

The Consortium for Enabling Technologies and Innovation

# *Virtual Summer Meeting for Young Researchers*

## Correlation of Irradiation Responses and Microstructures in AM 316 Stainless Steels

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Texas A&M University



# AM 316L SS preparation

Limited condition from Dr. Chen Sun at INL

316L SS **Direct Energy Deposition** process

LENS MTS 500 printer from Optomec Inc.

316L SS powders from John Galt Steel

Laser power: 400W

Scanning speed of 12.7 mm/s and

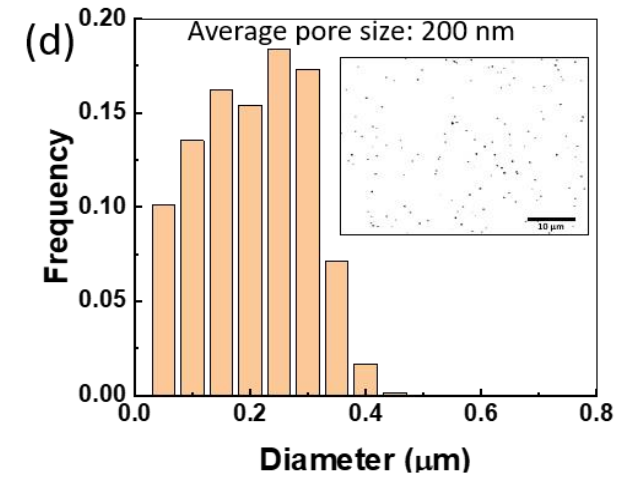
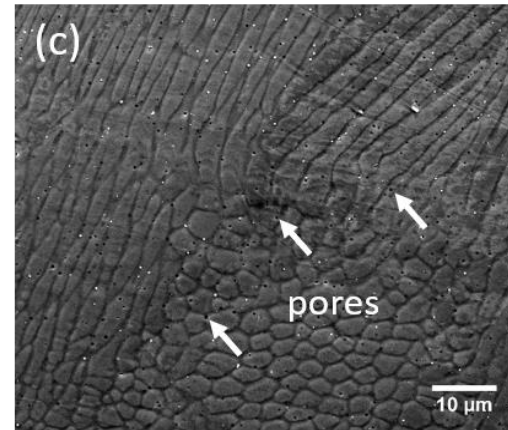
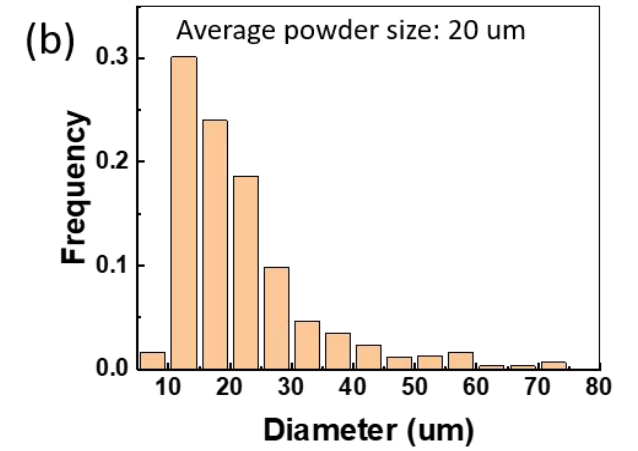
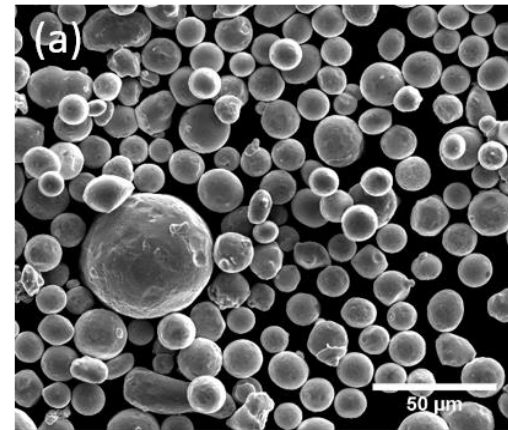
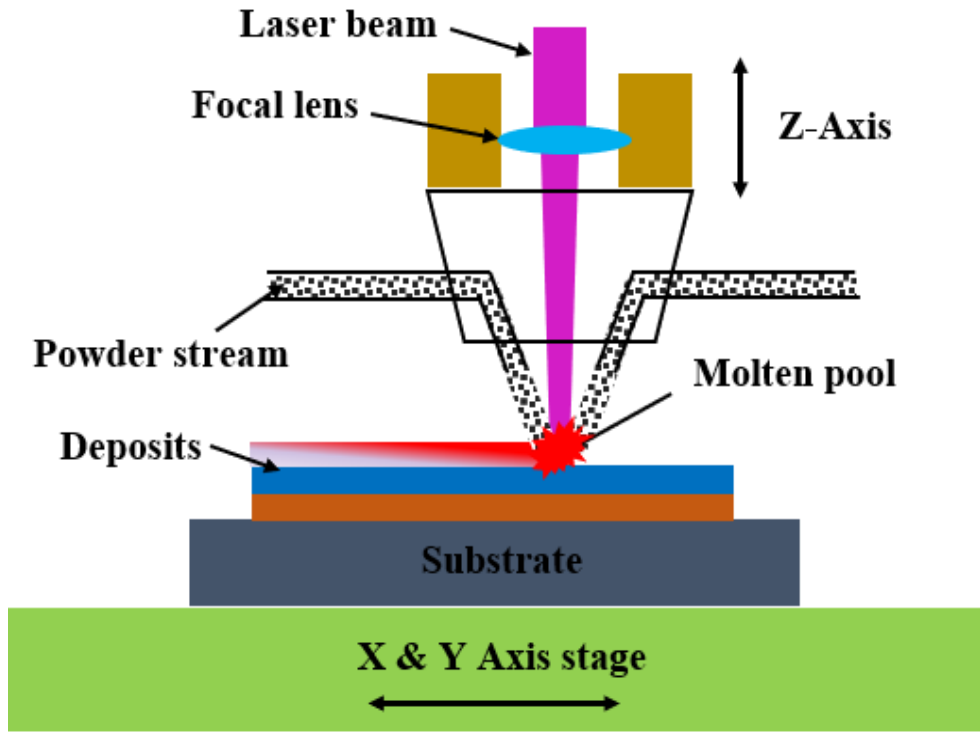
Spot size of 600  $\mu\text{m}$  in an argon atmosphere.

Systematic matrix from Dr. Dan Thomas at UW-Madison

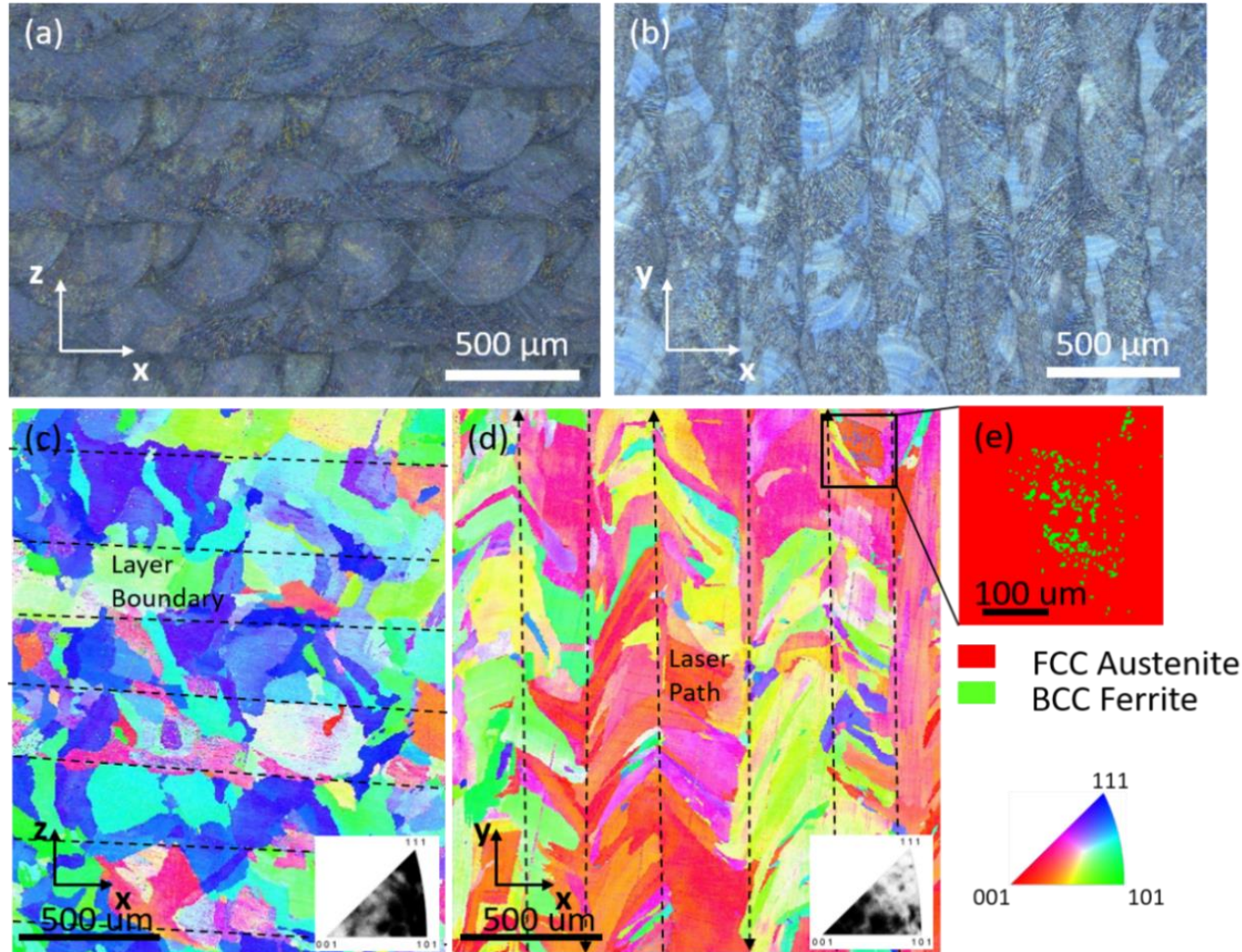
316L SS **Powder Bed Fusion** Process

S.No.	Laser Power (W)	Scanning Speed (mm/s)	Hatch Spacing (mm)	Layer Thickness (mm)	Volumetric Energy Density (J/mm <sup>3</sup> )
1	120	600	0.07	0.02	142.86
2	120	600	0.09	0.02	111.11
3	120	600	0.11	0.02	90.91
4	120	800	0.11	0.02	68.18
5	200	600	0.11	0.02	151.52
6	200	800	0.11	0.02	113.64
7	200	1300	0.11	0.02	69.93
8	260	800	0.11	0.02	147.73
9	260	1300	0.11	0.02	90.91

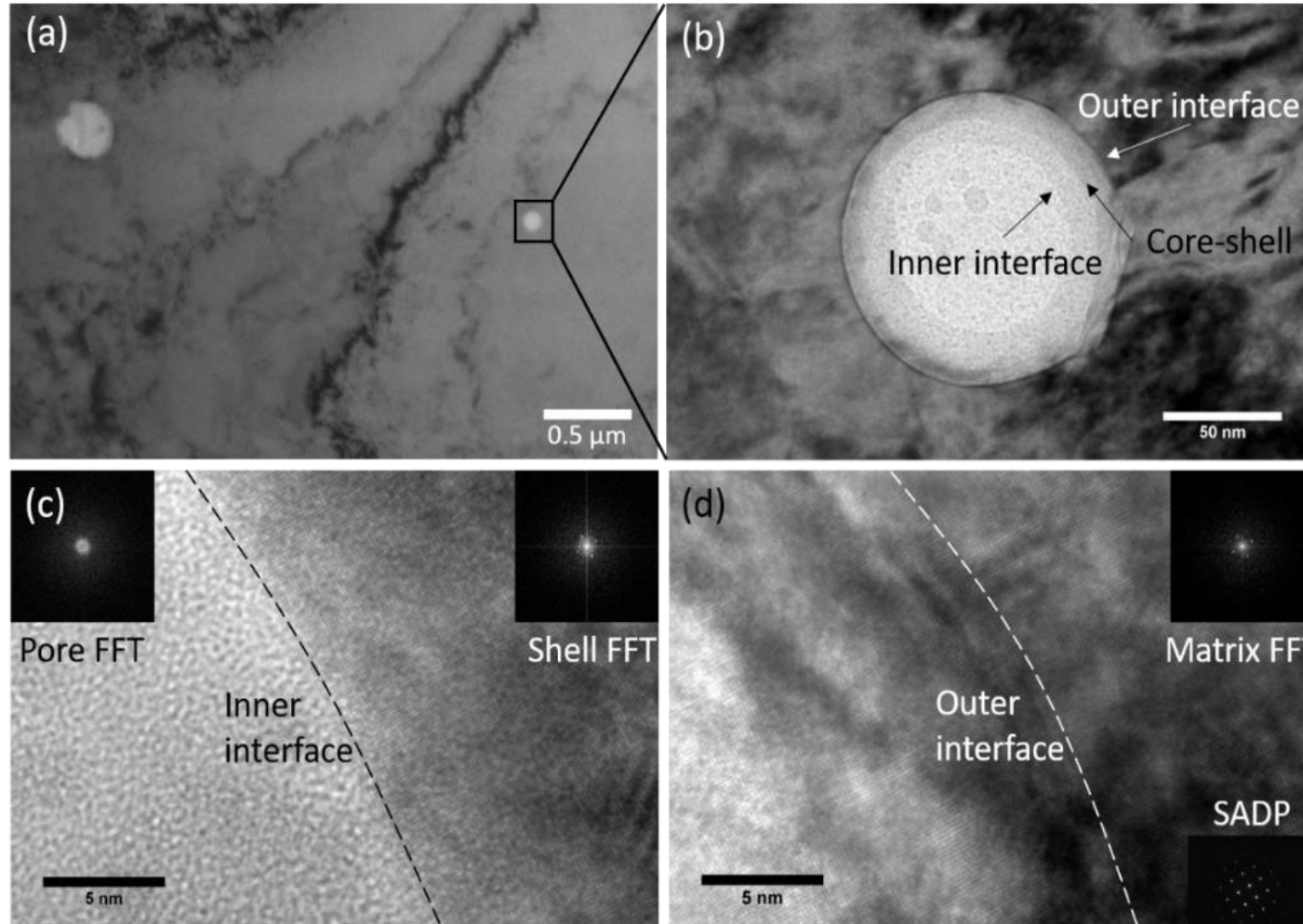
DED AM 316L



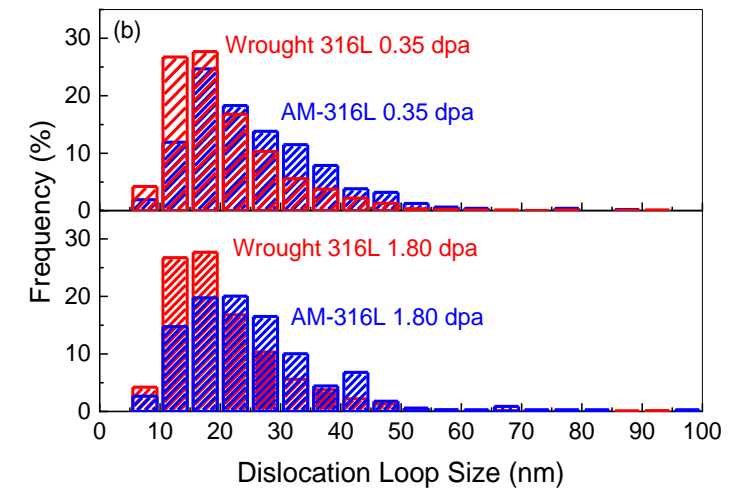
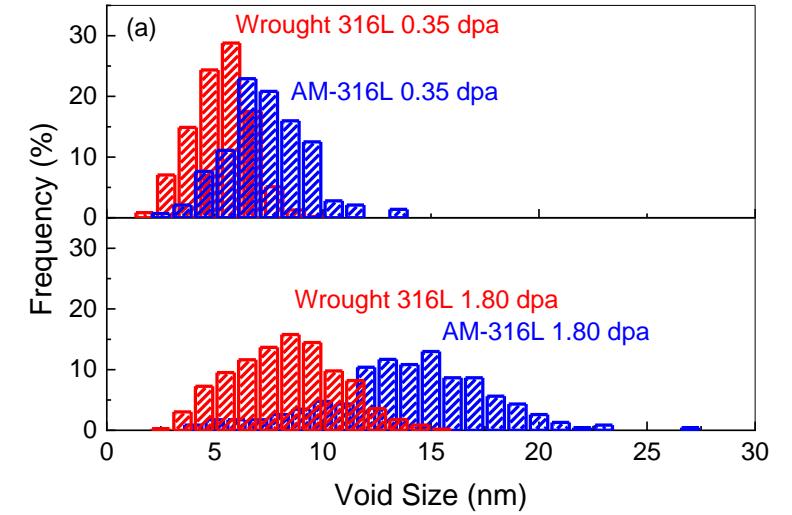
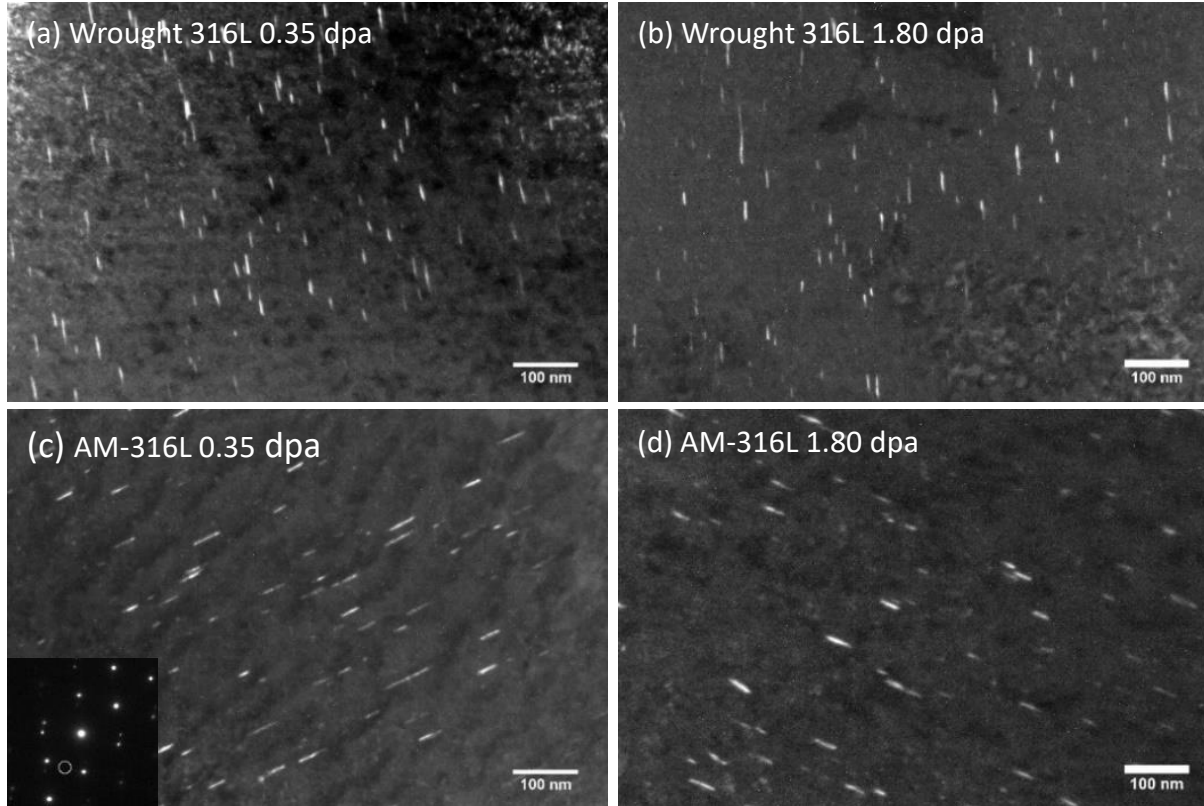
DED AM 316L



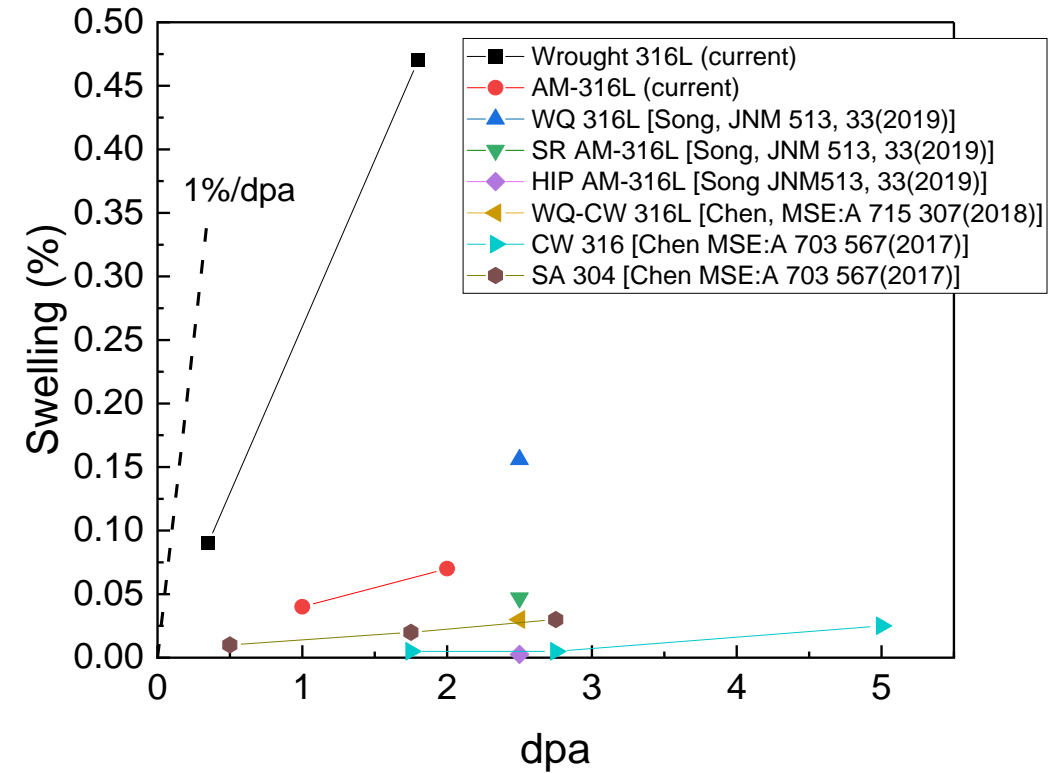
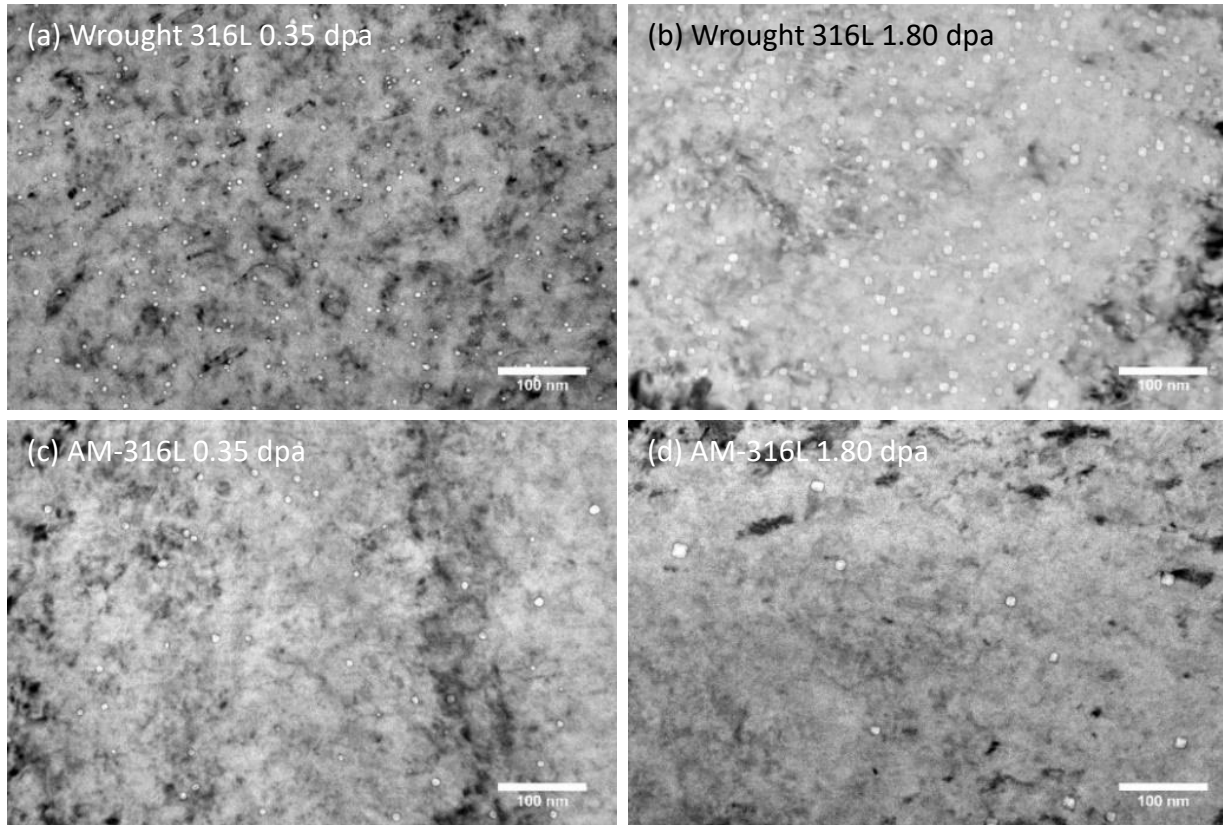
### DED AM 316L



Proton irradiated DED AM 316L



### Proton irradiated DED AM 316L



UW- Madison PBF AM 316L

### Printing parameters:

- **Laser Power:** 120 W
- **Scanning Speed:** 600 mm/s
- **Hatch Spacing:** 0.07 mm
- **Layer Thickness:** 0.02 mm



### Electropolishing parameters:

- **Solution:** 5% Perchloric Acid (HClO<sub>4</sub>) in Methanol
- **Applied Voltage:** 25 V
- **Temperature:** -35° C
- **Polishing time:** 12 s

Before Polishing



After Polishing





Helium implantation:  
to speed up void formation

## Ion beam condition

Beam: 100 keV He

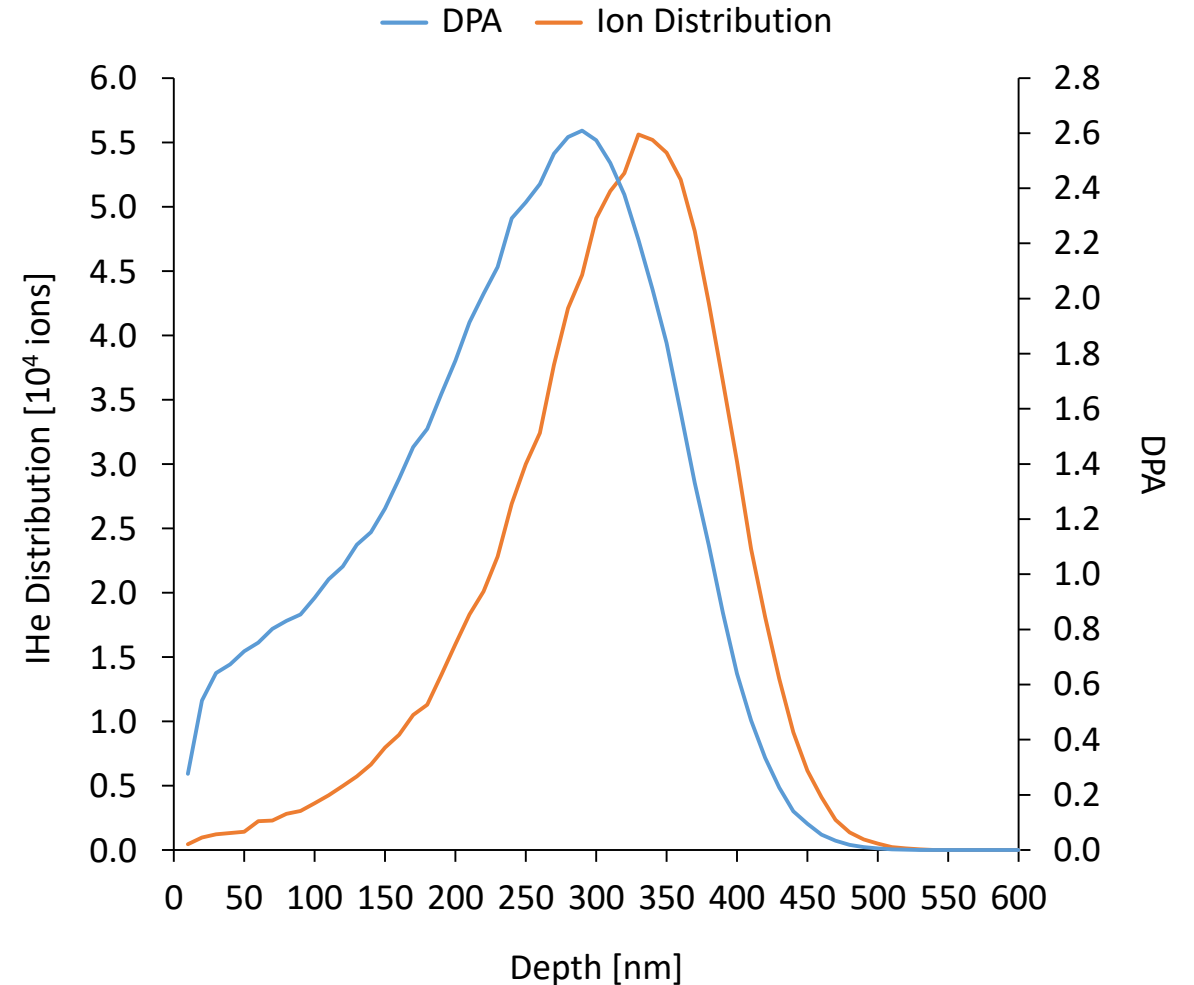
Raster beam (X: 0.25Hz, Y: 3.43 Hz)

Beam area: 7mm x 8.5mm

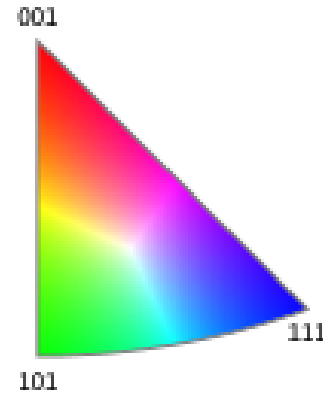
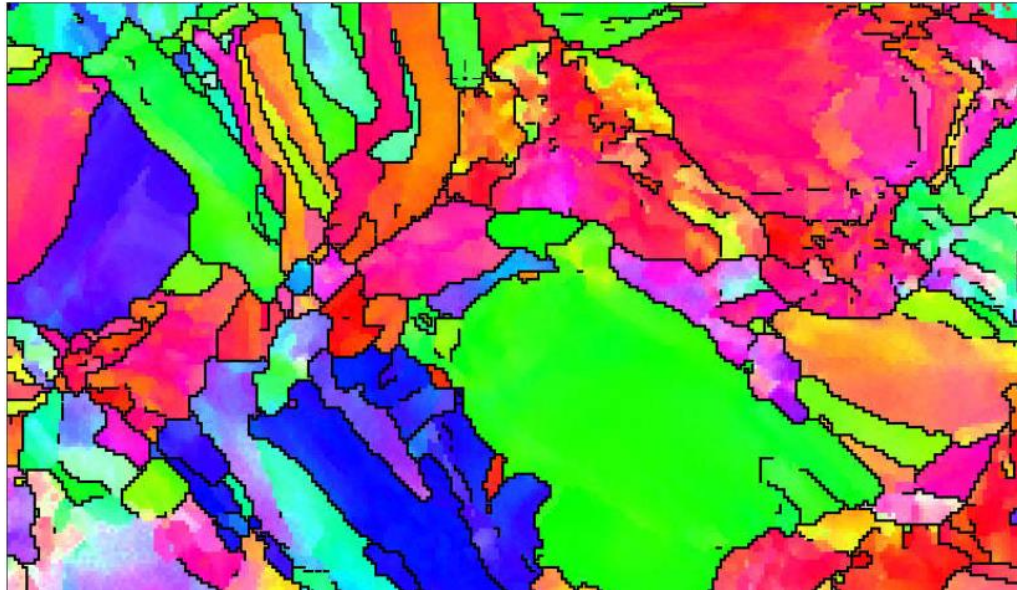
Beam current: 3  $\mu\text{A}$  ( $3.15 \times 10^{13}$  ions/cm<sup>2</sup>/s)

Temperature: 300° C

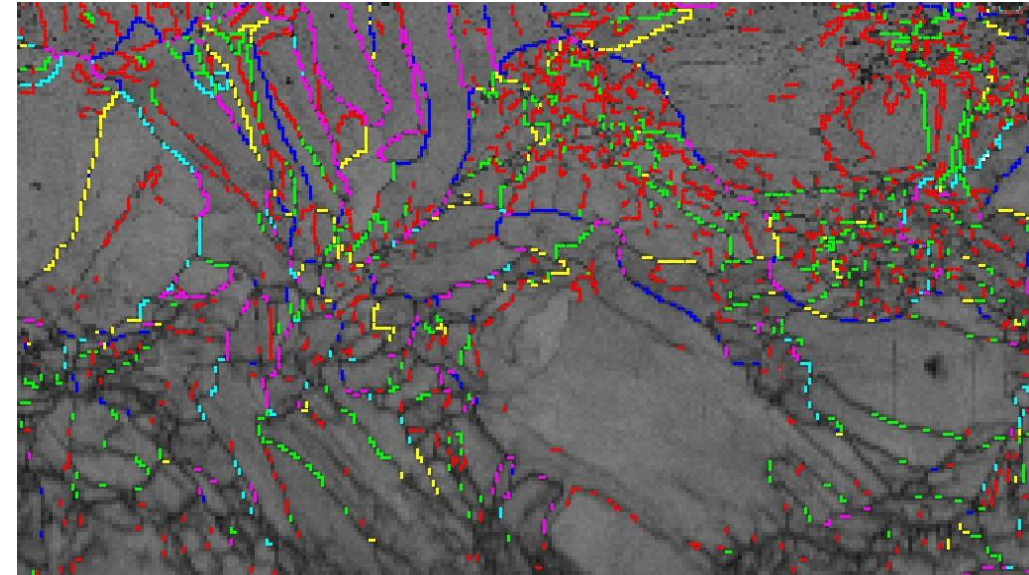
Fluence:  $1 \times 10^{17}$  ions/cm<sup>2</sup>



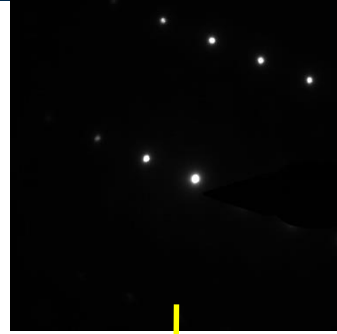
After helium ion irradiation, using EBSD to guide FIB location: numerous site selective characterization to study swelling dependence on grain boundaries



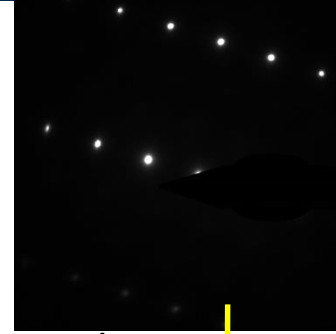
Colors:  
4° -10° Red  
10° -20° Green  
20° -30° Yellow  
30° -40° Blue  
40° -50° Fuchsia  
50° -60° Aqua



# TEM Results: Area without pore



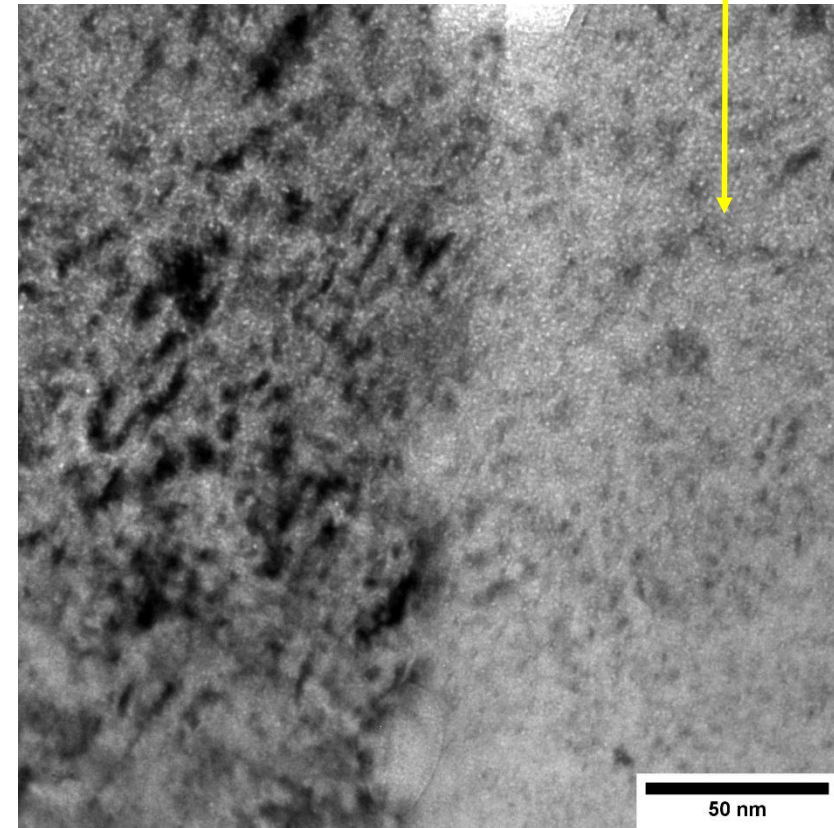
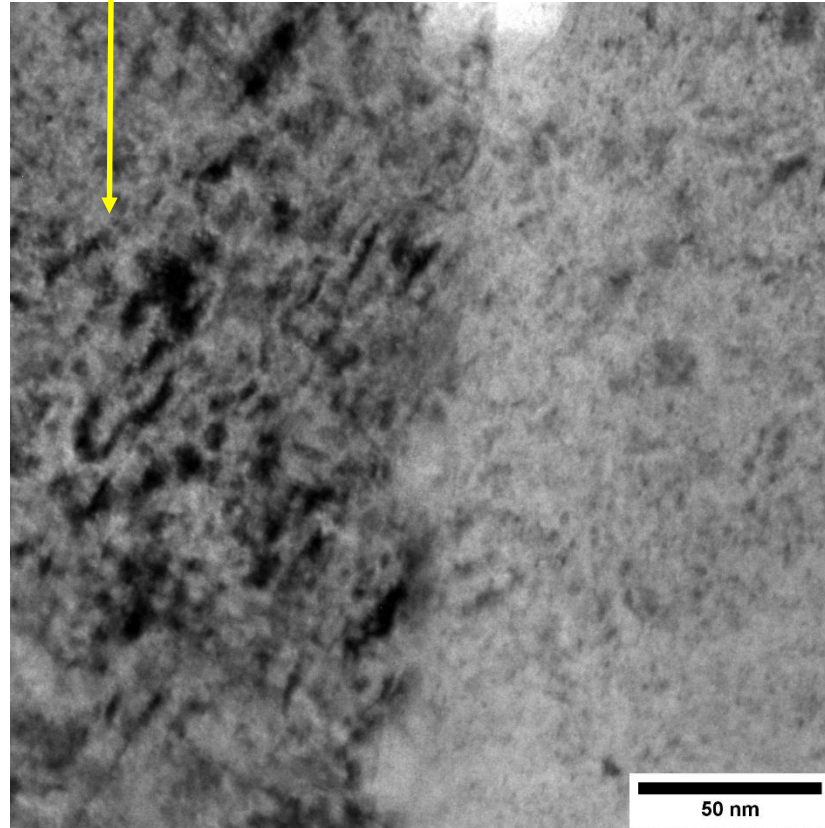
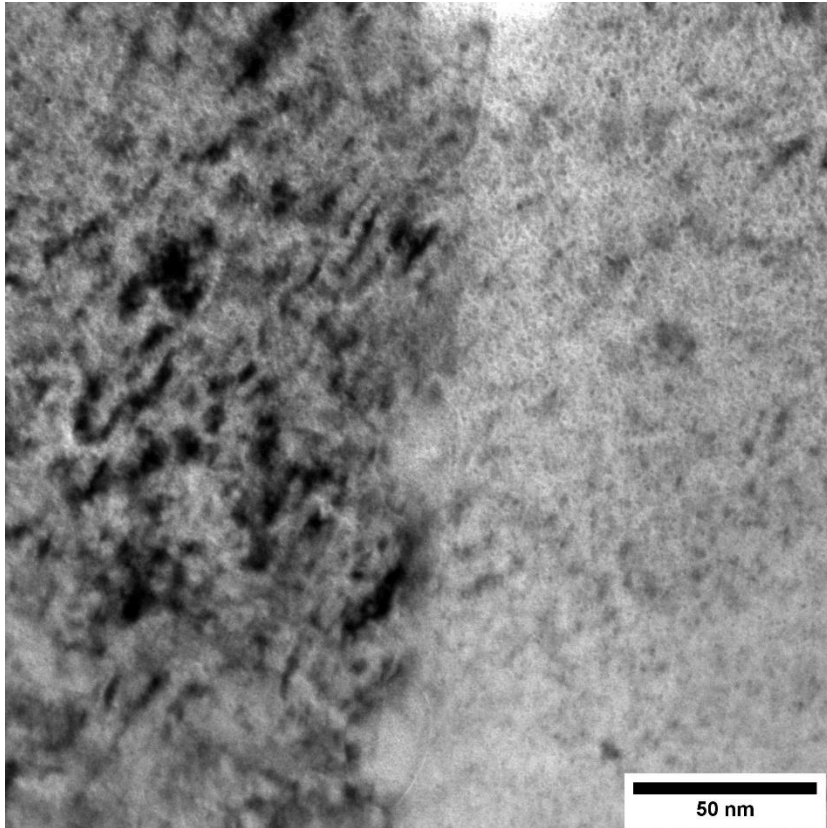
Left grain:  $\alpha = -7.09^\circ$ ,  $\beta = -4.73^\circ$   
Right grain:  $\alpha = 7.46^\circ$ ,  $\beta = 0.08^\circ$   
 $\Delta = 15.32^\circ$



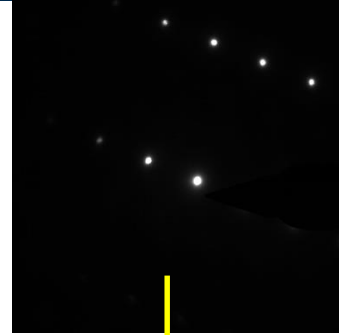
Over-Focused

Focused

Under-Focused

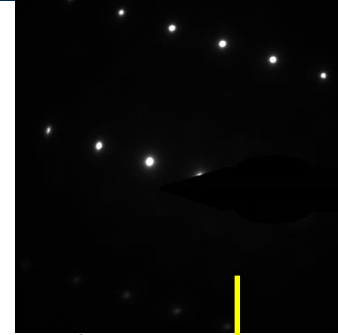


# TEM Results: Area with pore



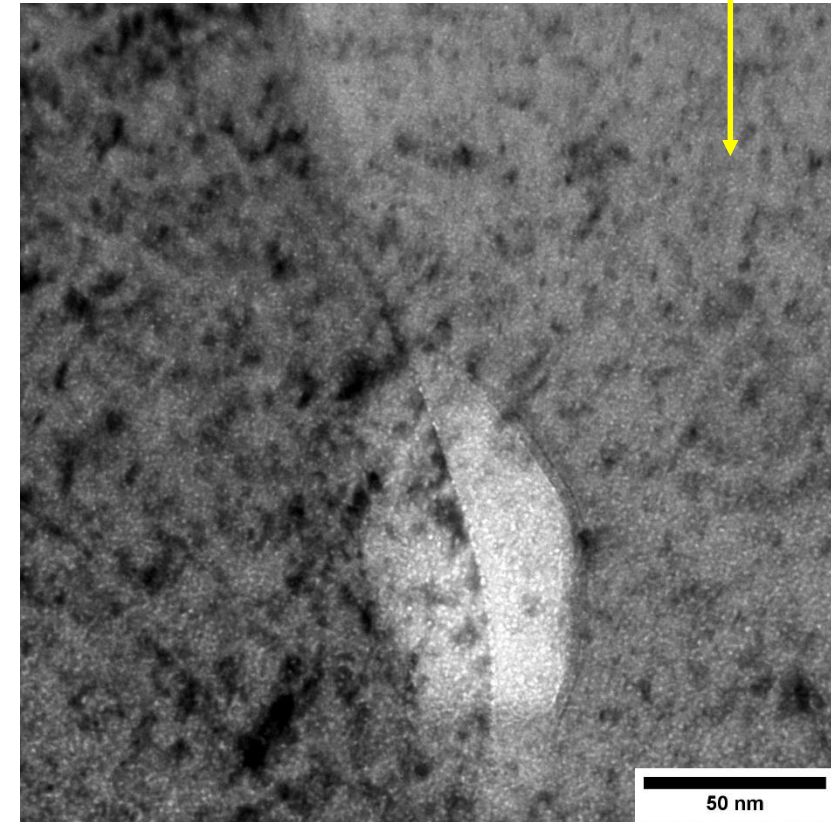
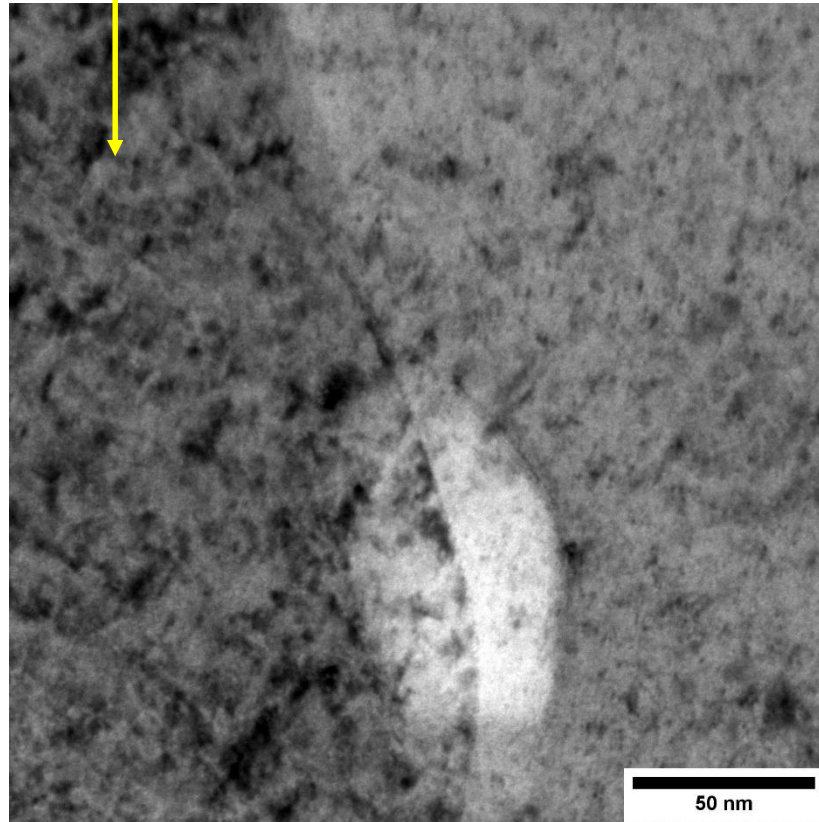
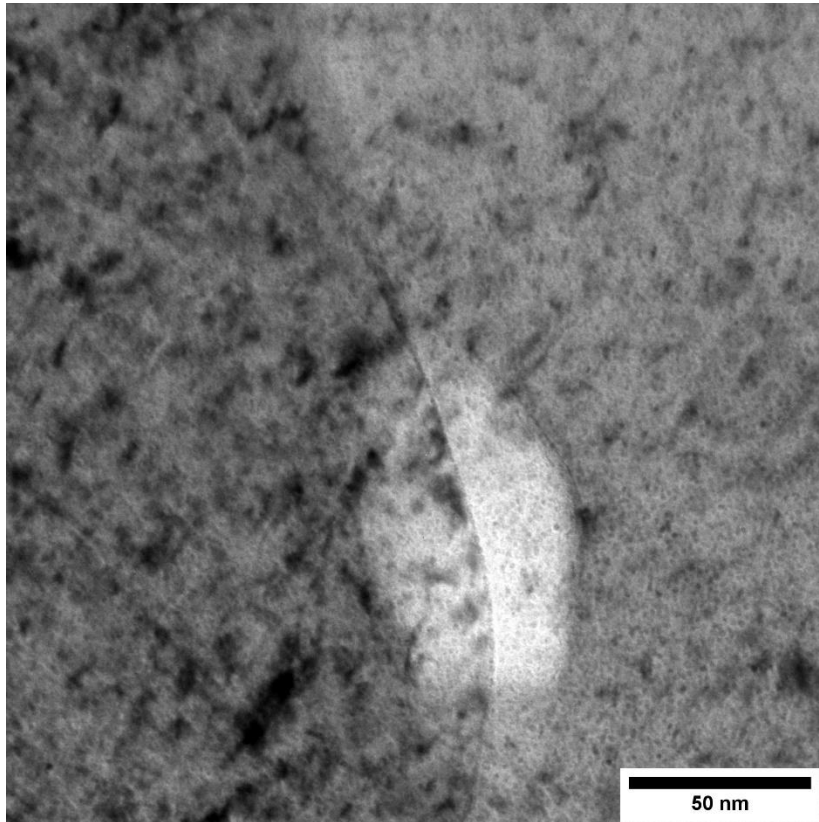
Over-Focused

Focused



Under-Focused

Left grain:  $\alpha = -7.09^\circ$ ,  $\beta = -4.73^\circ$   
Right grain:  $\alpha = 7.46^\circ$ ,  $\beta = 0.08^\circ$   
 $\Delta = 15.32^\circ$

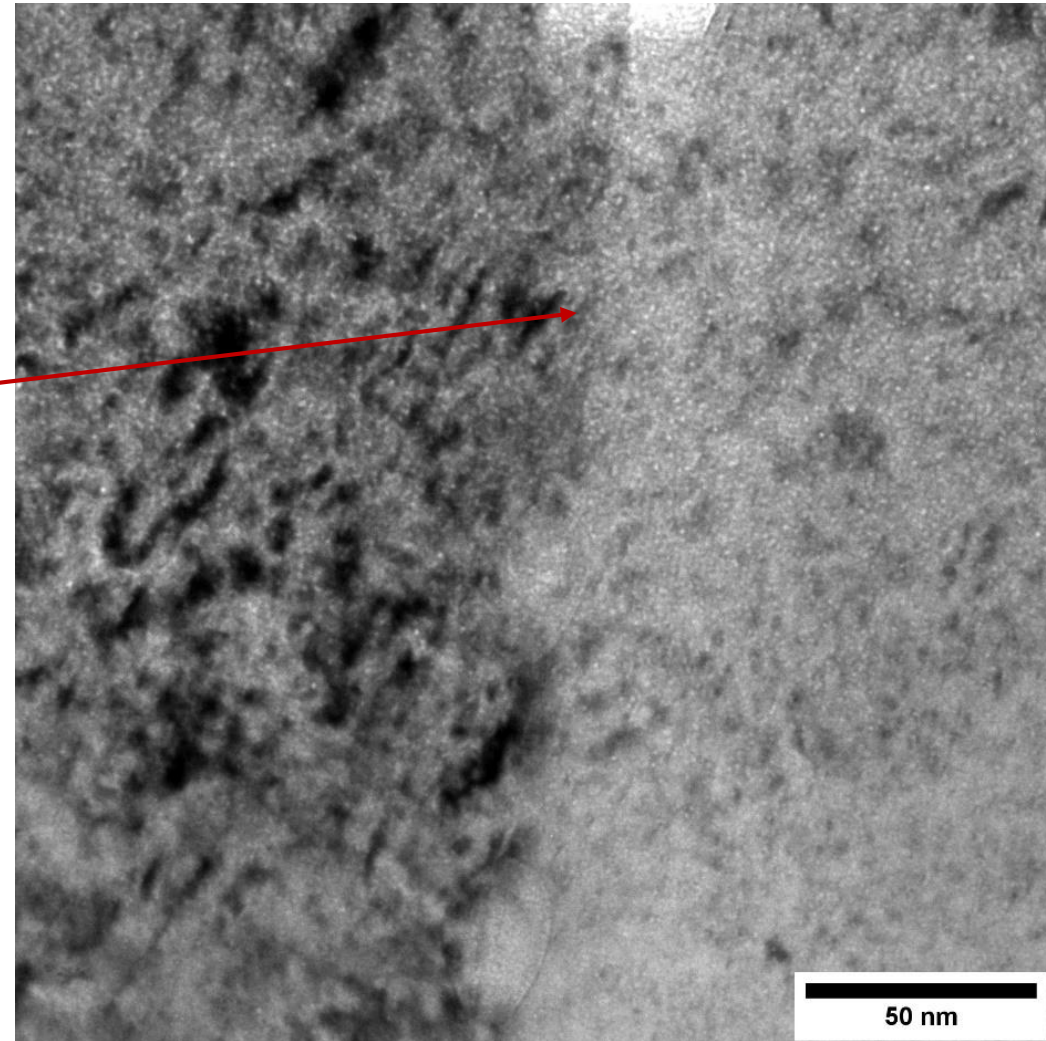


# TEM Results:

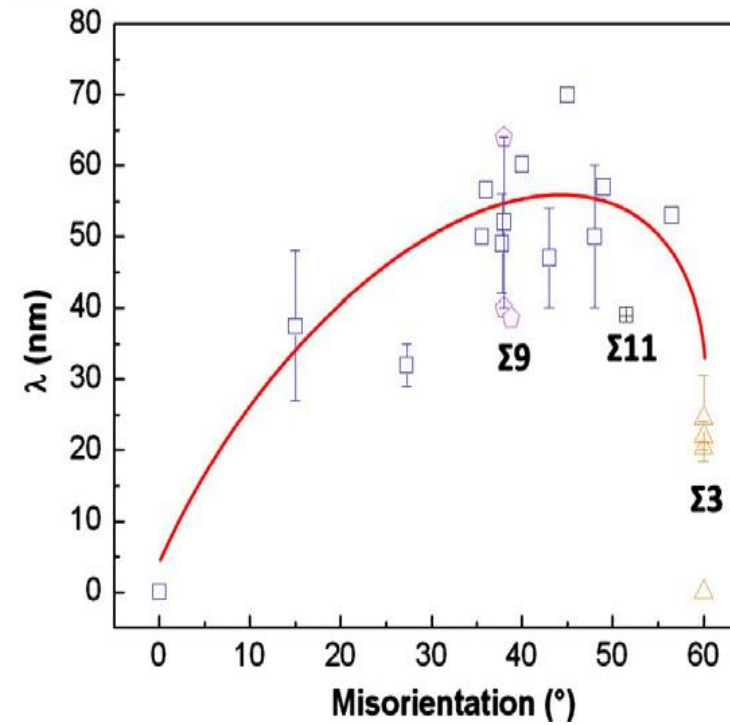
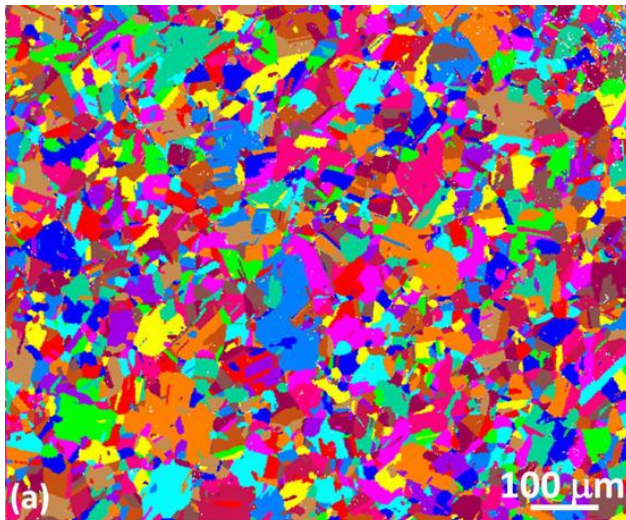
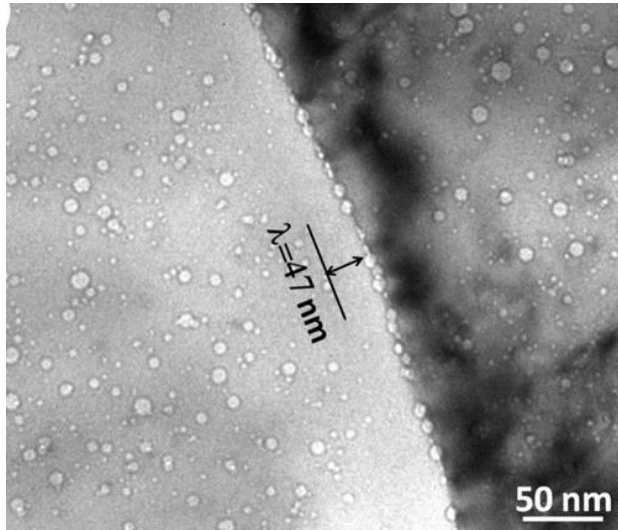
Under-Focused

Bubble aggregation can be observed at boundary in both areas.

No noticeable depletion zone: expected for small angle boundaries



He into Cu



Data taken from Han et al., 2012

Boundary sink efficiencies depend on boundary angles: to be tested in AM steels

# Testing Matrix of UW-Madison 316L AM samples

Temperature	Irradiation	Status	Goal
300 C	Helium ion implantation to $1 \times 10^{17} / \text{cm}^2$	Irradiation completed, FIB/TEM delayed due to the virus	The key is to <b>study void depletion as a function of boundary misorientation angles</b> . The irradiation temperature is low avoid grain growth.
400 C	1 dpa proton irradiation.	Irradiation finished FIB/TEM delayed due to the virus	The key is <b>to study boundary segregation as a function of misorientation angles</b> . The temperature is high enough to induce segregation, but low enough to avoid grain growth)
	3 dpa proton		
	5 dpa proton		
575 C	50 dpa Fe	Scheduled in July	The key is to <b>swelling tolerance</b> for multiple samples manufactured under different conditions. The temperature is at maximum swelling temperature.
	100 dpa Fe		
	150 dpa Fe		



70 YEARS OF SCIENCE & INNOVATION



# Acknowledgement

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