

TriBeam System for Multimodal Data

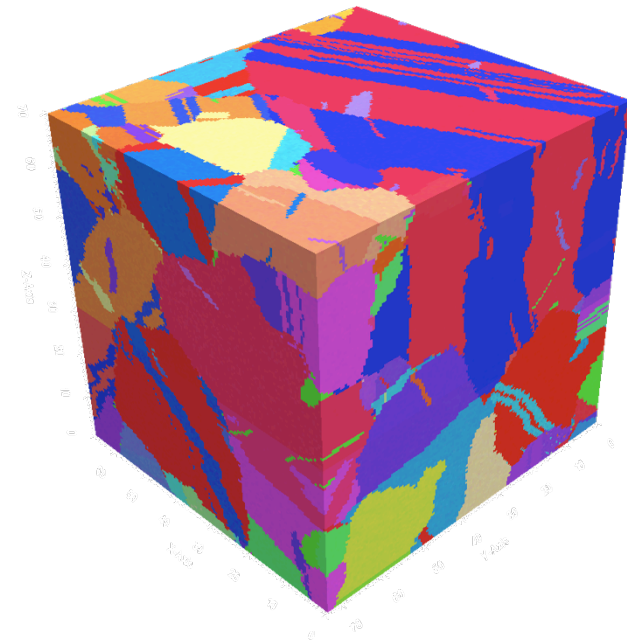
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² *School of Metallurgy and Materials, University of Birmingham, UK*

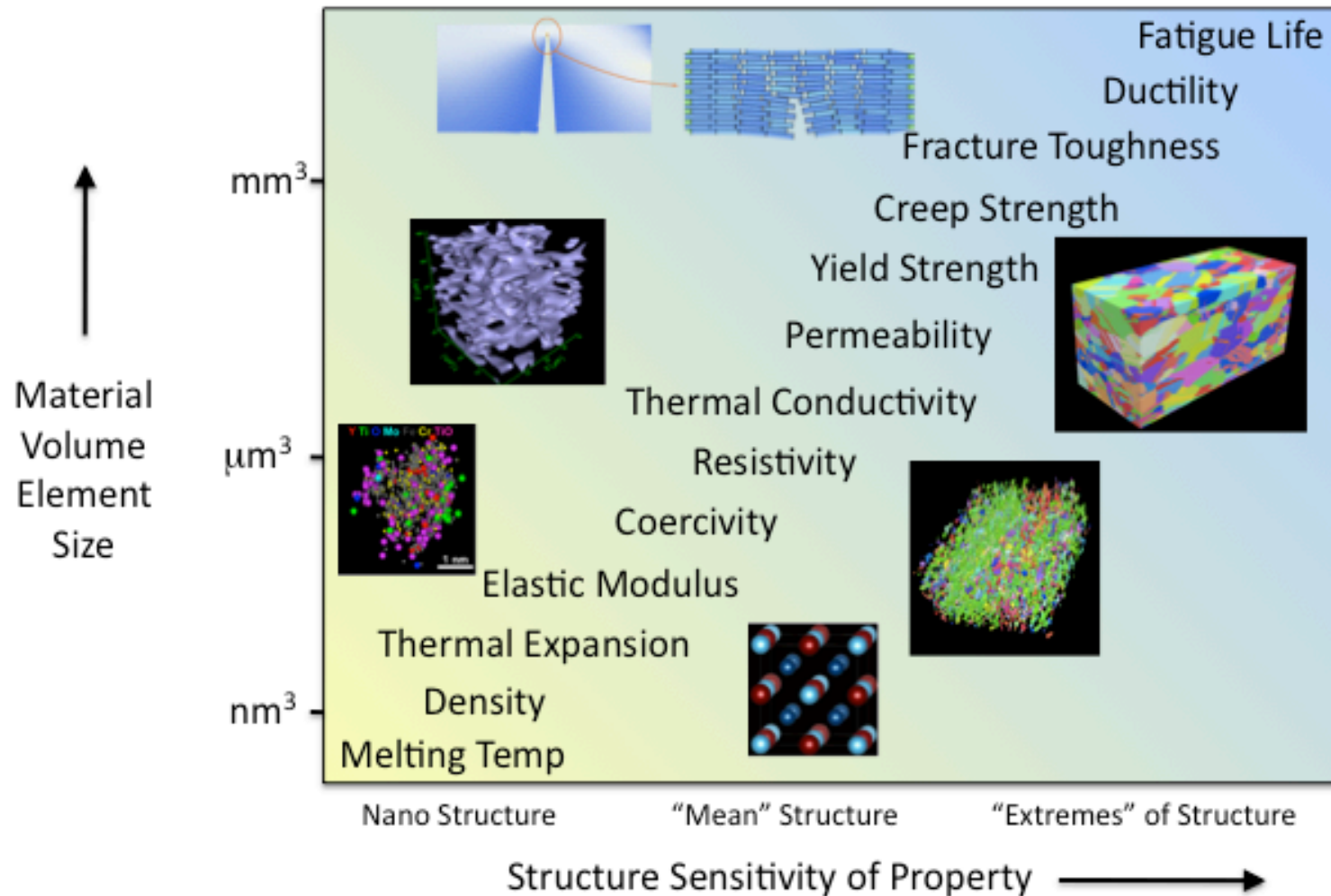
³ *Carnegie Mellon University, Pittsburgh, PA*

⁴ *Karlsruhe Institute of Technology, Karlsruhe, Germany*



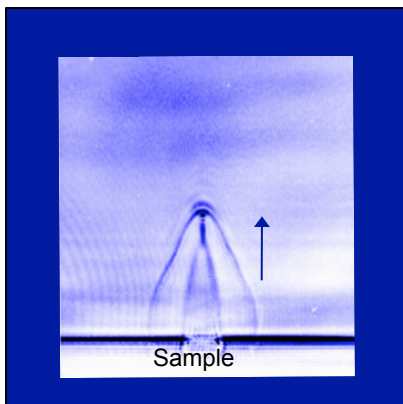
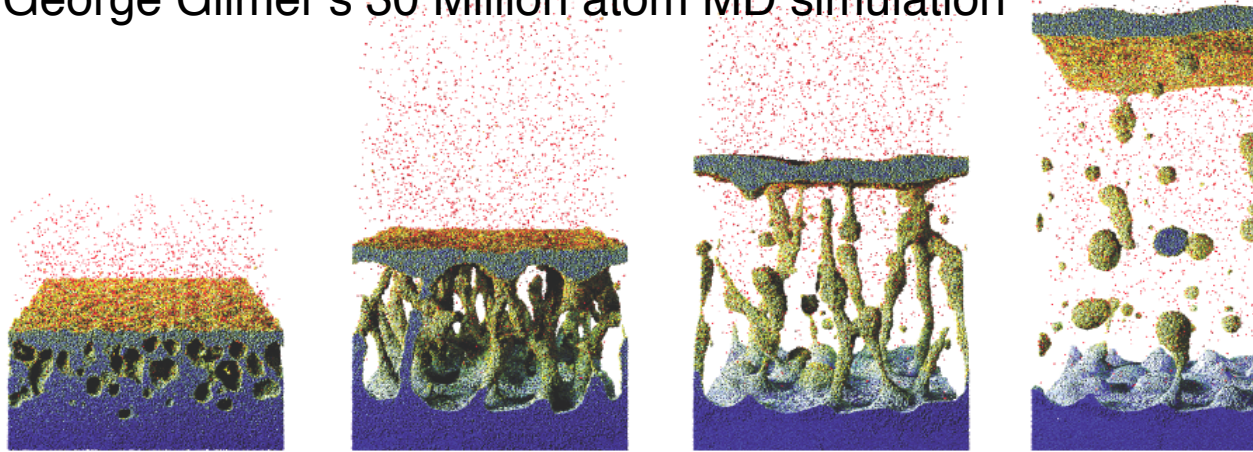
Acknowledgements:

M. Straw, M. Groeber (AFRL), M. Jackson (BlueQuartz), J.P. McDonald (Dow), S.M. Yalisove (UM), Pollock Group, GE, FEI Company



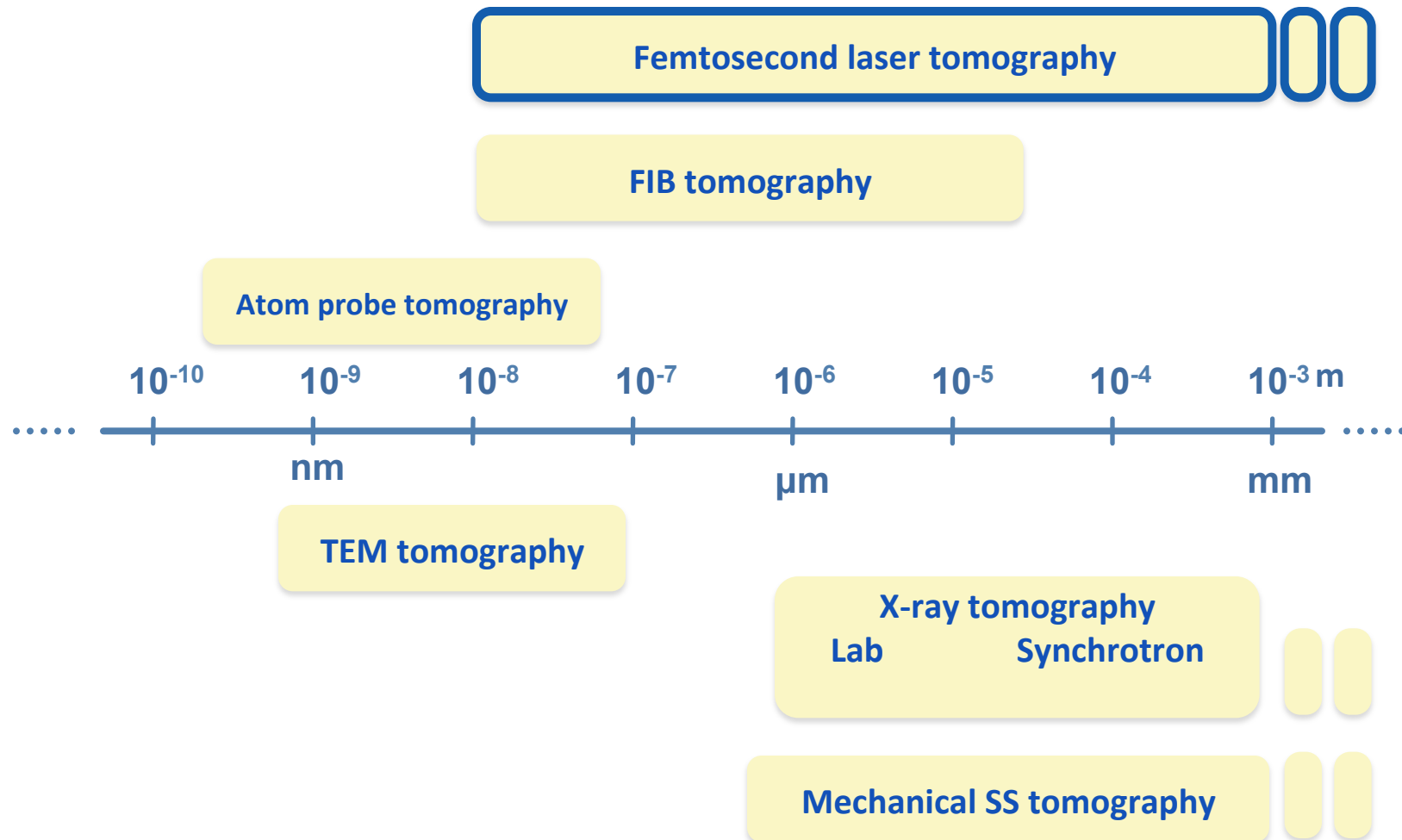
We *need* more volume(s)!

George Gilmer's 30 Million atom MD simulation



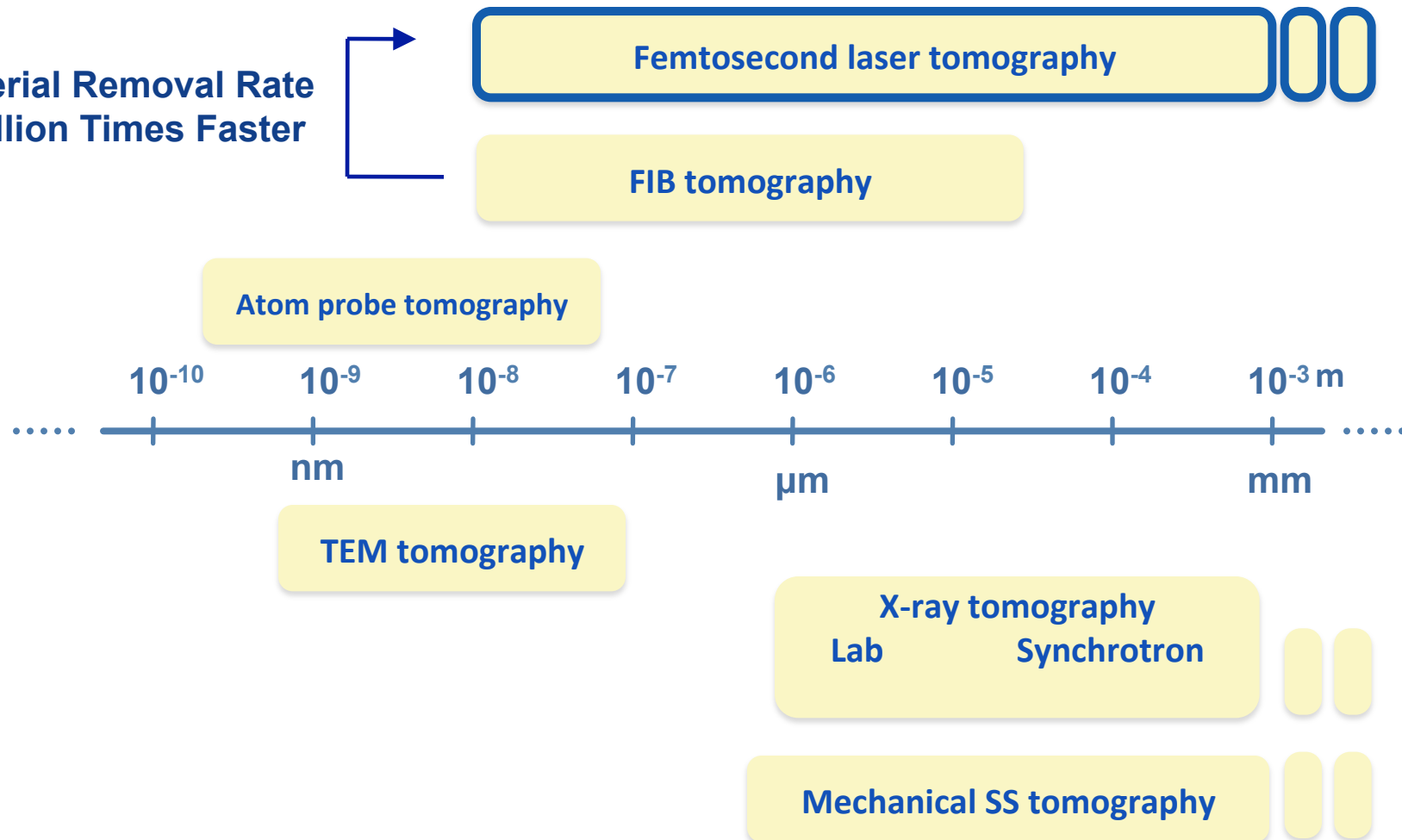
- 150fs laser pulse; ablation occurs much later (ns)
- Ablated layer 30 – 50nm in thickness, kHz repetition
- Collateral damage very limited (no melting)
- Same process occurs in wide range of materials
(metals, semiconductors, ceramics, polymers)
- Layer by layer ablation *ex-situ* in air or *in-situ* in vacuum

3-D Data Acquisition Techniques



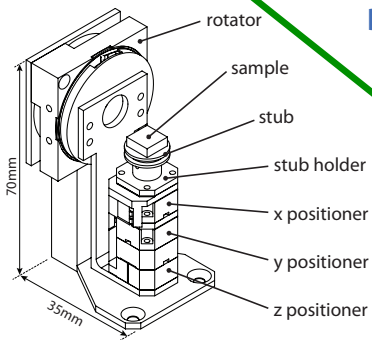
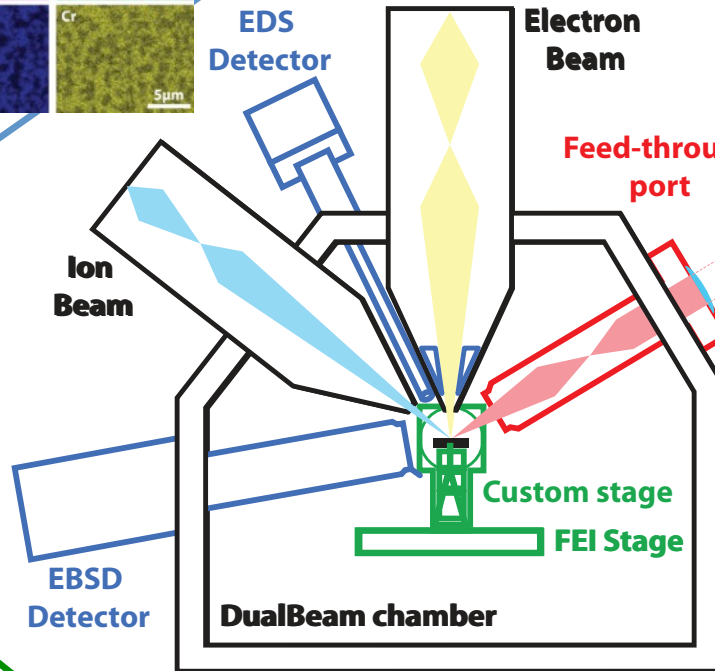
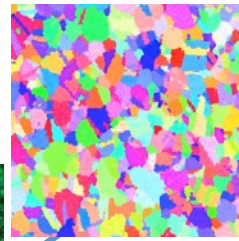
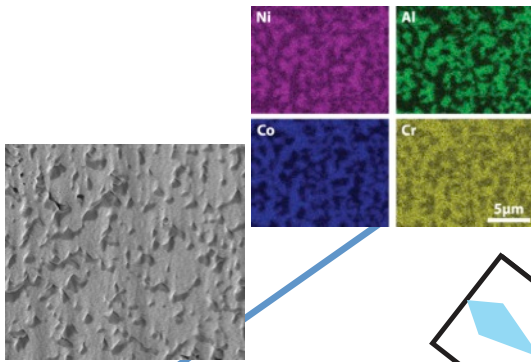
3-D Data Acquisition Techniques

Material Removal Rate
≈ Million Times Faster



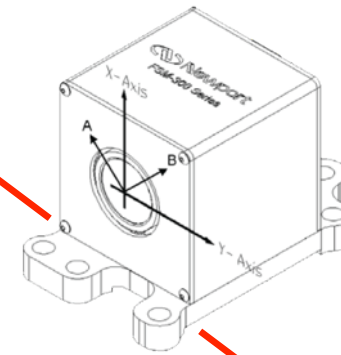
DETECTORS AND IMAGING

- SE, BSE, EDS, EBSD

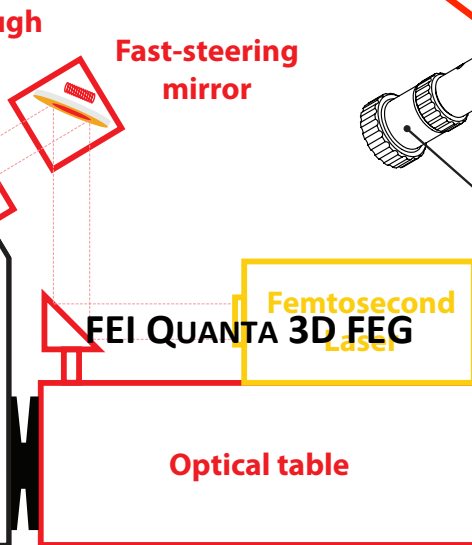
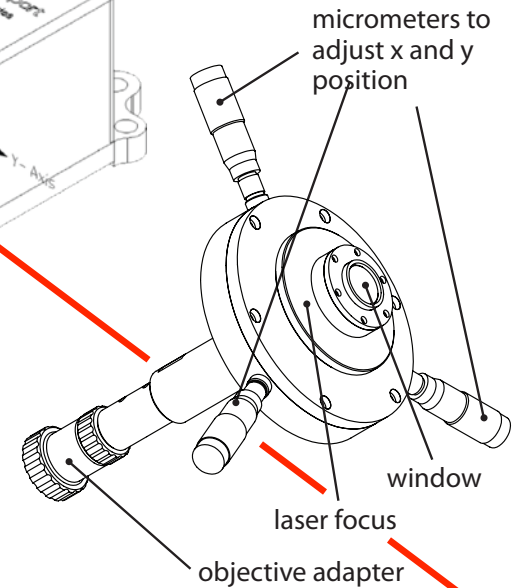


- Full eucentric rotation
- nm-resolution

SAMPLE POSITIONING



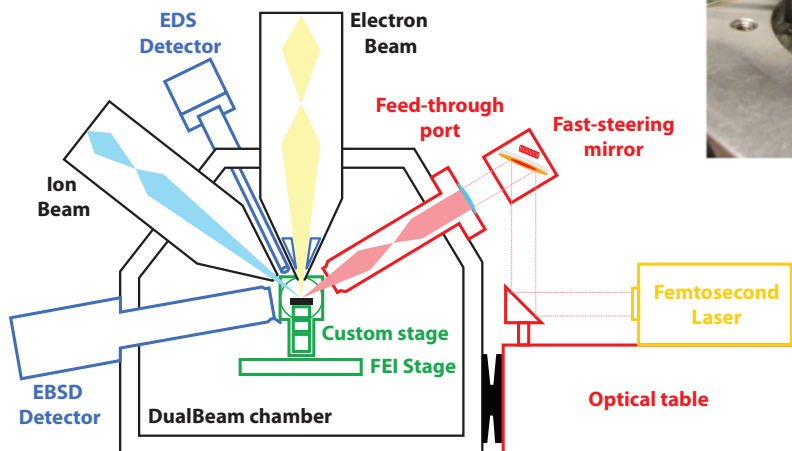
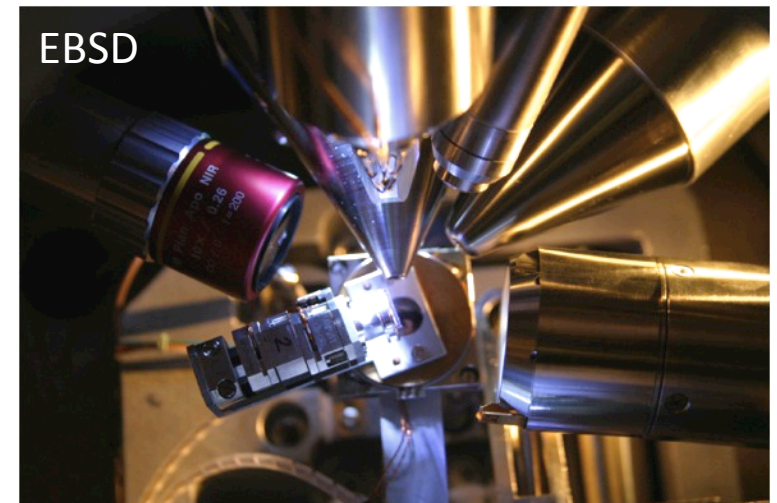
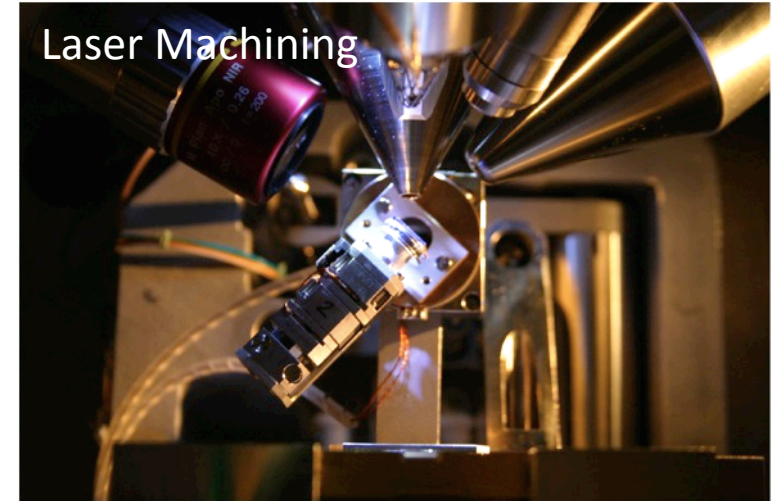
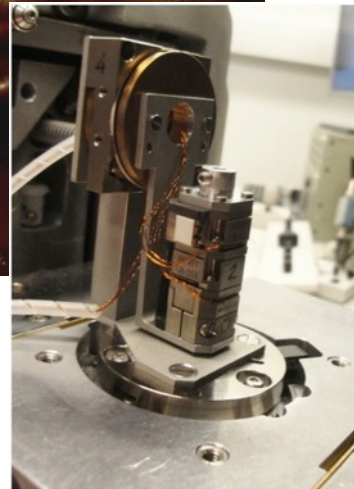
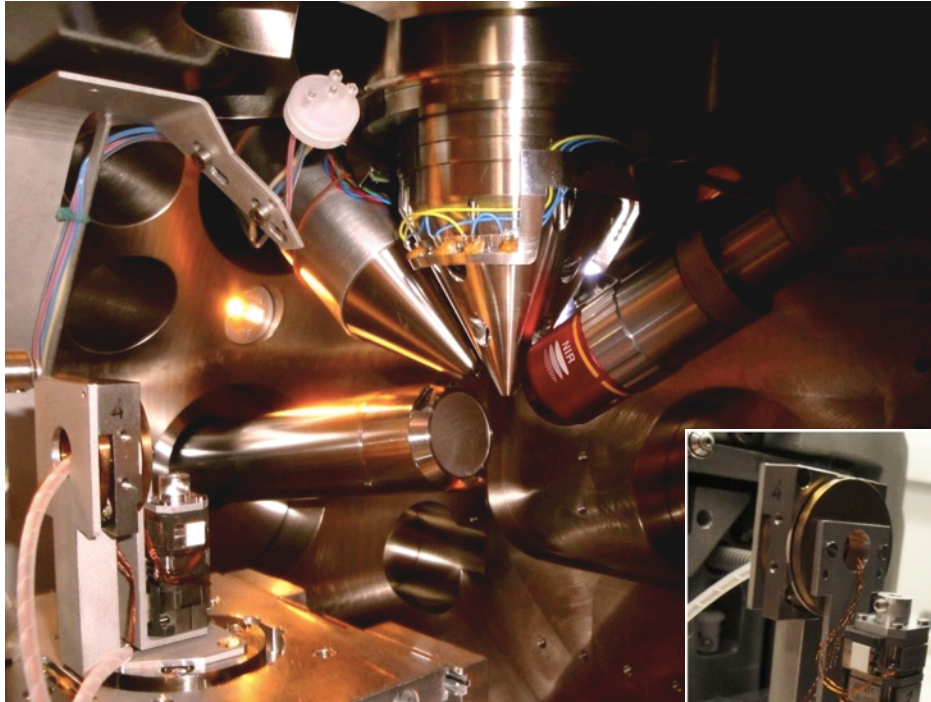
LASER DELIVERY



- 780 nm
- 150fs pulses
- 1kHz repetition rate
- Material removal with negligible heat affected zone
- 10^5 - 10^6 faster at removing material compared to ion beam

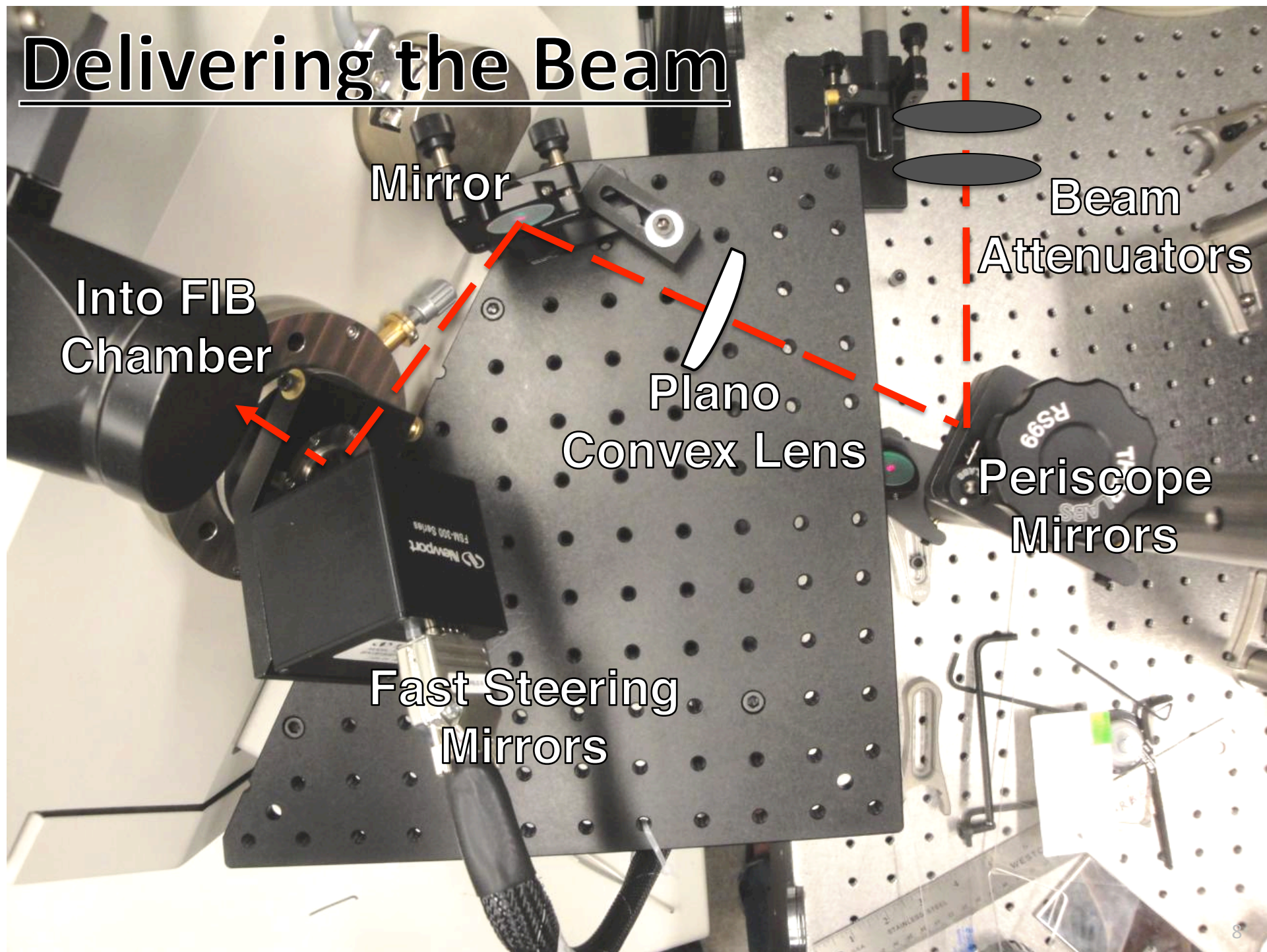
FEMTOSECOND LASER

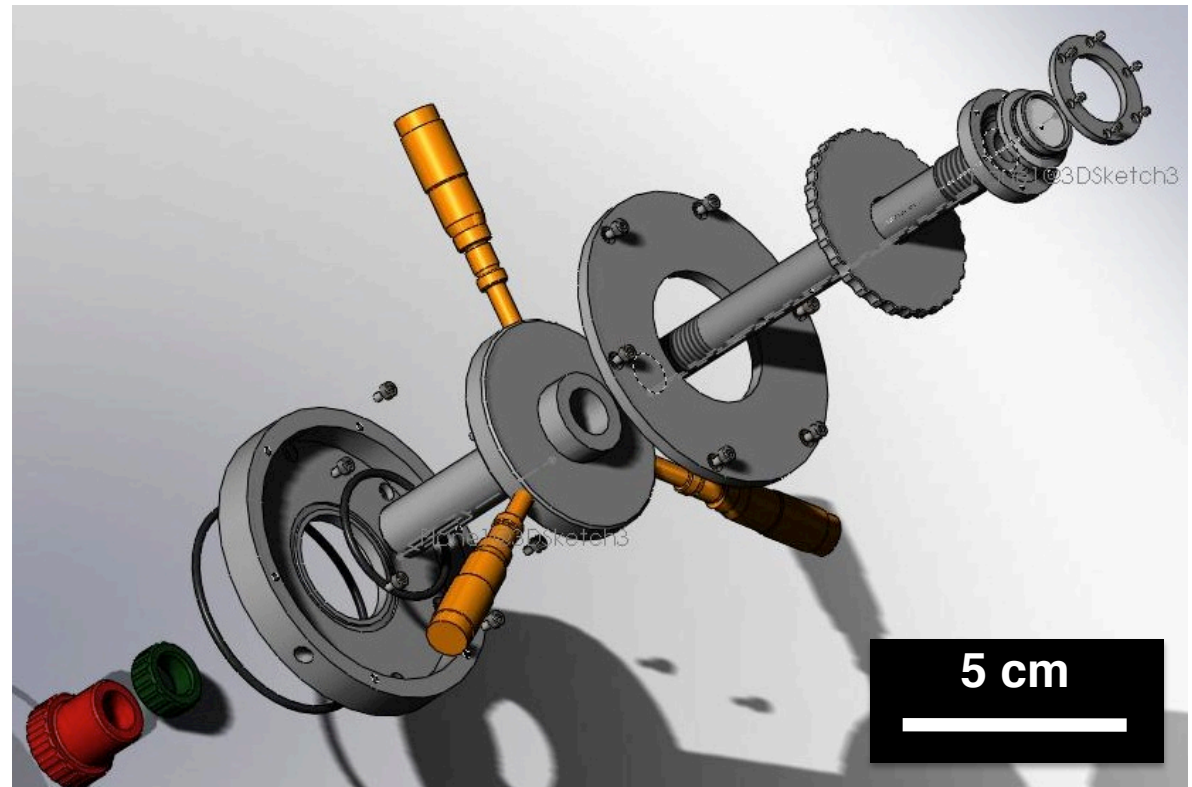
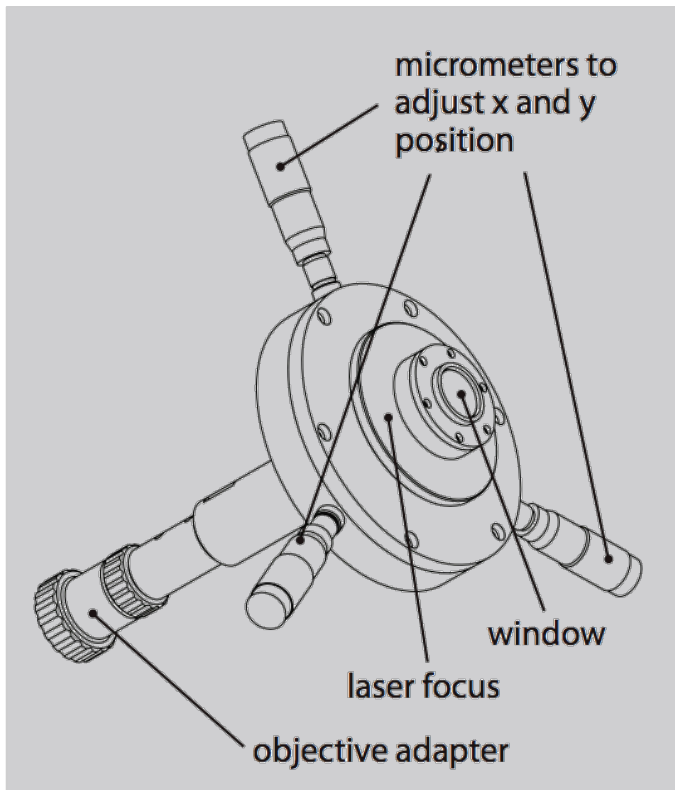
TriBeam System



M. Echlin, A. Mottura, C. Torbet, T.M. Pollock. *RSI* (2012)

Delivering the Beam

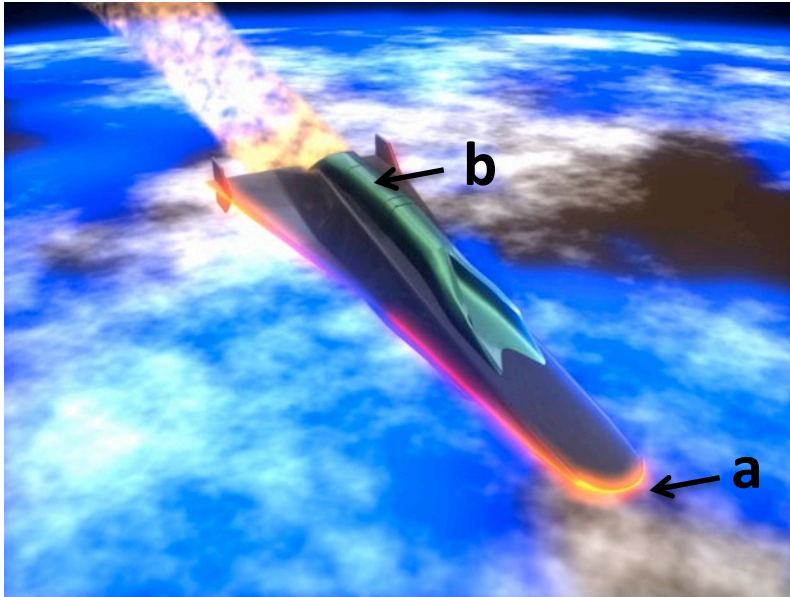




- Micrometer adjustments for X and Y positioning
 - Target laser to eucentric position
- Microscope objective position can be changed
 - Focus laser beam to eucentric position
- Adjustments can be made in vacuum

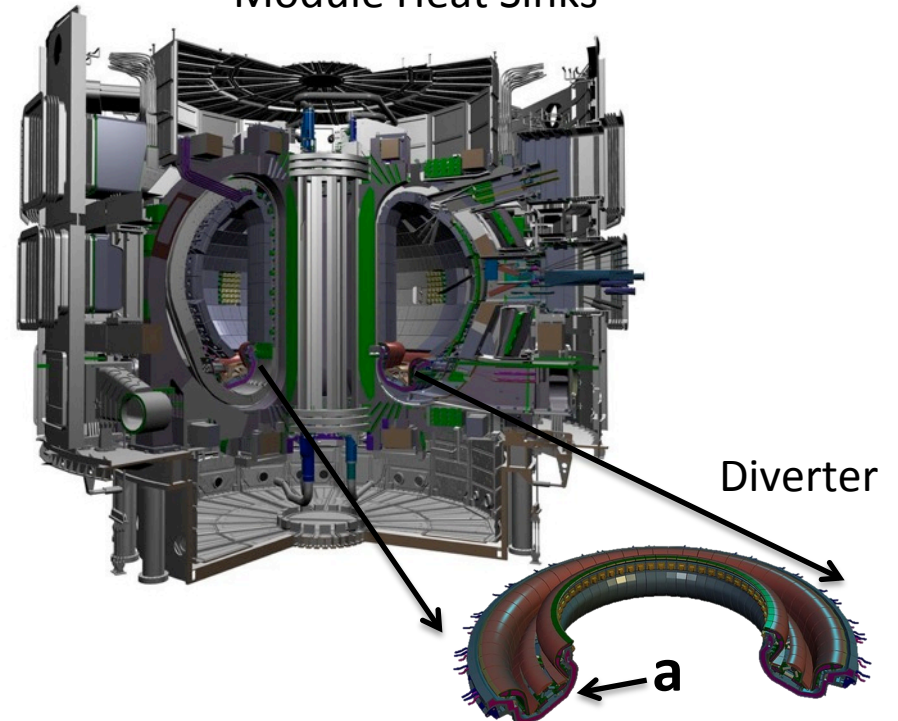
W-Cu Applications

Aerospace – Mach 5+ Hypersonic Vehicles

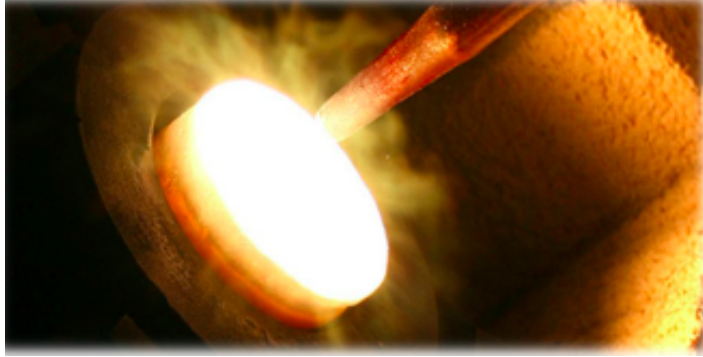


- a) Leading edge - Ablative structural material. 2700°C at leading edge.
- b) Combustor – thermal management via ablative cooling. 3000°C inside combustor

Fusion – Power Generation ITER Tokamak Blanket Module Heat Sinks

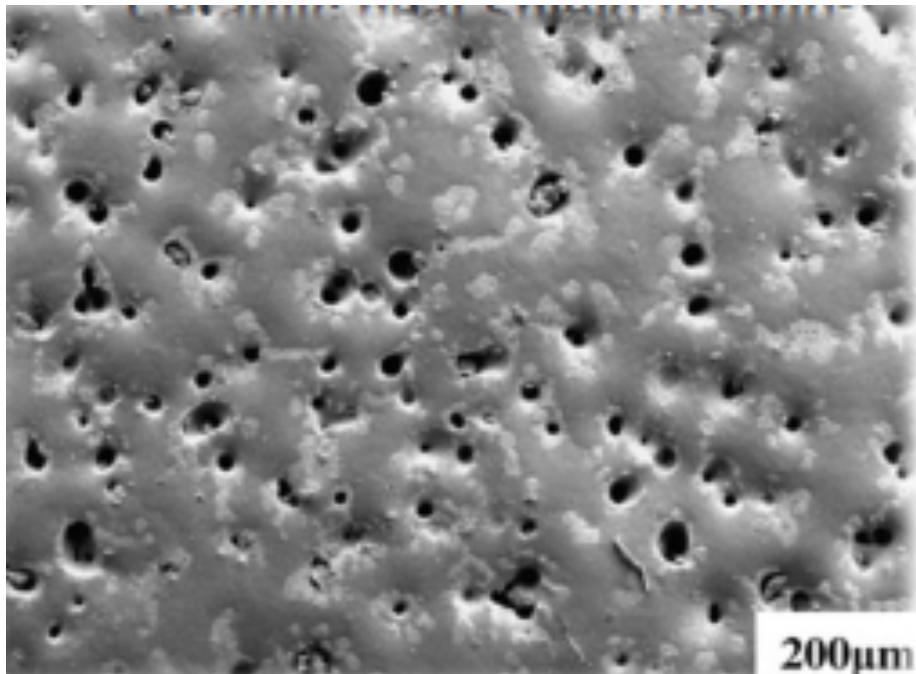


- a) Thermal management material – heat sinking below plasma facing material or use under pulsed fusion events, extracts heat withstand up to 3000°C over a 20 year lifetime.

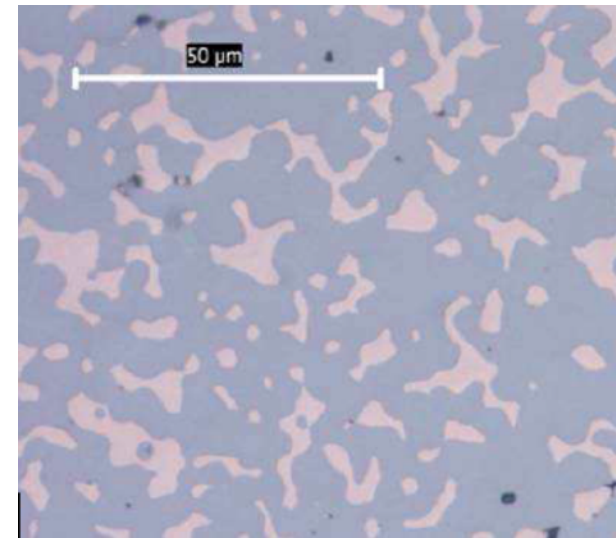


Interpenetrating W – Cu Composites

- Ablation of Cu phase provides cooling under high heat fluxes
- CuW used for thrust vectoring, experience extremely high temperatures ($>2000^{\circ}\text{C}$)
- Want to Measure Permeability, S_v , V_f directly in 3D



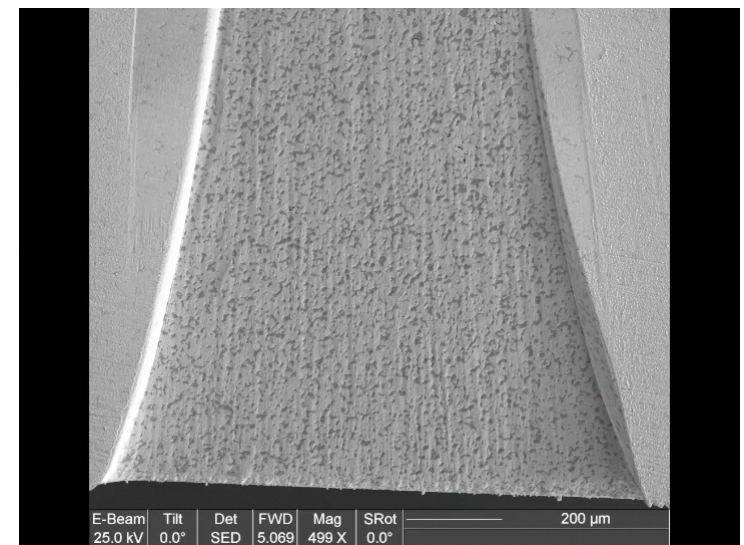
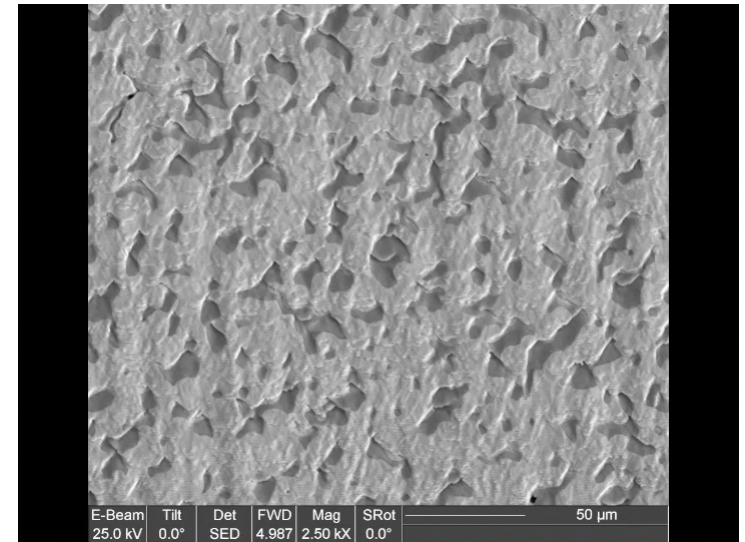
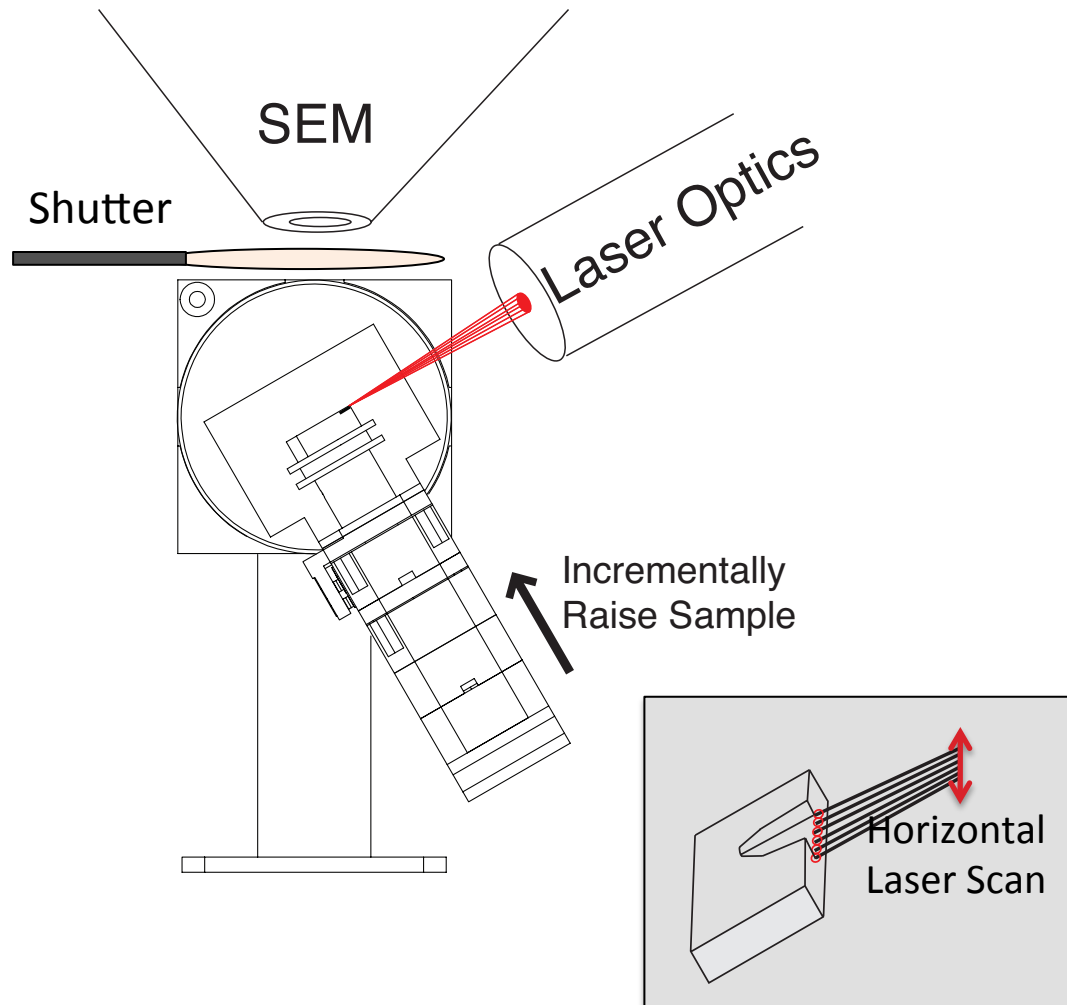
Following High Heat Flux Ablation



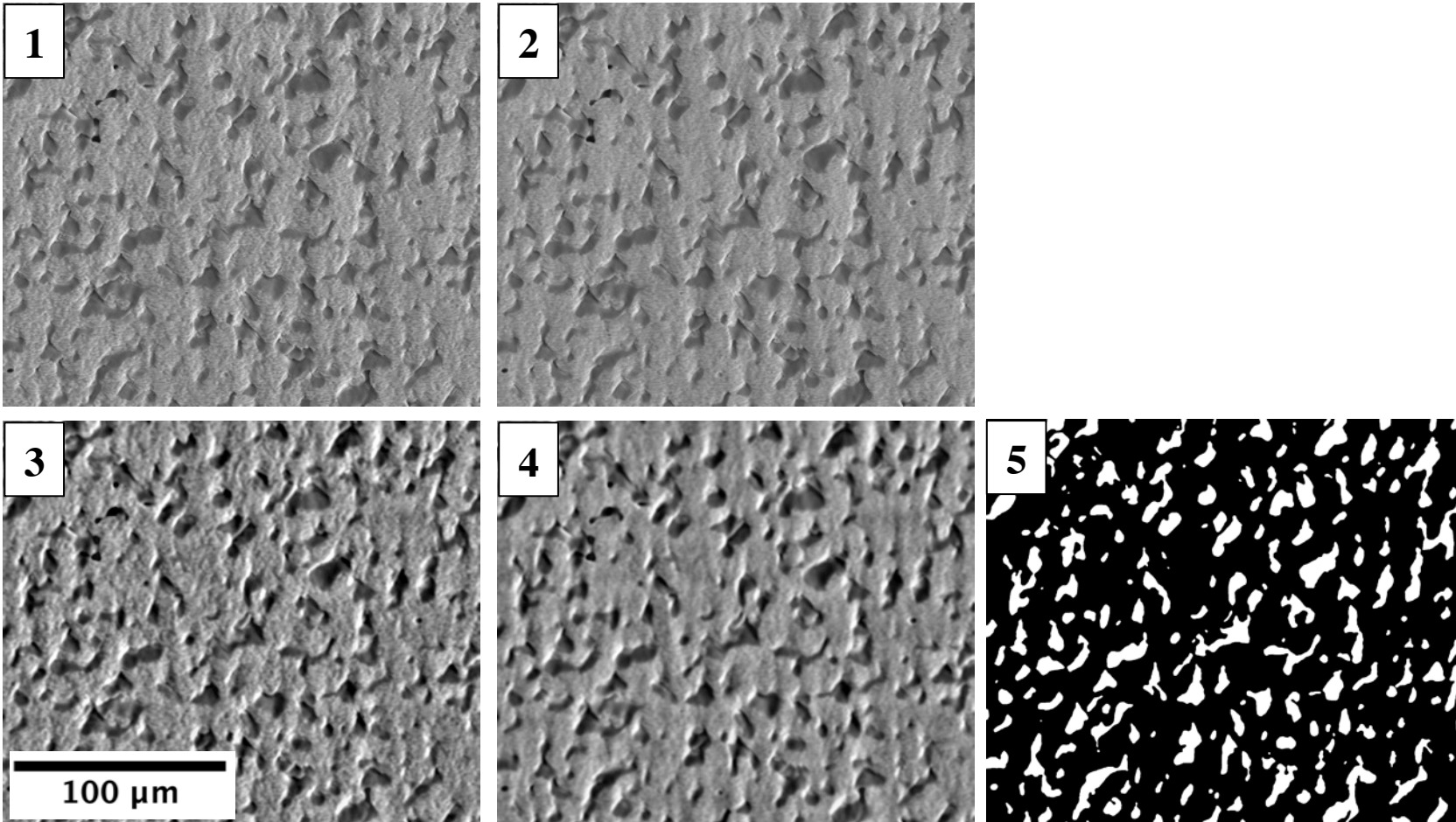
W-Cu Composite

Schematic of Laser Ablation

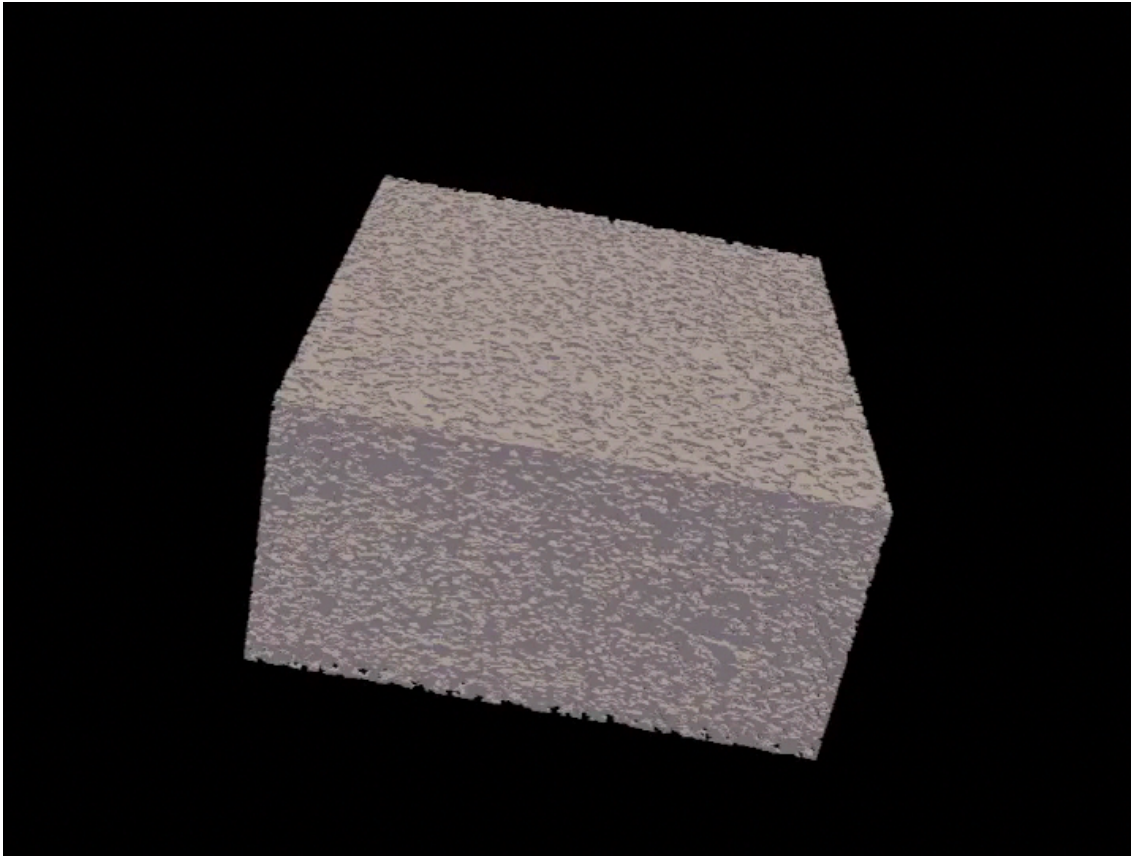
The convergence of research and innovation.



Machining time per slice = 25 s
3-D dataset acquisition time = 48 h.

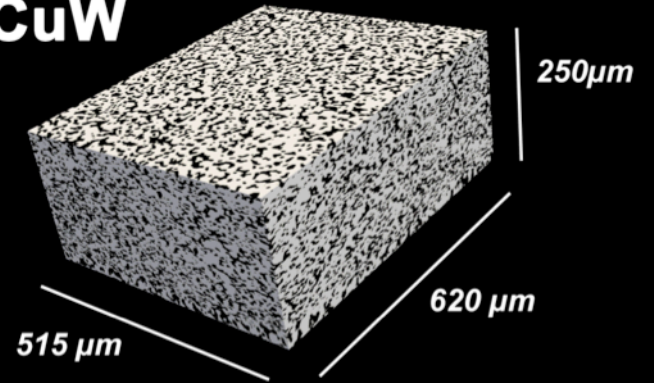


- (1) Raw secondary electron image as captured by the TriBeam
- (2) 3D median filter
- (3) FFT bandpass filters for horiz. high frequency LIPSS and vert. low frequency curtaining
- (4) 3D Gaussian filtering
- (5) Batch EM-MPM segmentation using initialized areas

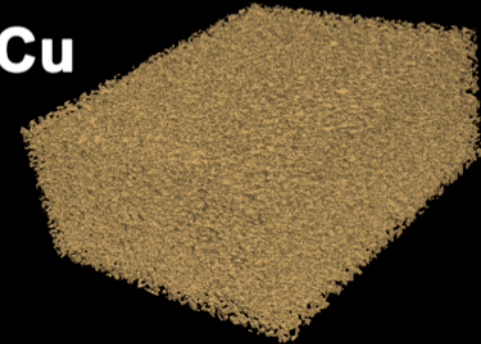


- Cu volume fraction measured 28 vol%
- Nominal Cu composition is 15 wt% (27.5 vol%)
- 1000 SE images collected
- Interconnected Cu phase
- 2D Dilation / Erosion shows potential segmentation error of less than +/-4%

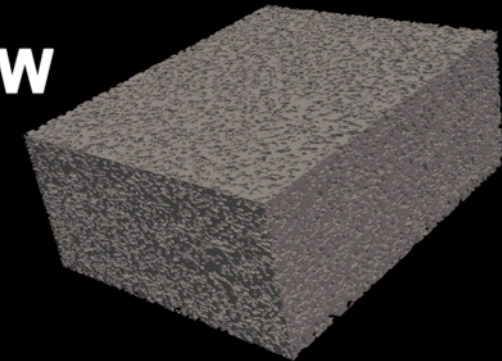
CuW



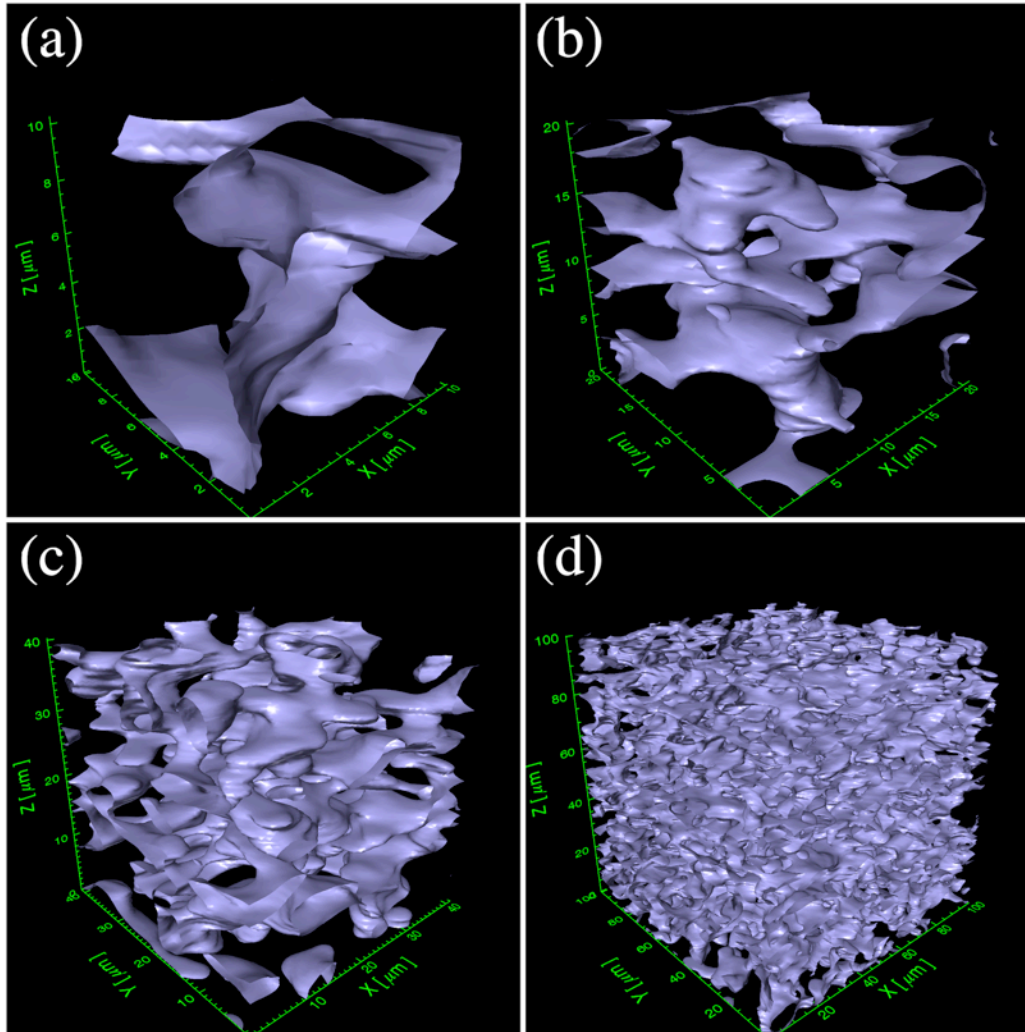
Cu



W

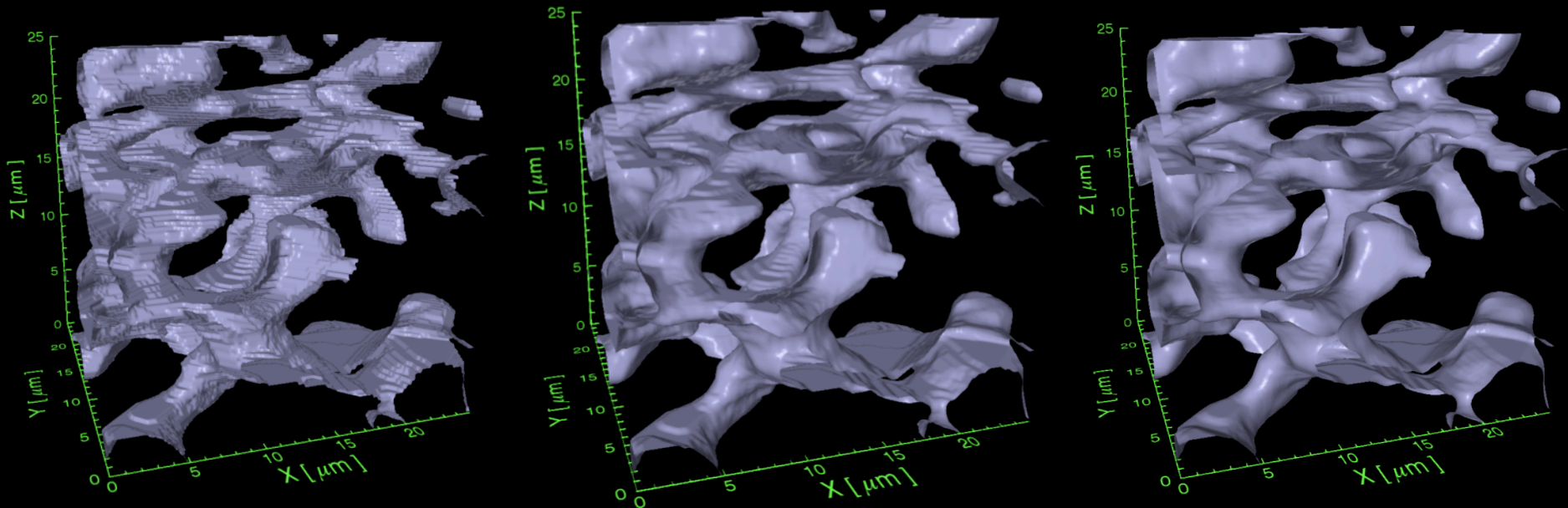


Example Volume Element Samplings



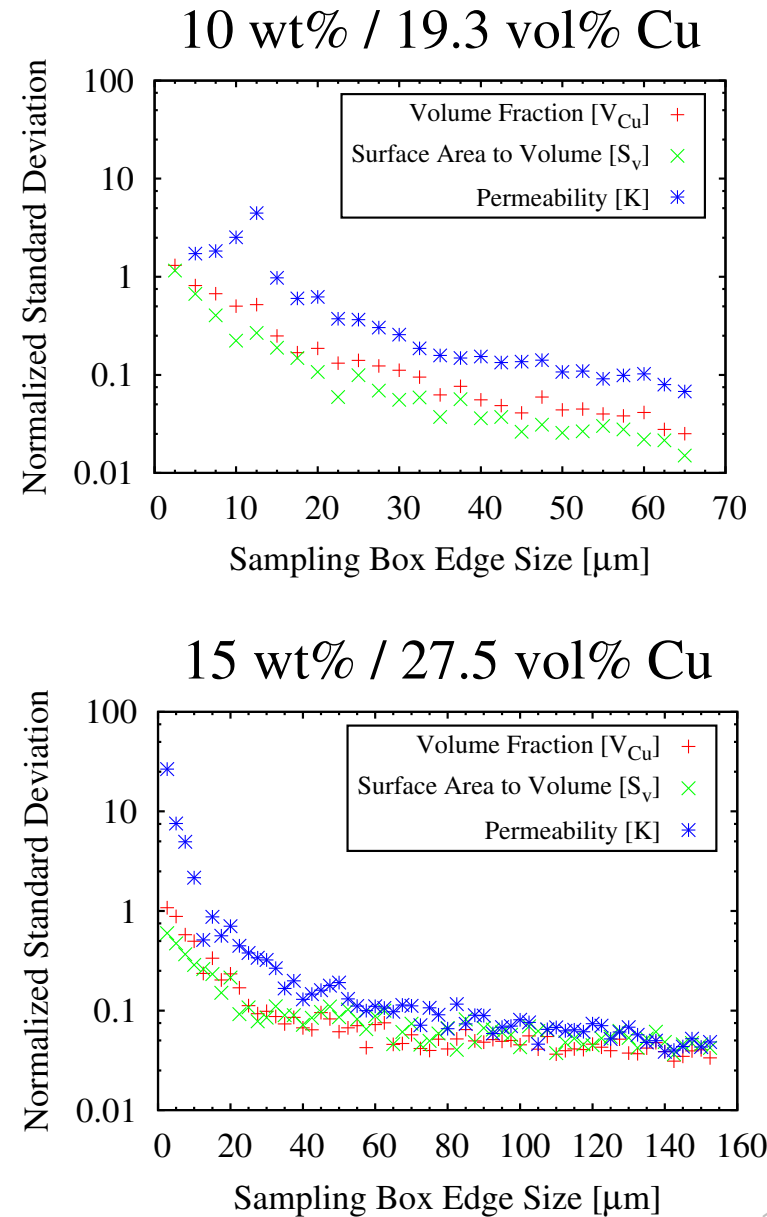
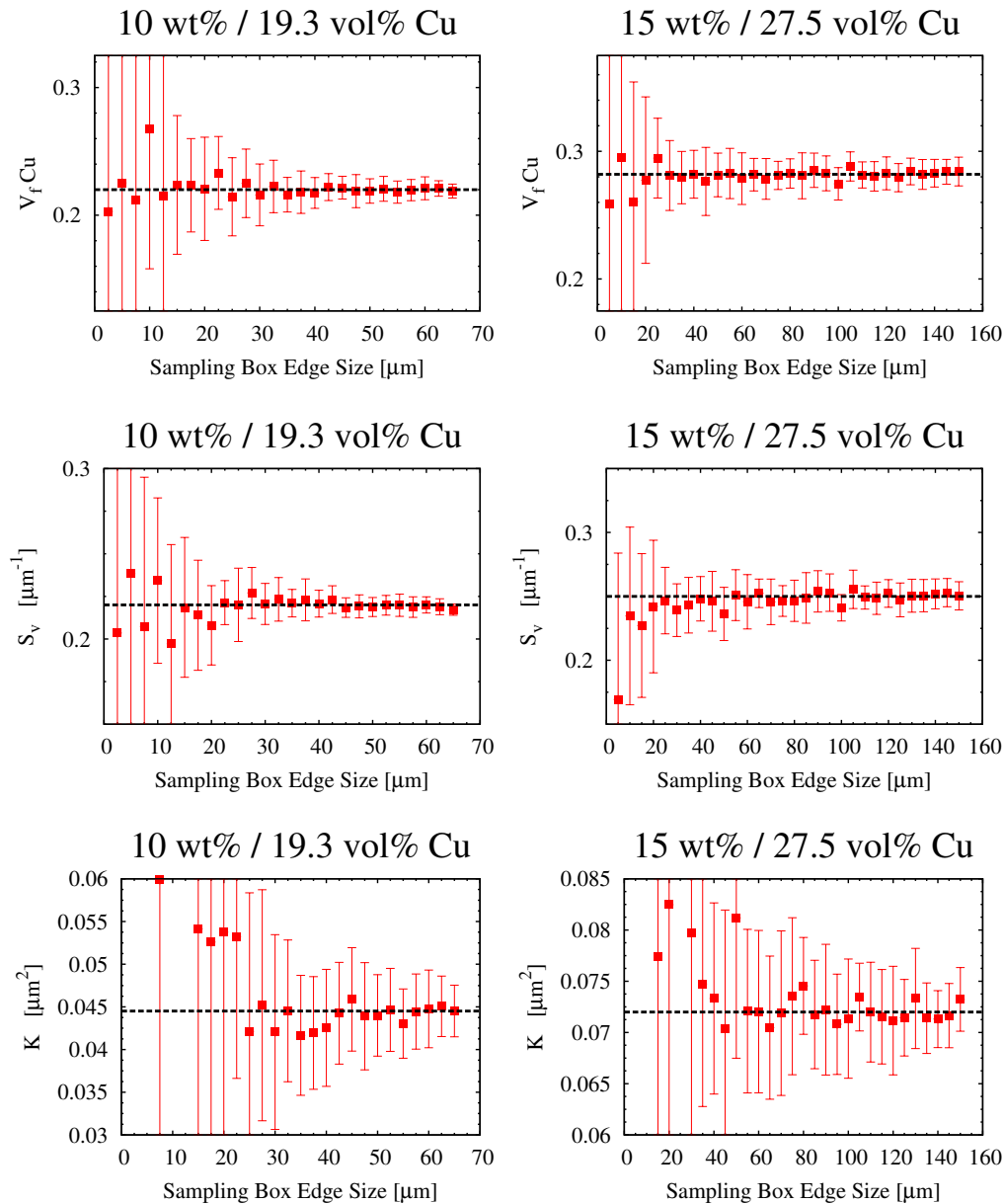
Sampling Methodology

- Random volumes selected from with edge sizes ranging from $10\mu\text{m}$ - $160\mu\text{m}$
- V_f , S_v measured for each sample
- K calculated from V_f , S_v using the Kozeny-Carmen model
- Compute variability in V_f , S_v , K to determine MVE size and PVE dependency
- Select desired bounds on K to set the PVE size which in turn sets the MVE size



- S_v has dependence on smoothing
- Convergence is not affected by smoothing
- Small regions of connectivity unperturbed

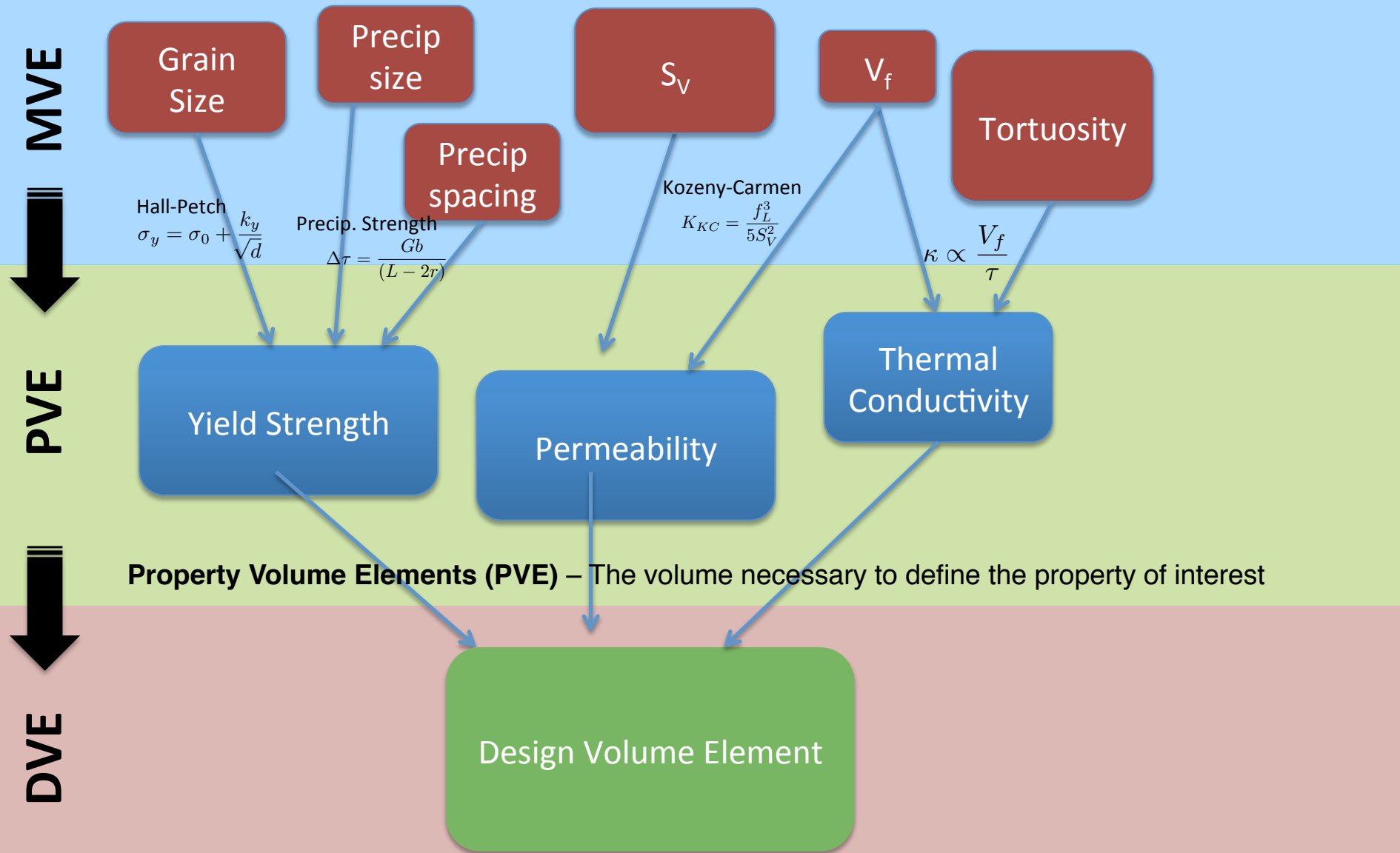
MVE Sampling for Property Convergence



Representative Volumes

The convergence of research and innovation.

Microstructural Volume Elements (MVE) – The volume necessary to define the microstructure of interest



Property Volume Elements (PVE) – The volume necessary to define the property of interest

Design Volume Element (DVE) – Volume necessary for convergence of all relevant PVEs

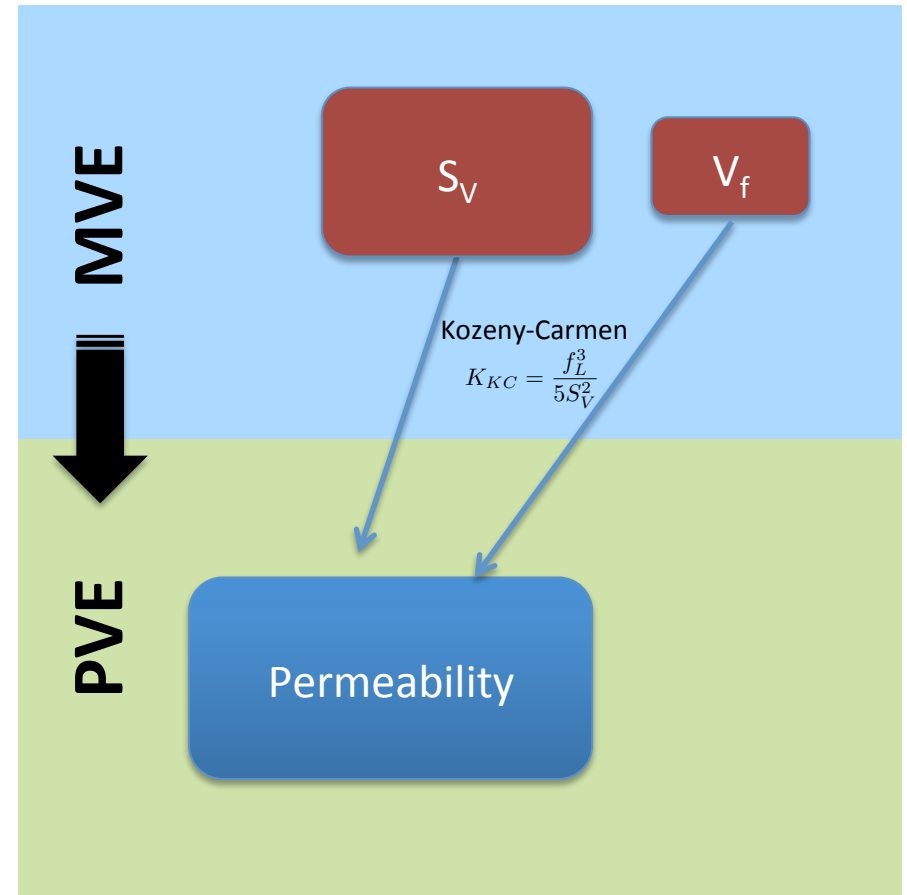
Fluid Flow (Darcy's law):

$$Q = \frac{-KA}{\mu} \frac{P_b - P_a}{L}$$

$$q = \frac{-K}{\mu} \nabla P$$

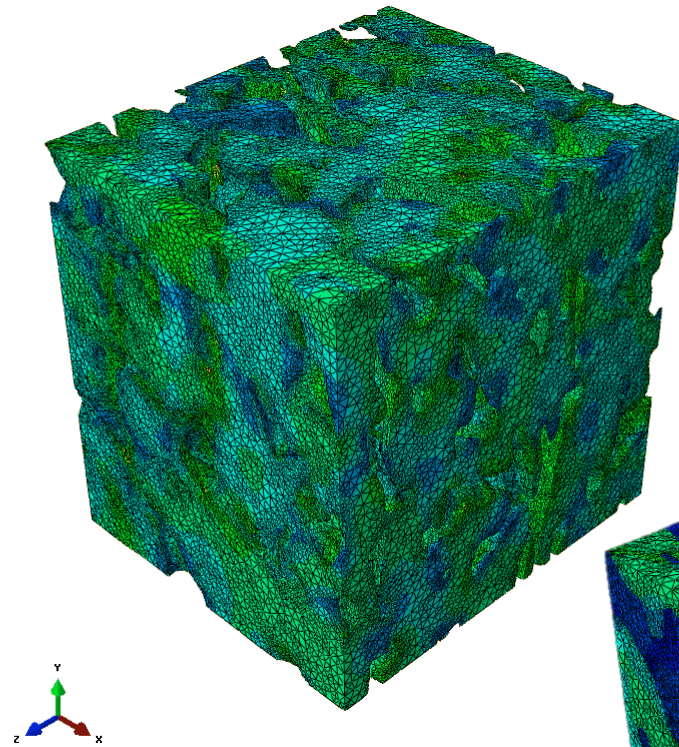
Permeability Calculation (Kozeny-Carmen):

$$K_{KC} = \frac{f_L^3}{5S_{GV}^2(1 - f_L)^2} = \frac{f_L^3}{5S_V^2}$$

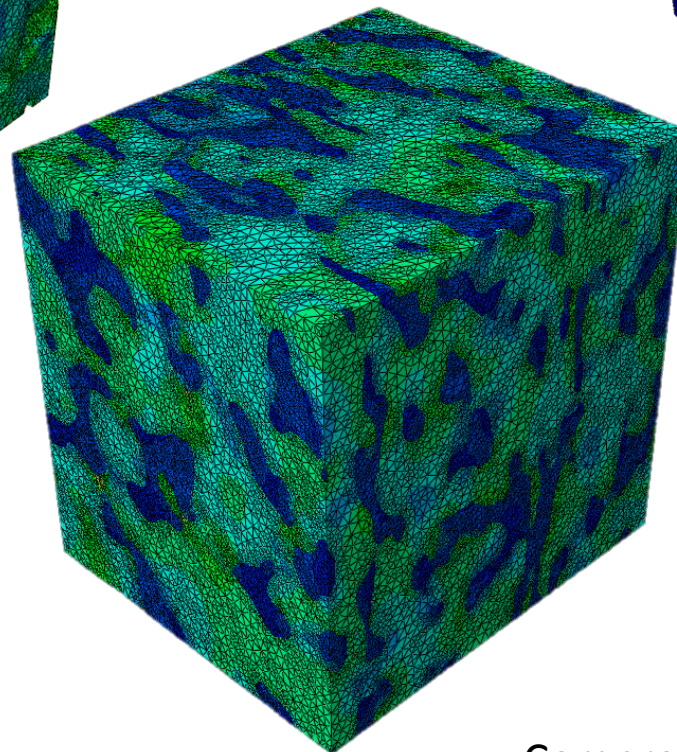
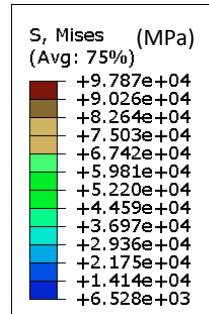


The convergence of research and innovation.

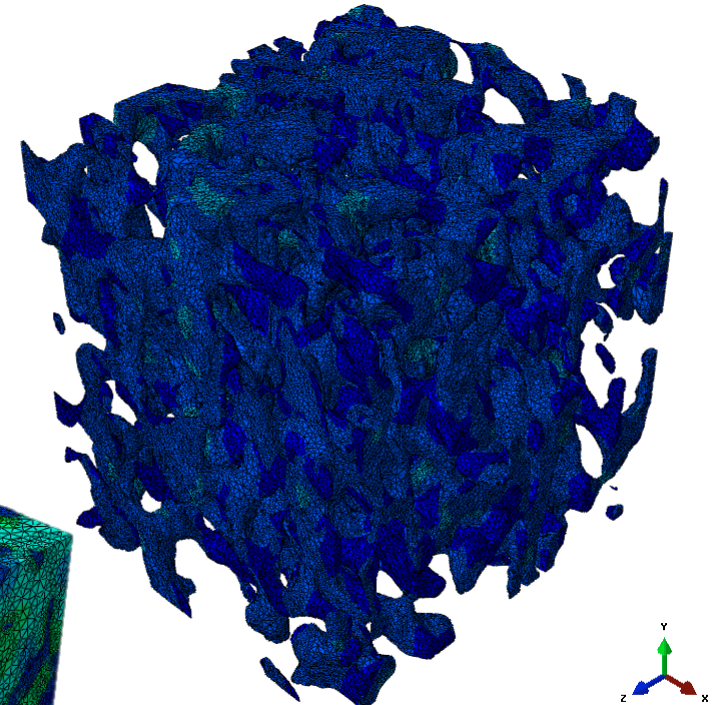
Mises stresses in the W Matrix



Most of compressive load carried in W matrix due to higher stresses in stiffer material.



Mises stresses in the Cu

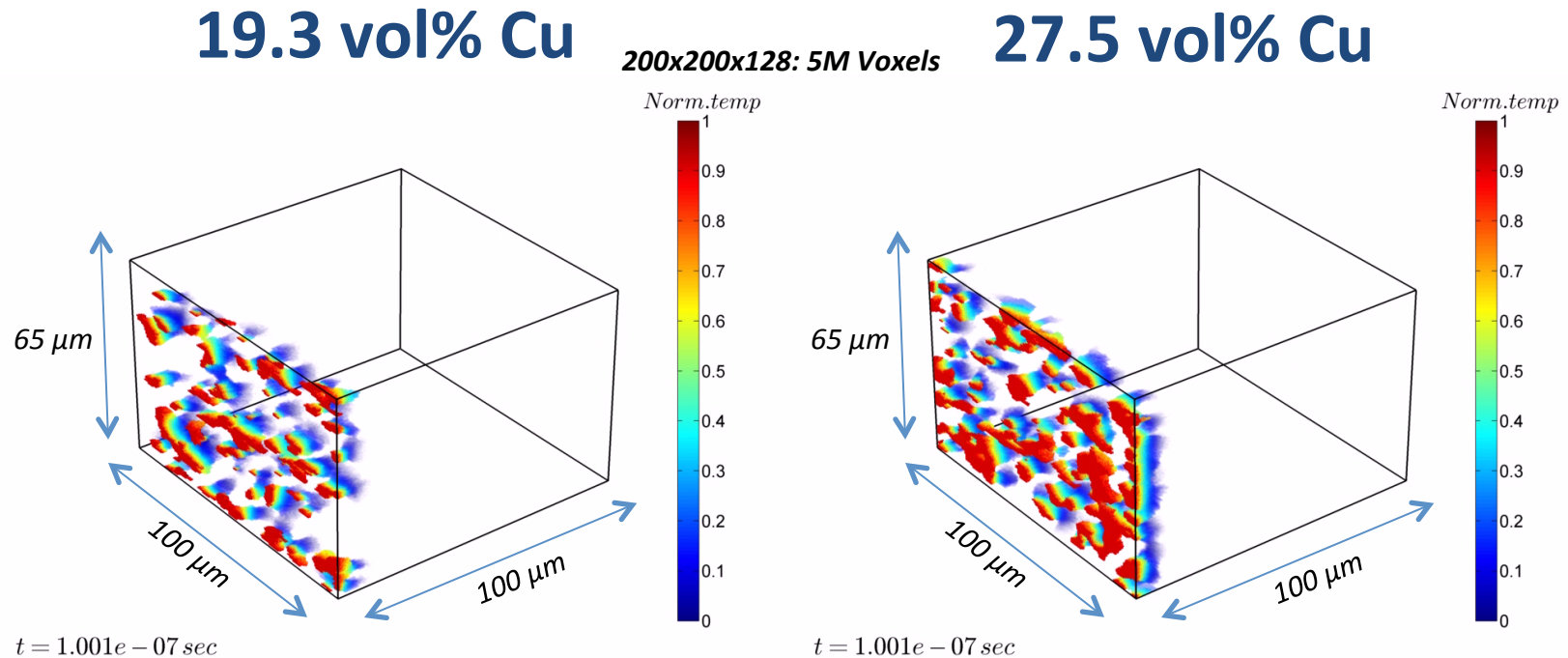


Significant strength reduction expected past the melting point of copper

WCu 15wt% Cu
60um x 60um x 60um

Compressed in X direction to 10% strain

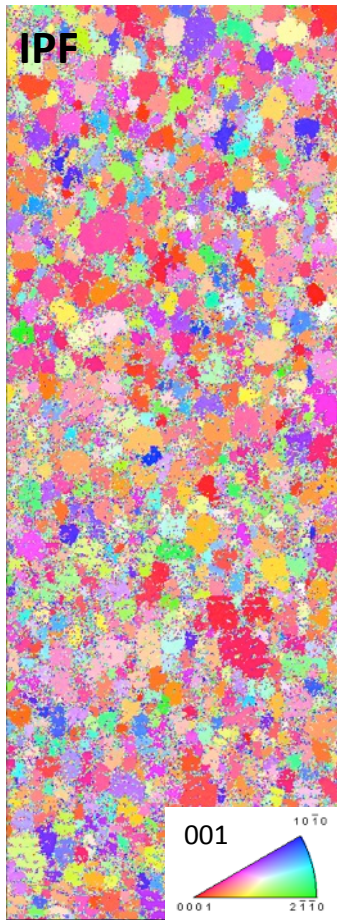
Meshless Method: Lattice Monte Carlo



- Modeled thermal diffusion using random walk of heat particles
- Segmented datasets (binary, ternary, ...) inherently suited for cubic/cuboid grid-based simulation methods
 - No meshing required → faster iterative workflow
- Novel GPU-based LMC code, **cudaLMC**, speeds up transient LMC conduction analysis by 160x over a single CPU core*

*Comparison between 3.0GHz Intel CPU vs cudaLMC v1.2 running on Tesla K20 GPU (2013), M. Wang et al (publication in progress)

Laser Ablated Mg Alloy - AXJ-530



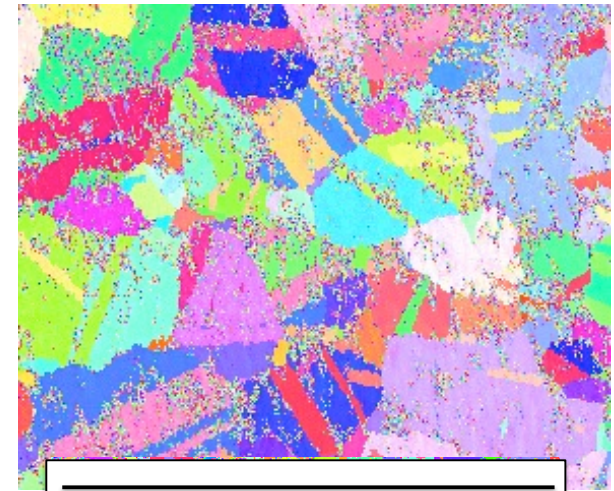
Laser Ablated Ni Alloy - Rene88DT



Laser Ablated Strontium Titanate

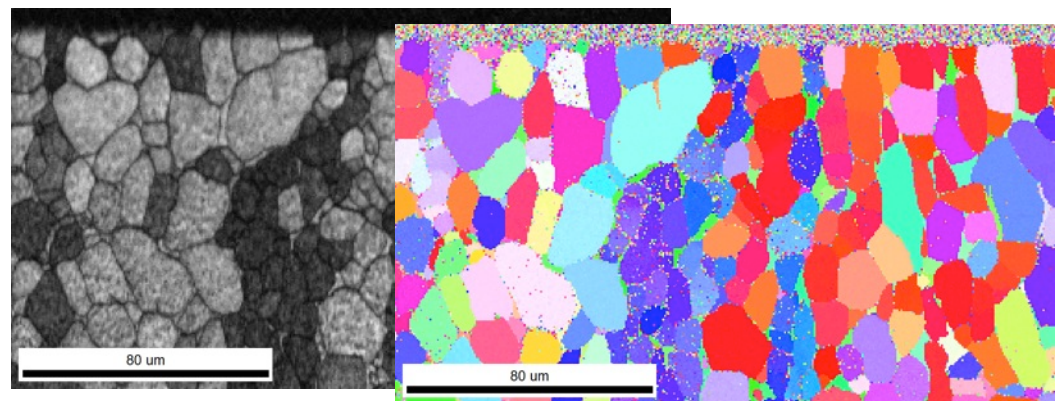
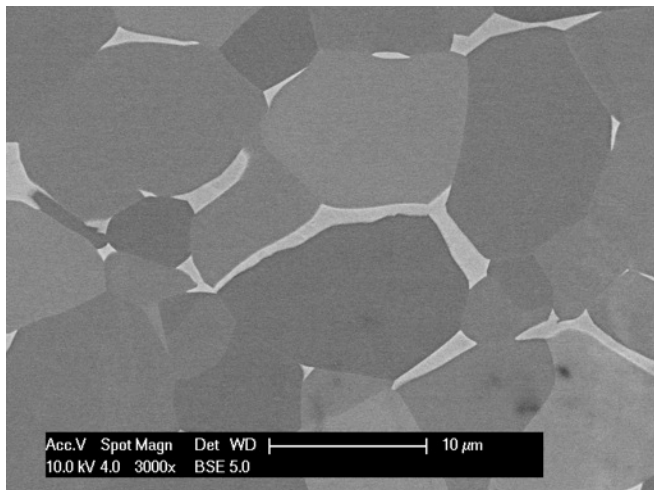
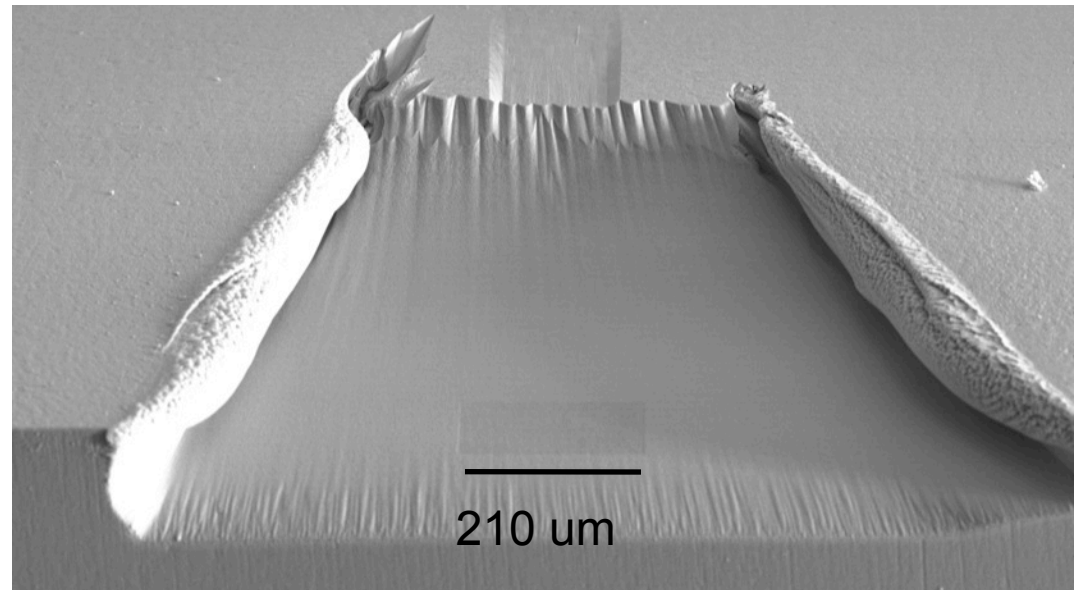
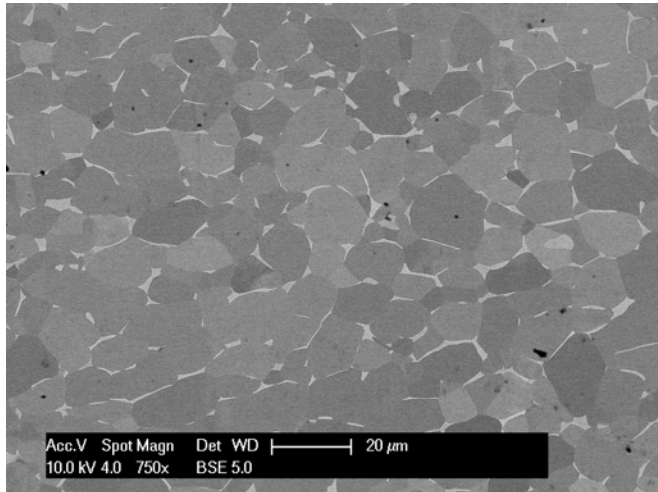


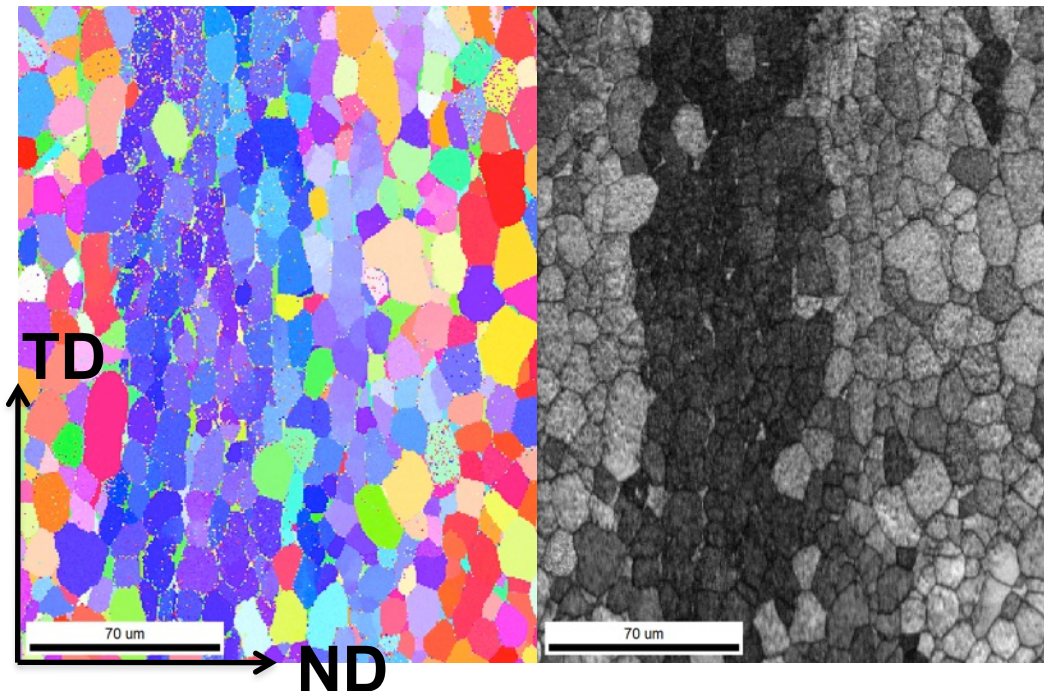
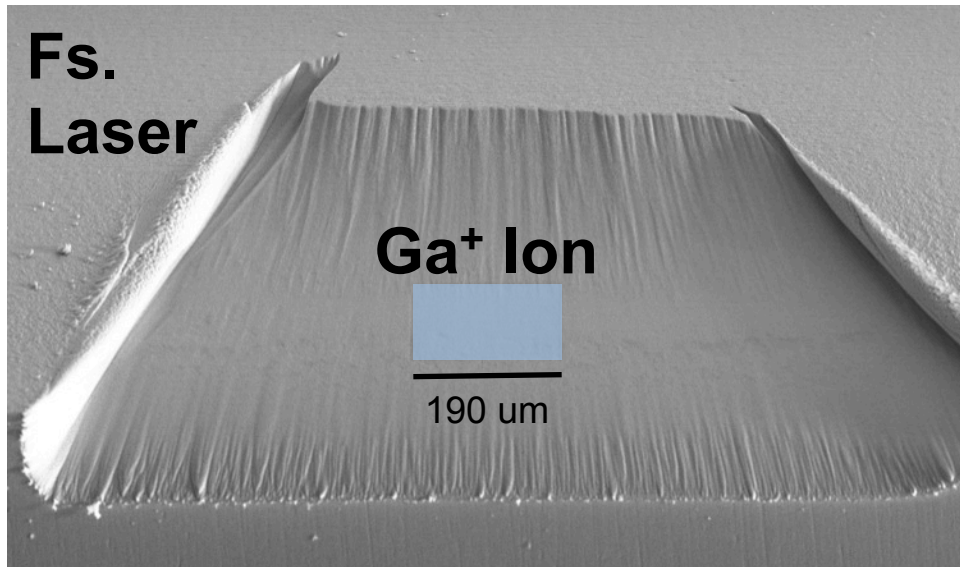
Laser Ablated Rene88DT



Laser Ablated + FIB Milled



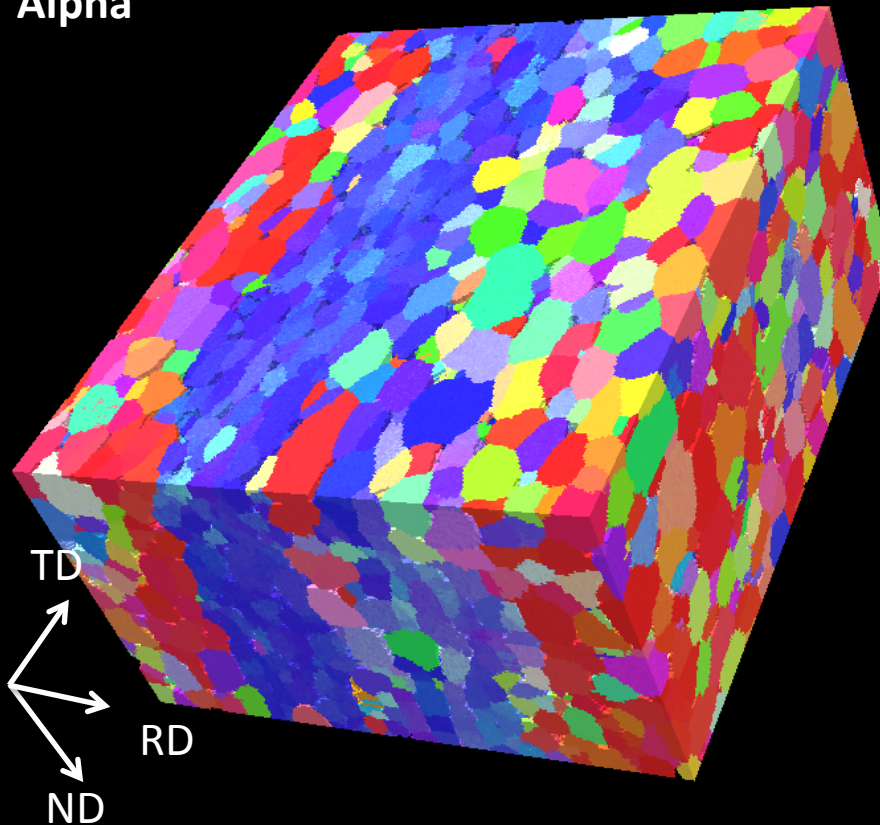




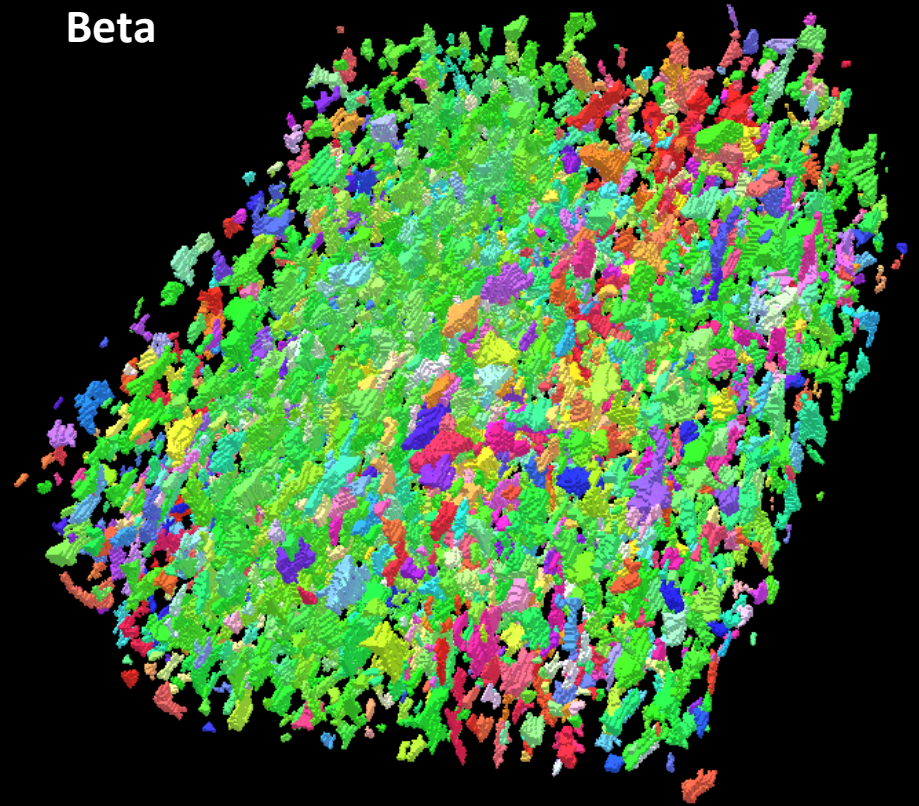
- Fs. laser ablated Ti64 surfaces do not produce high contrast diffraction patterns
- $<10^\circ$ surface parallel 20 nA ion cleaning was performed (5-10 minutes)
- Diffraction pattern contrast dependent on ion polishing angle (Ga⁺ damage)
- Diffraction patterns are collected from ion polished surface
- Microtextured region of alpha grains was collected
- Beta grain are indexed

Dataset size: 190 x 250 x 108 μm

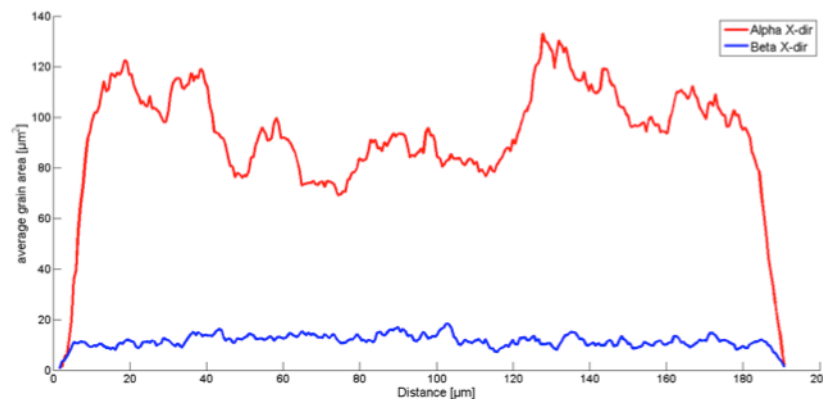
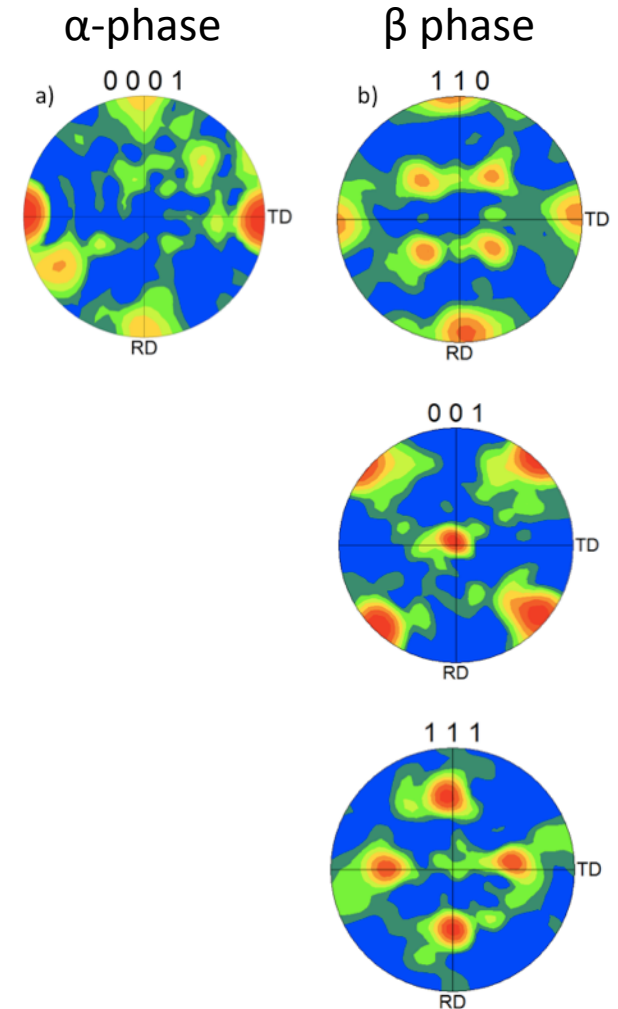
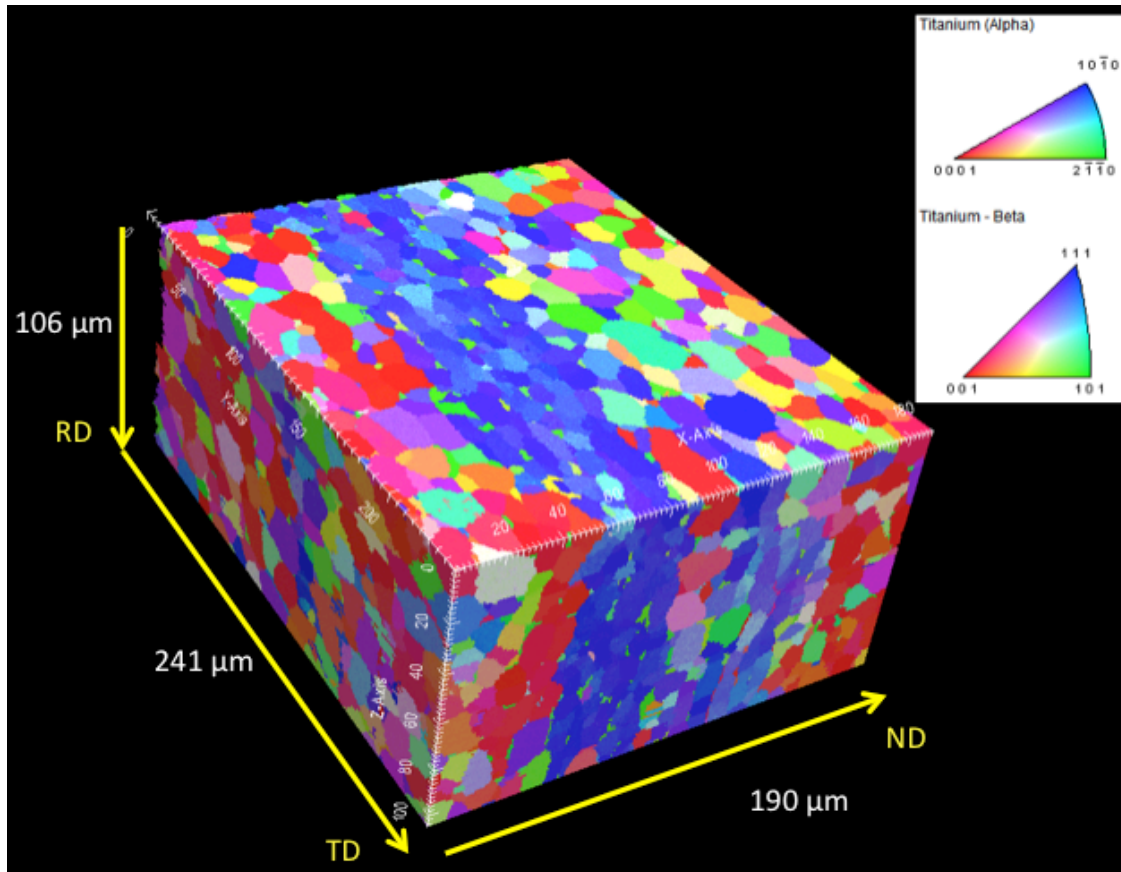
Alpha



Beta

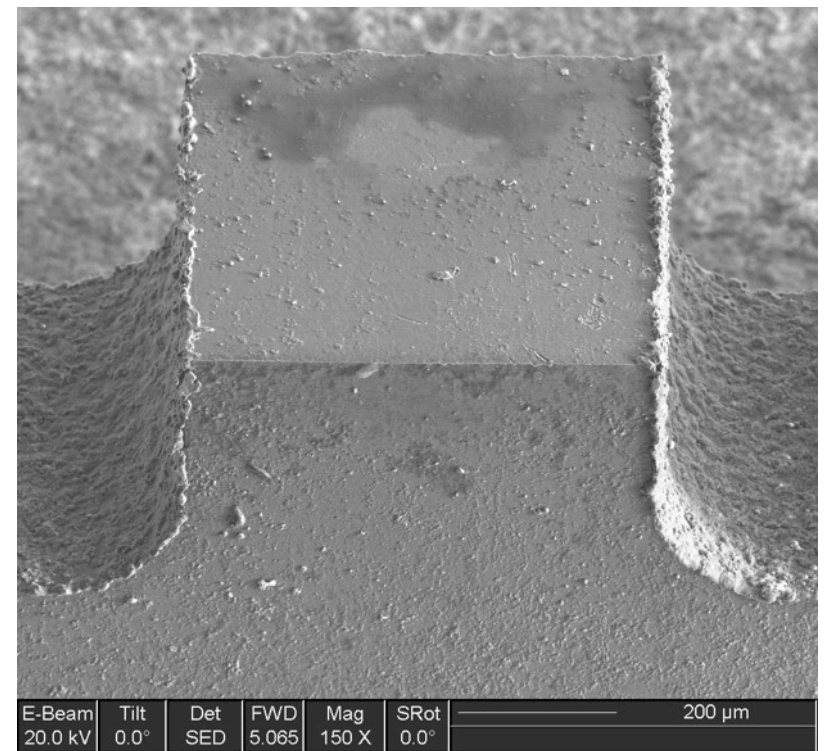
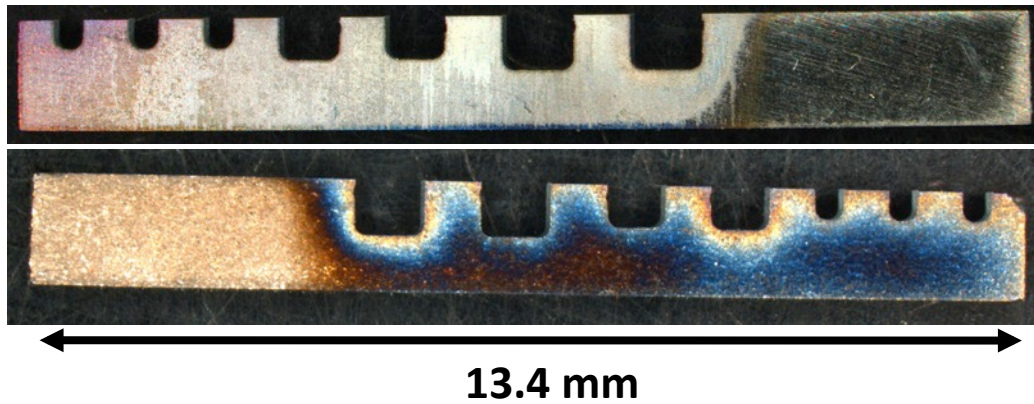
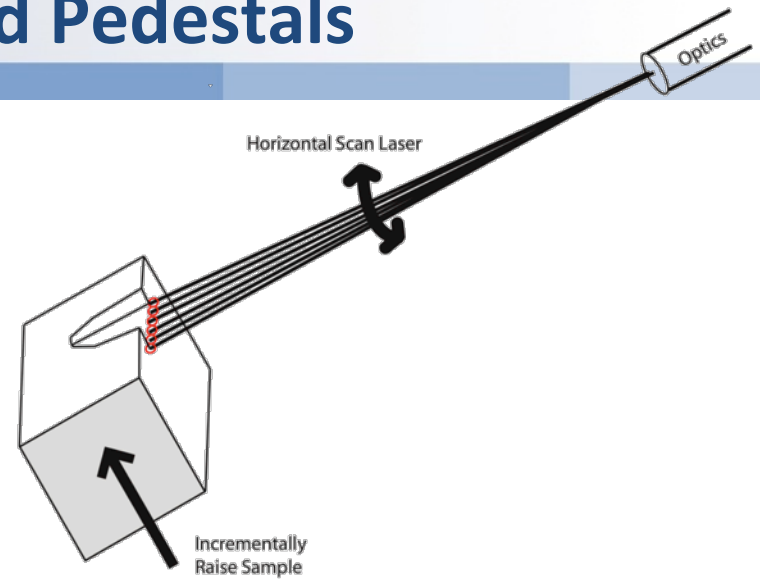
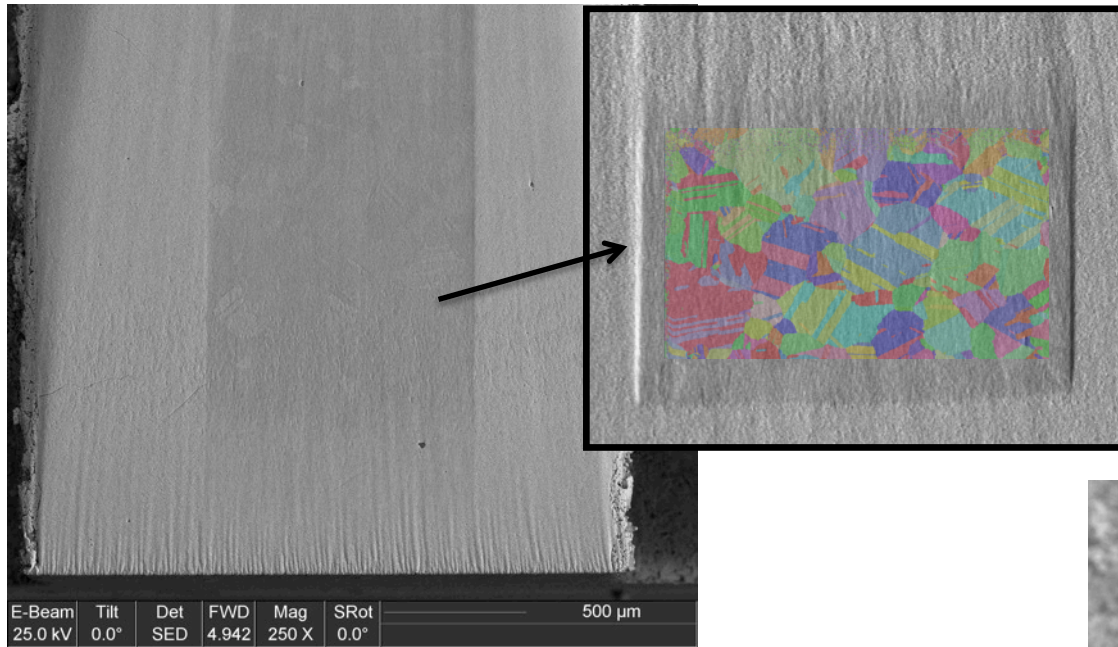


Dataset size: 190 x 250 x 108 μm
Z-resolution: 1 μm XY-resolution: 0.6 μm



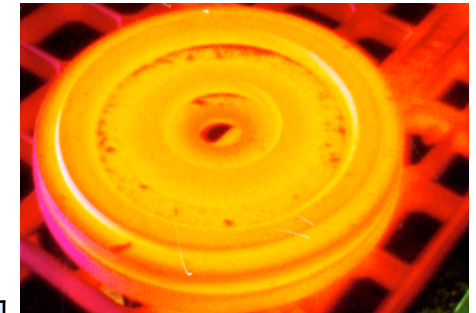
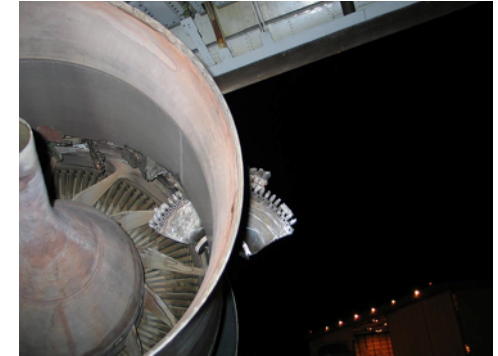
Total No of grains	No. of grains used for statistics	No. α grains	No. of β grains	Volume fraction α phase	Volume fraction β phase	Grain fraction α phase	Grain fraction β phase
8893	6645	2484	4161	0.9353	0.0647	0.3738	0.6262

Laser Ablated Pedestals



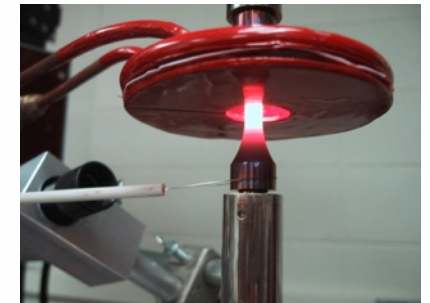
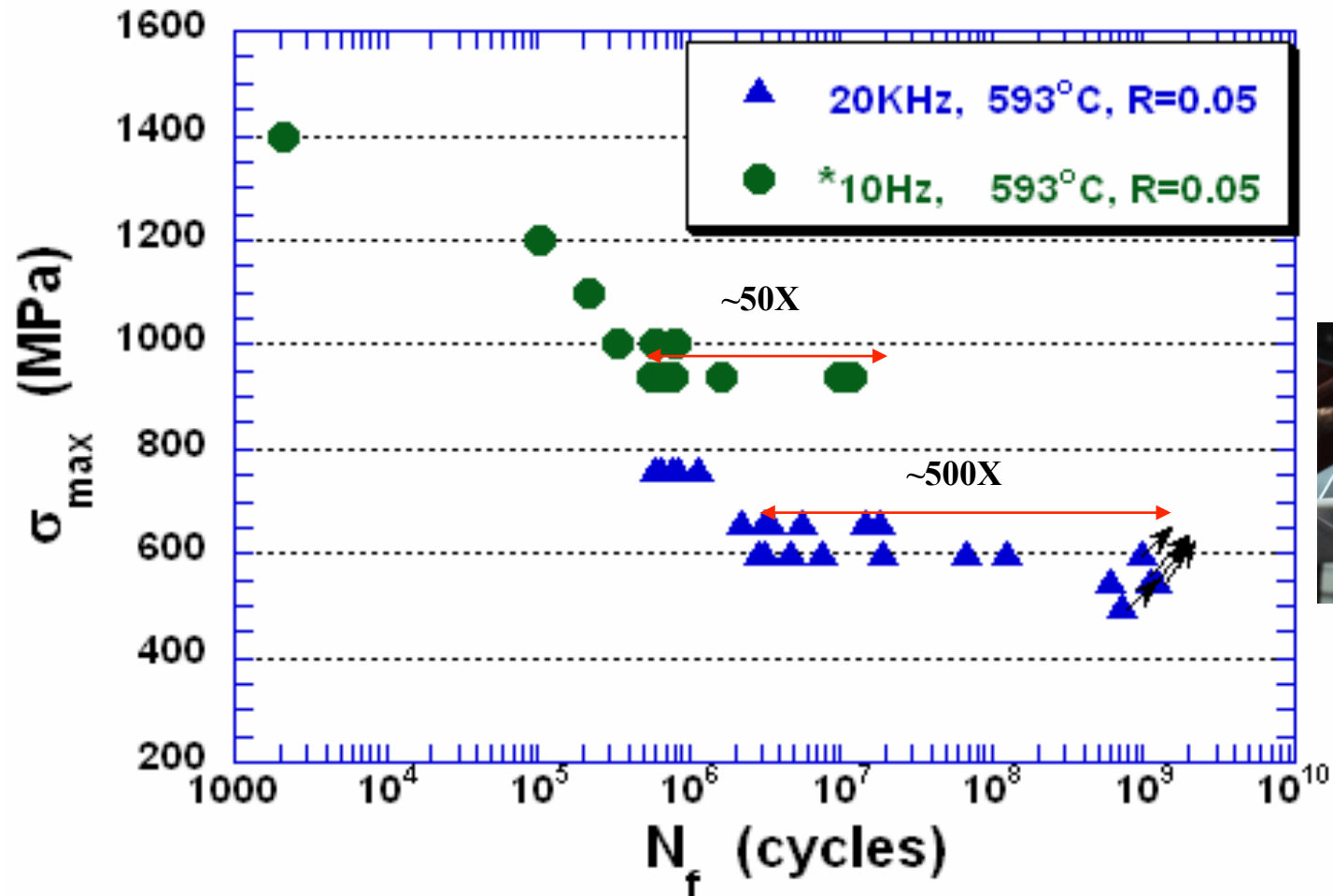


- Turbine Blades and Disks of Ni-base alloys are critical components
- Properties that limit their performance are “Structure Sensitive” Properties such as Fatigue and Fracture
- Submicron features in mm^3 to cm^3 volumes of material

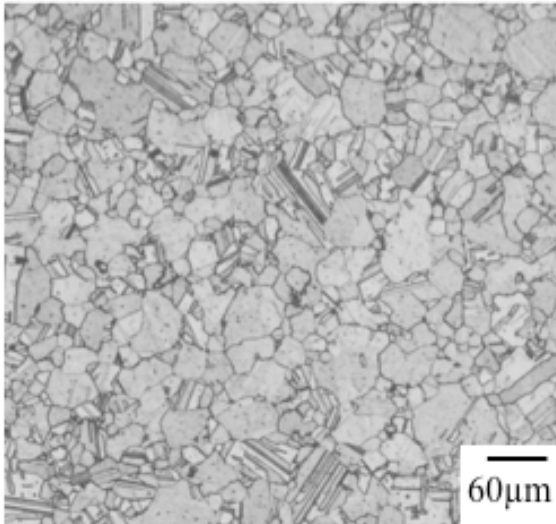


Chemical composition of René 88DT (weight percent)

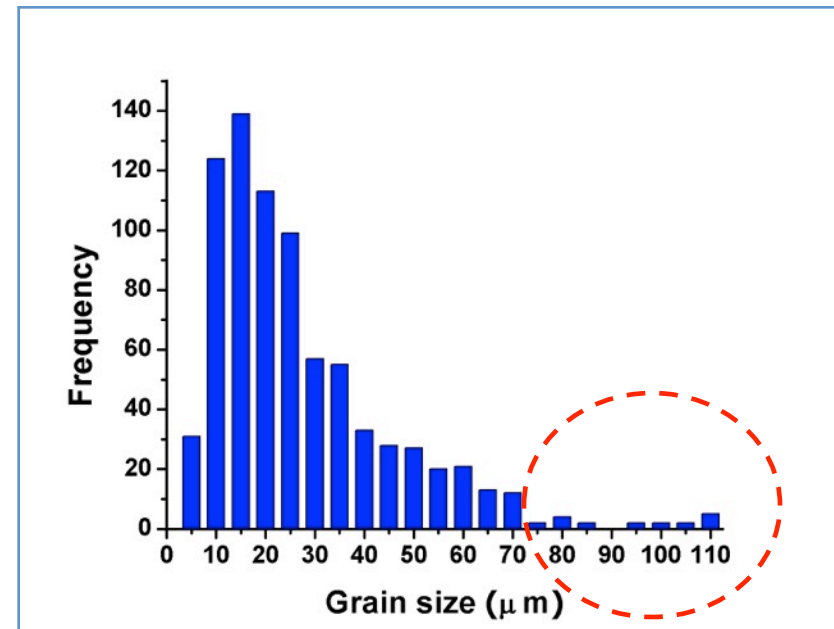
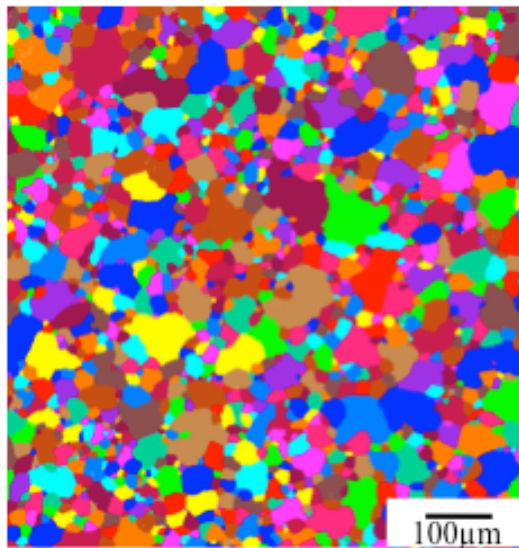
Cr	Co	Mo	W	Al	Ti	Nb	C	B	Ni
16	13	4	4	2.1	3.7	0.7	0.03	0.015	Bal

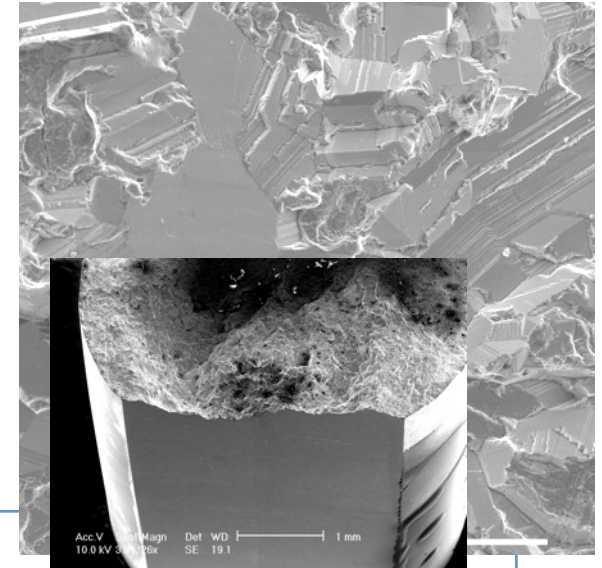
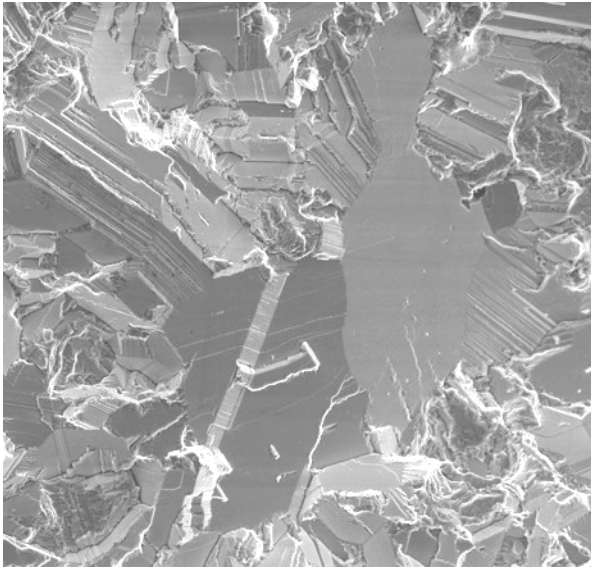


J. Miao, T.M. Pollock and J.W. Jones, *Acta Materialia* 60, 2840, (2012).



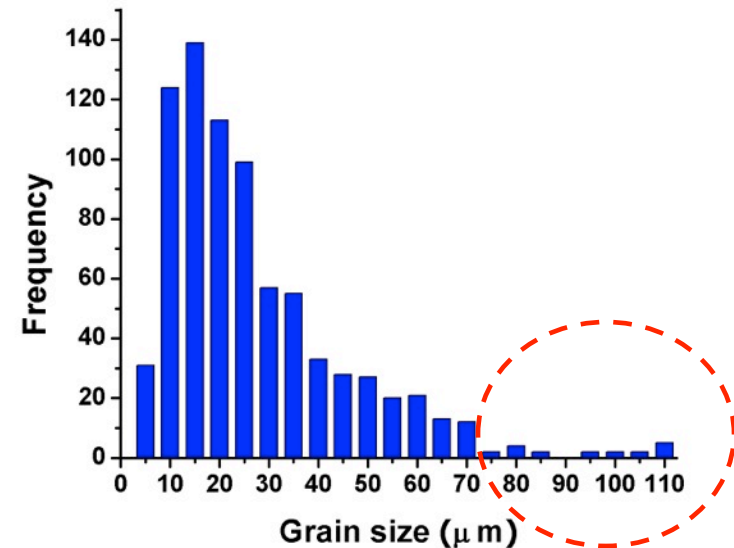
- Forged pancake
- Average grain size ~20µm
- Large grains at the tail of the distribution



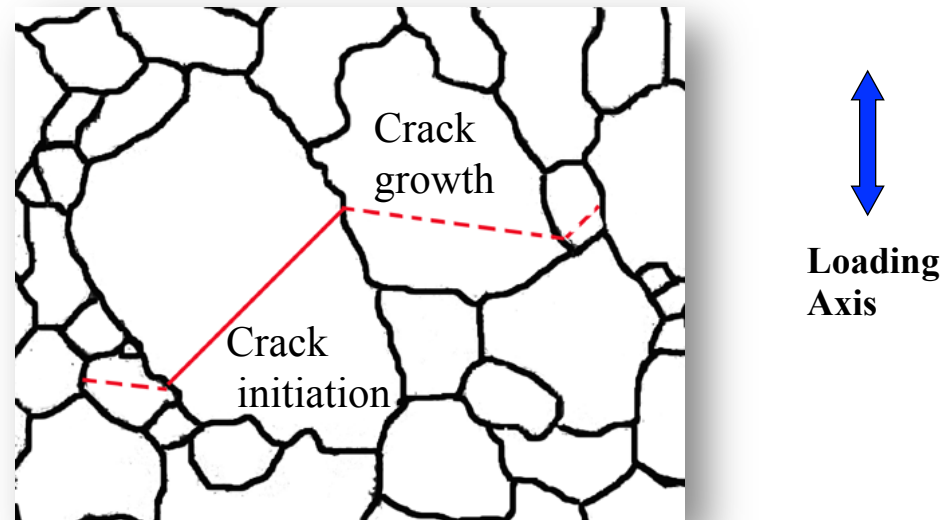


$$\sigma_{\max} = 600 \text{ MPa}, N_f = 3.16 \times 10^6$$

Large crystallographic facets at crack initiation sites are transgranular and produced by strain localization on slip planes prior to crack formation



Fatigue Crack Initiation



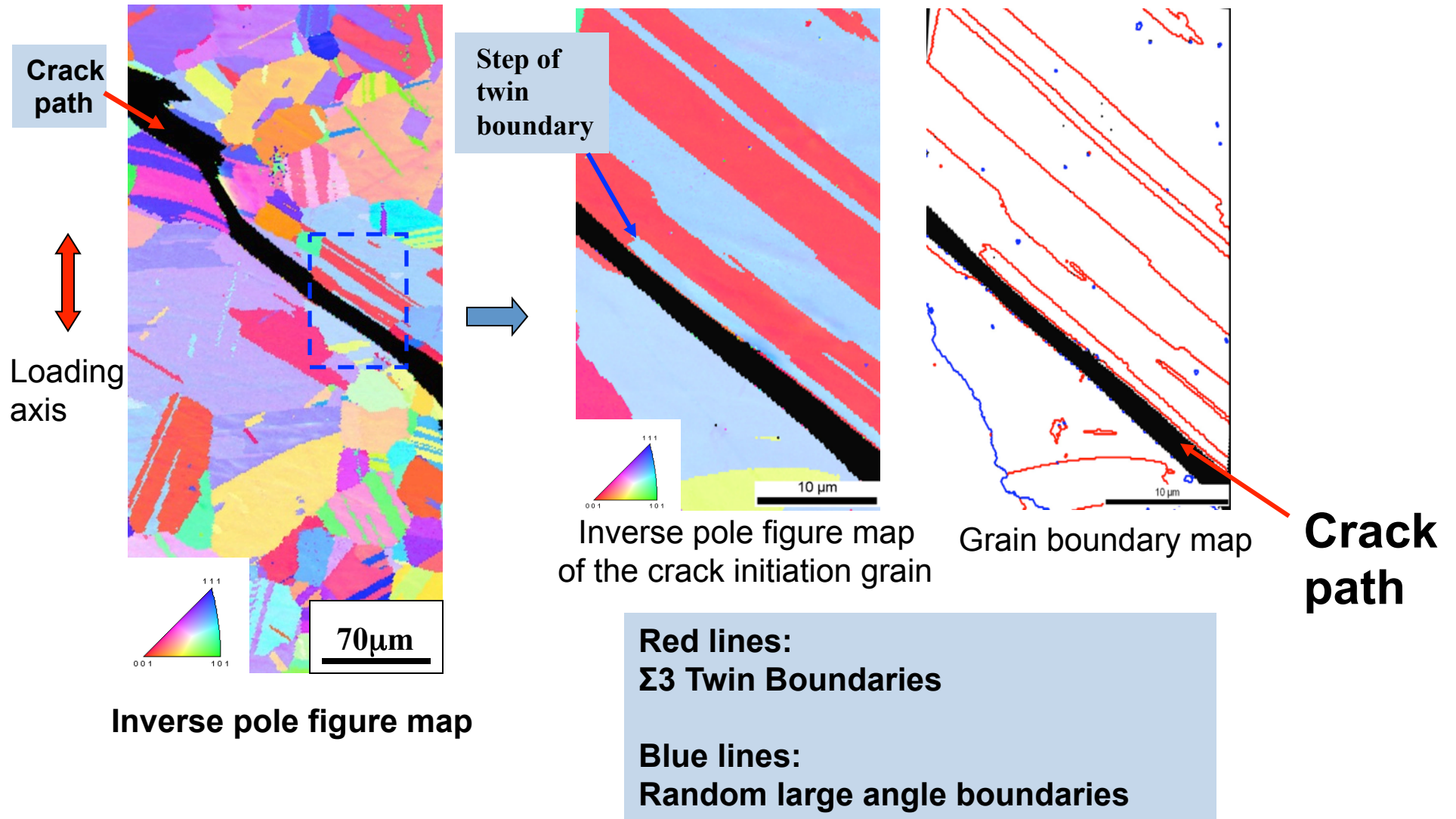
Critical microstructure features:

- Large grain (more than two times larger than the average grain size).
- Favorably Oriented for Slip (Schmid Factor > 0.45)
- Favorably Oriented Twin boundaries

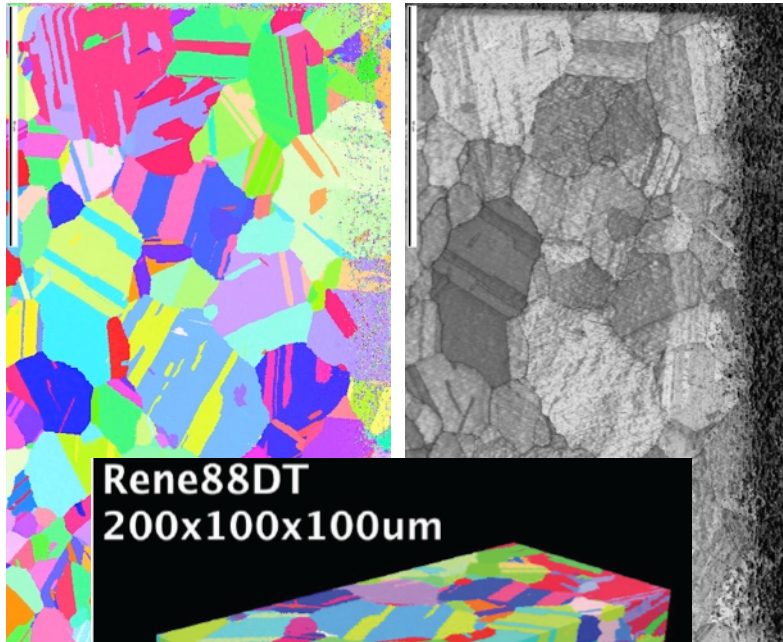
AND

- Neighboring grains misoriented by < 20 degrees with high SF

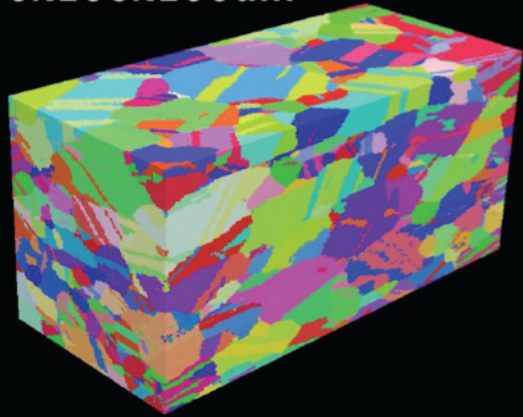
Cracks Initiate Parallel to Twins



Step size 0.3 μm in X-Y and 0.75 in Z
(240 \times 150 \times 200 μm^3)

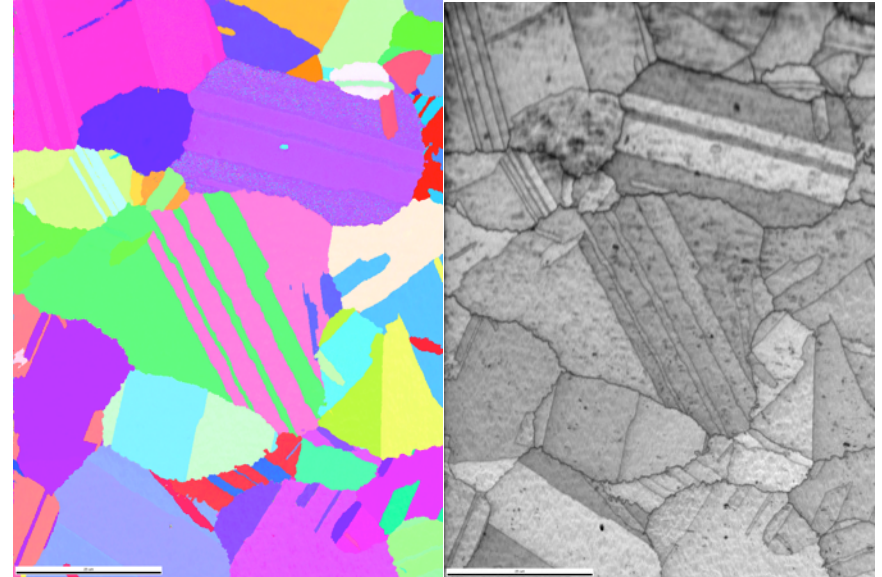


Rene88DT
200 \times 100 \times 100 μm

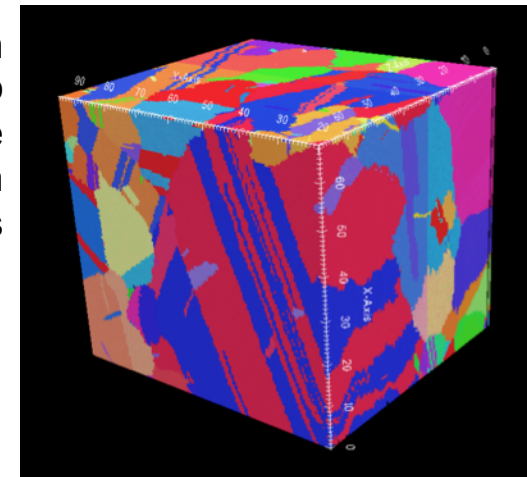


Medium resolution to have a huge volume

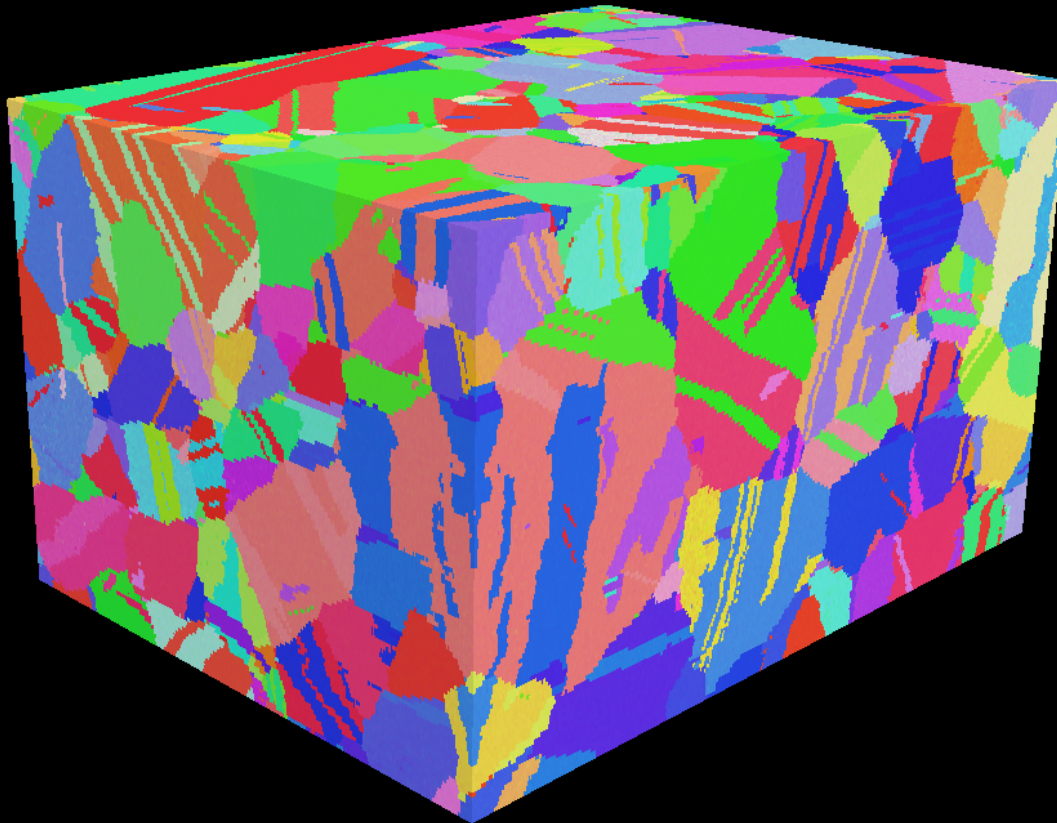
Step size 0.1 μm in X-Y and 0.5 in Z
(70 \times 100 \times 75 μm^3)



High resolution to describe the grain-twin boundaries



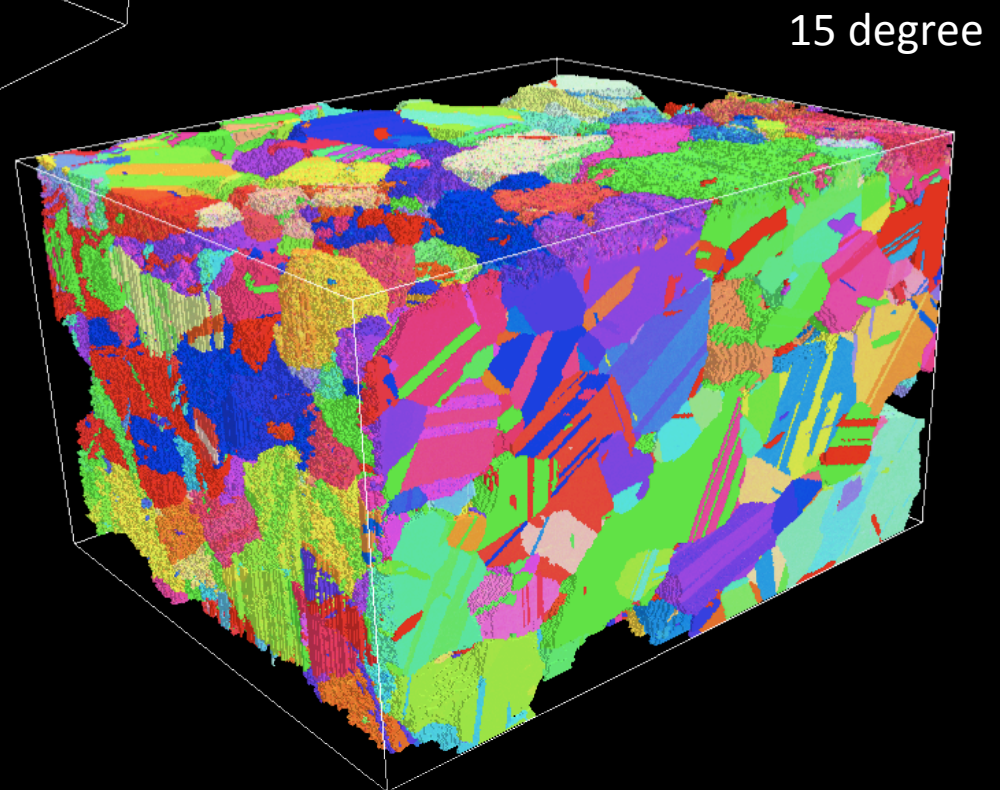
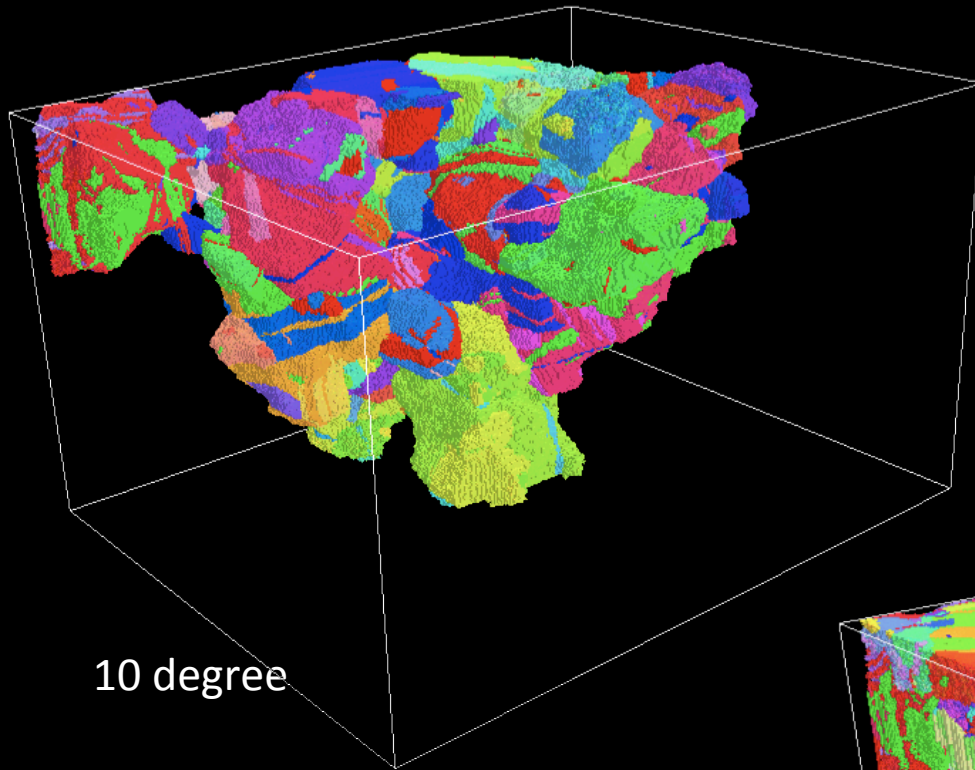
Dataset size: 160 x 120 x 90 μm
Rene88DT – Nickel Disk Alloy



- 100h Dataset
 - Laser + FIB (30 kV, 10°, 20nA)
- 8x8 camera binning @ 375indexed pts/s
 - Avg CI \approx 0.5

Rene88DT Clustering: Highly Connected

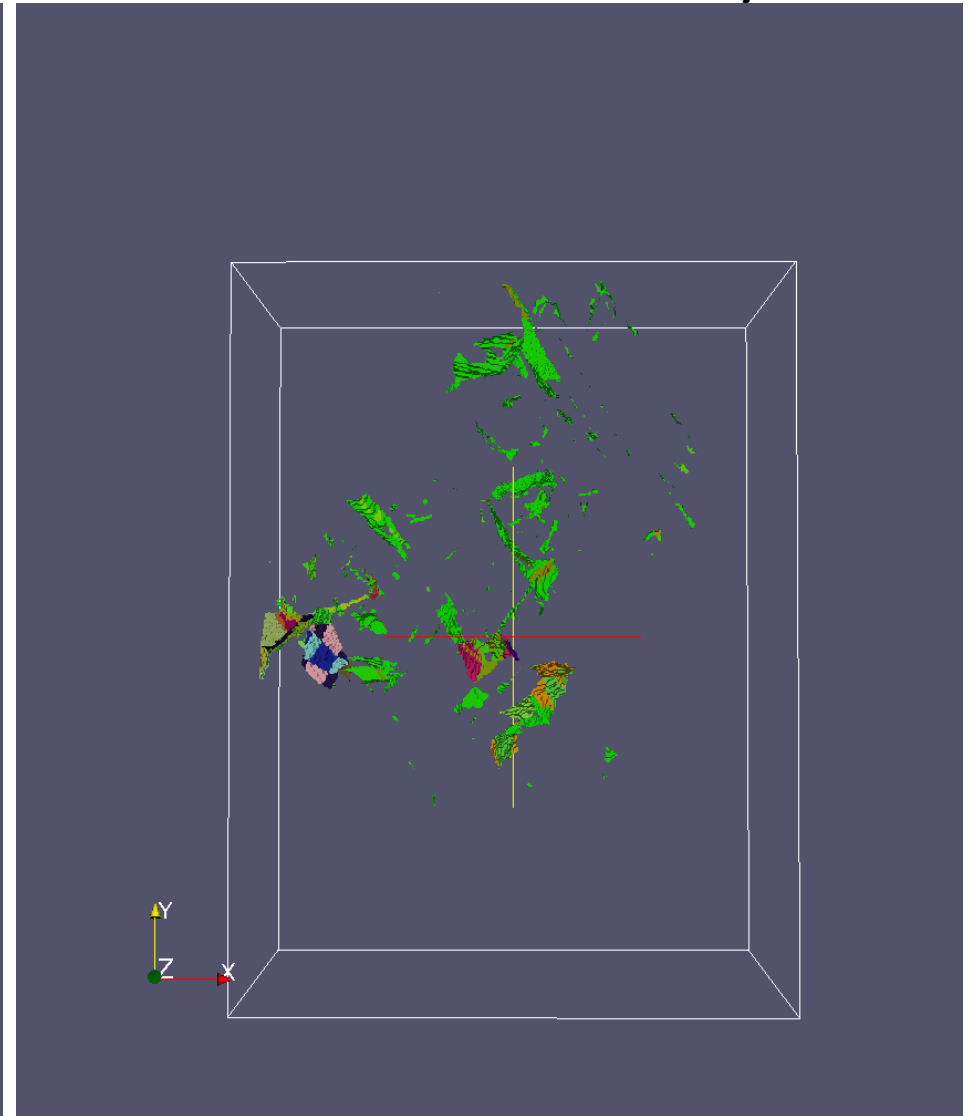
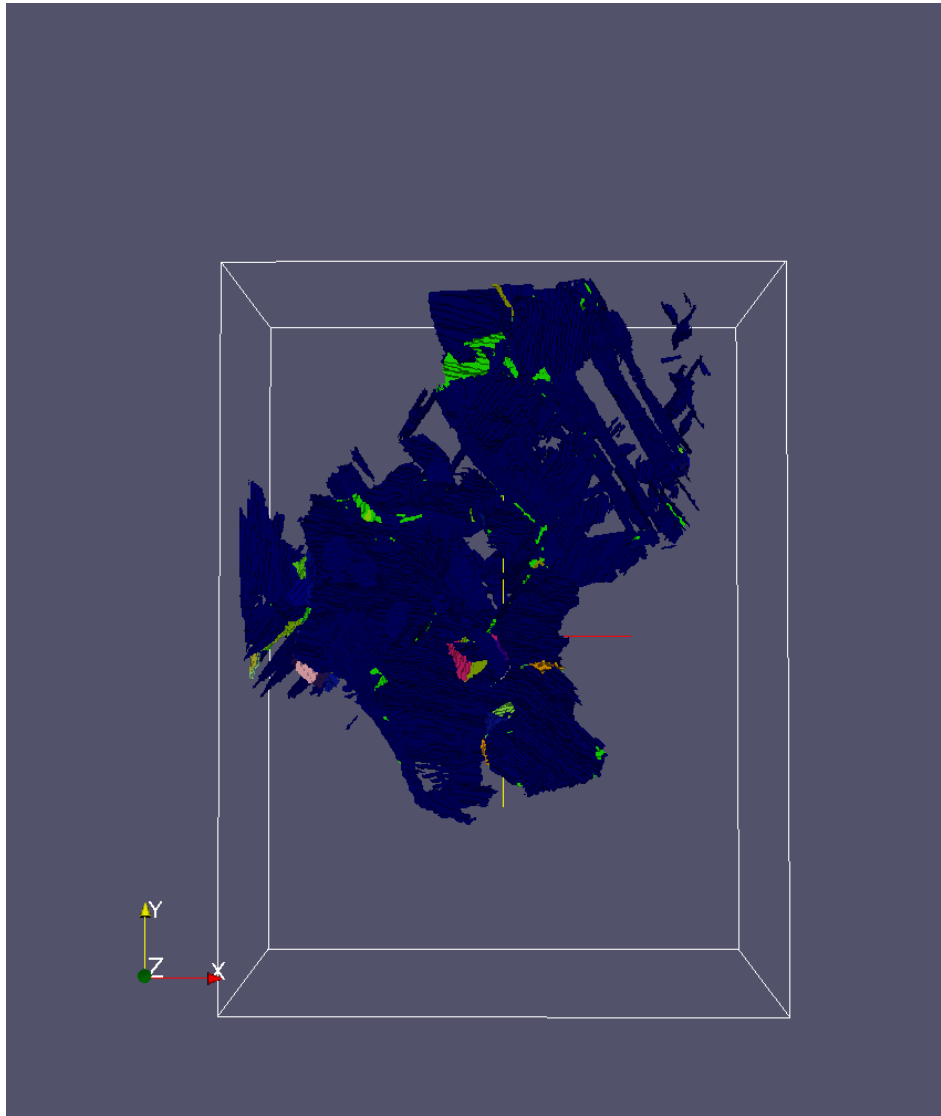
The convergence of research and innovation.



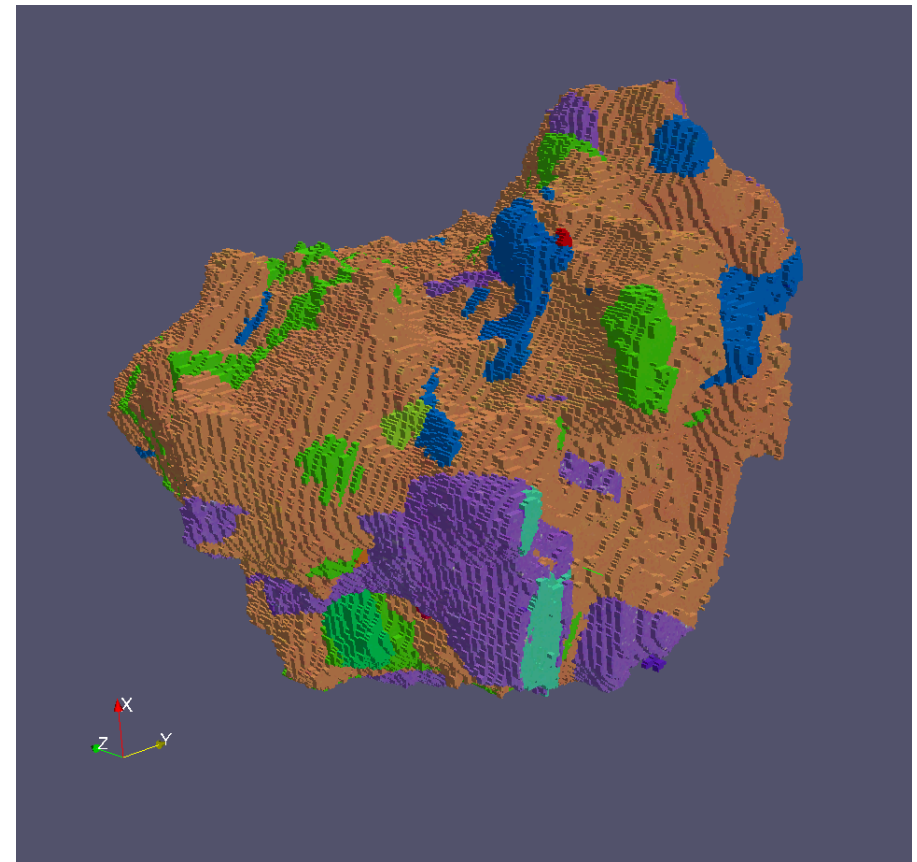
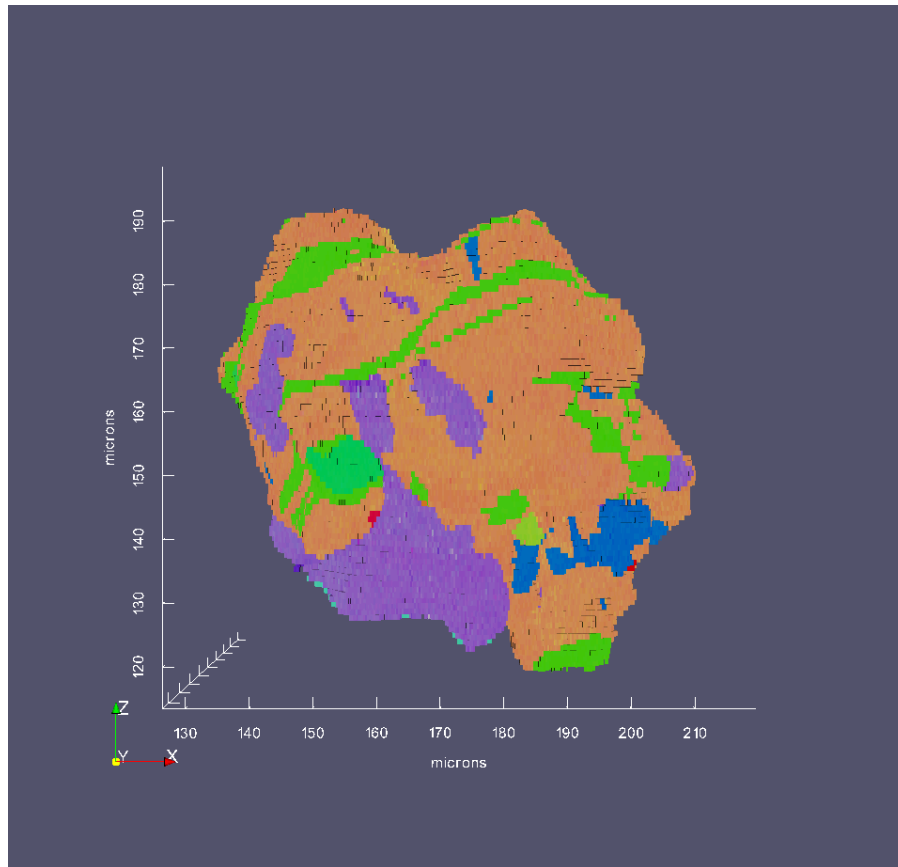
Identifying Large Grains and Large Twins

The convergence of research and innovation.

Dataset size: 160 x 120 x 90 μm
Rene88DT – Nickel Disk Alloy

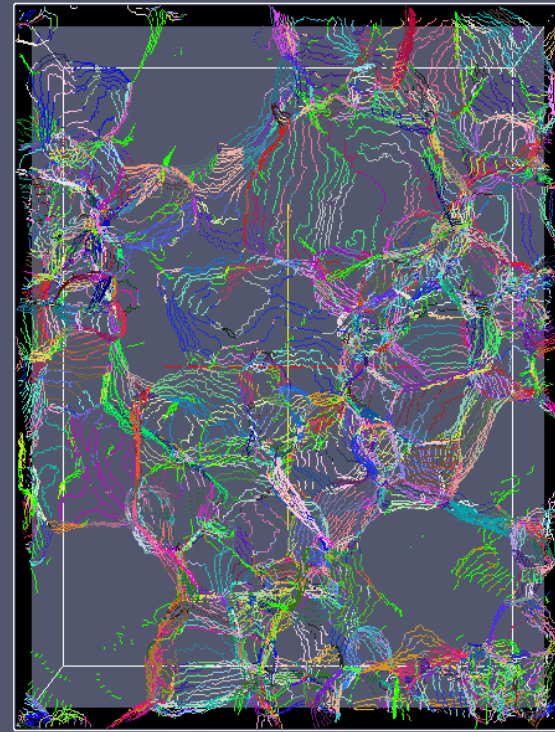
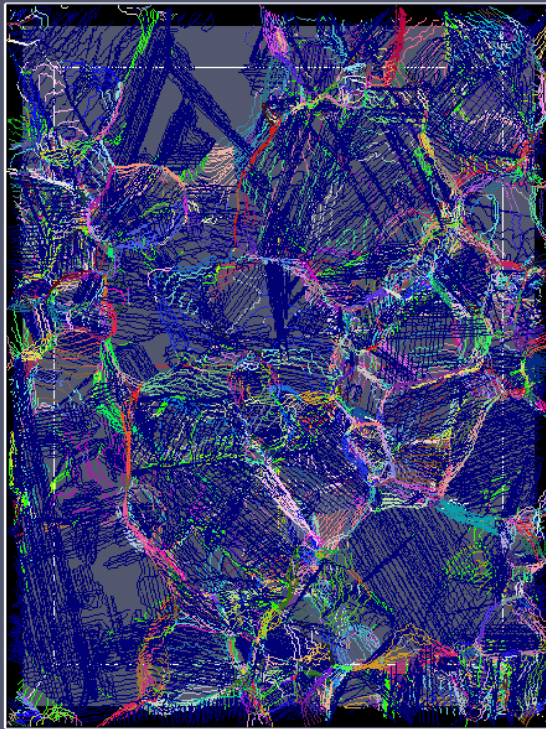


Length of Slip Band? Single Grain, Twins Oriented Regions in Color

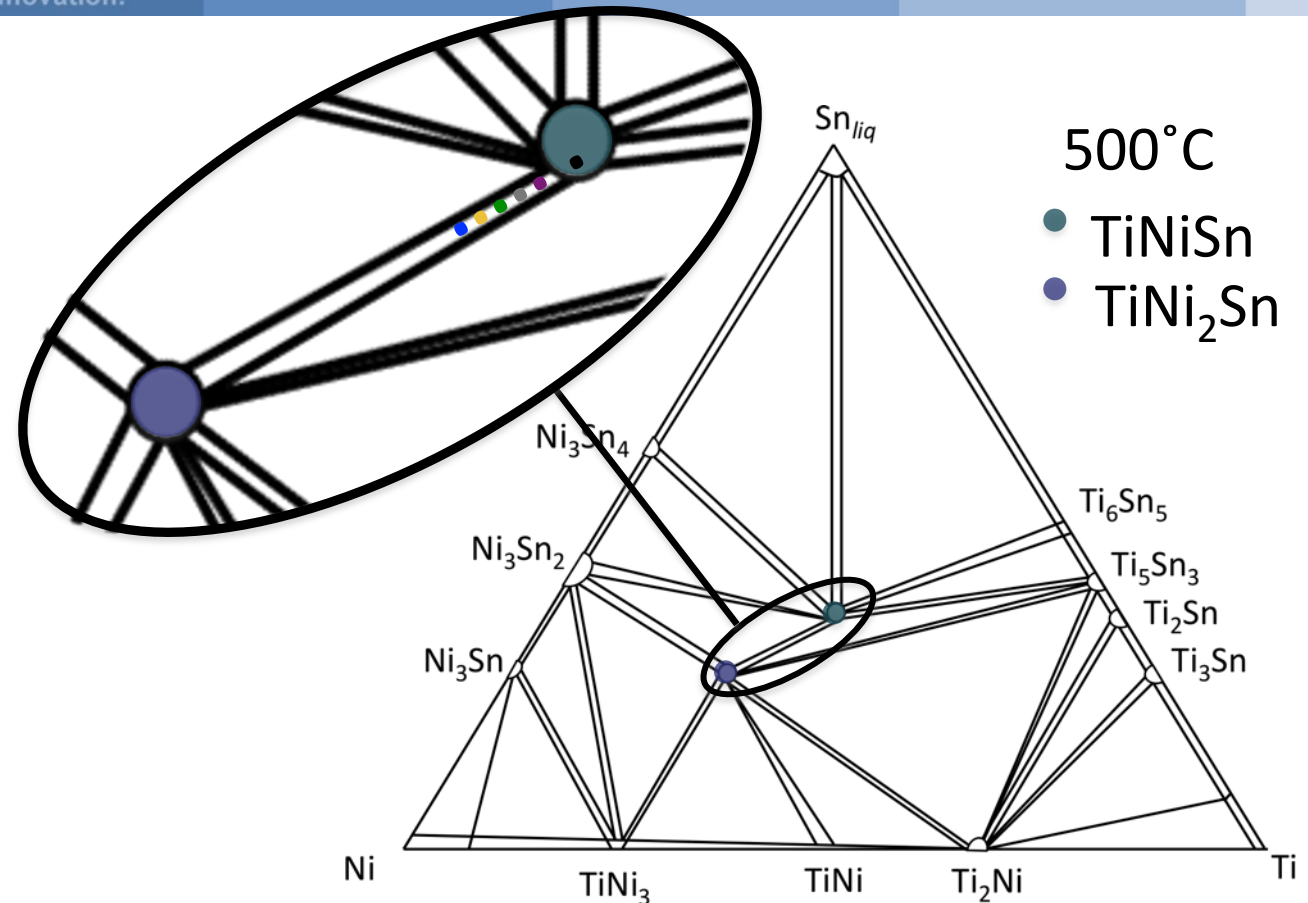
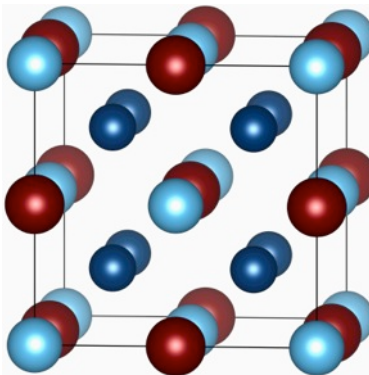
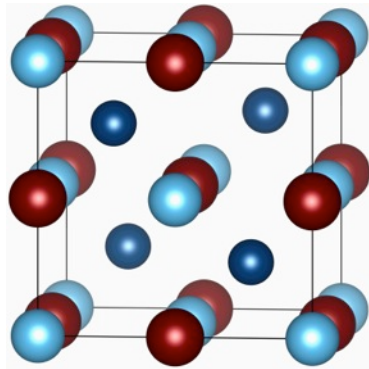


Dataset size: 160 x 120 x 90 μm
Rene88DT – Nickel Disk Alloy

Twins Removed



Identify large twin boundary surface favorably oriented for slip AND adjacent grains that crack can propagate into



- Half Heusler TiNiSn is thermoelectric and the Heusler is metallic TiNi₂Sn
- There is a two-phase field between them
- Similar structures, 3% lattice mismatch

The Full Heusler Phase Improves Thermoelectric ZT

The convergence of research and innovation.

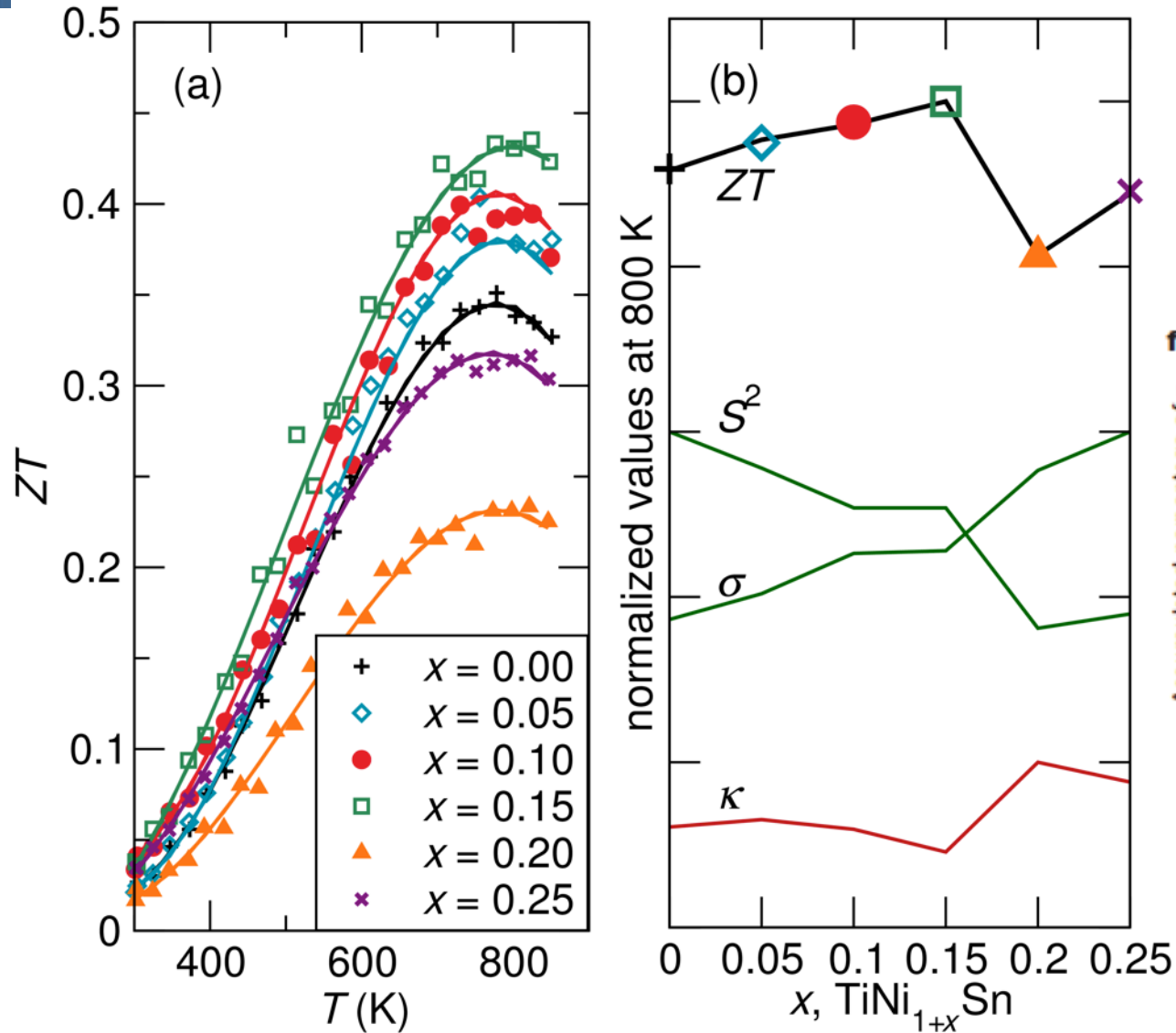
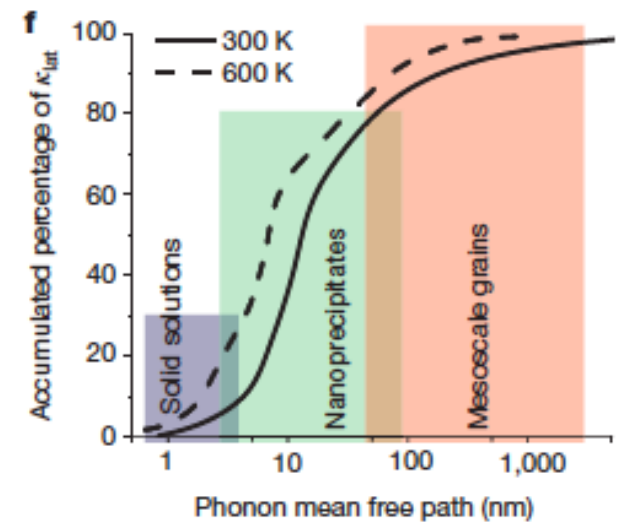


Figure of Merit

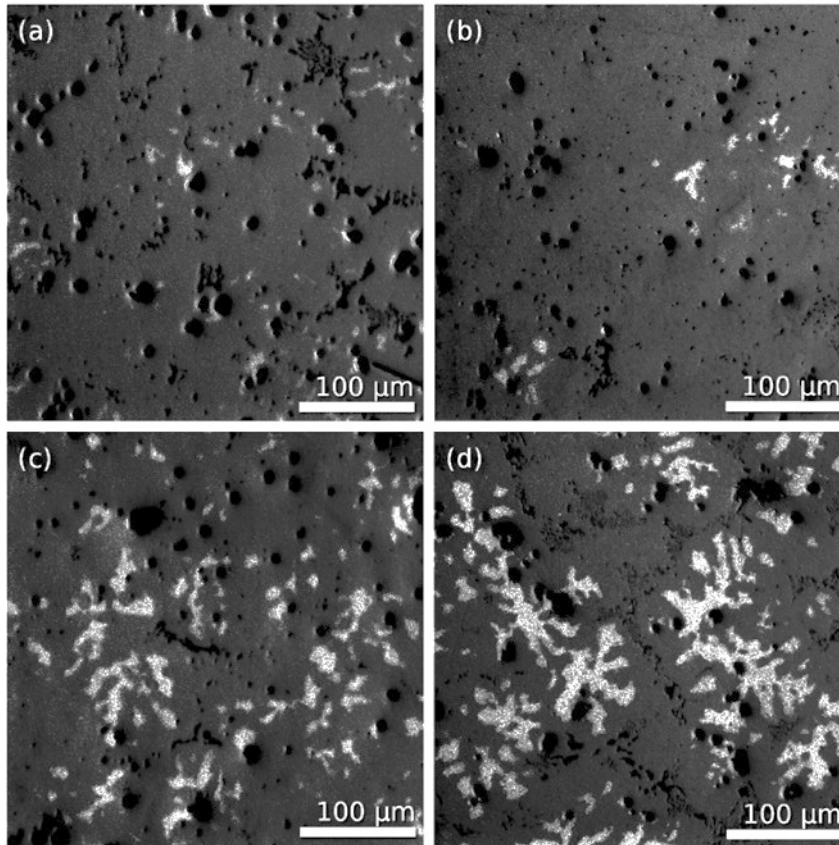
$$ZT = \frac{S^2 \sigma}{\kappa} T$$



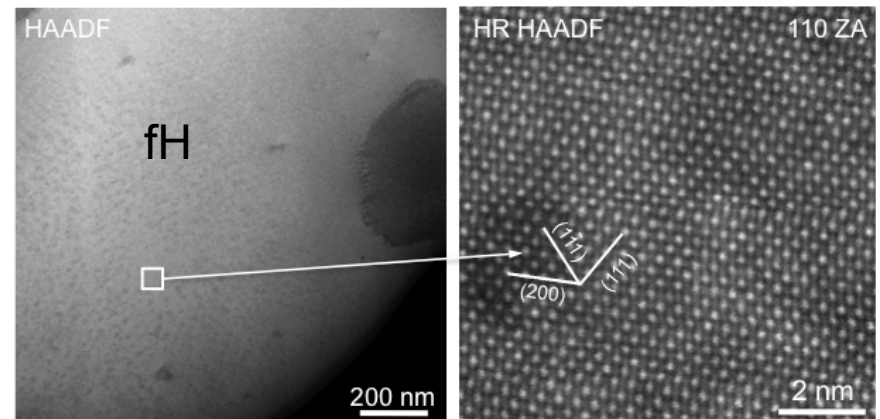
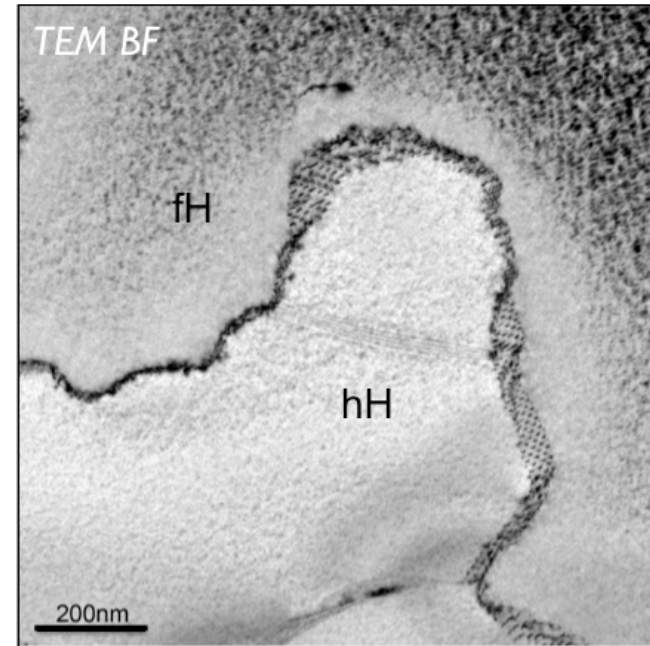
Biswas *et al*, *Nature* 489, 2012

Hierarchy of Interfaces and Lengthscales TiNi_{1+x}Sn

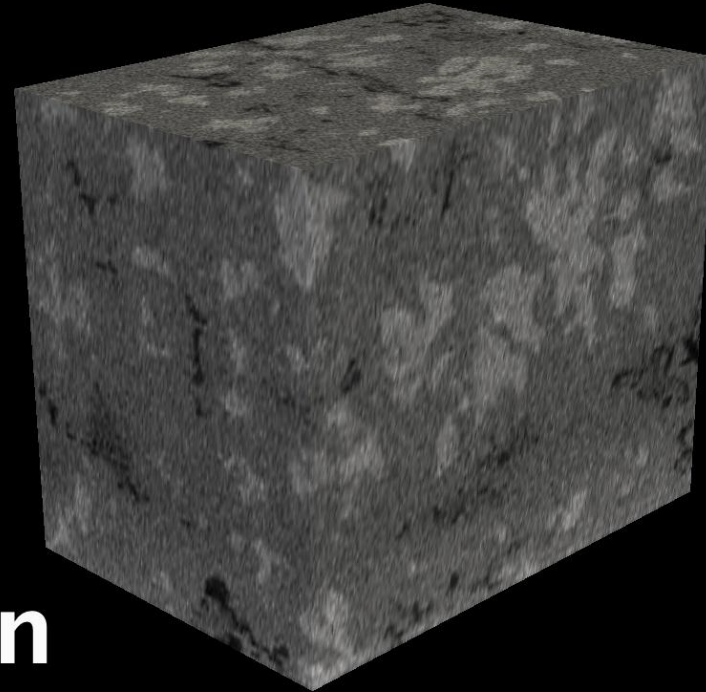
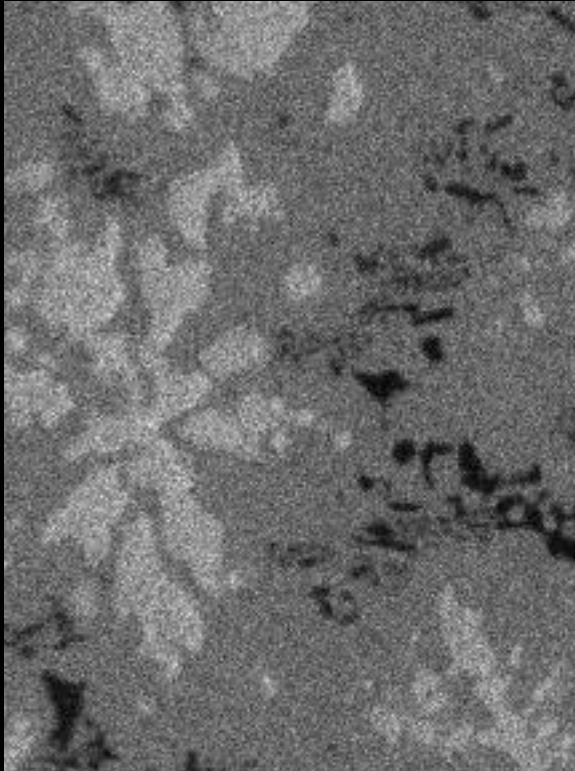
[Dark grey: half-Heusler; light grey: Heusler]



Dendritic Heusler phase

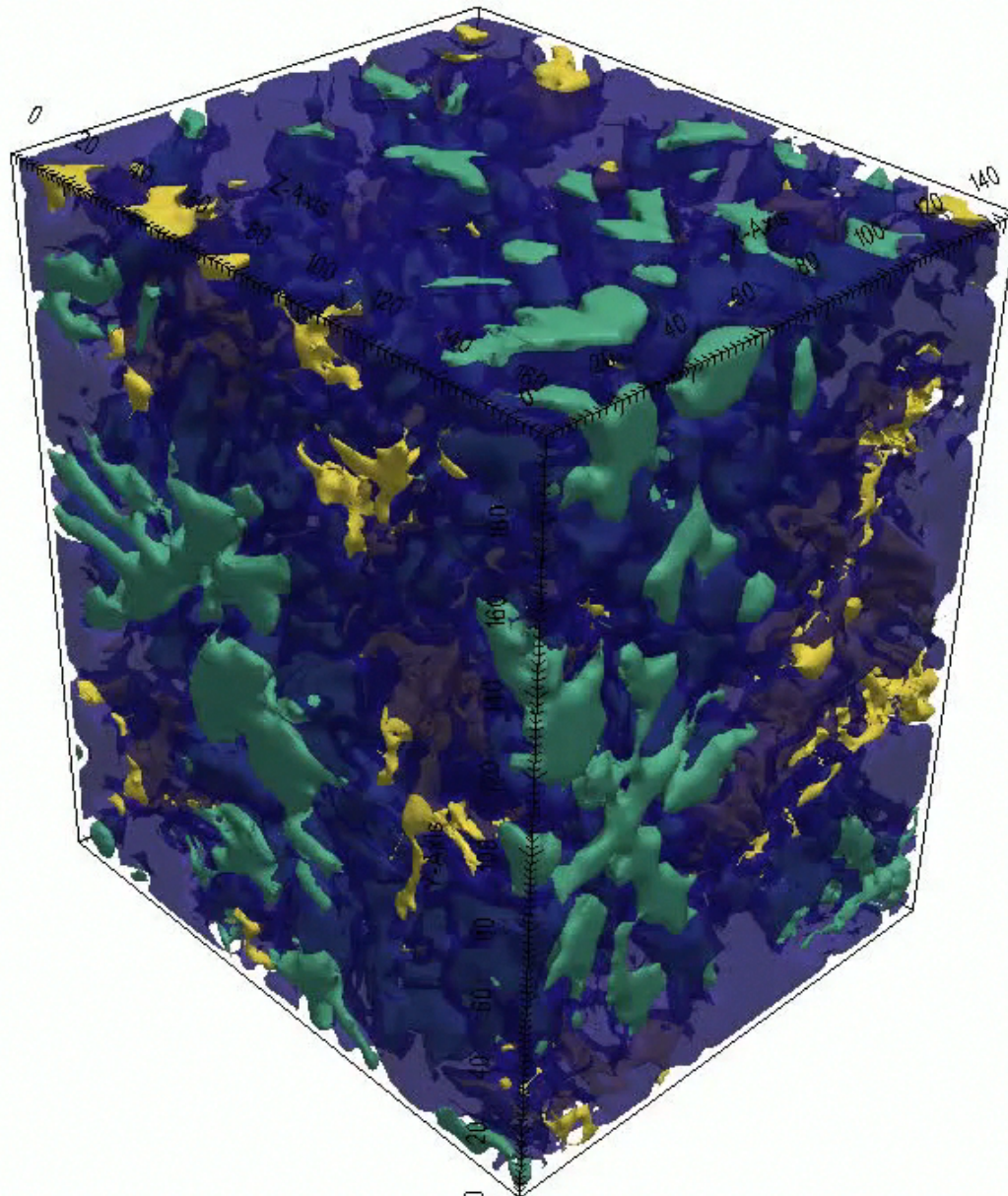


Small coherent precipitates



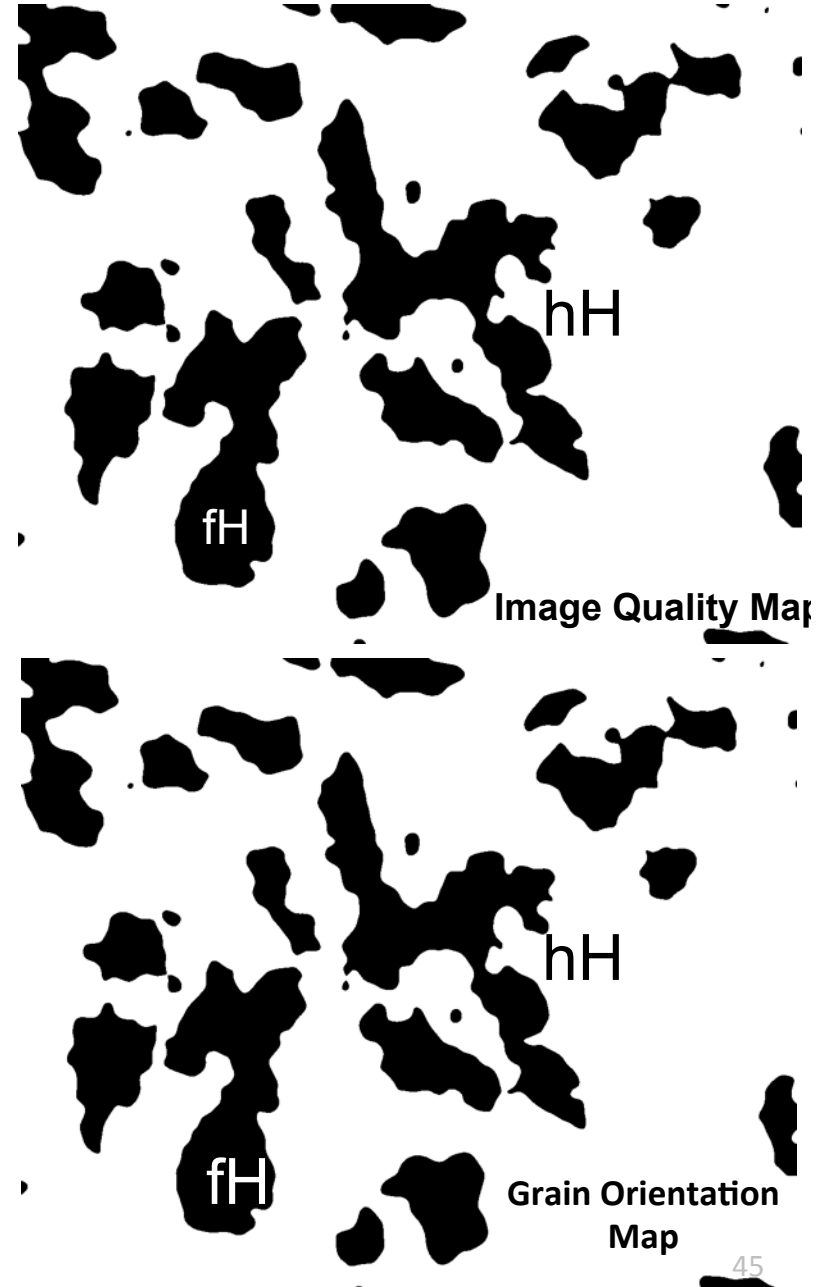
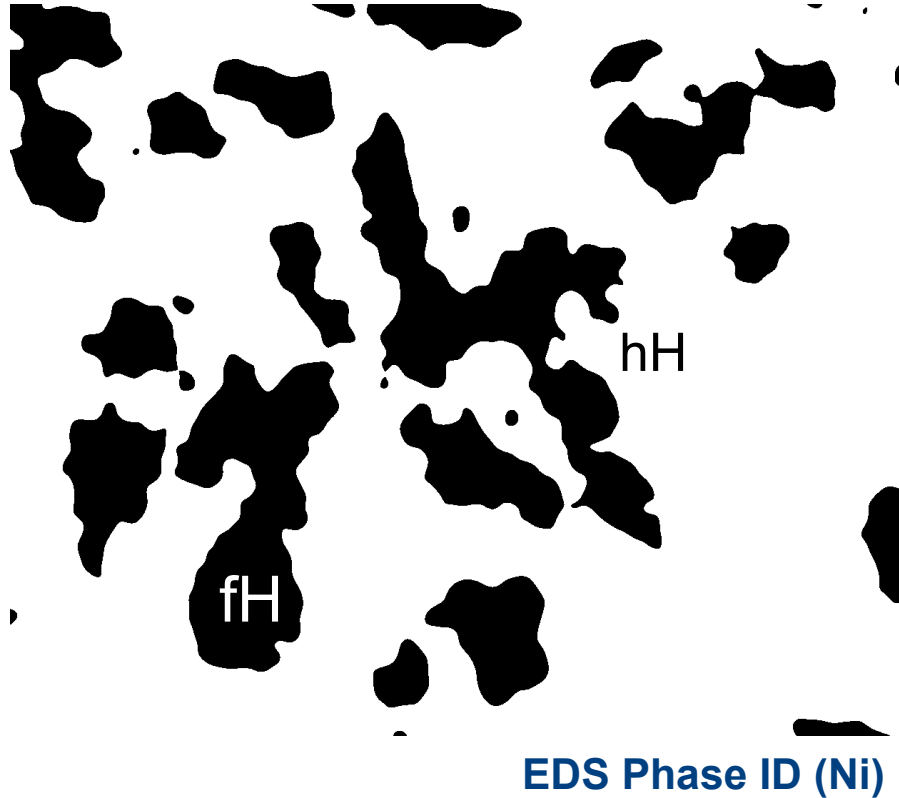
NiTiSn
200x170x140um

FH = Light phase; Mid tone Gray = hH; TiSn = Dark



Y – TiSn
(7%)
G – Heusler
(20%)
B – Half-Heusler
(73%)

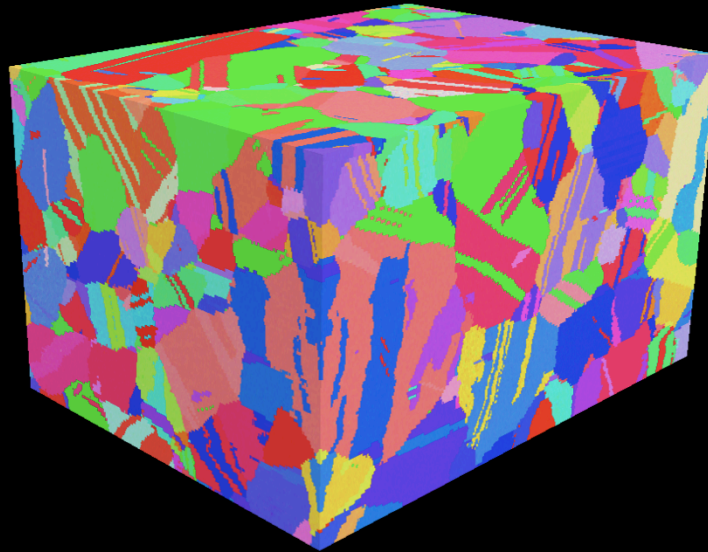
Tribeam: Multimodal information



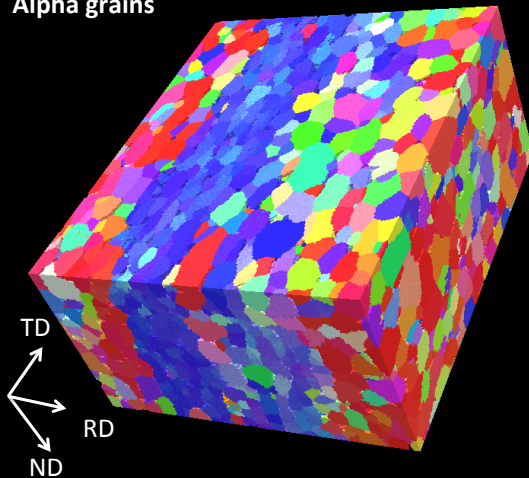
- Measurement of connectivity of full-Heusler phase requires multimodal EDS/EBSD mapping to distinguish phases
- New algorithms for merging datasets being developed
- Low misorientation (semicoherent) boundaries between fH and hH in many regions

The convergence of research and innovation.

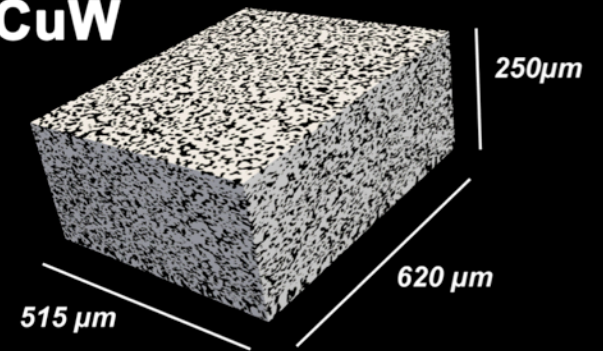
Dataset size: 160 x 120 x 90 μm
Rene88DT – Nickel Disk Alloy



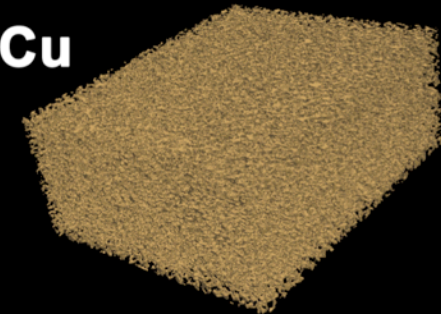
Dataset size: 190 x 250 x 108 μm
Alpha grains



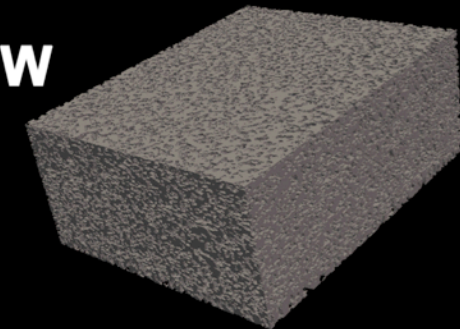
CuW

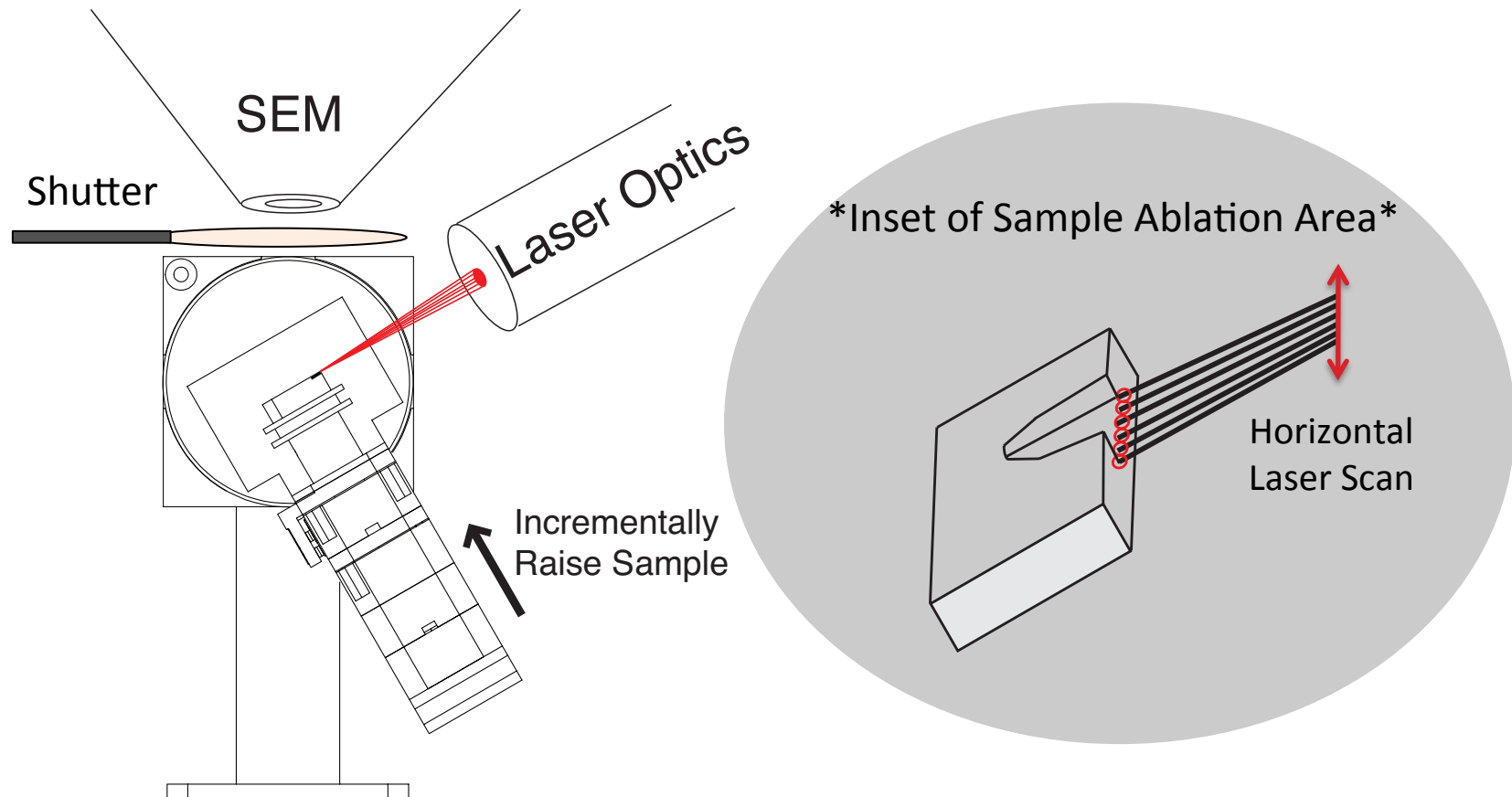


Cu



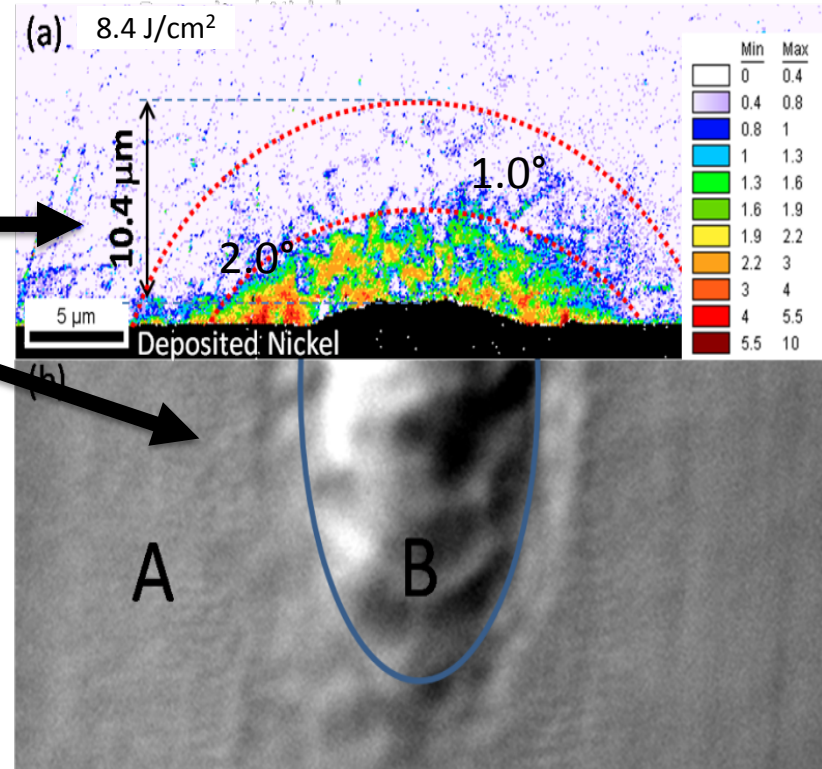
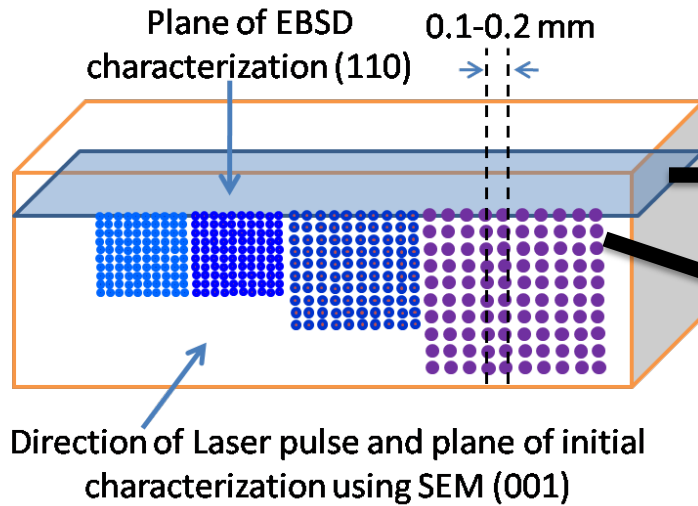
W



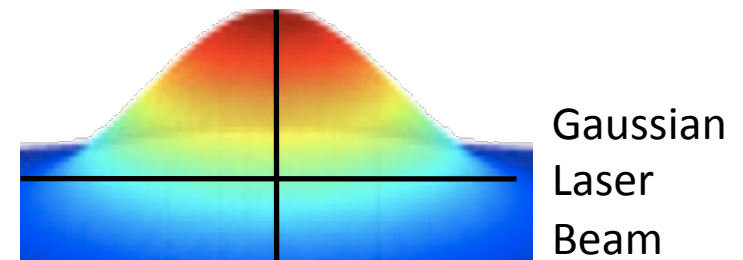


- Sample is incrementally raised into the focus of the beam
- The pole piece is blocked from redeposition by a shutter
- Surface parallel ablation decouples phase removal rate dependence

SCHEMATIC OF LASER MACHINING! – DIFFERENT THAN TRIBEAM

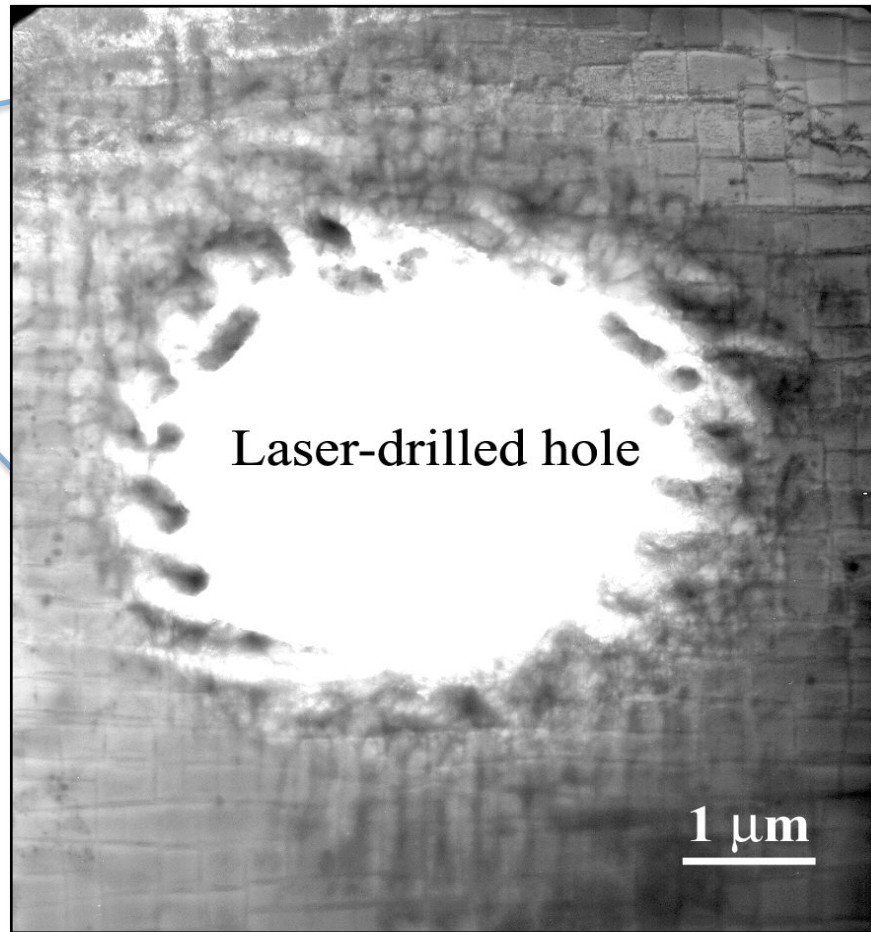
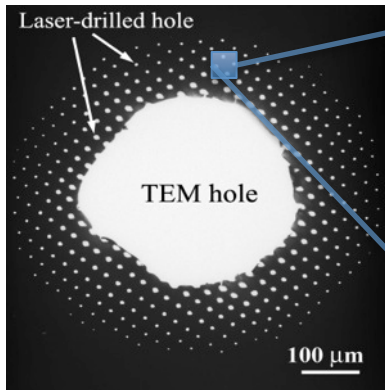


- High Fluence pulses, sample perpendicular to beam
- Most damage limited to less than 3-4 μ m in depth
- Highest damage < 5.5°
- EBSD patterns can still be obtained
- Dislocation densities of $\sim 2.8 \times 10^{10}$ per cm^3

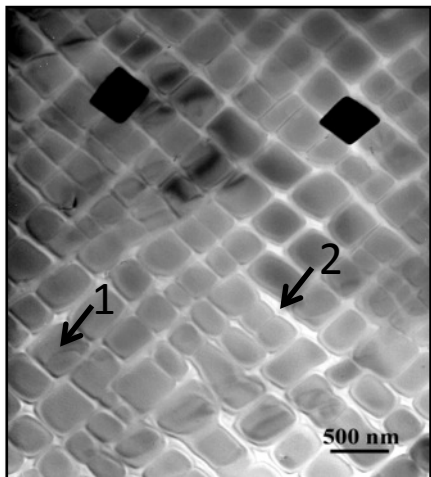


The convergence of research and innovation.

Single crystal Ni-base
Superalloy TEM foil

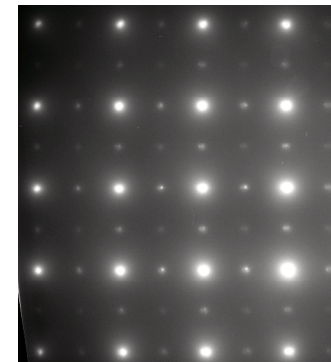


Ni-base Superalloy
Unperturbed microstructure

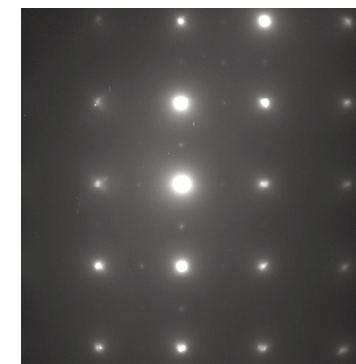


1. $L1_2$ ordered γ' precipitates
2. γ phase matrix

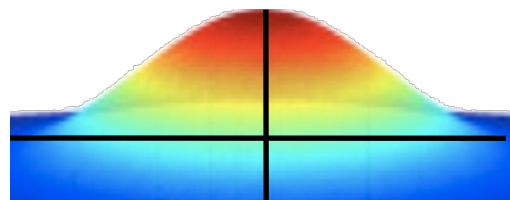
Diffraction patterns
sampled near edge
of laser-drilled hole



γ' precipitates

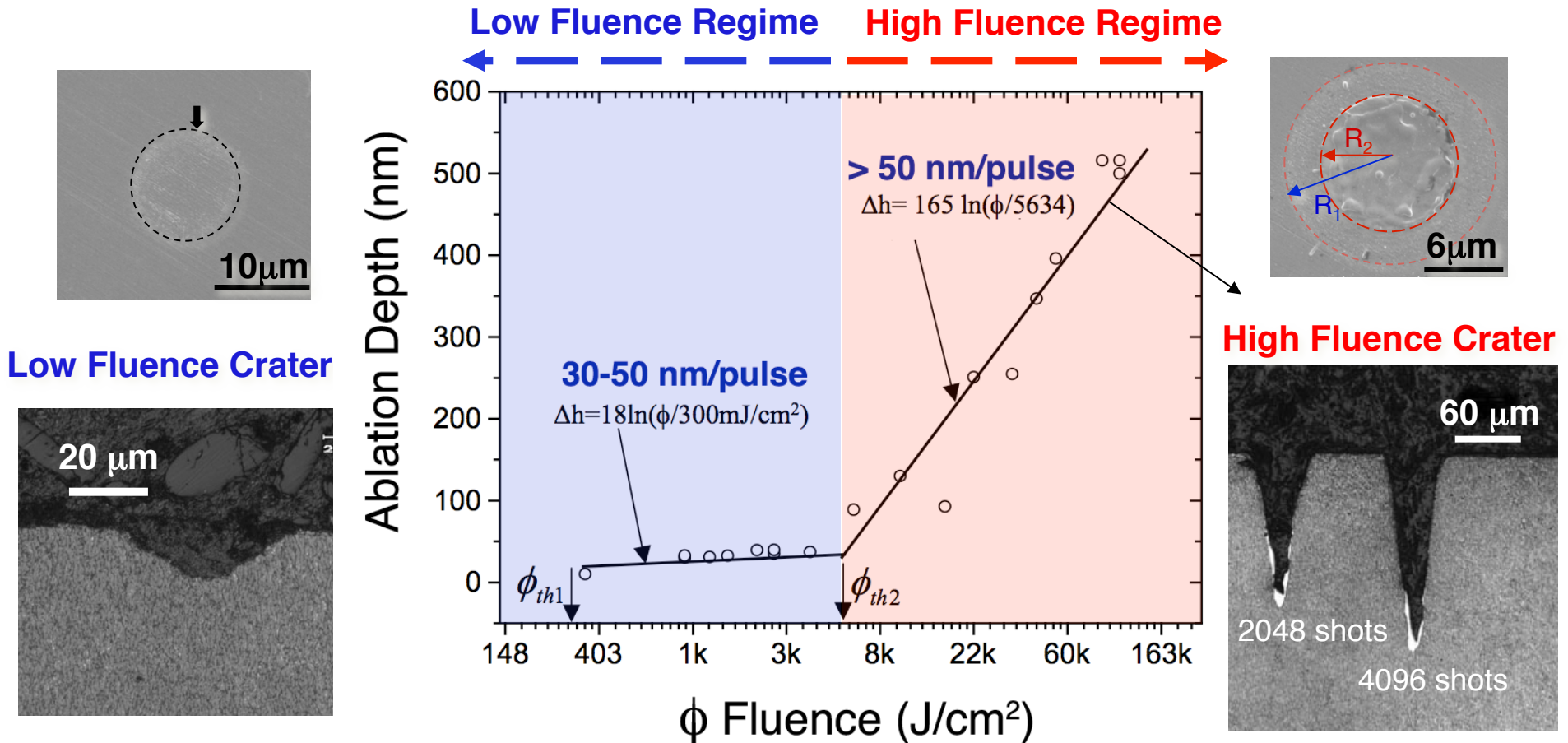


γ matrix



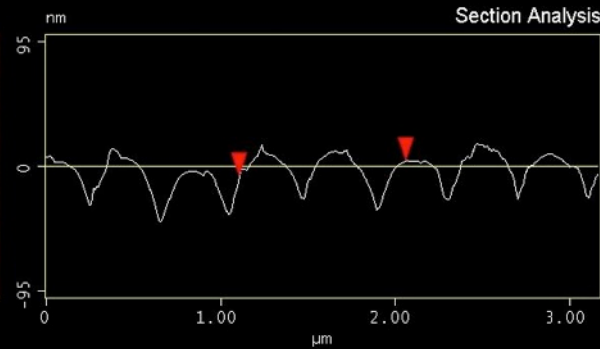
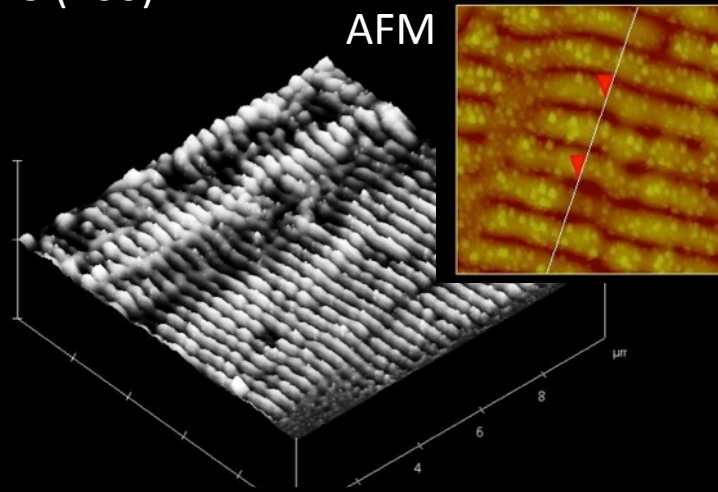
Two Distinct Ablation Rates in Superalloy

The convergence of research and innovation.

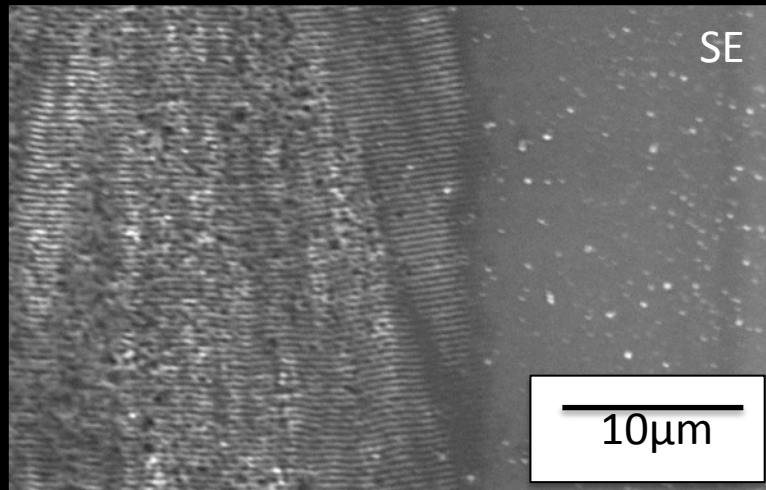


- Single pulse ablation of CMSX-4 Superalloy
- Depth and ablation morphology characterized with AFM
- Two distinct ablation rates and damage

Si(100)

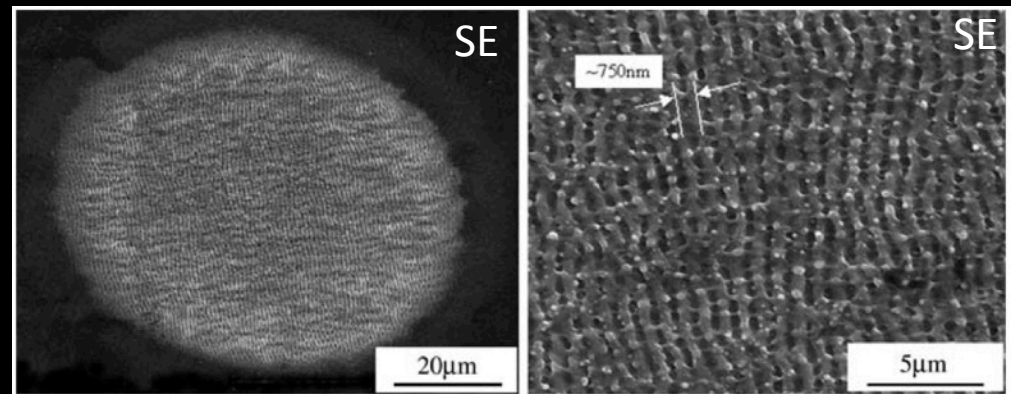


Periodicity dependent on:
Wavelength, Material, Laser Fluence



J.P. McDonald, S.M. Yalisove, *et al.* MRS Bulletin, 31, 626, (2006).

Ni Alloy - CMSX-4



S. Ma *et al.* Metall. Mater. Trans. A 38A, 2349 (2007).

Laser Ablated Mg Alloy - AXJ-530



100 μ m

Laser Ablated Ni Alloy - Rene88DT



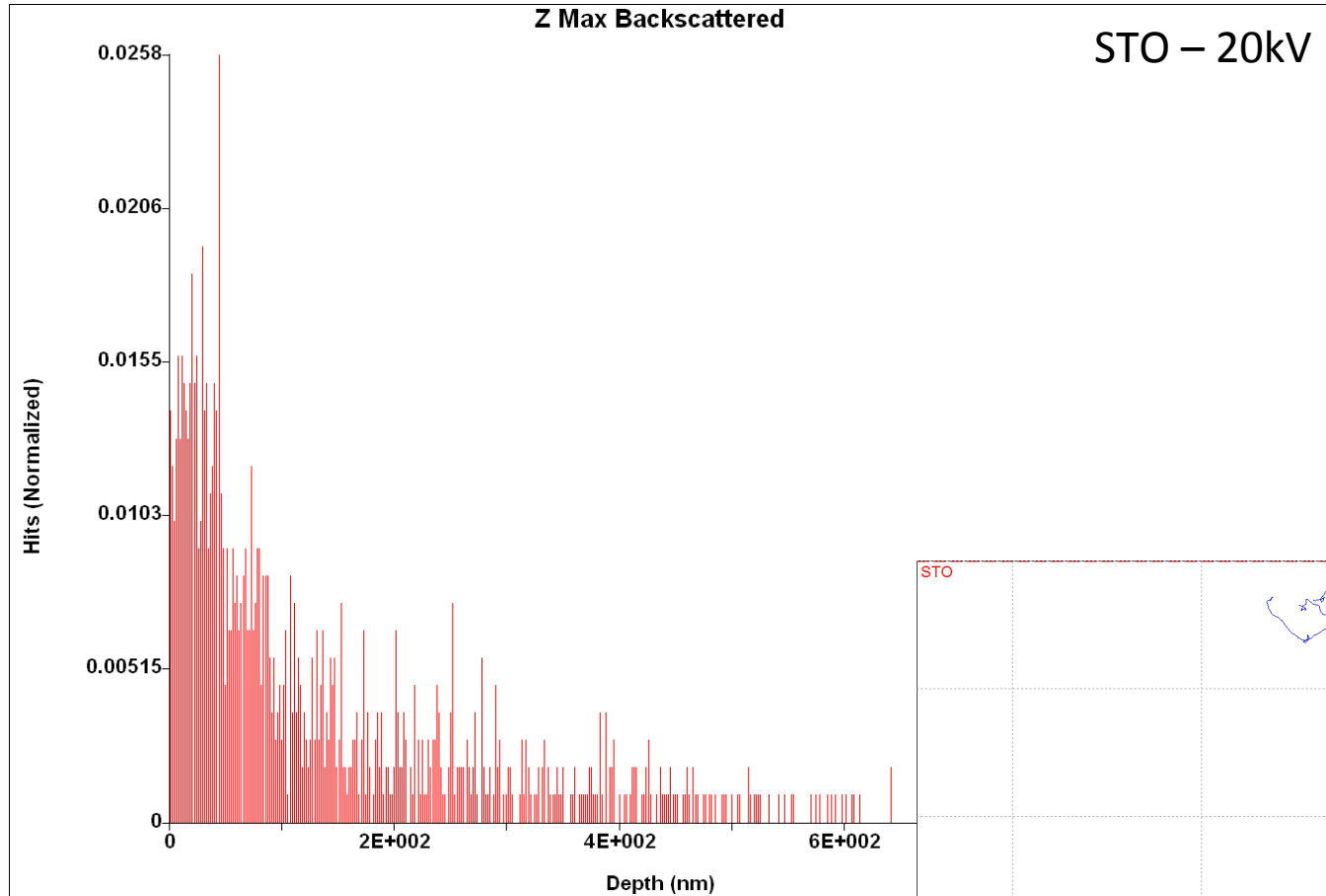
100 μ m

Laser Ablated Strontium Titanate

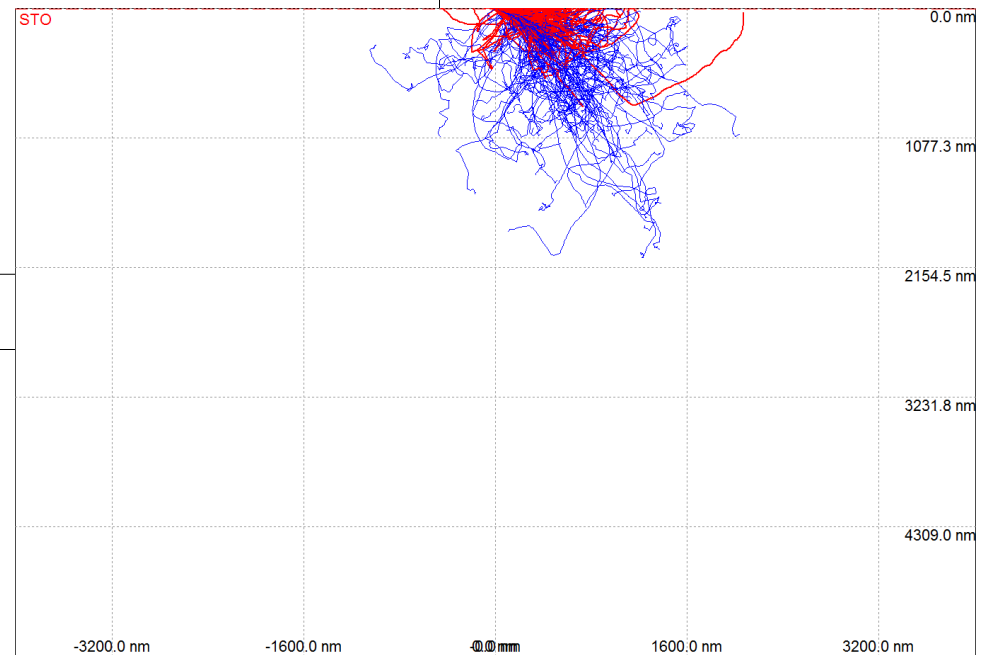


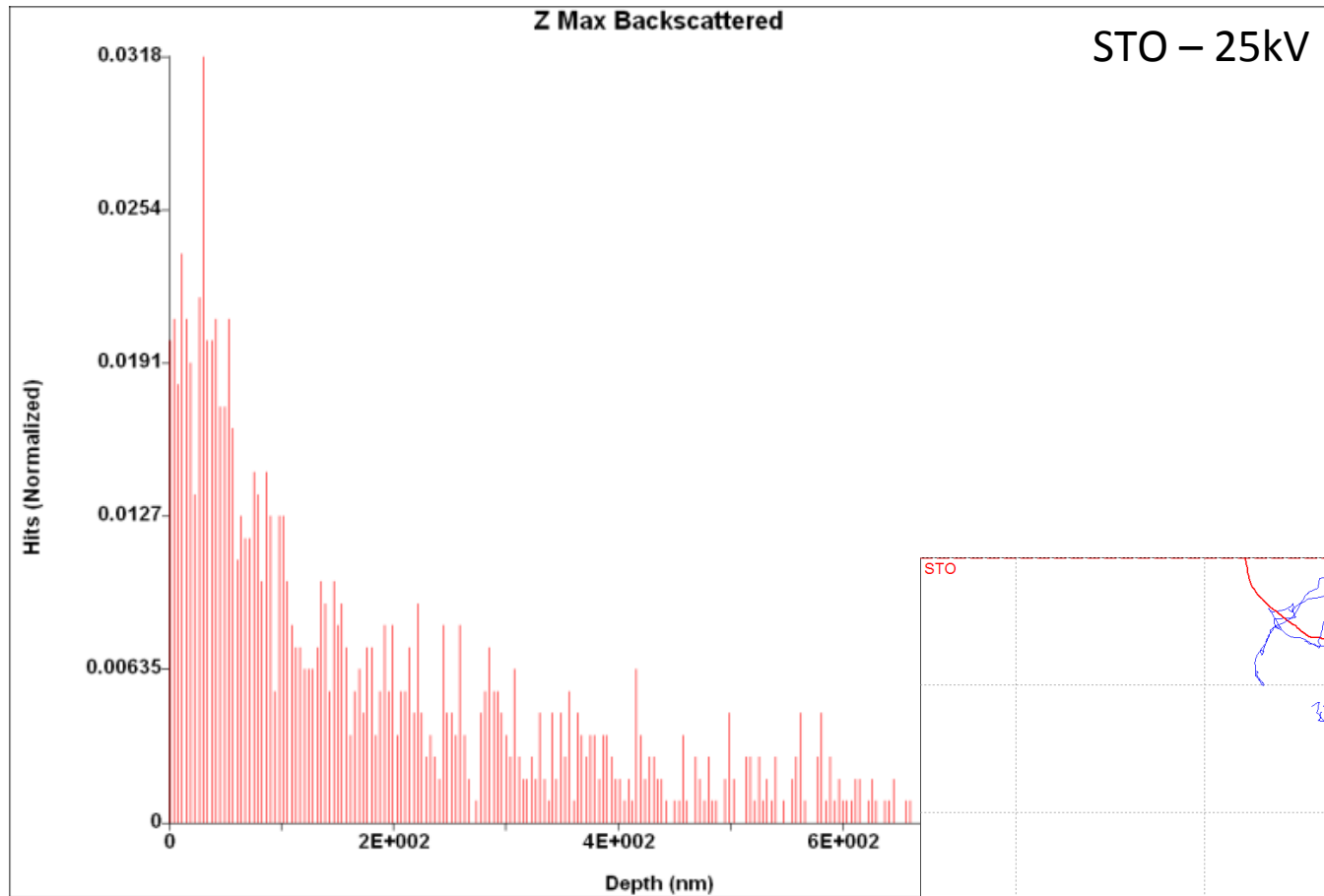
100 μ m

- Direct EBSD mapping of laser ablated surfaces in Ni, Mg, Ti, STO
- Surface laser damage is limited to $<$ the sampling depth for EBSD
- Non-systematic locations of pixels / pixel groups that do not index
- EBSD resolution $<$ 0.5 μ m/pixel

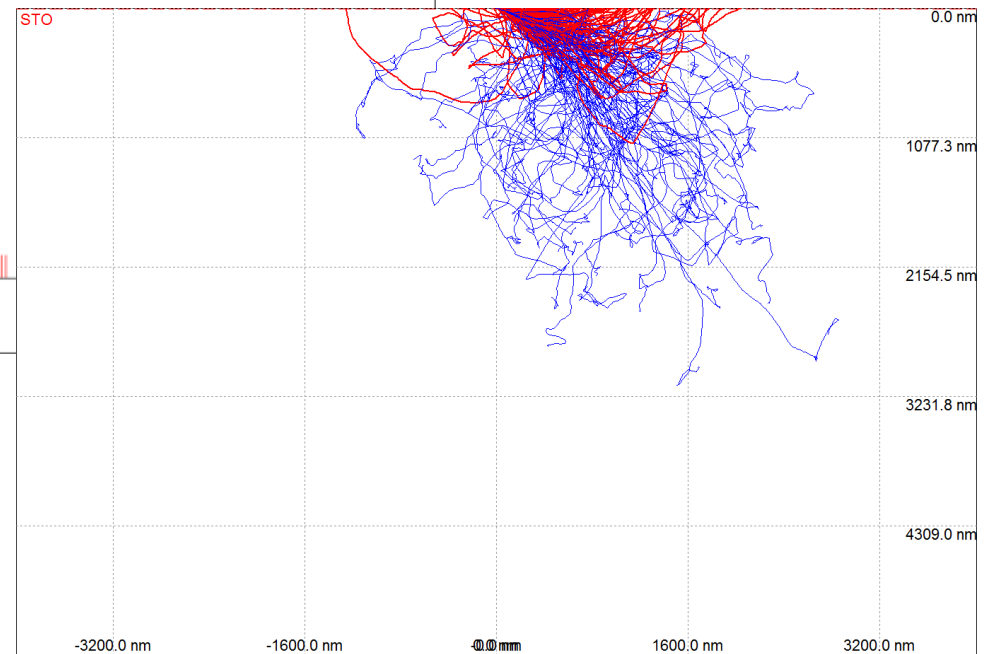


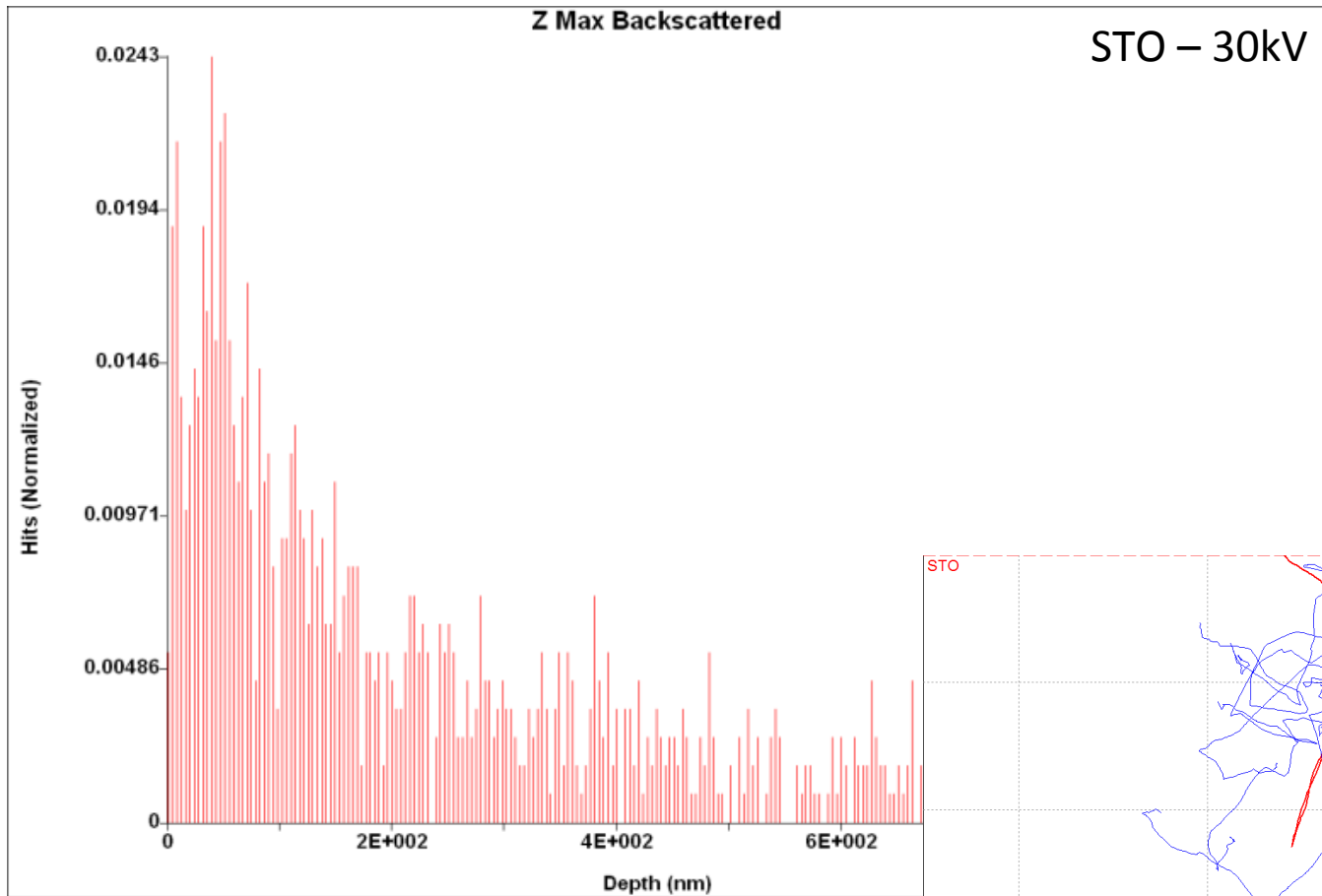
Depth < 150 nm



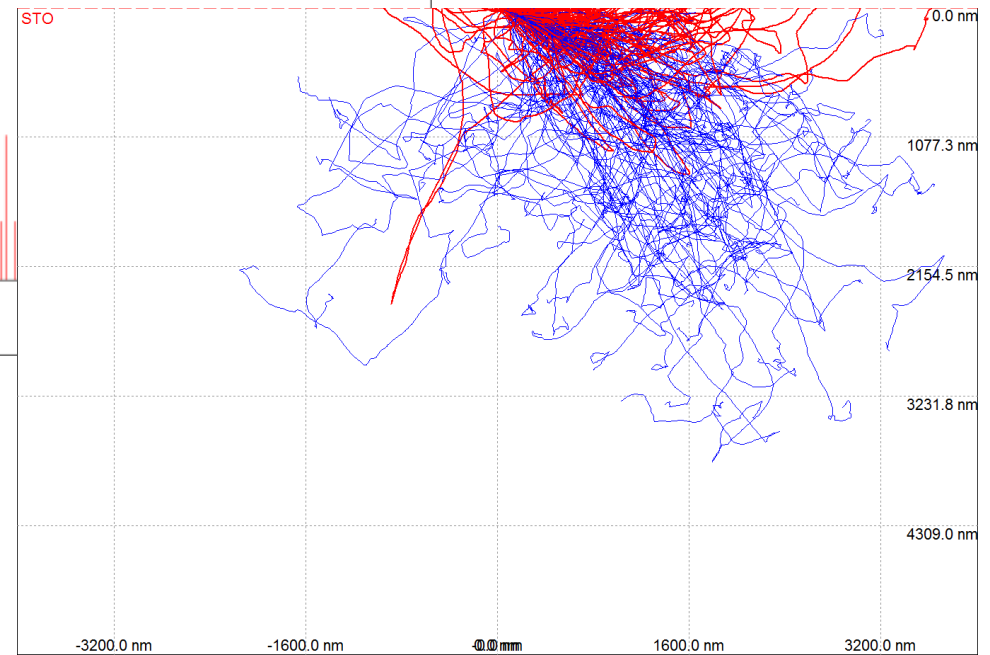


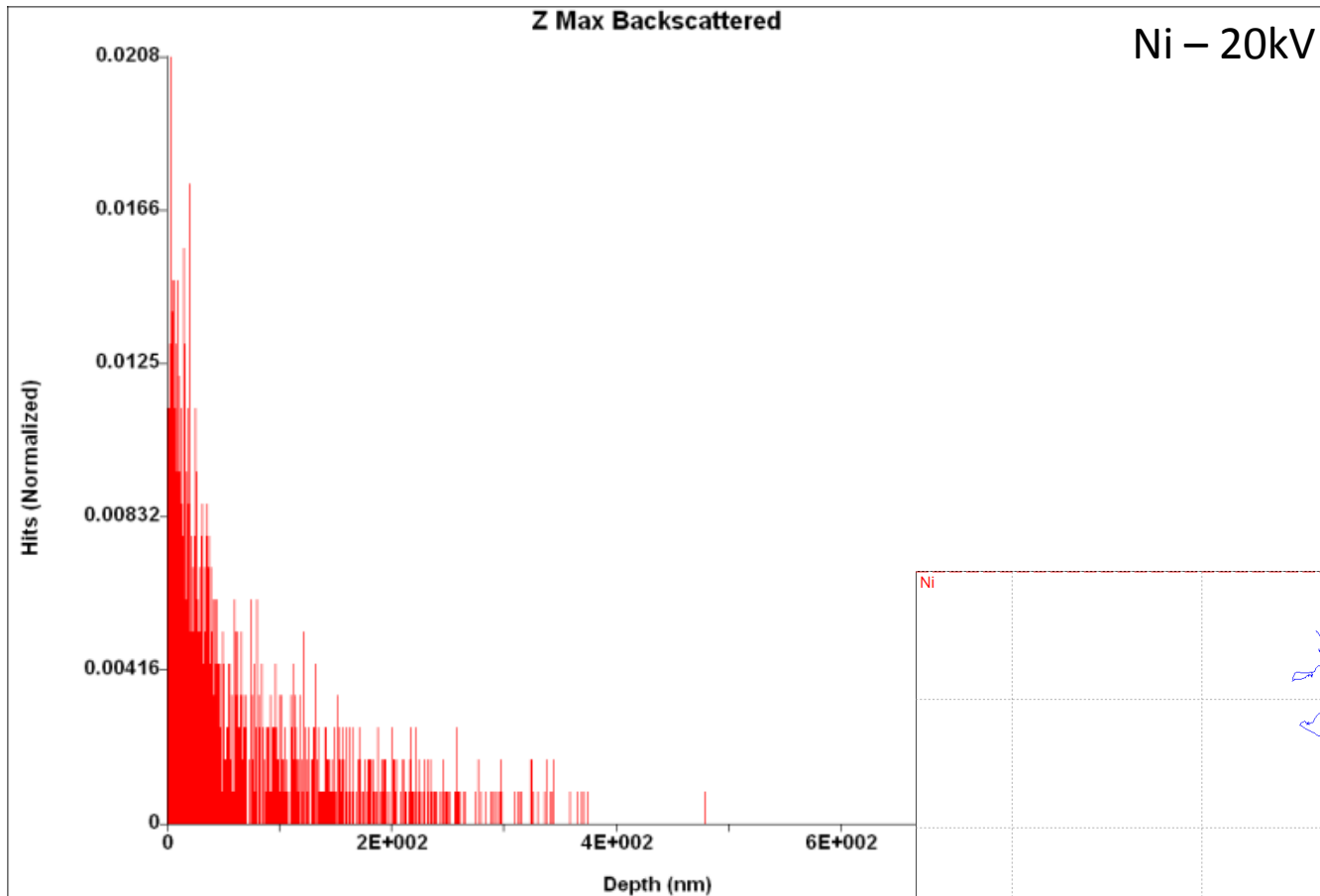
Depth < 200 nm



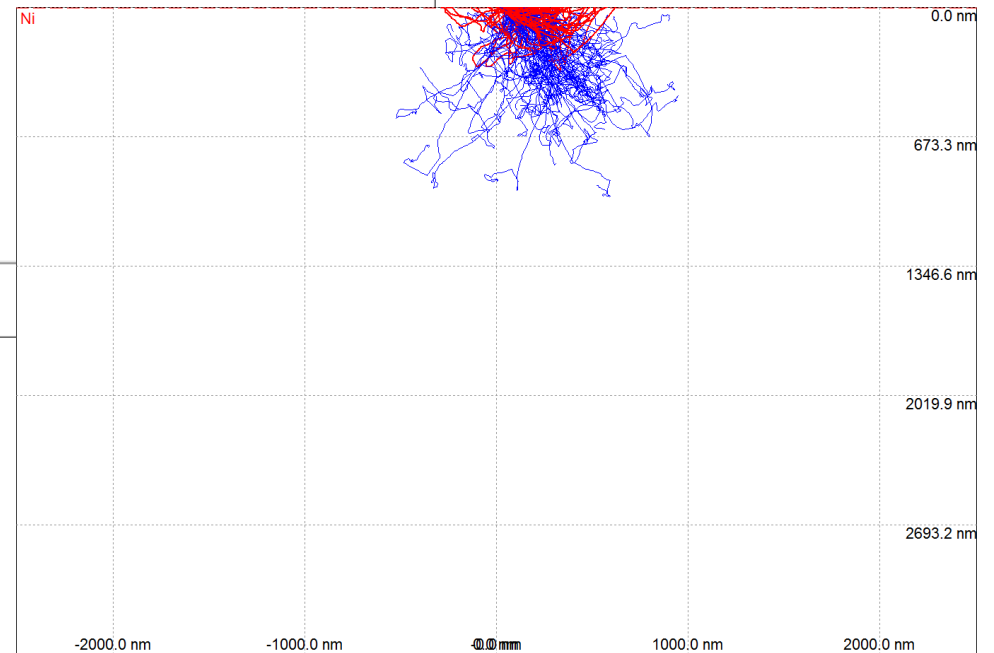


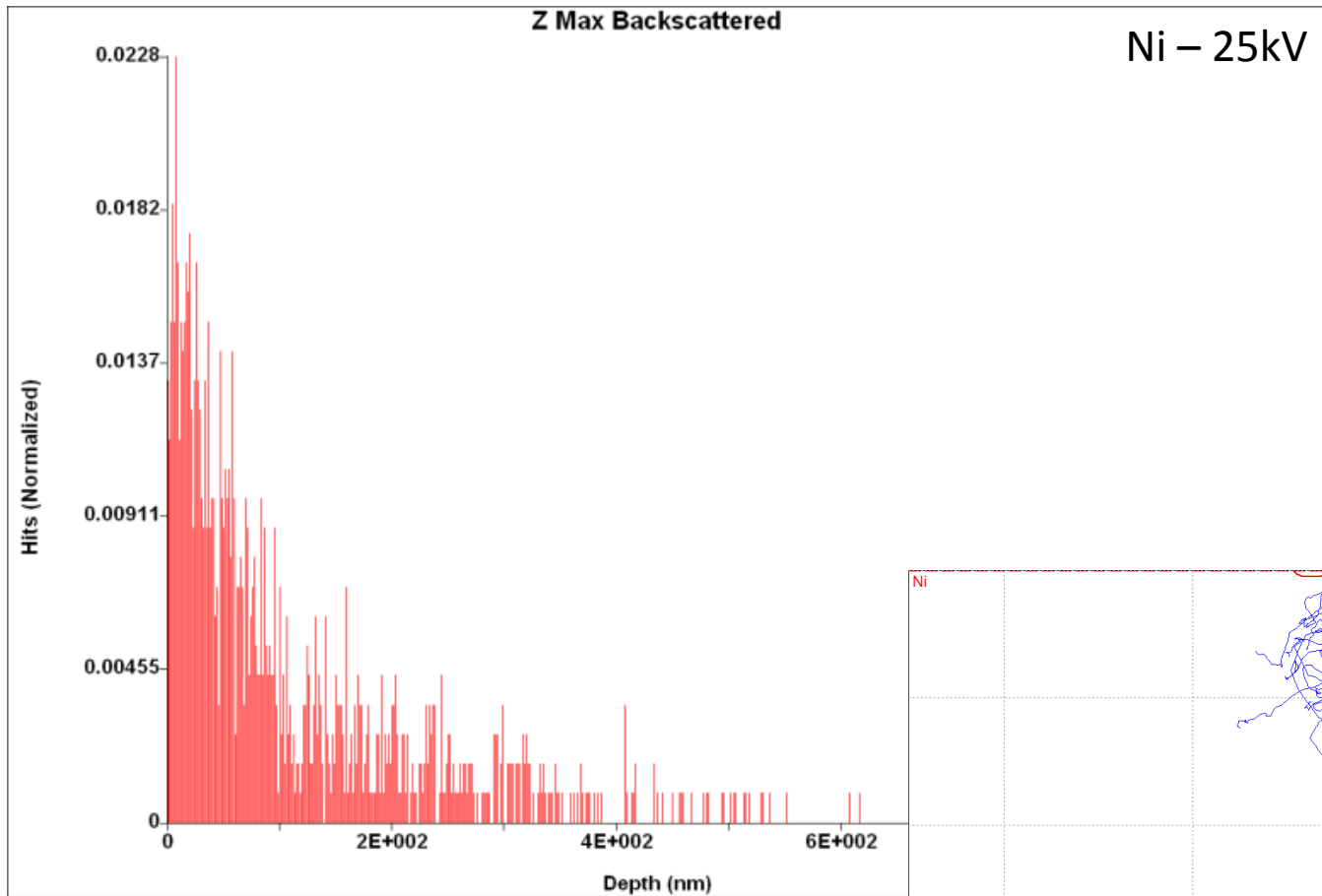
Depth < 250 nm



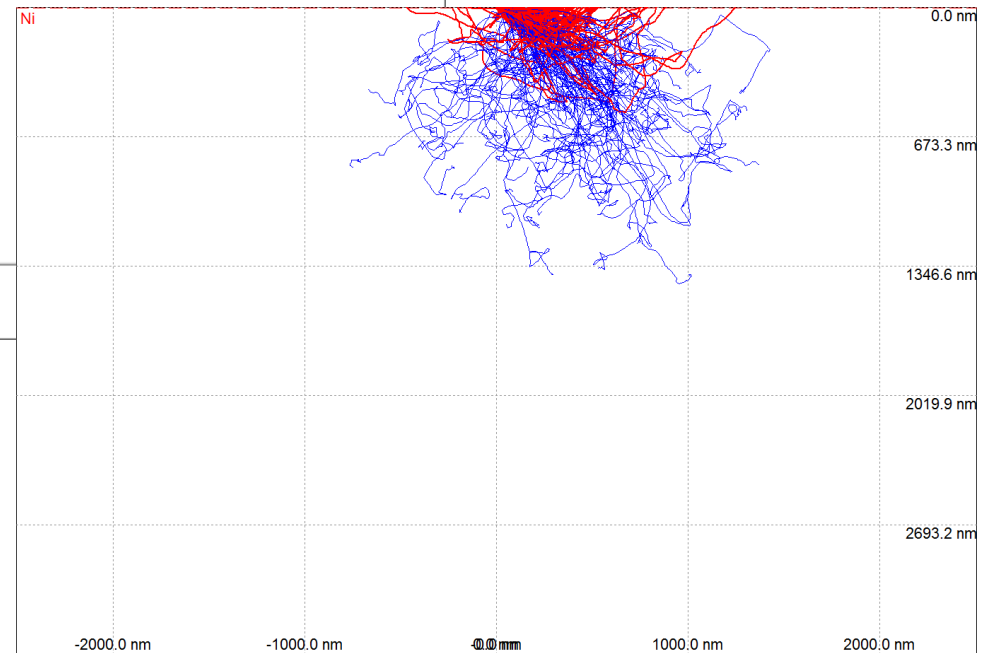


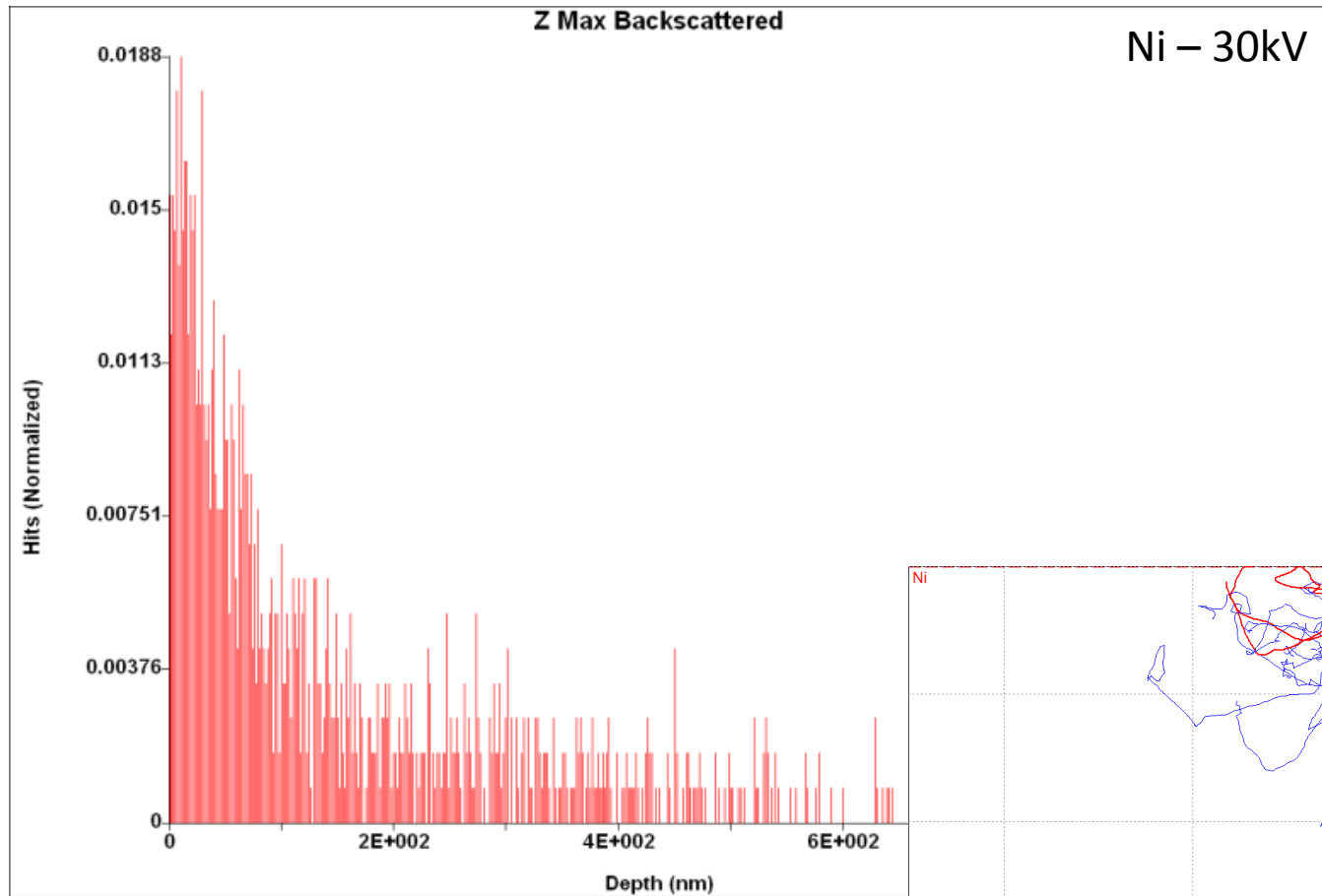
Depth < 100 nm



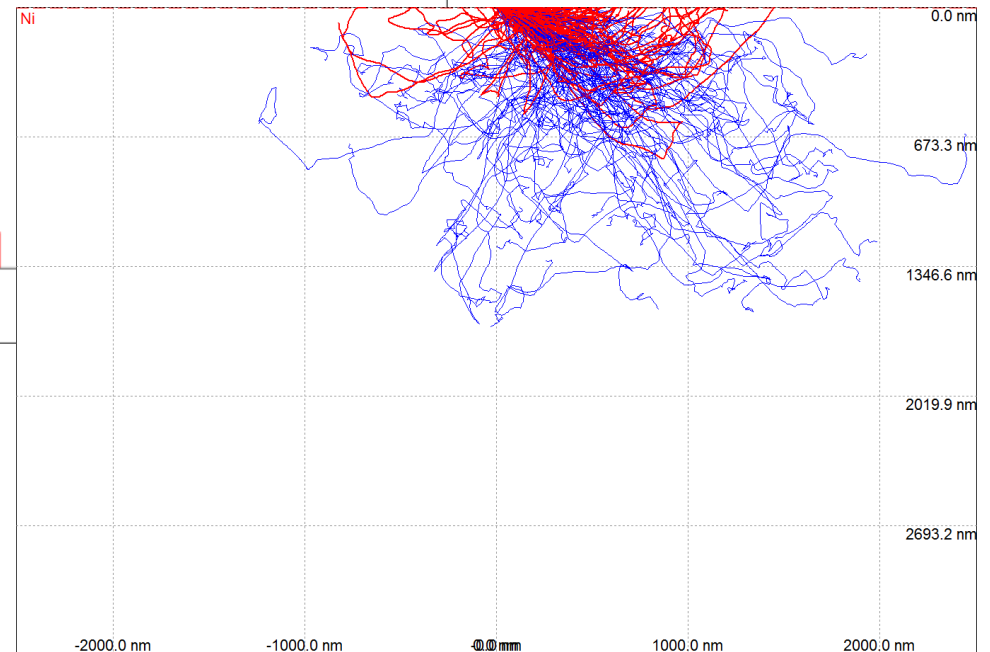


Depth < 150 nm

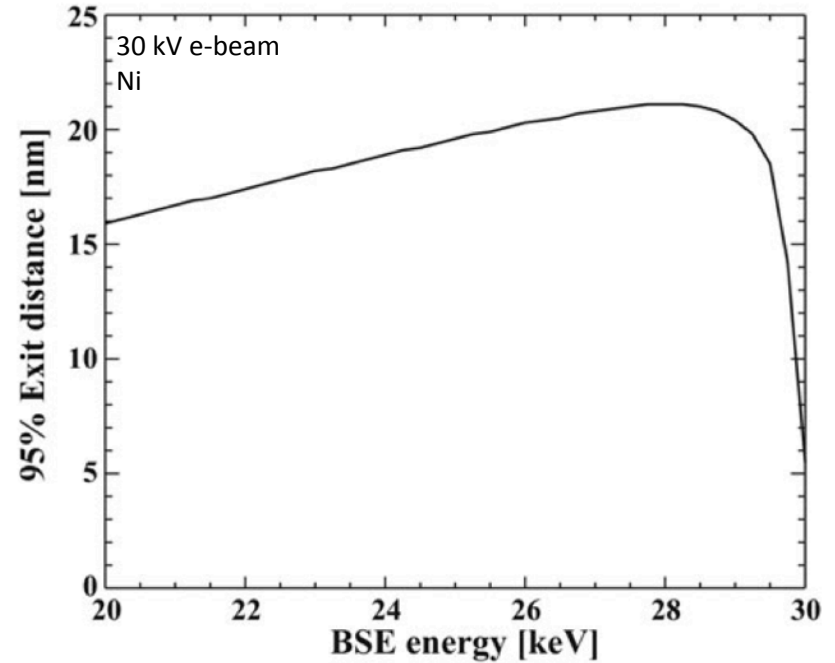
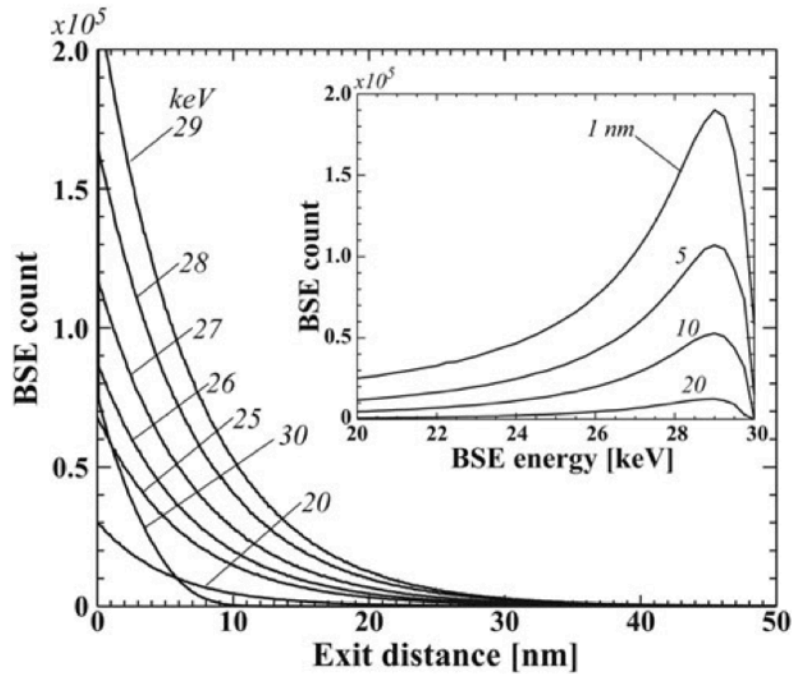


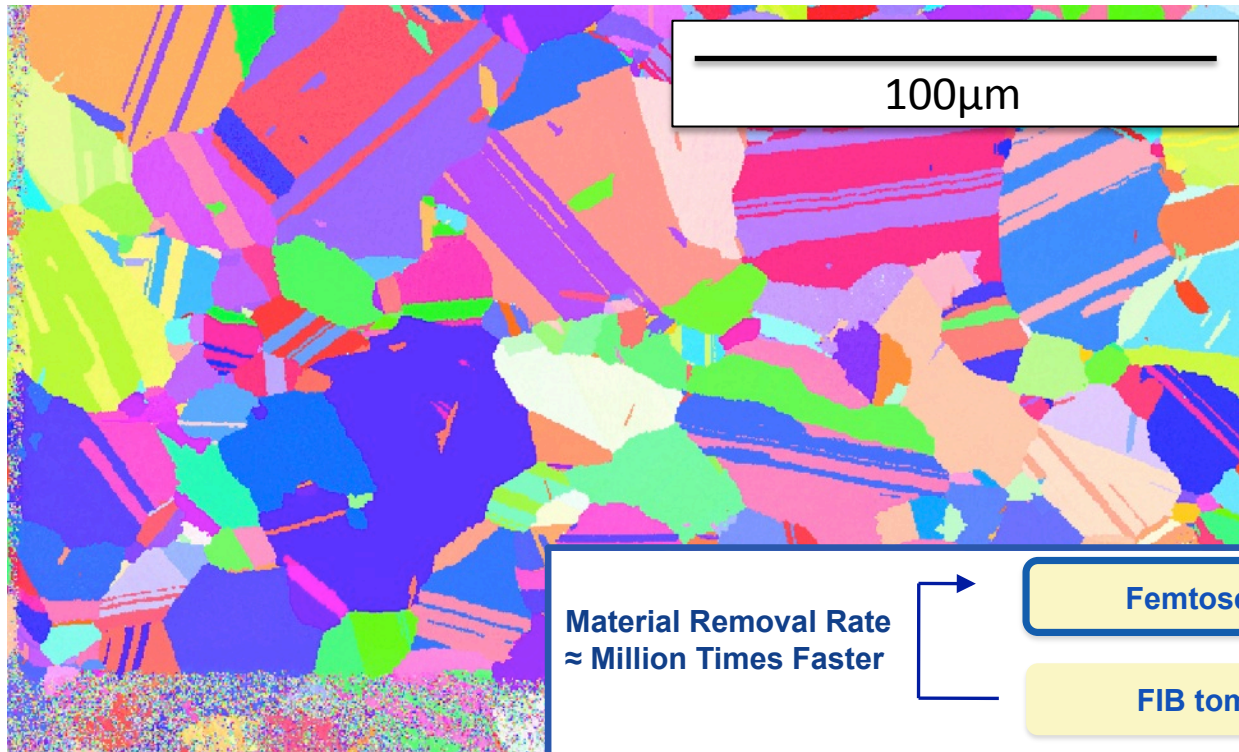


Depth < 200 nm



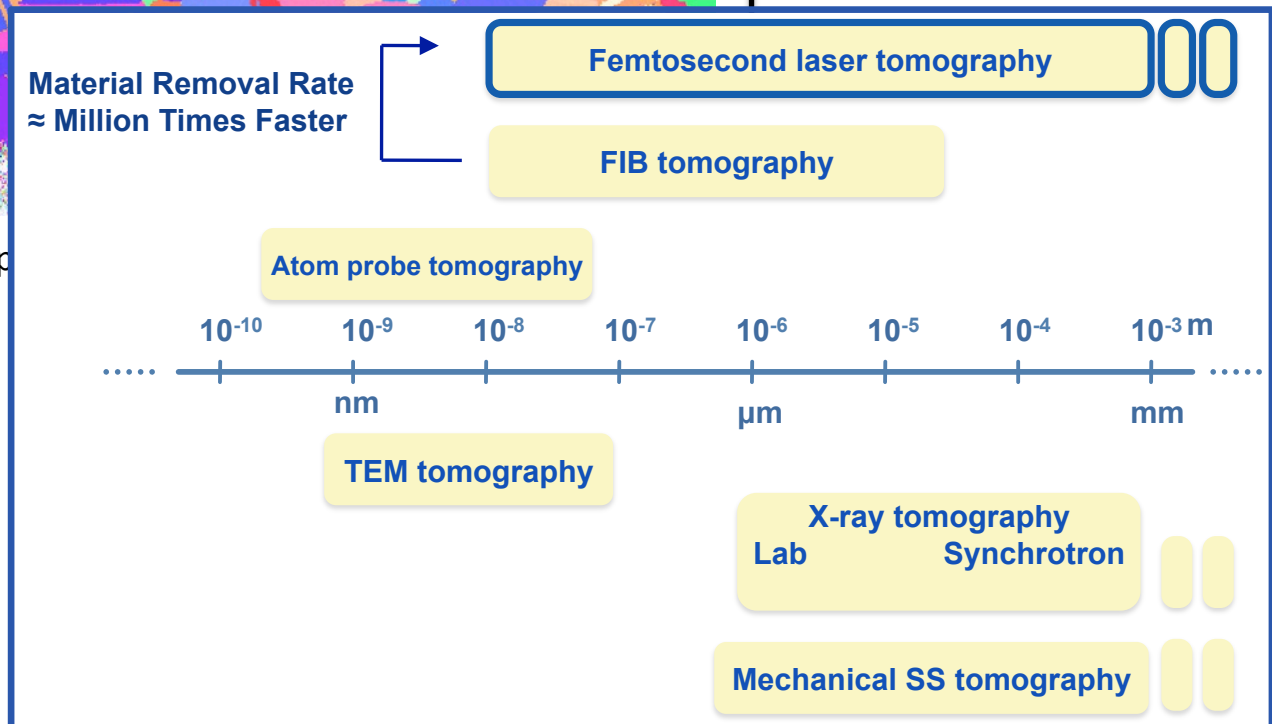
Ni





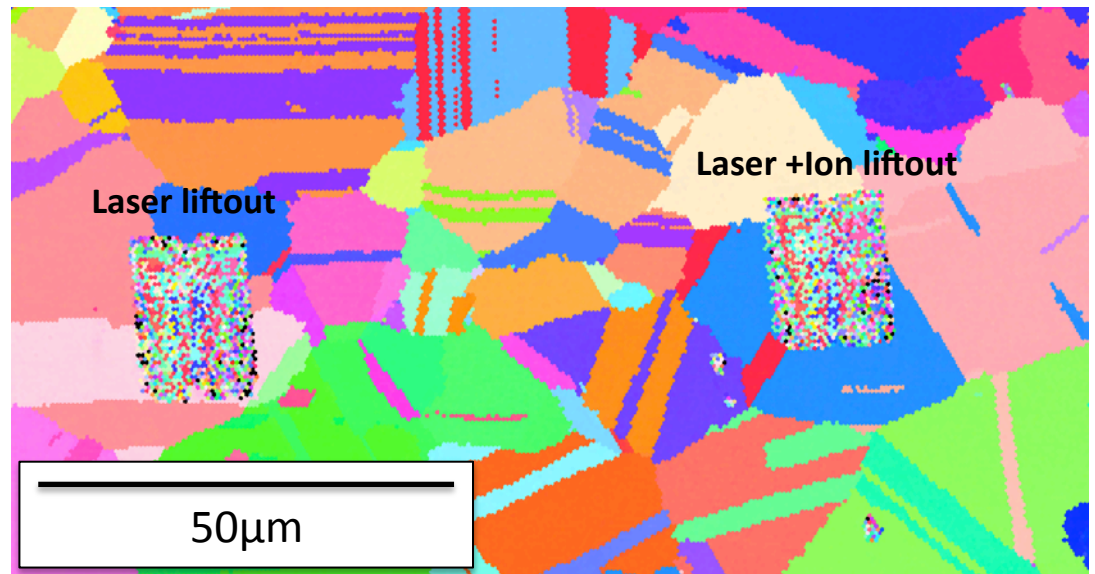
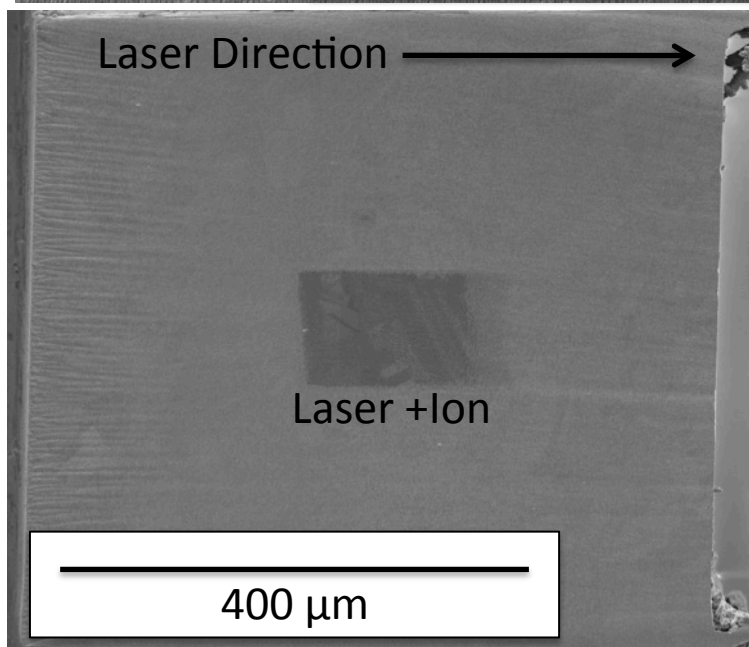
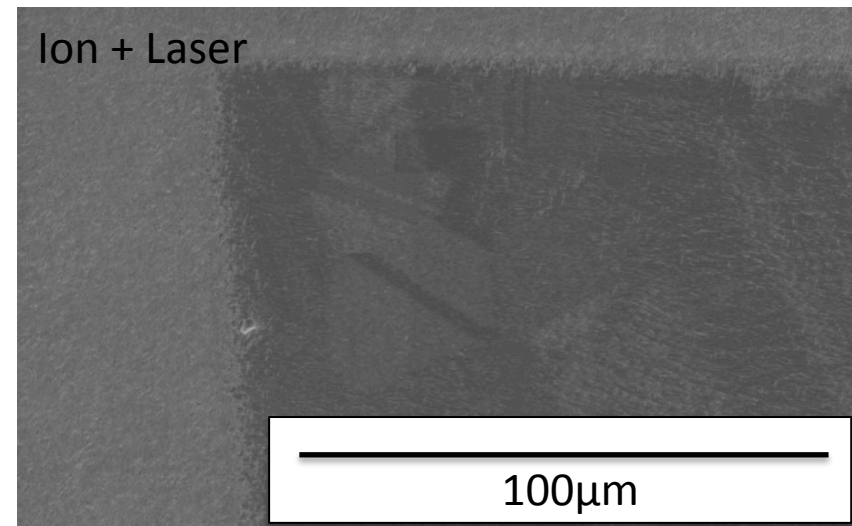
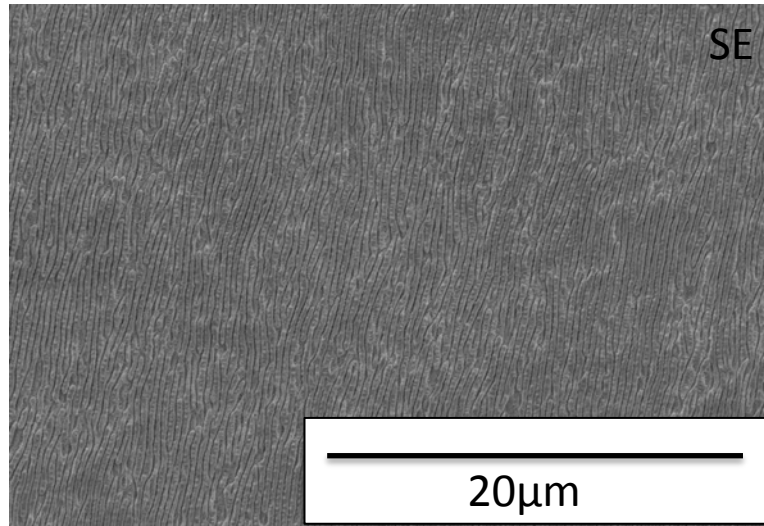
Fs. Laser + FIB

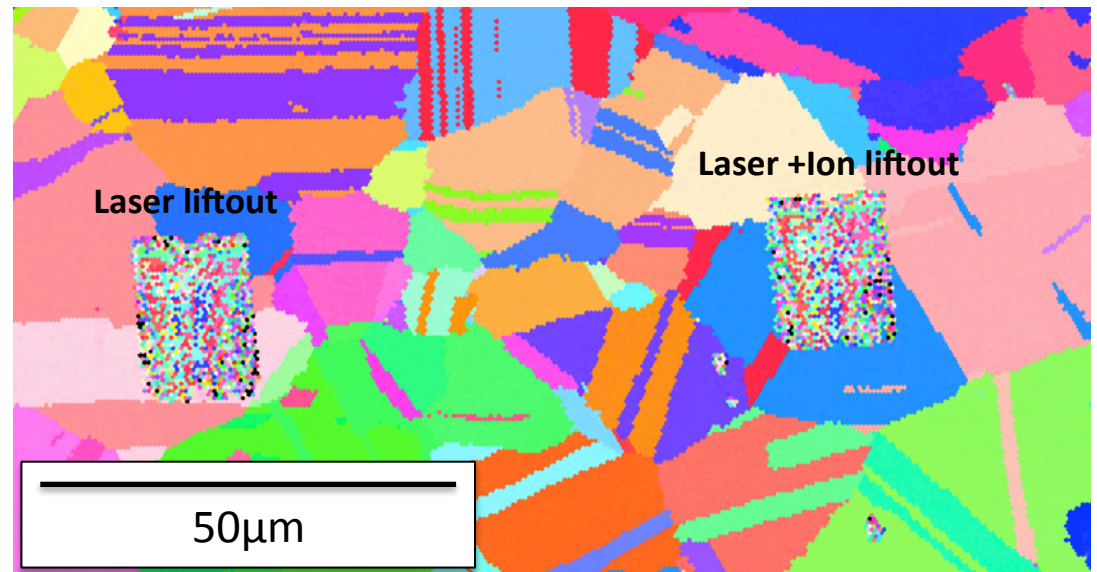
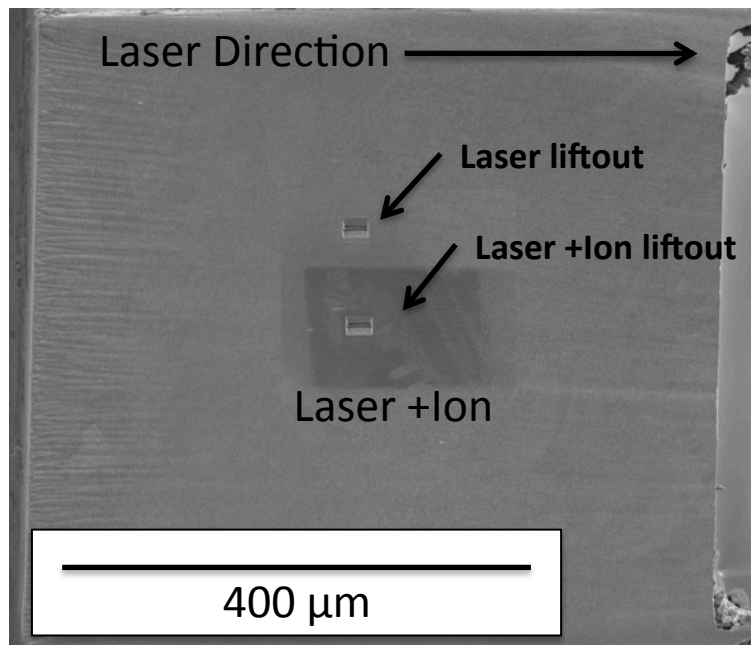
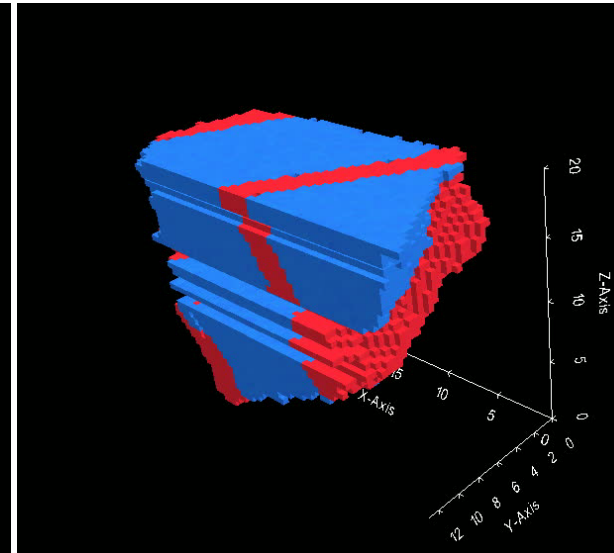
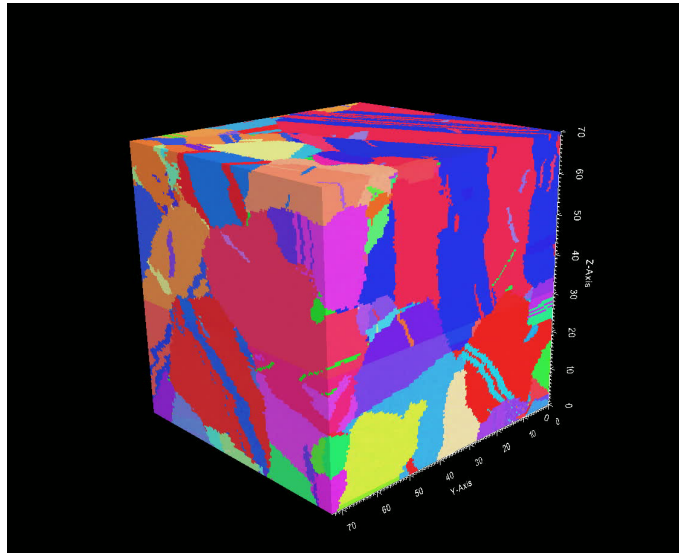
Ion Milling: 30kV, 20nA, 10° p



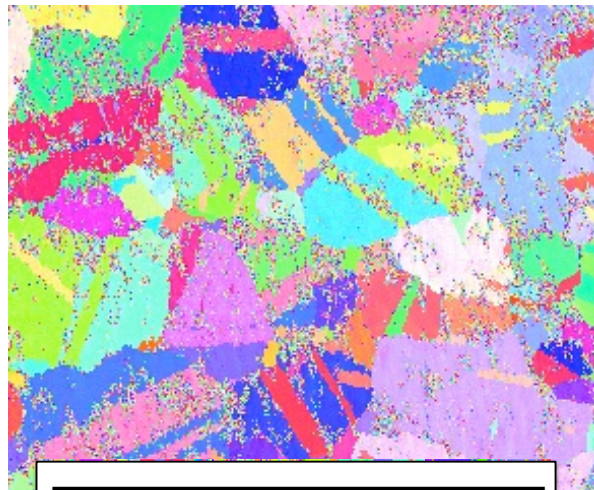
Fs. Laser + FIB in Rene88DT Ni Alloy

Laser Induced Periodic Structures



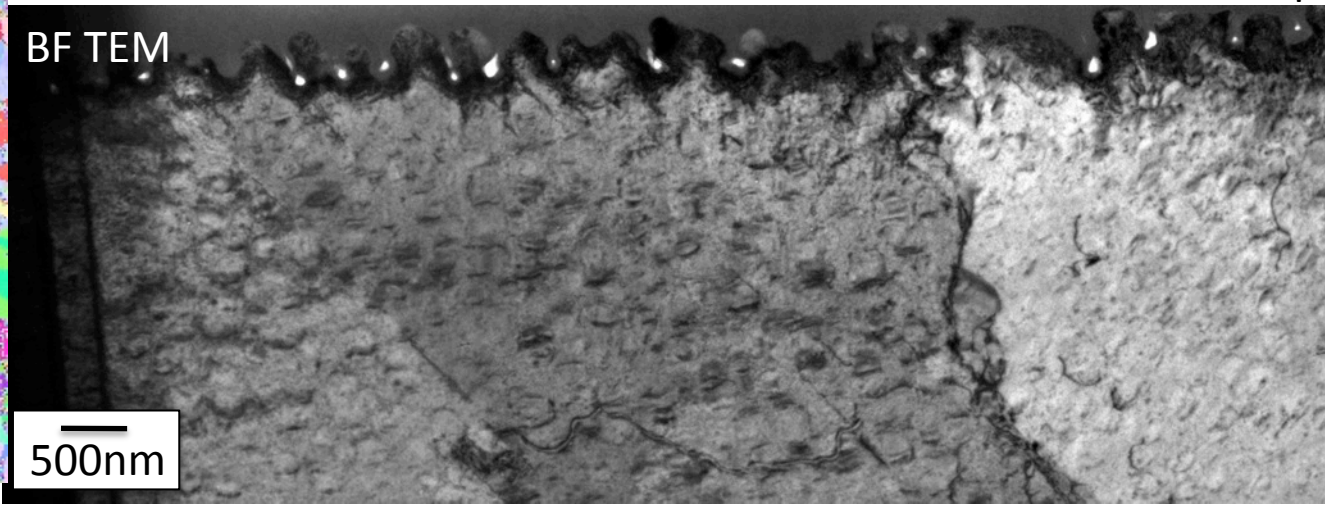


Laser Ablated



100 μ m

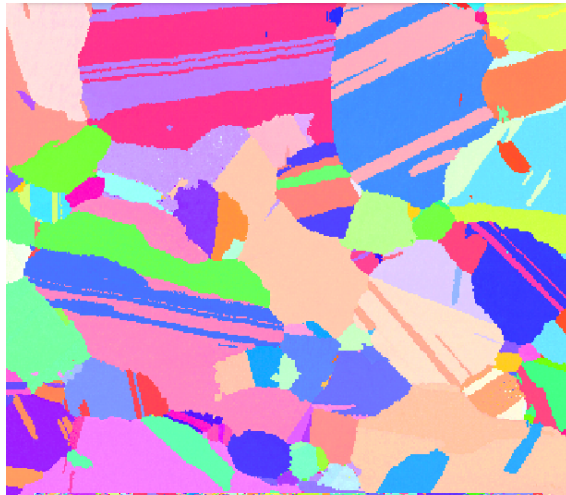
BF TEM



500nm

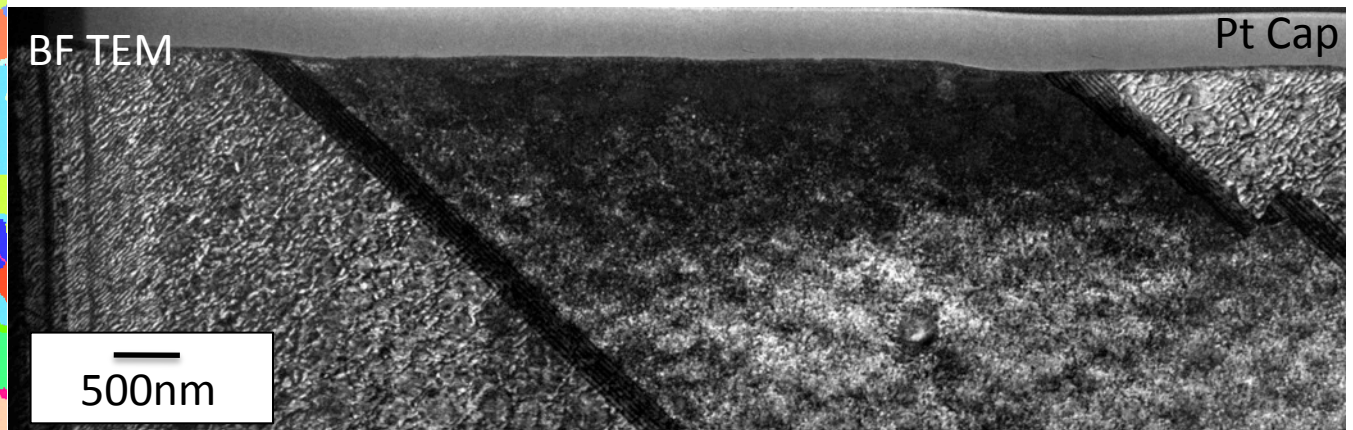
Pt Cap

Laser Ablated + FIB Milled



100 μ m

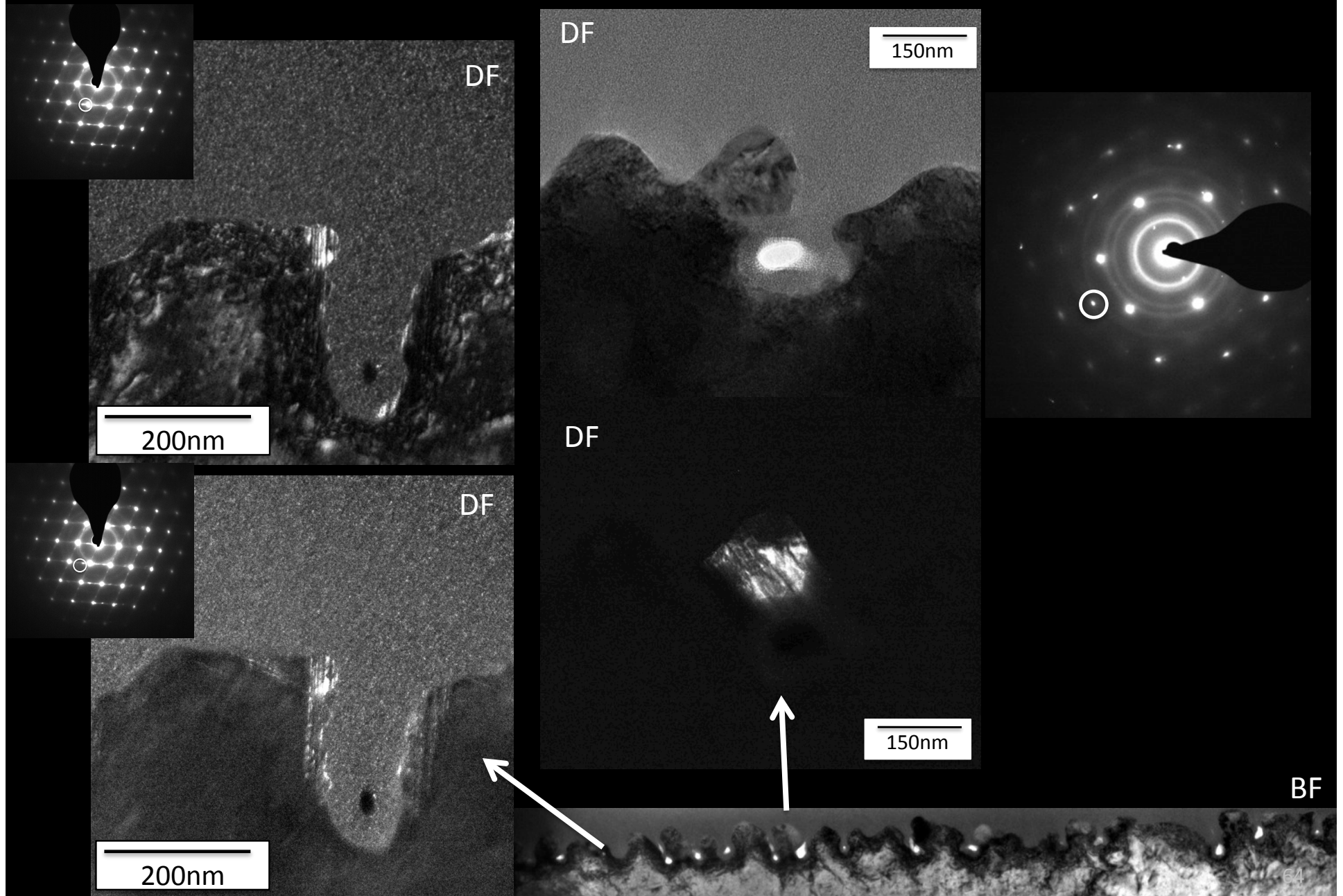
BF TEM



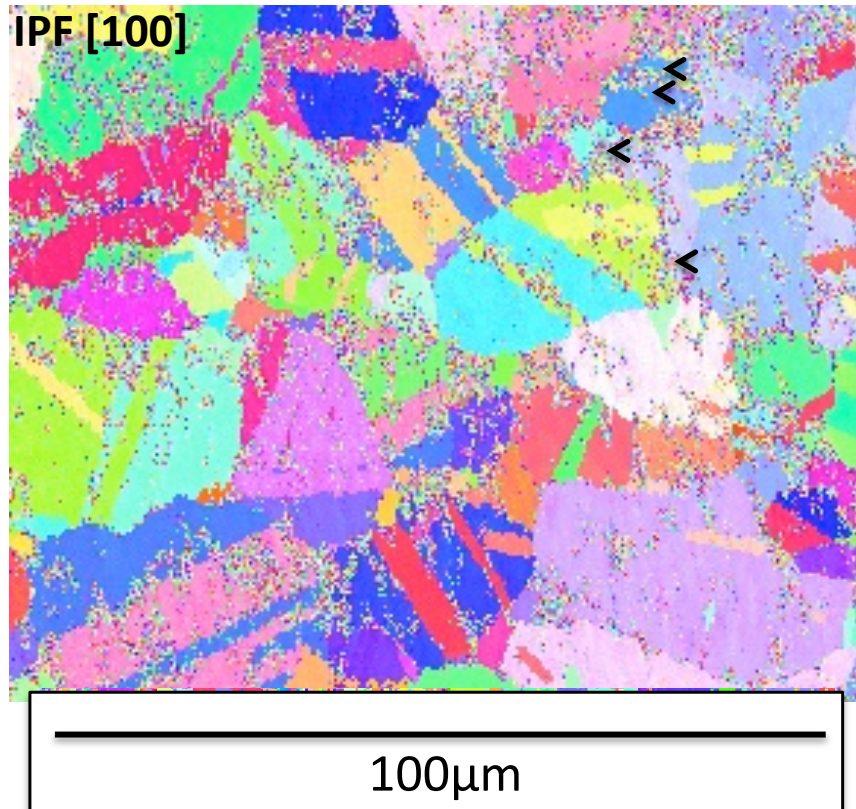
500nm

Pt Cap

FIB parameters: 30kV, 20nA, <10° glancing angle



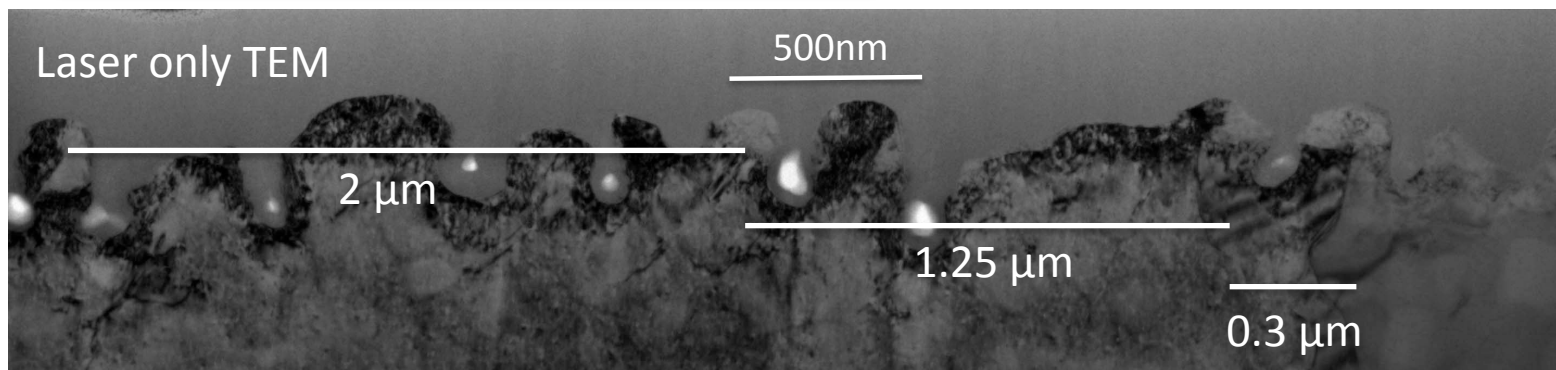
Rene88DT – Fs. Laser Ablated Surface



Examples of non-indexed or poorly indexed EBSD patterns

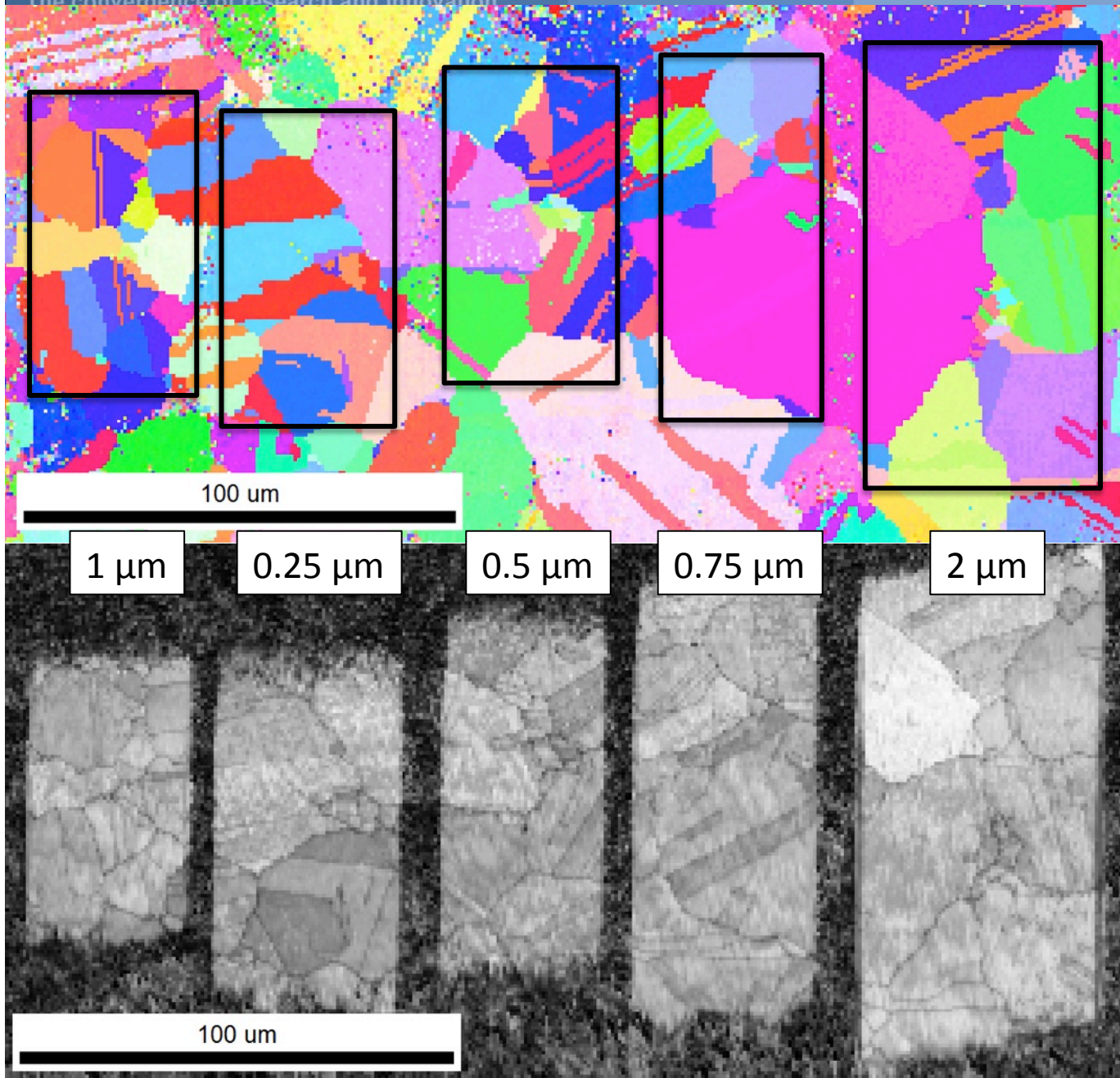
Spatial periodicity of non-indexed points correlates with LIPSS and recrystallization

Need to confirm by EBSD mapping a surface, and liftout TEM from a known location



Optimizing FIB Milling Parameters for EBSD

The convergence of research and innovation



FIB Conditions:
30kV @ 20 nA

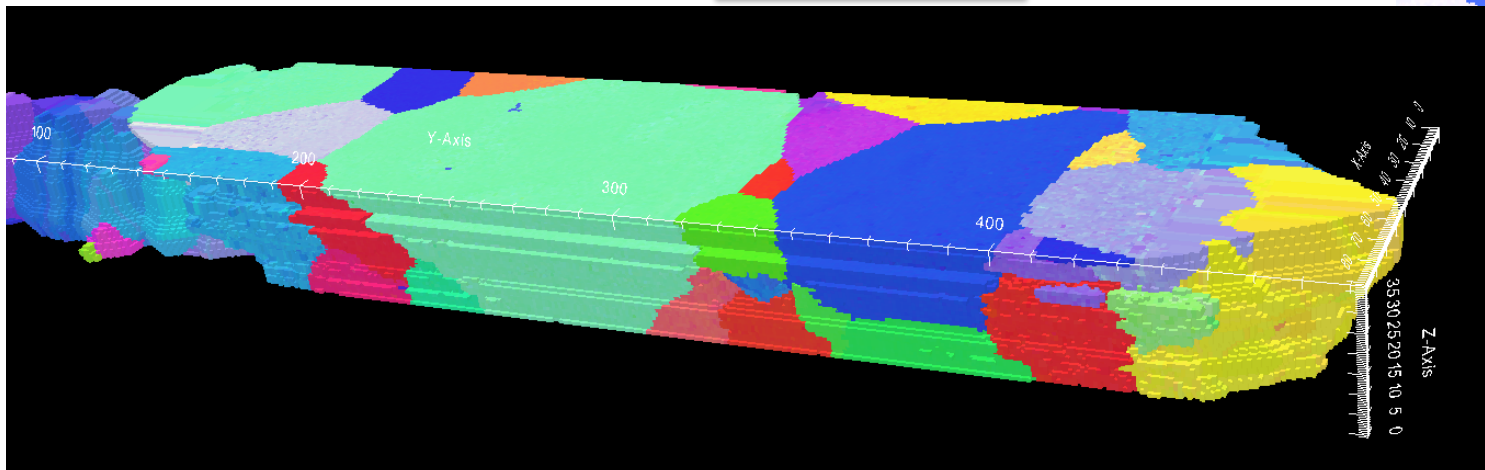
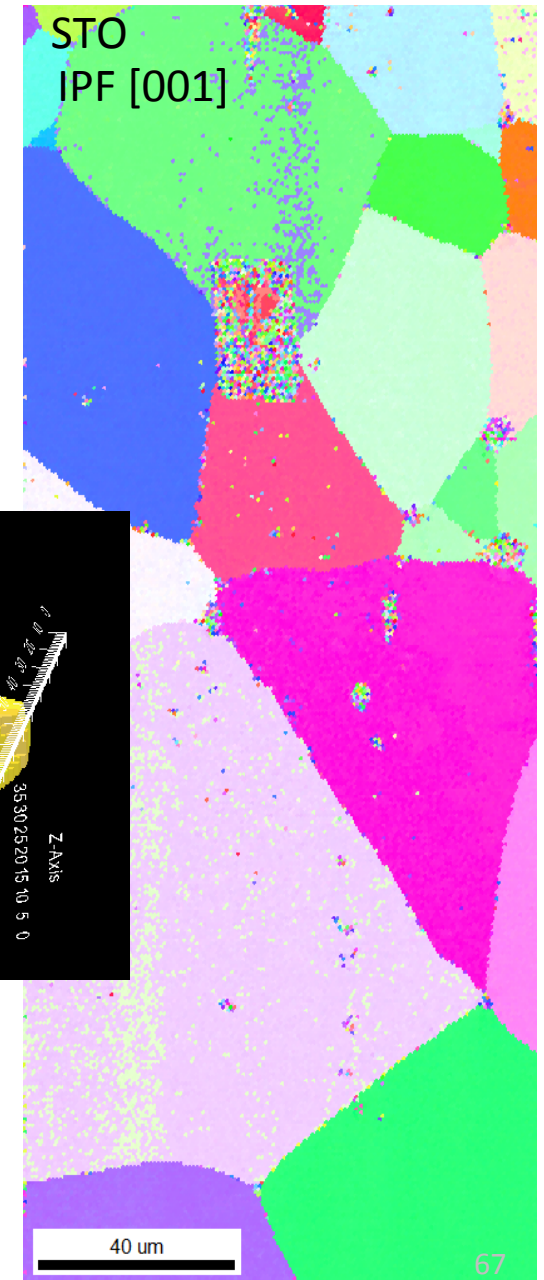
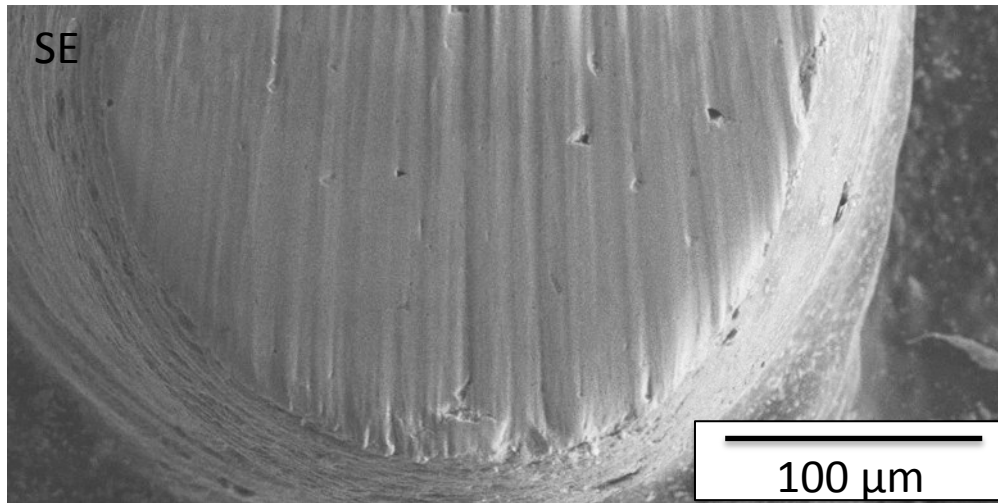
Depths calibrated for Si
with Normal FIB beam
Incidence

Ion mill depth
independent of EBSD
image quality

< 0.1-0.2 μm threshold
FIB mill depth for
dramatic improvement
in EBSD

Why?

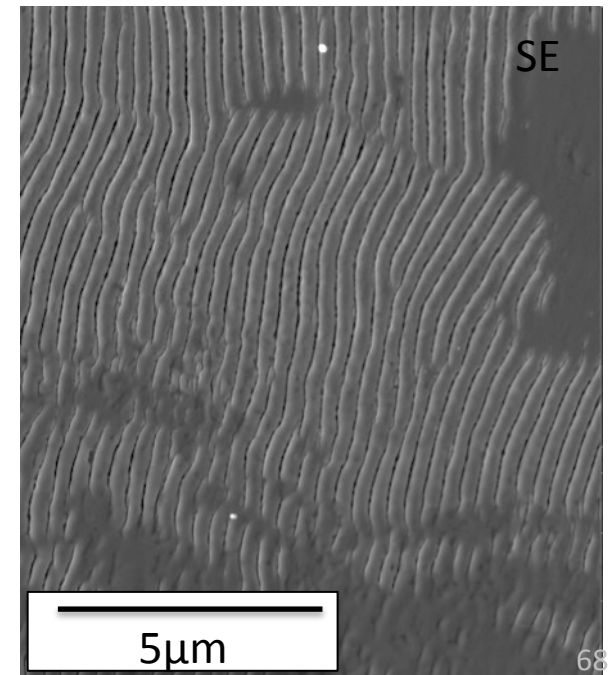
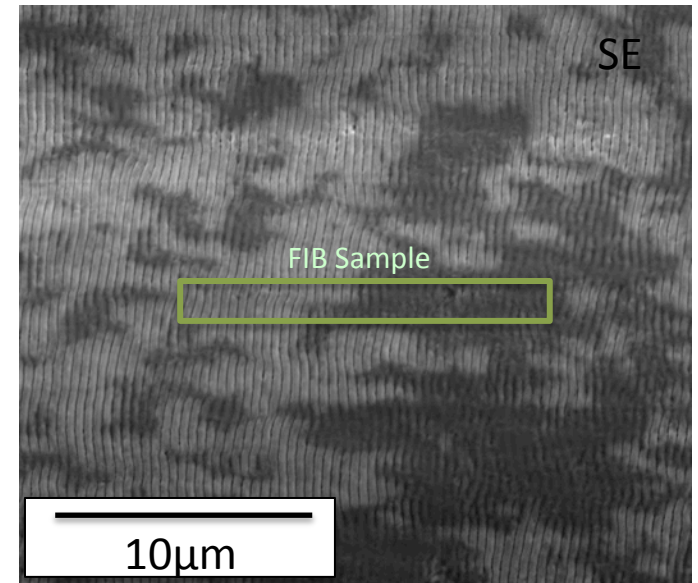
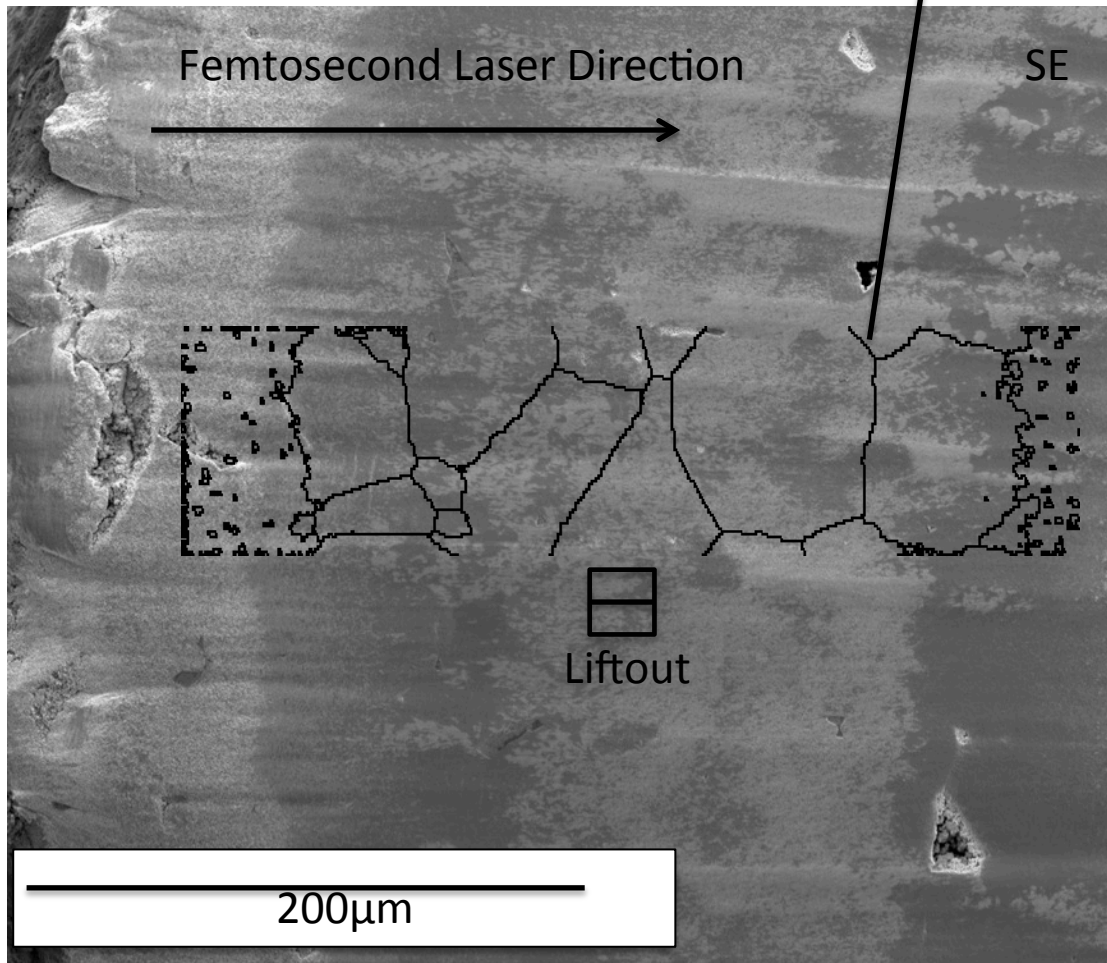
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- EBSD patterns can be imaged from a relatively narrow range of Fs. laser machining fluences
- Would like to expand the area over which EBSD patterns can be indexed to the entire pedestal

LIPSS depth:
darker contrast – smoother
lighter contrast – rougher

EBSD grain
boundaries



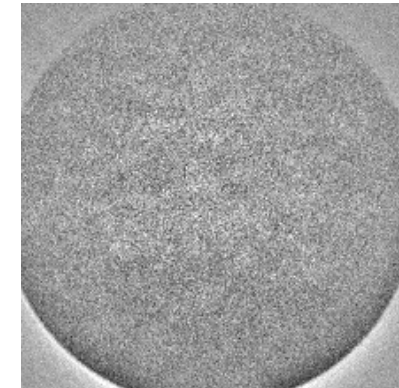
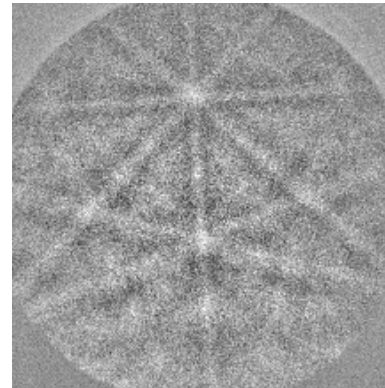
Near glancing angle FIB milling in STO

5kV 13 nA FIB mil

30 kV 65nA FIB mill

30kV

5kV



- Laser ablated surface milled at $<10^\circ$ at both 5kV and 30kV
- Diffraction patterns only able to be indexed after 5kV FIB mill
- Current optimized to be highest possible for each accelerating voltage
- TEM foil liftouts required to discern STO Ga+ damage mechanisms

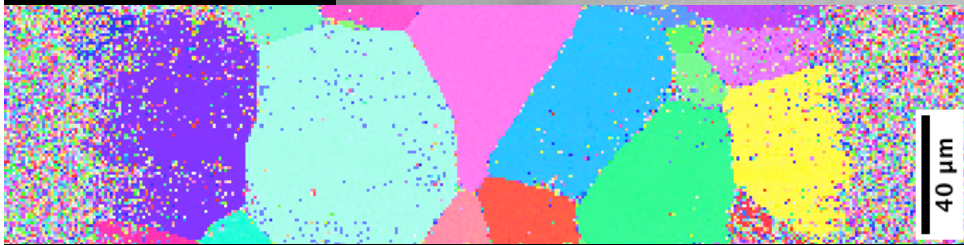
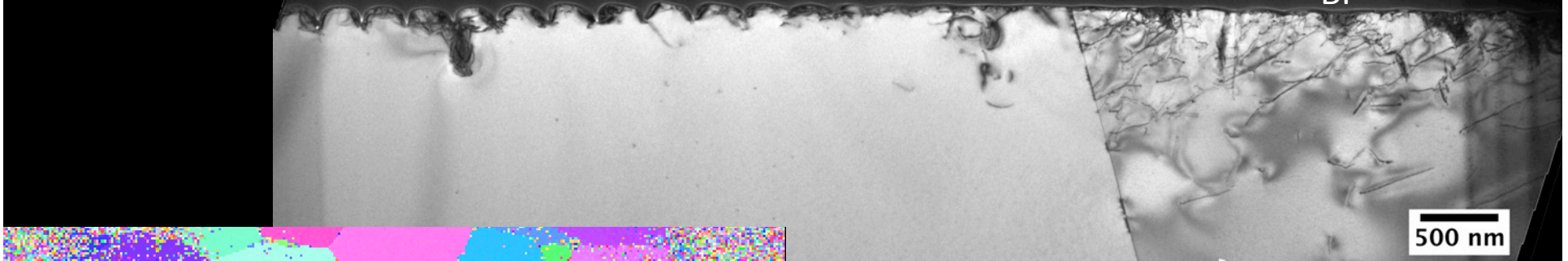
Removing Roughness with FIB in STO

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Fs Laser

BF

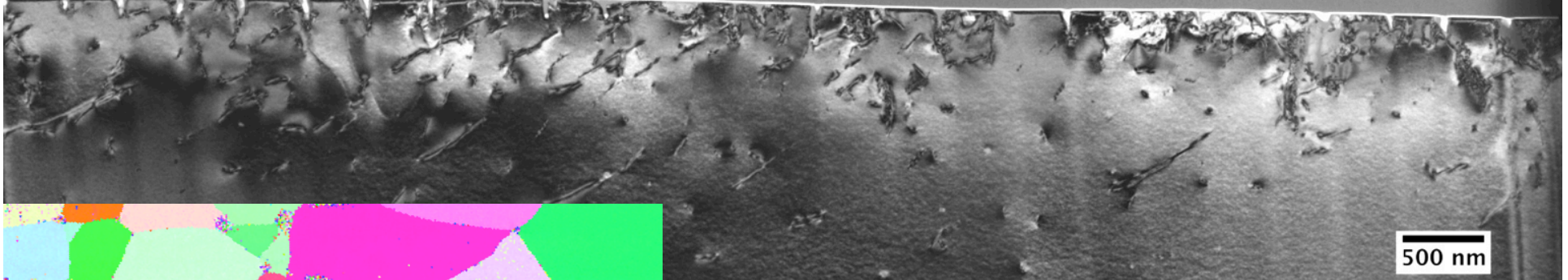
Pt



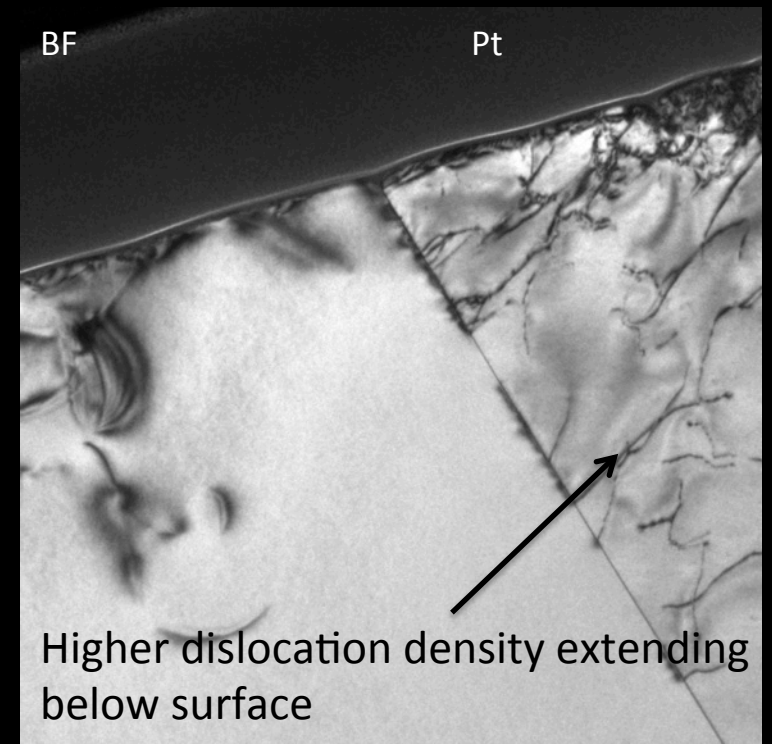
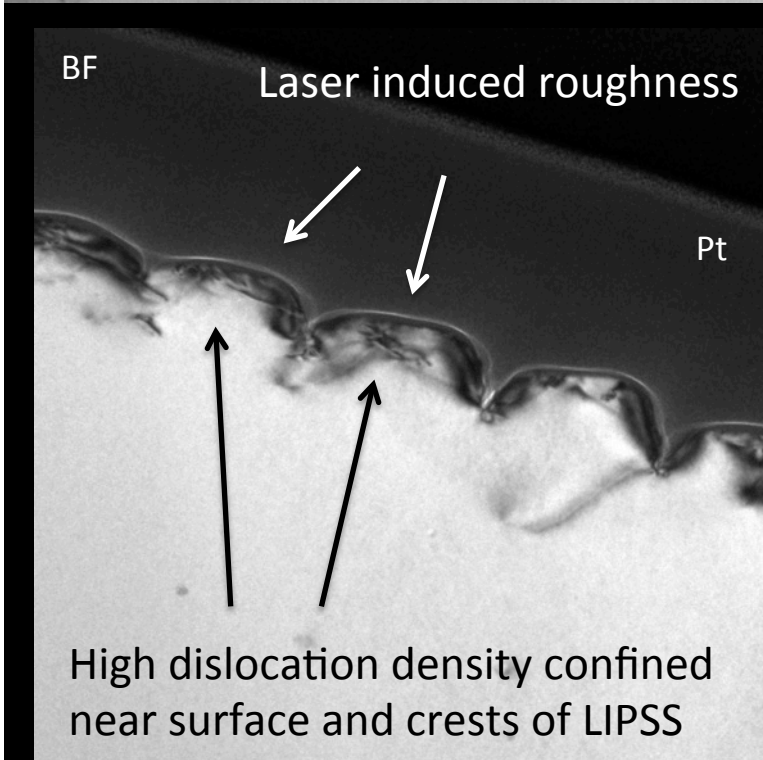
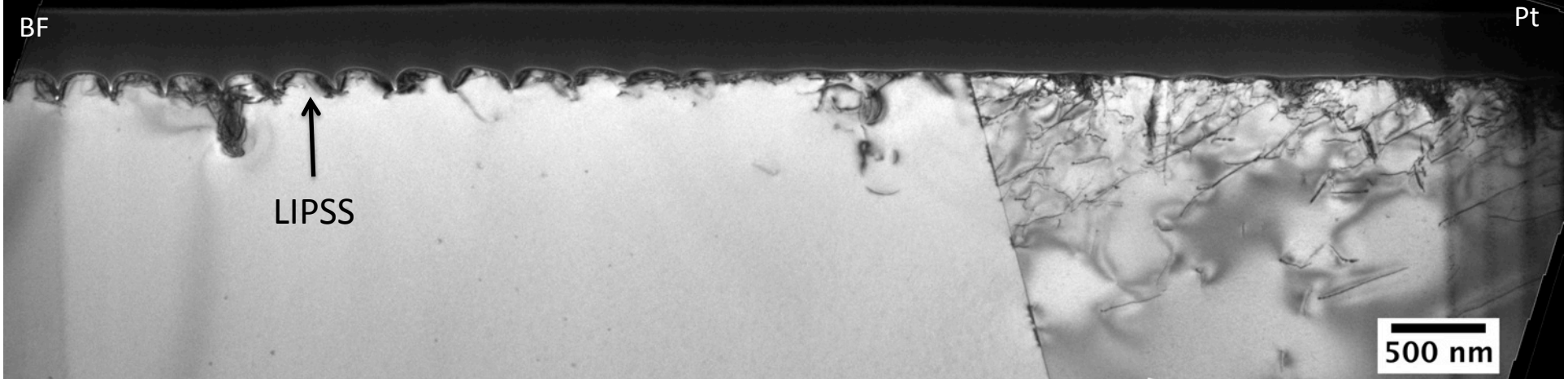
Fs Laser + 5kV FIB

BF

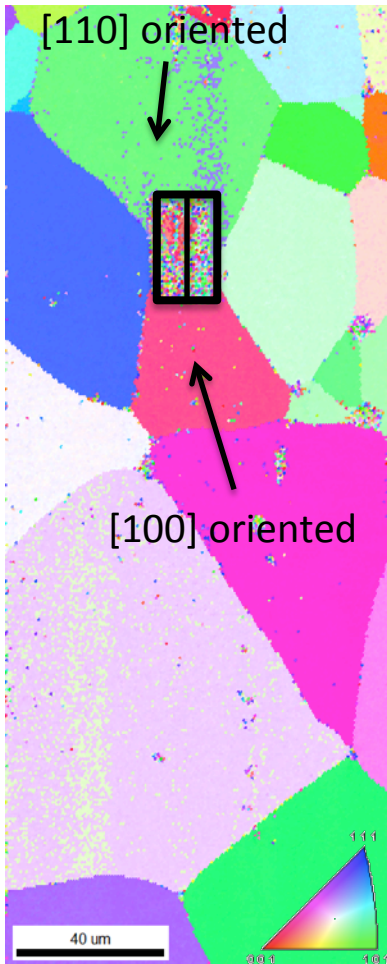
Pt



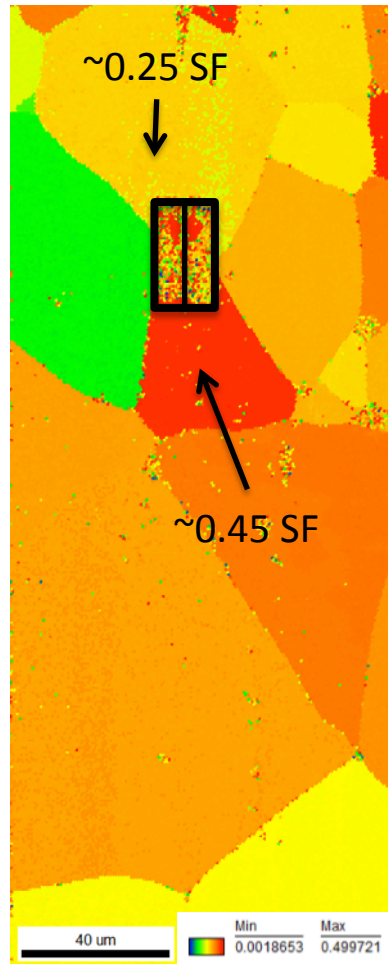
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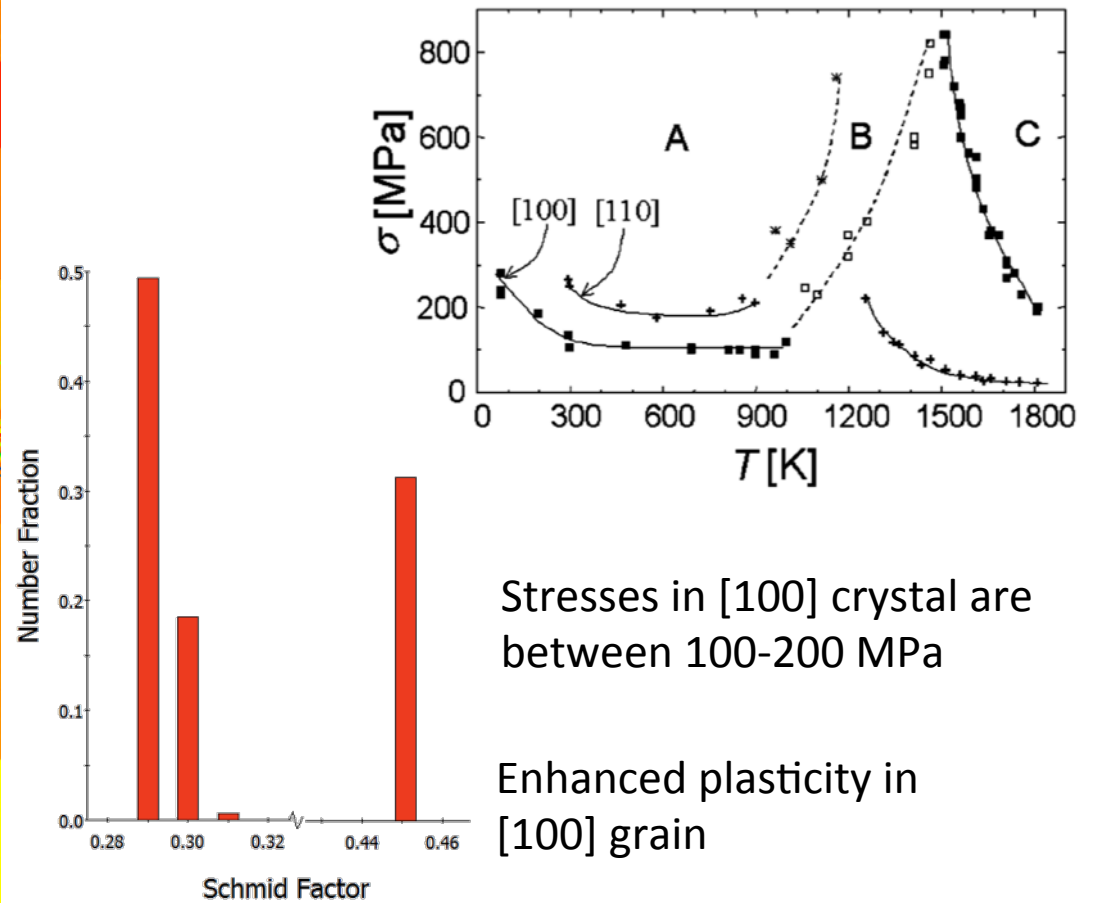
IPF Map



Schmid Factor



Flow Stress vs. Temperature in STO



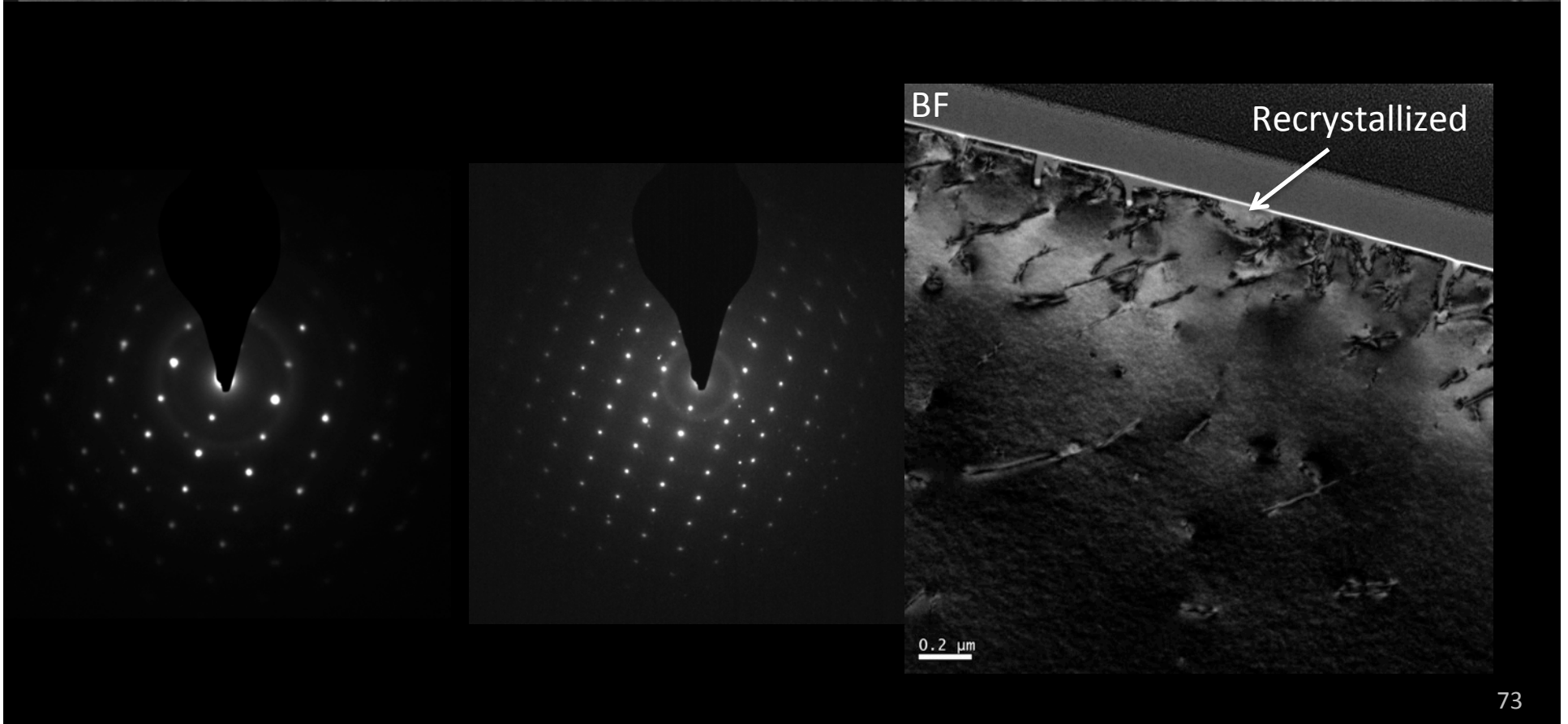
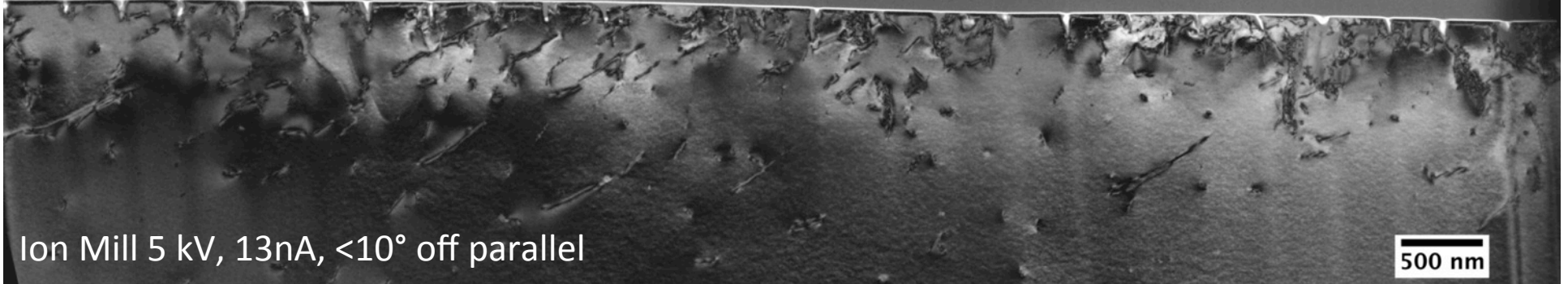
P. Gumbsch, M. Ruhle *et al.* APS. 87(2001) 8.

S. Taeri, D. Brunner, W. Sigle, M. Ruhle. Z. Metallkd. 6(2004) 95

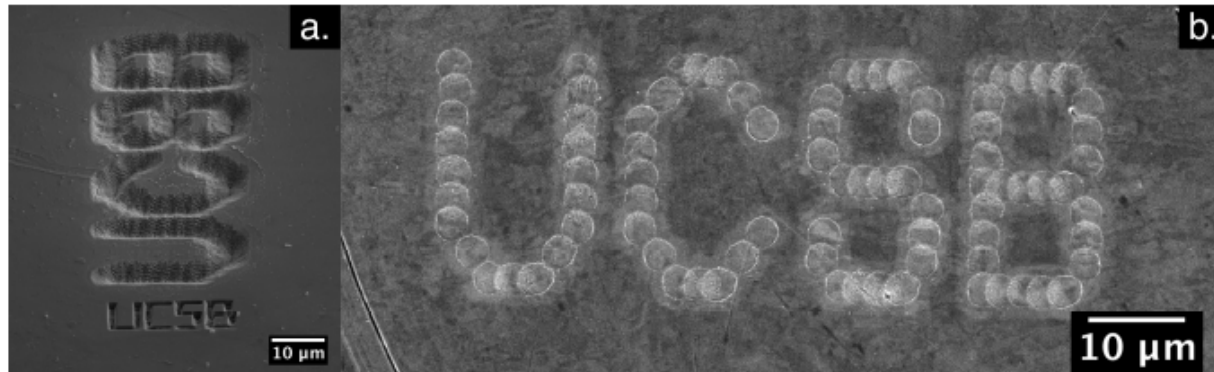
Orientation	Regime A		Regime C	
	Slip system	Schmid factor	Slip system	Schmid factor
<100>	<110>{110}	0.5	<110>{110}	0.5
<110>	<110>{110}	0.25	<100>{100}	0.5

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BF



- Bulk ablation with fs. laser can yield low damage surfaces capable of producing EBSD maps in STO, Ni, Mg, Ti
- FIB milling at near glancing angles in conjunction with fs. laser ablation can produce lower damage surfaces with higher EBSD map quality in both STO and Ni (and Ti)
- In STO dislocation damage is more extensive in grains with preferred orientations for slip, allowing us to characterize the stresses being imparted on the sample



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