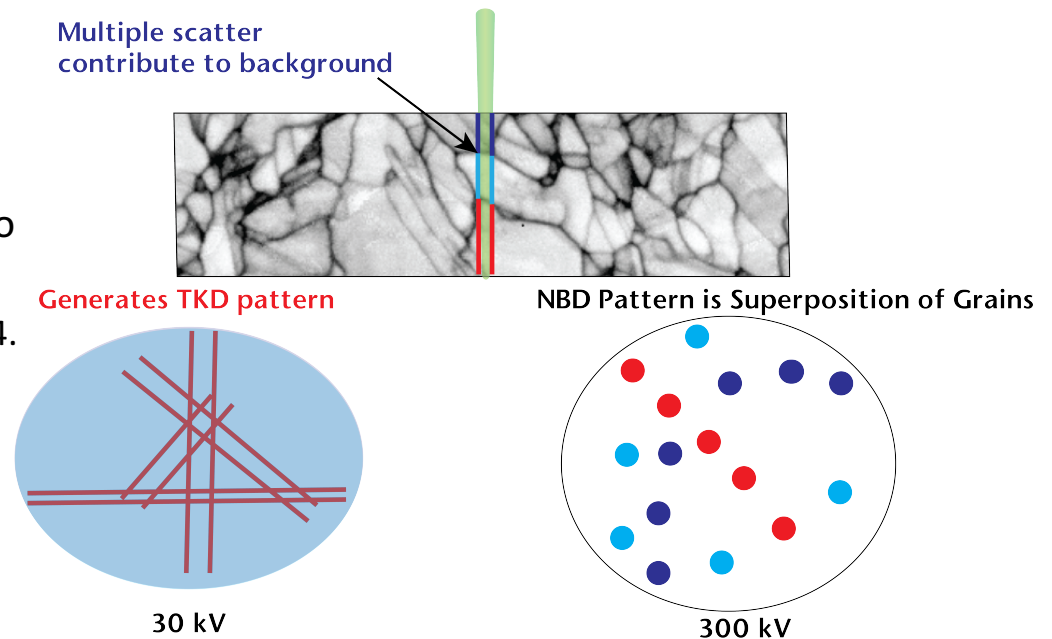- Electron Energy

500 nm

Carbon Contamination thickness = t

Specimen

Horizontal Detector
Effective Contamination
Thickness t

Carbon Contamination thickness = t

Specimen

Effective Contamination
Thickness t

θ

Vertical Detector
Effective Contamination
Thickness $(t/\sin\theta) > t$

What Causes the Observed Differences in These Techniques?

- Drift

- Electron Optics Mode

- Contamination

- Electron Energy
  - Thickness does not vary here
  - For TKD the near-exit surface contributes to diffraction pattern
    - Rice, K.P. et al.. Journal of Microscopy, 2014. **254**(3): p. 129-136.

- At 300 kV in TEM, longer mean free path means diffraction occurs through sample thickness

Multiple scatter contribute to background

Generates TKD pattern

NBD Pattern is Superposition of Grains

30 kV

300 kV

Elimination of Contamination

- Plasma Cleaning of Chamber
- Plasma Cleaning of Sample
- Cold Finger in Chamber to condense contaminants

Lens Mode Does Not Matter
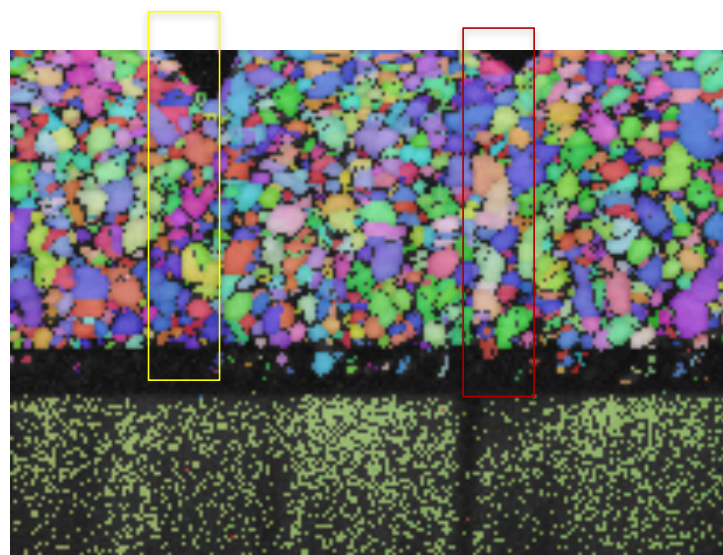
200 nm

Vertical Detector
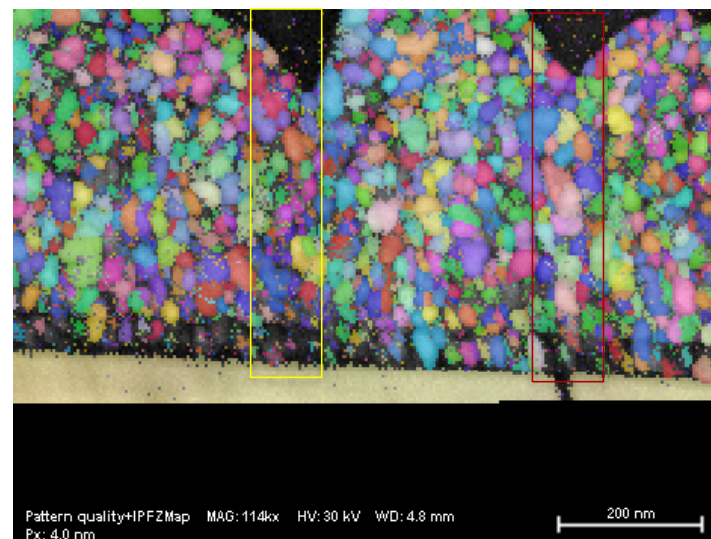
Horizontal Detector

Multiple Grains Through Thickness Not Singularly Indexable at 300 kV
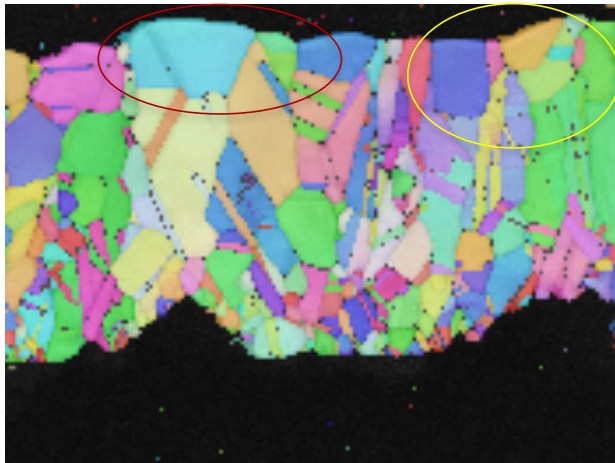
30 kV Vertical Detector
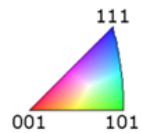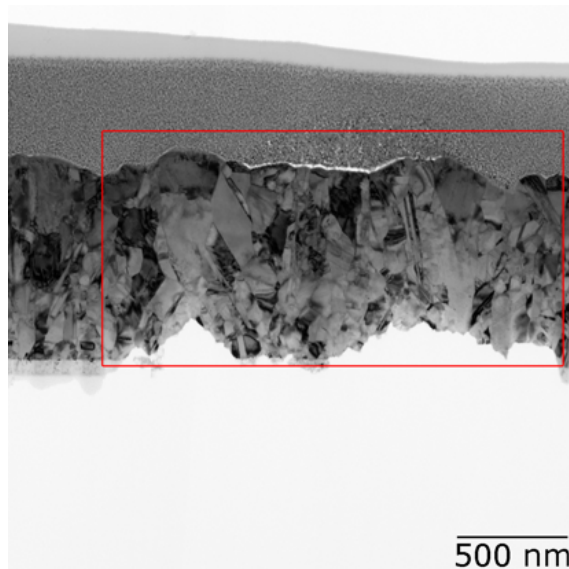"Wrong side up"
Data flipped
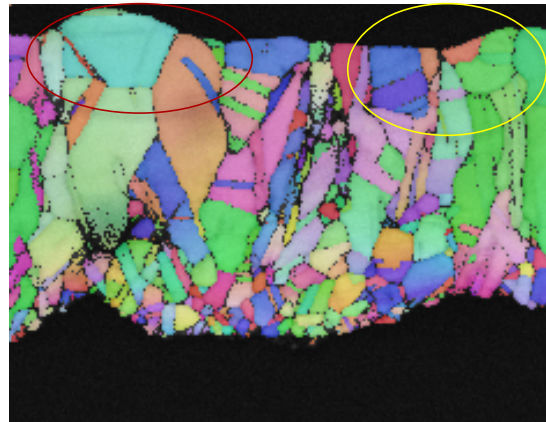
30 kV Vertical Detector
"Right Side Up"
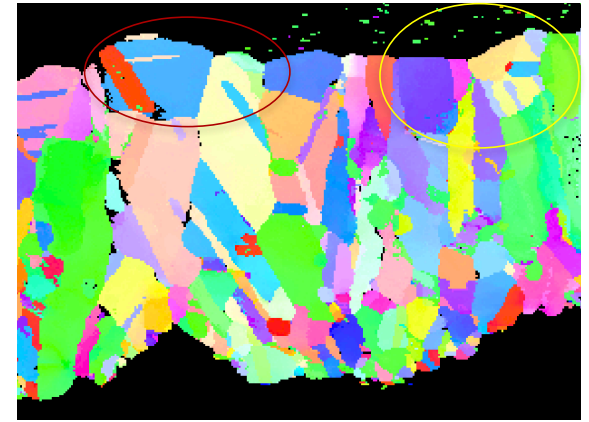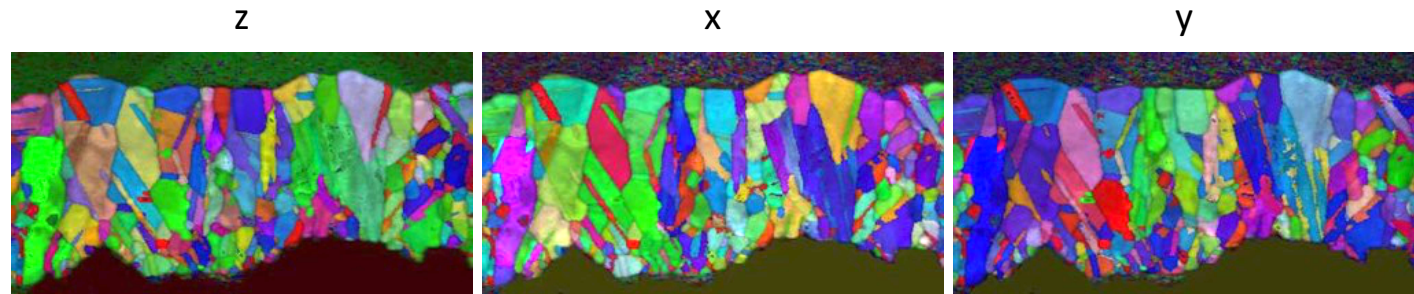

300 kV

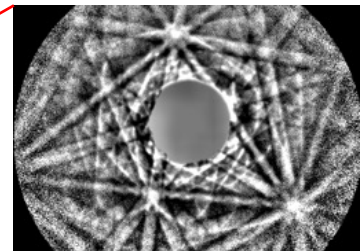# TKD Indexability is Insensitive to Orientation

2017-0524-Dbanga Au TKD
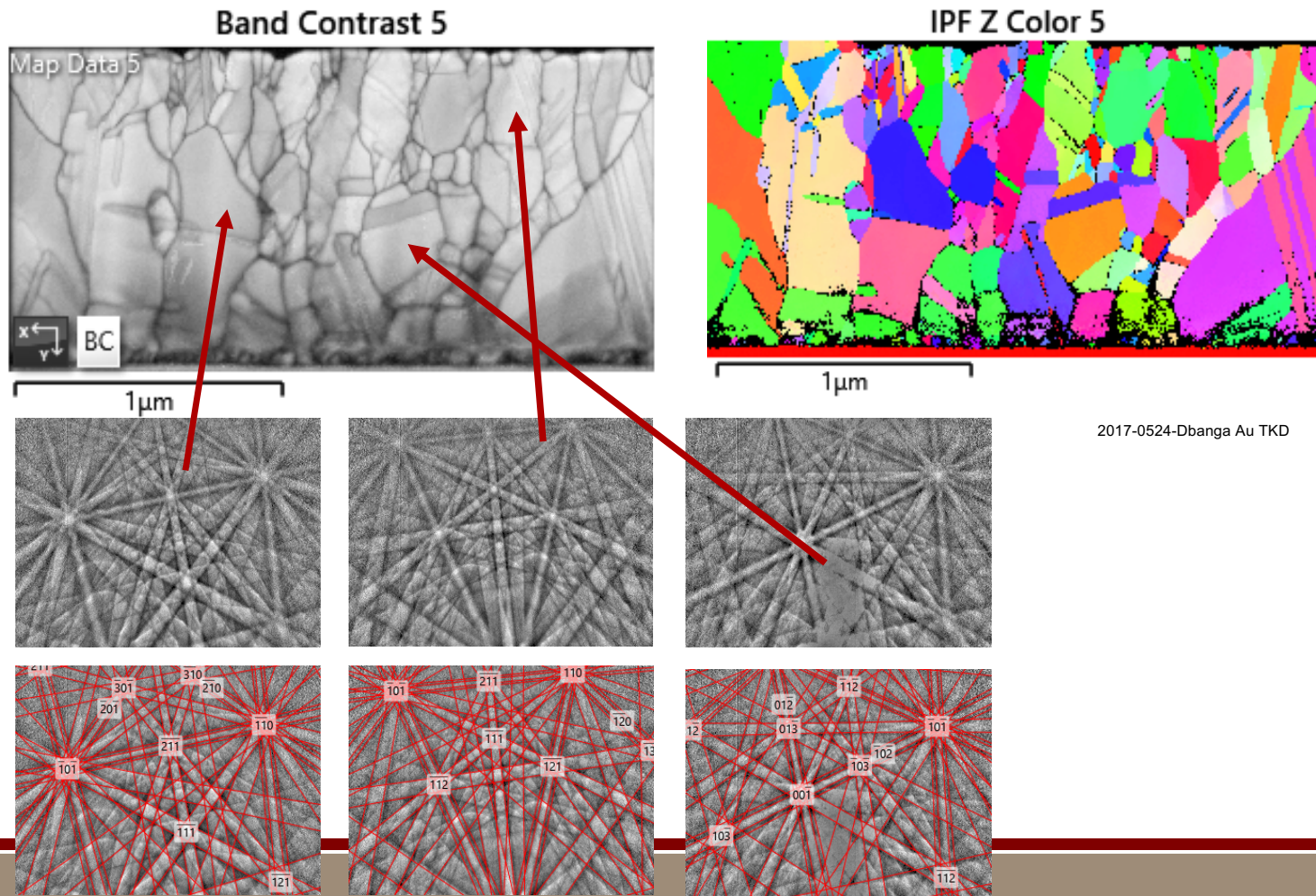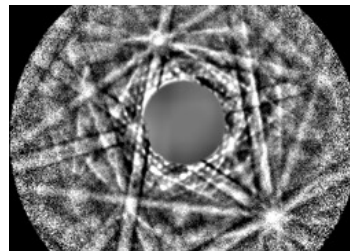
PED Singular Solutions More Difficult Far From Zone

z   x   y

500 nm

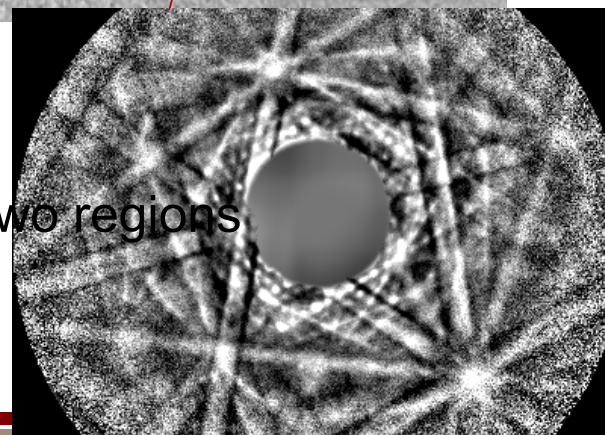Low-angle GB comparison for TKD and PED

111

001   101

Off-zone pattern: 2 bright spots

200 nm
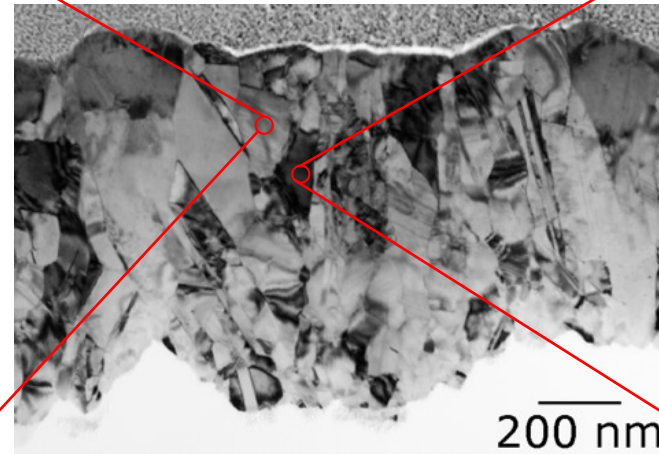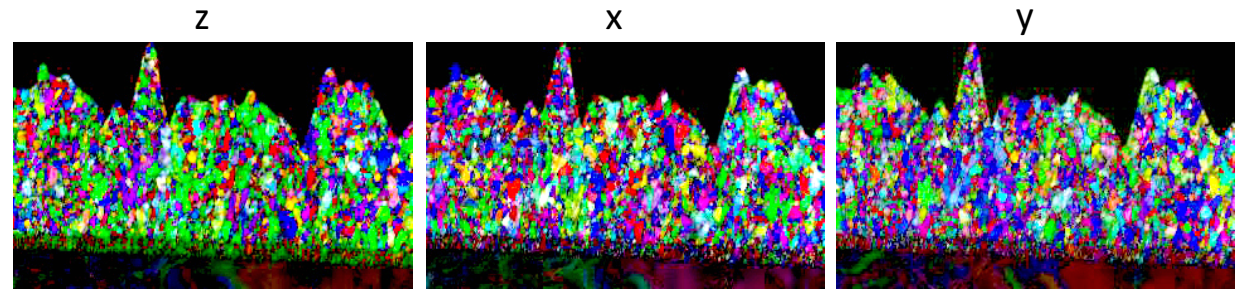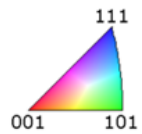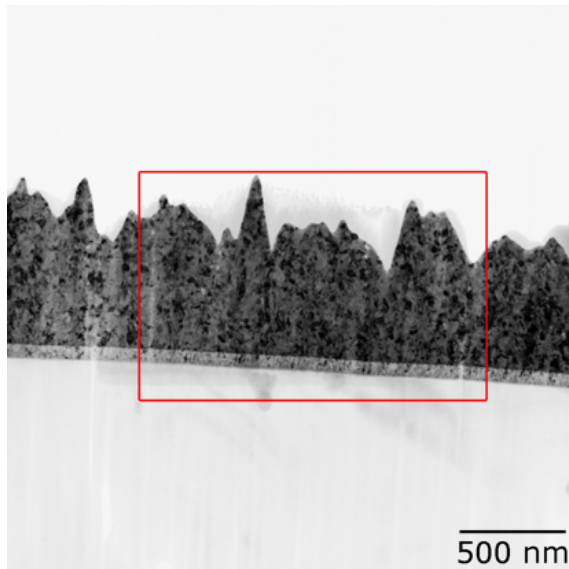
Close to Zone Pattern
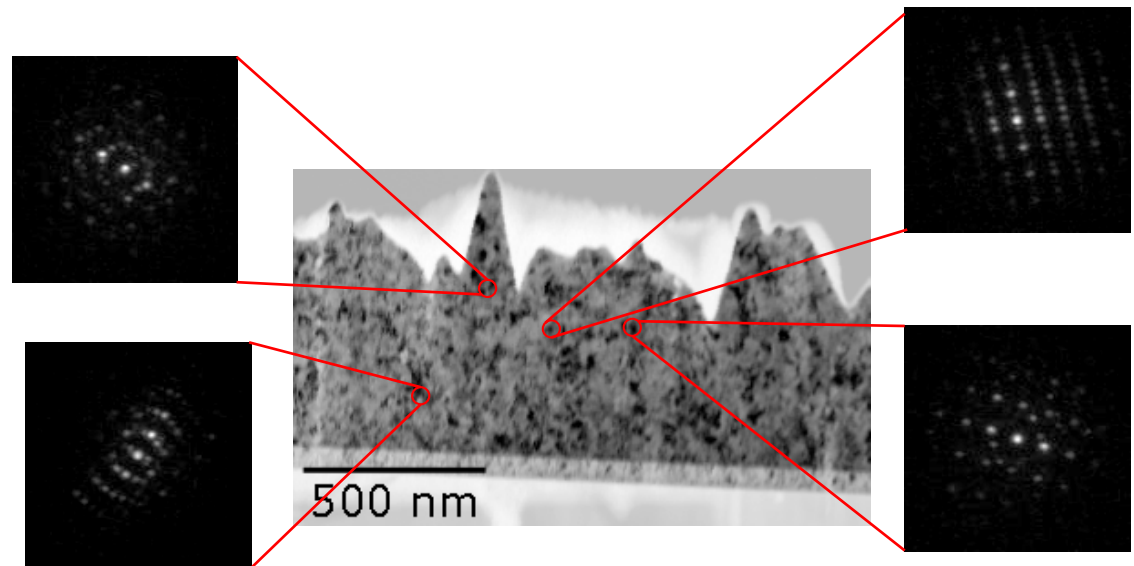
Dark grain
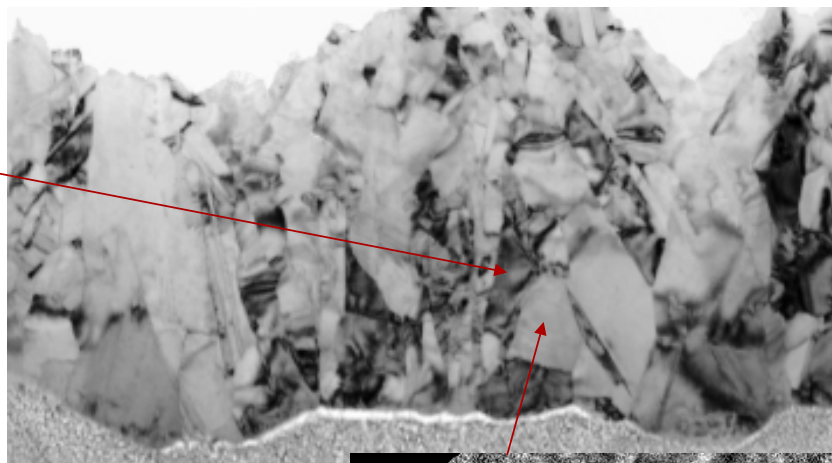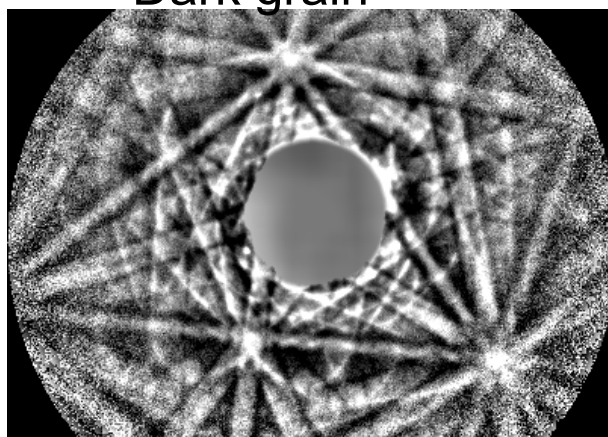
Light grain

A low angle boundary is between these two regions

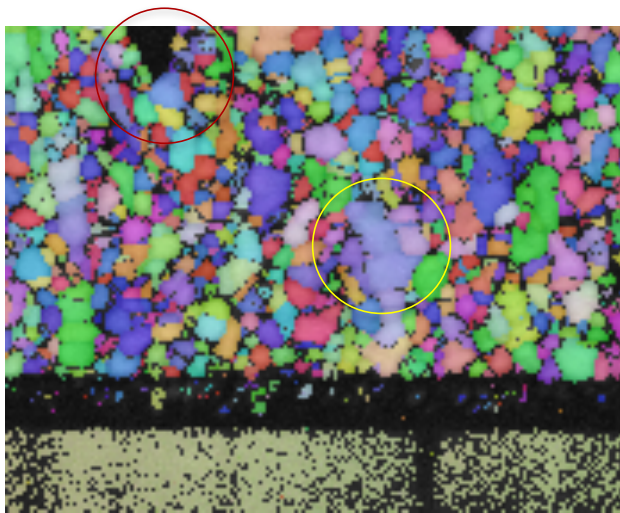# Multiple Grains Through Thickness Not Singularly Indexable at 300 kV

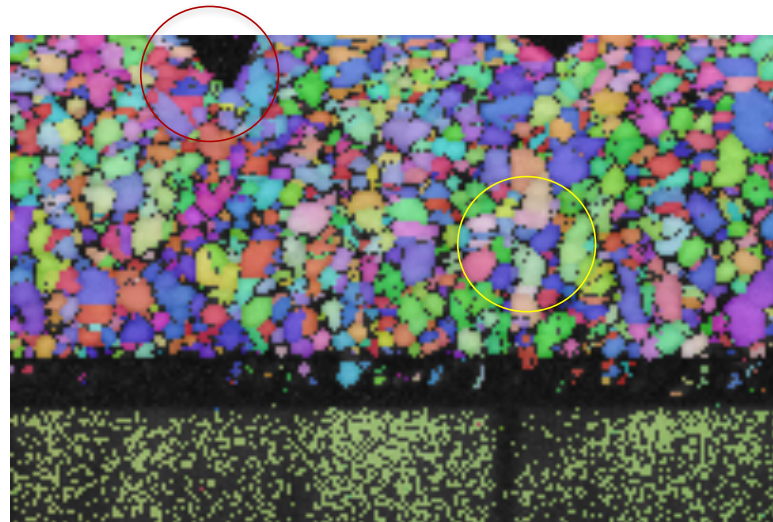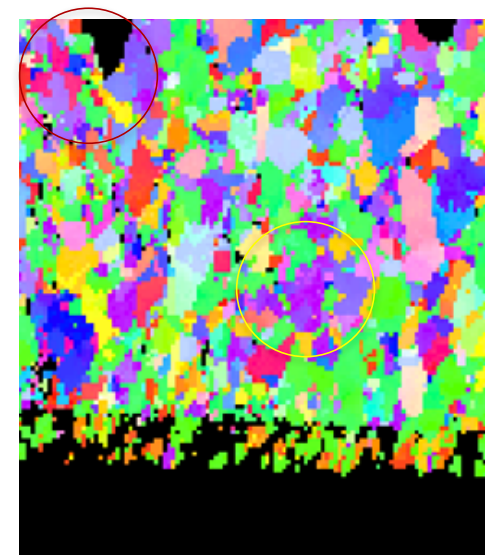Diffraction patterns showing overlap of grains (multiple orientations)

Multiple Grains Through Thickness Not Singularly Indexable at 300 kV

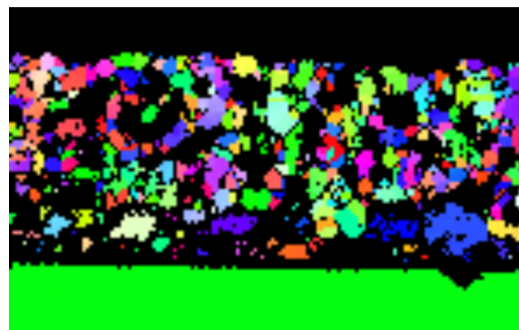30 kV Vertical Detector
"Wrong side up"
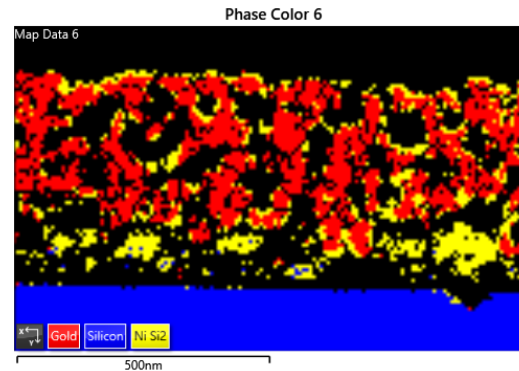Data flipped

30 kV Vertical Detector
"Right Side Up"

300 kV
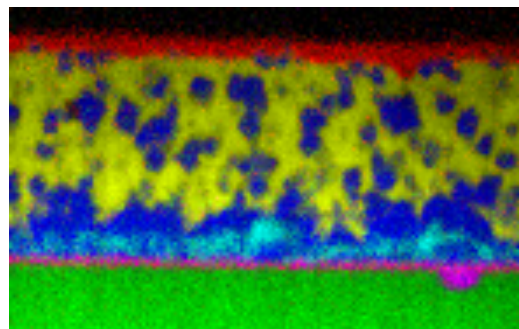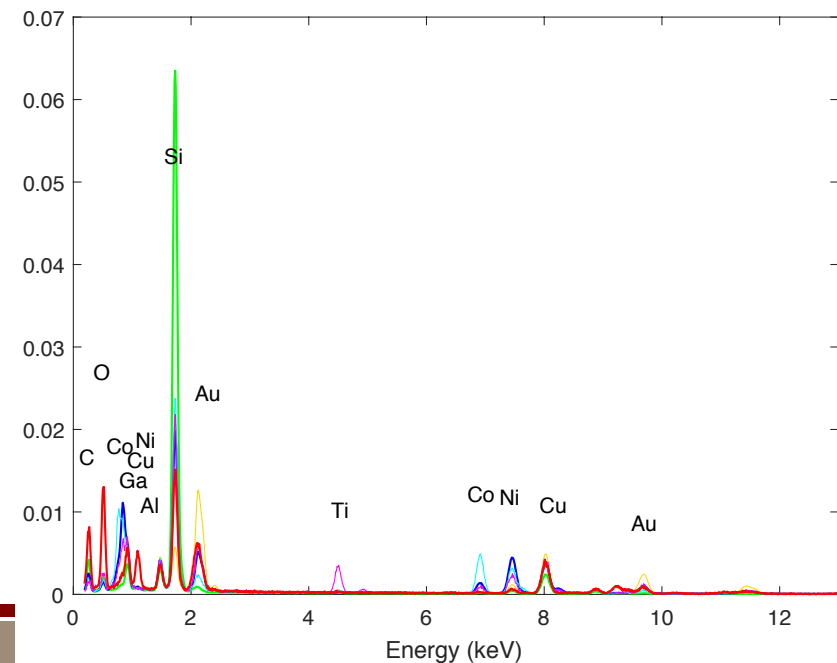
SEM Can Combine TKD with EDS

Au
Si
NiSi$_2$

500 nm

Si
Au
Ni-Si
Co
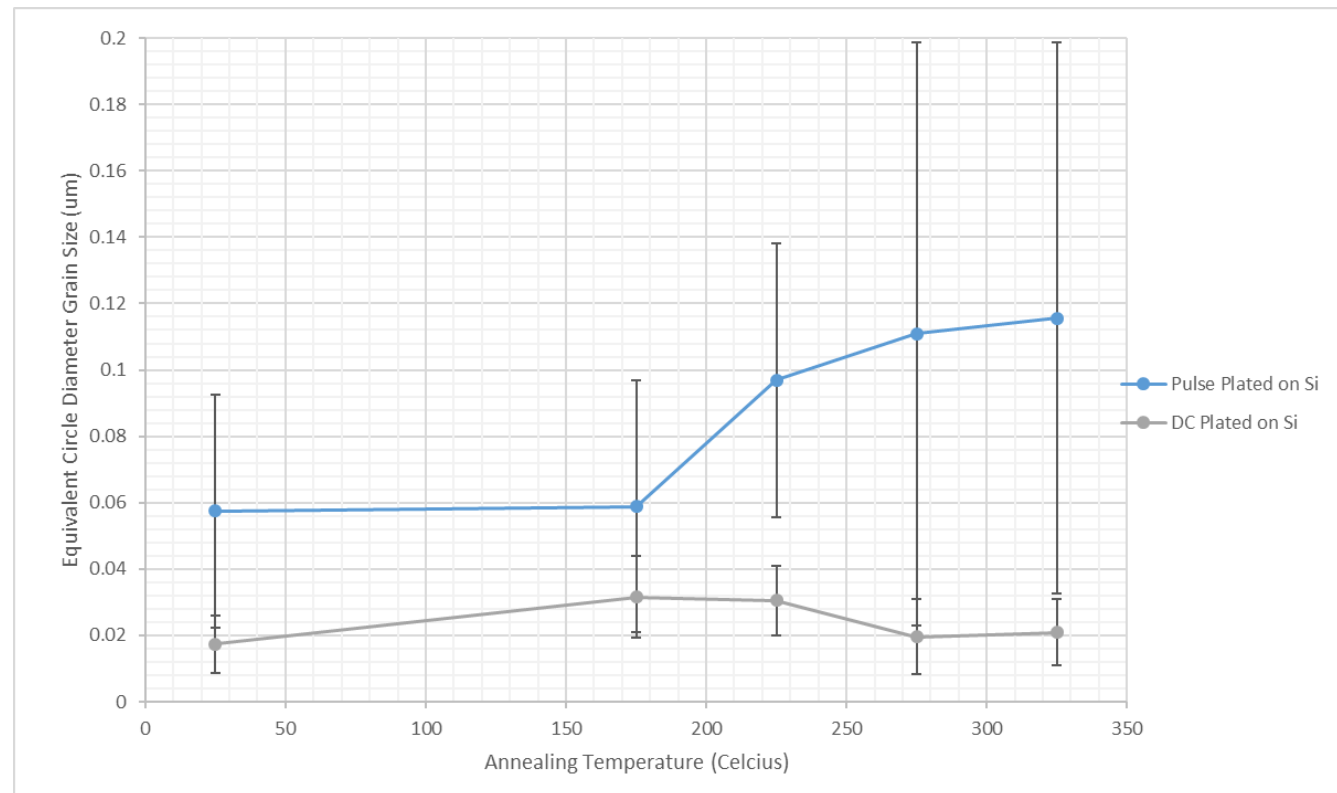Ti
C-O

Aged at 325°C for 2 weeks

EDS can be used to identify missing phases and fill in missing data in TKD map

The Effect of Aging Can be Predicted for Pulsed-Plated and DC-Plated Au films

- TKD allows quantification of grain morphology and orientation evolution
- EDS measures compositional evolution

Summary and Conclusions

- Orientation image mapping can be performed at 30 kV using TKD in an SEM or at 300 kV using NBD in a TEM
- The current state of detectors enables useful information to be gathered in all experimental configurations
- The orientation of the TKD detector does not limit the spatial resolution of TKD
- The electron lens configuration does not limit the spatial resolution of TKD
- Contamination is extremely detrimental to obtaining high quality data in TKD
- We can combine TKD with EDS in the SEM to characterize structure and chemistry

Summary and Conclusions

- If you can clean your chamber and your sample to prevent contamination, TKD at 30 kV is a nice compliment to TEM