



# Effect of Friction Stir Welding on Microstructure Evolution of Self-Ion Irradiated MA956

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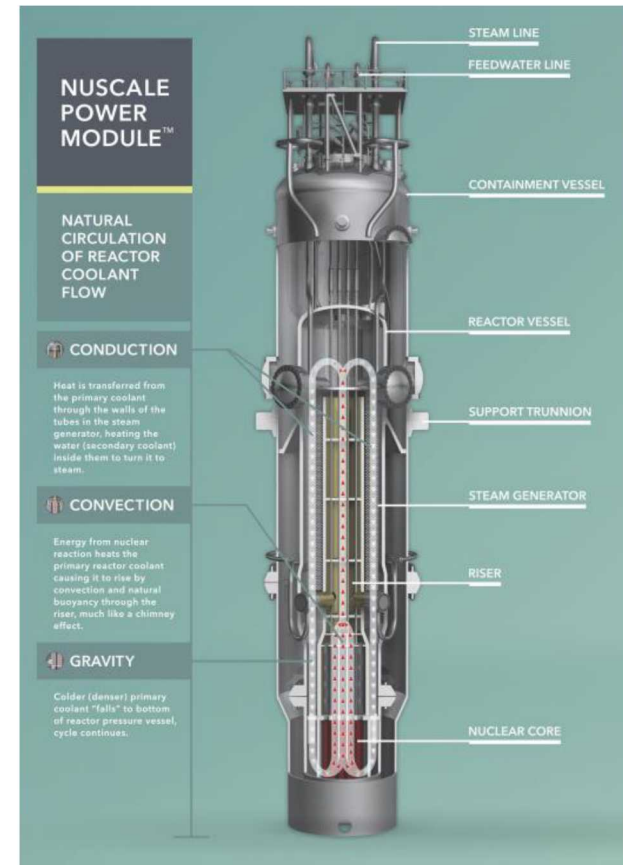
# Motivation- Materials Challenges for DOE and DOD

## Sodium Fast Reactors (SFR)

- Swelling of fuel cladding and duct at high dose
- Radiation-induced embrittlement by precipitation of brittle phases
- Corrosion from sodium
- Fuel-clad chemical interaction (FCCI)

## Small Modular Reactors

- High damage due to non refuel design
- Stress corrosion cracking/Irradiation assisted SCC



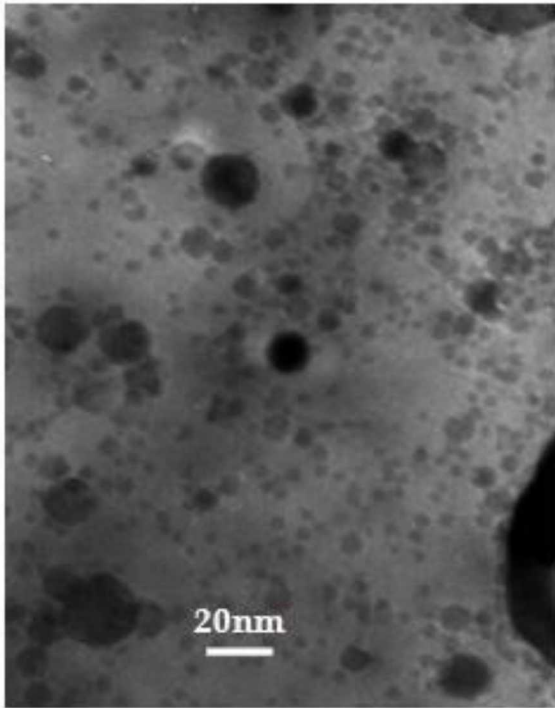
50 MWe modular reactor design

Courtesy of NuScale Power





# ODS Steel- A possible reactor material?



Typical 9Cr ODS ferritic steel

- Ideal candidates for clad material
  - Strength
  - Corrosion resistance
  - High temperature mechanical properties
  - Radiation tolerance
- A limited data set at high damage in both previous neutron and ion-irradiated ODS inhibits mechanistic understanding of the microstructure behavior at very high damage levels
- A key issue to be resolved is the challenge of **welding** and the resulting **radiation tolerance**
- Applicable to non refuel reactors:
  - Traveling Wave Reactor
  - Small Modular Reactor concepts

*S. Ukai, Chapter 14, "Microstructure and High-Temperature Strength of 9CrODS Ferritic Steel, Metals, Ceramic and Polymeric Composites for Various Uses, (2014)*

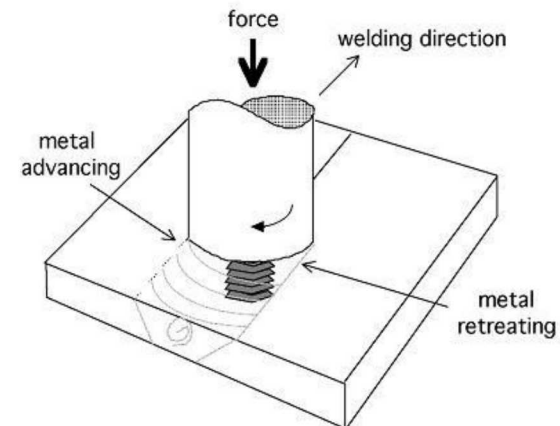
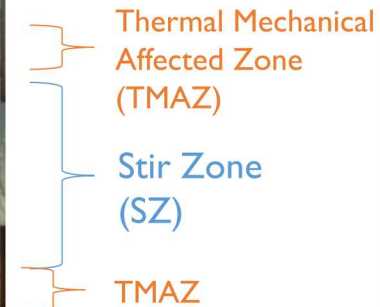




# Friction Stir Welding (FSW)

- Traditional fusion welding melt both pieces to be joined and fuse upon cooling
  - Has a large heat affected zone and cause heterogeneous dispersoid distribution
- Friction stir welding- a solid state joining technique that doesn't melt the workpiece and mechanically intermixes the joint
- A systematic comparison of effect of welding and irradiation is lacking in literature

FSW MA956



H. Bhadeshia, "Friction Stir Welding," University of Cambridge

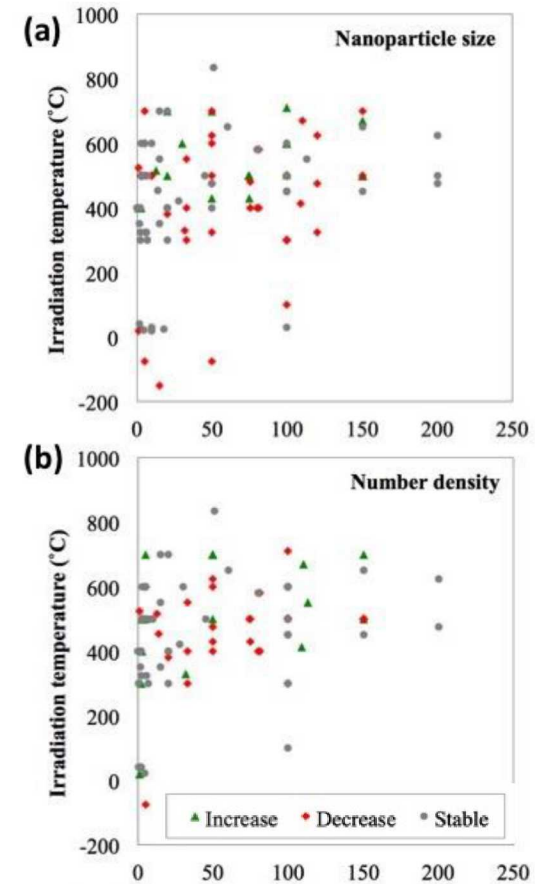




# Dispersoid Behavior Inconsistent throughout Literature

## Proposed Mechanisms:

- Ballistic dissolution: Radiation dissolves dispersoids
- Ostwald ripening: Dissolution of smaller dispersoids and redeposit on larger dispersoids
- Irradiation-enhanced diffusion: solute in solution redeposits on larger dispersoids or forms new dispersoids



J.P. Wharry et al. / Journal of Nuclear Materials  
486 (2017) 11-20



# Research Objective

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To understand how welding and irradiation affect the microstructure and mechanical properties of ODS steel MA956 in reactor relevant operating envelope

To understand how welding and irradiation affect the microstructure of ODS steel MA956 at 450°C up to 200 dpa





# Approach

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1. Comparison of as received base material (BM) and stir zone (SZ)
2. Assessment of microstructure at low dose (up to 25 dpa)
3. Behavior of BM and SZ up to high dose (up to 200 dpa)

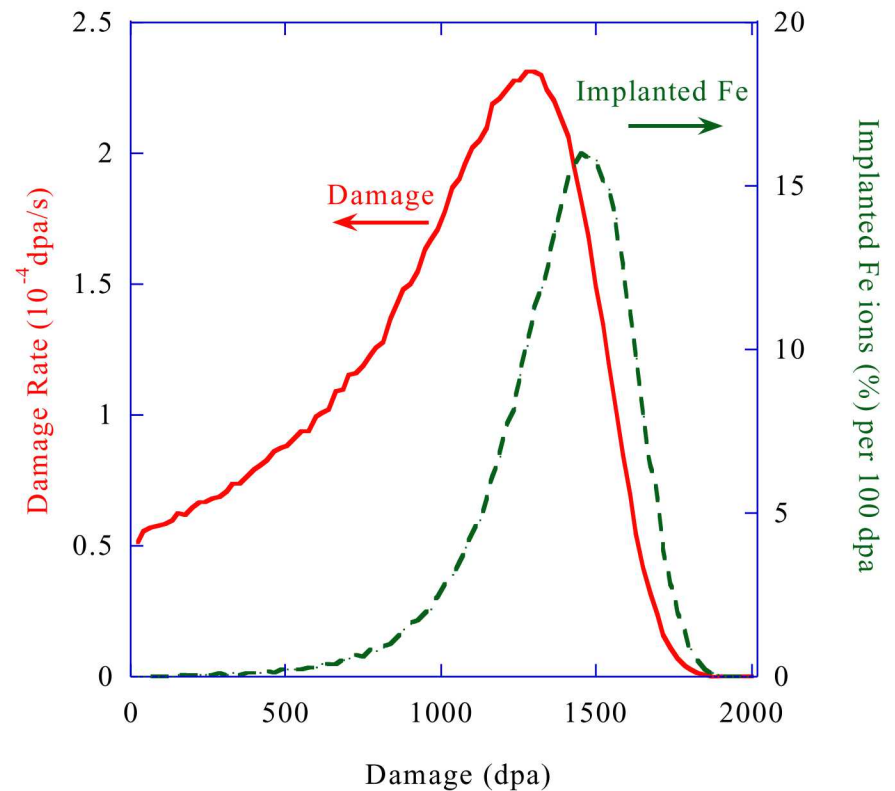




# Ion Experiment Design

Alloy	Fe	Cr	Al	Y <sub>2</sub> O <sub>3</sub>	Ti	Mn	Si	Ni	C	Mo	S	P
MA956 (wt%)	Bal	19.93	4.75	0.51	0.39	0.09	0.08	0.04	0.023	0.02	0.008	0.006

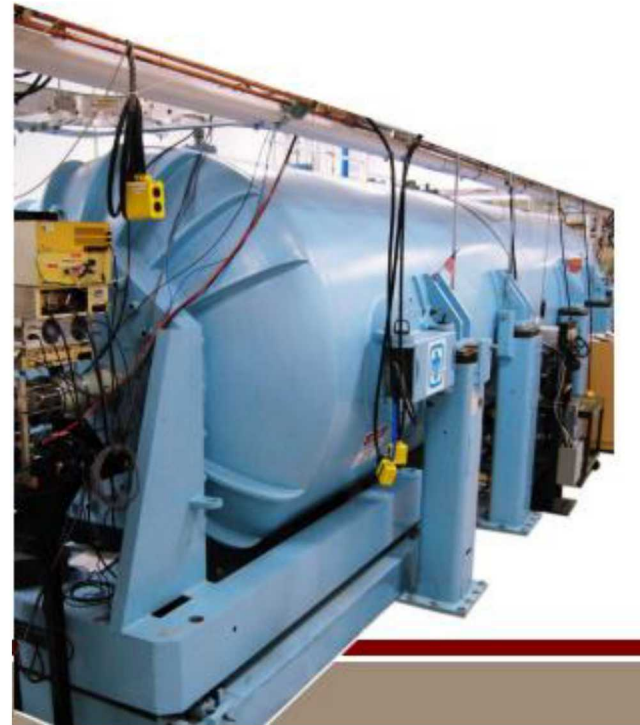
- MA956
- Irradiation of tem bar samples with 5 MeV Fe<sup>2+</sup> ions performed with 6 MV Tandem at Sandia National Laboratories or Michigan Ion Beam Laboratory
  - Damage measured at 600 nm using Quick Kinchin-Pease with E<sub>d</sub>=40 eV
  - Raster Scanning





# Ion Experiment Design (SNL)

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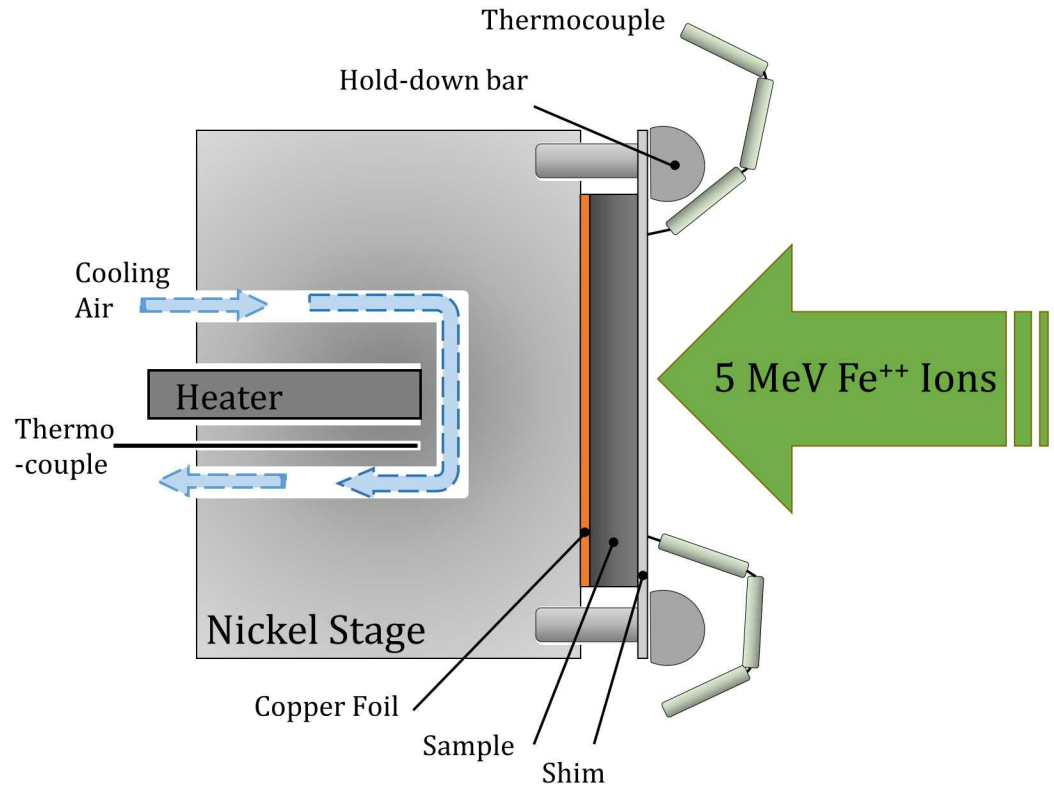
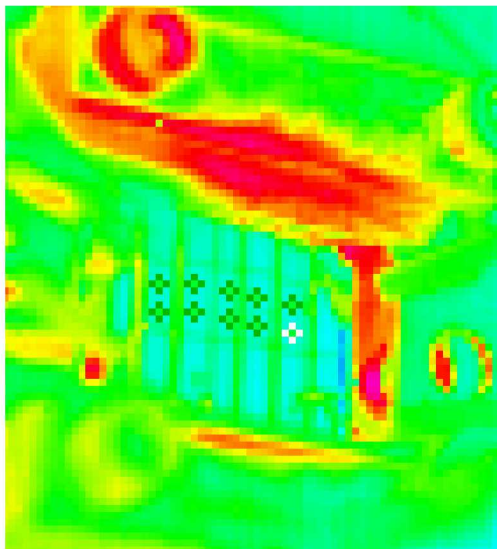


- Irradiations performed at SNL on the 6 MV Tandem
- LabView controlled button heater used to maintain temperature



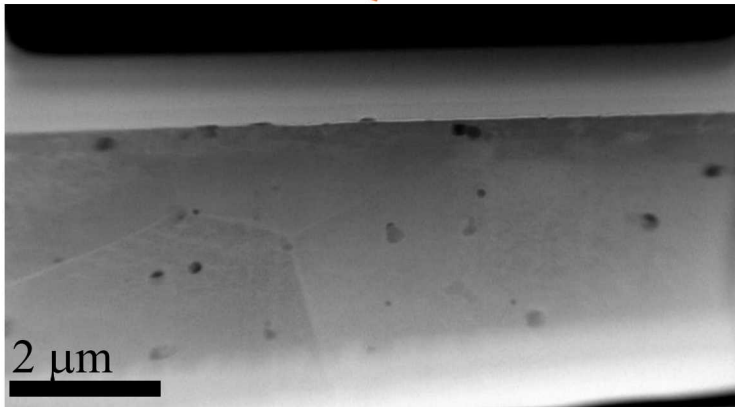
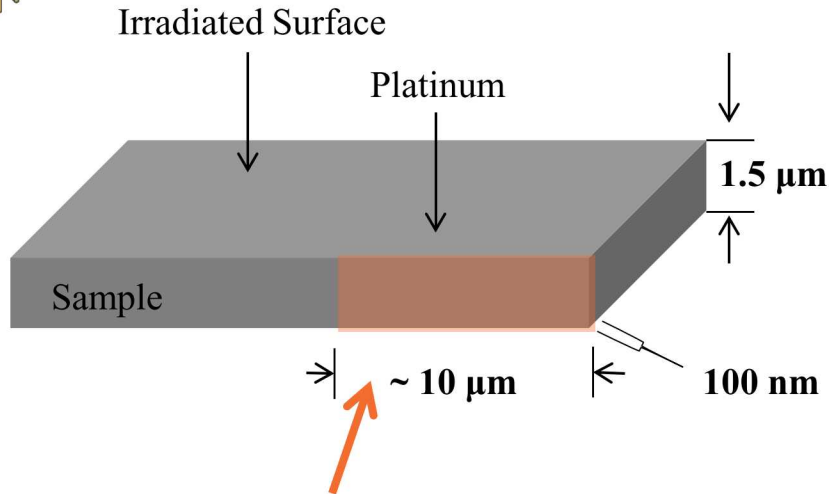


# Ion Experiment Design (MIBL)





# Microstructure Analysis



MA956-Friction Stir Welded

- Samples prepared via liftout method using Focused Ion Beam (FIB) at MaCS at CAES and at Naval Research Laboratory (NRL)
- Cross section liftout maintains integrity of surface while allowing depth profiling of dispersoid distribution
- Dispersoids imaged in high angular annular dark field in STEM mode from 500-700 nm
- Dislocation loops and network imaged in STEM BF
- Complementary Atom probe performed at MaCS and NRL



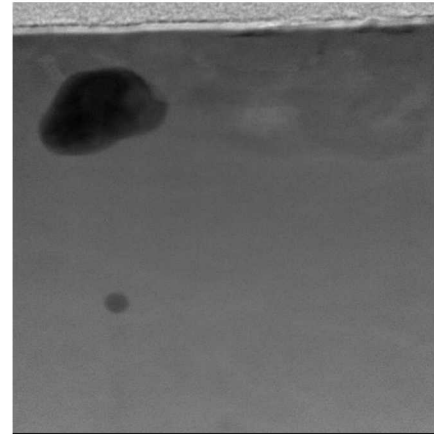
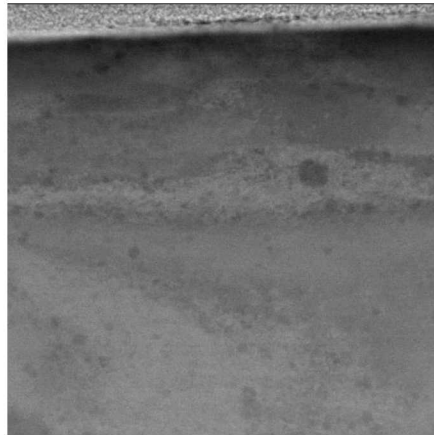


# MA956: As received microstructure

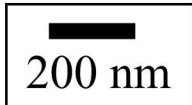
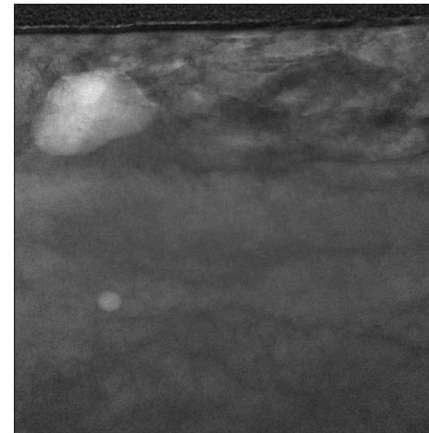
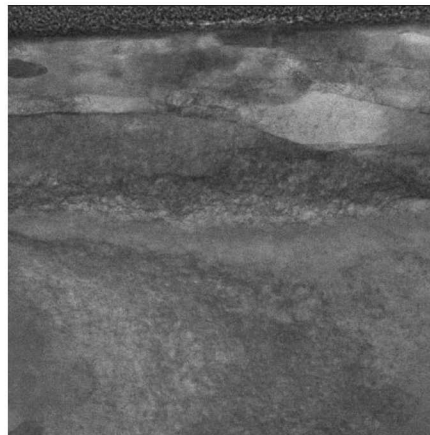
Base Material (BM)

SZ

HAADF



BF

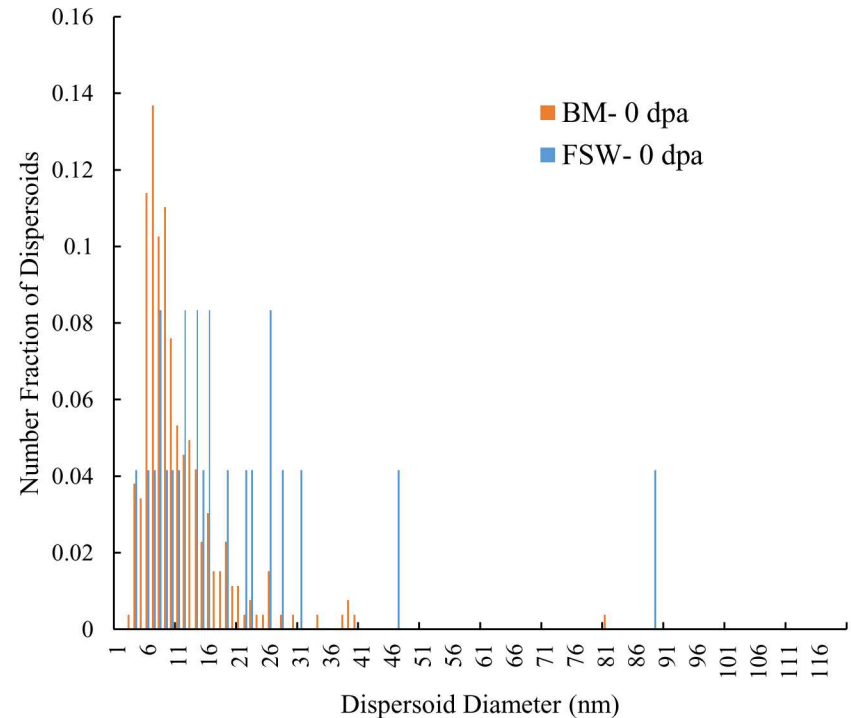
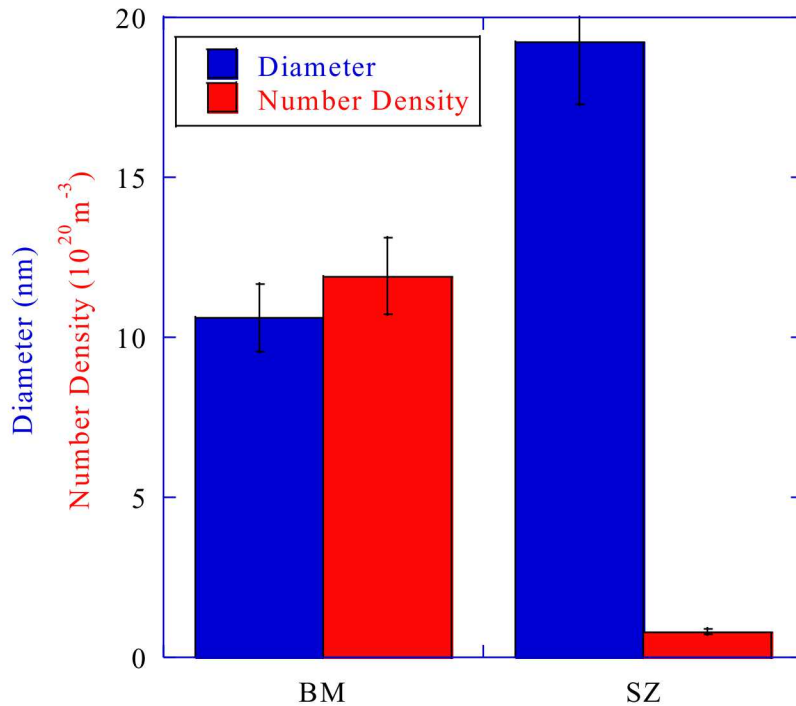


- Dispersoids much more visible with z contrast in HAADF images
- Qualitatively, fewer and larger dispersoids





# Quantitative Comparison of Dispersoids, 0 dpa



- Coarsening mechanisms (increased diameter, decreased number density and flatter distribution) consistent with previous results
- Mechanical testing reported softening with friction stir welding
  - Larger grains, fewer dispersoids





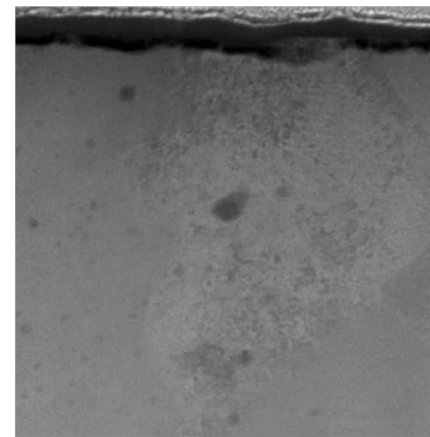
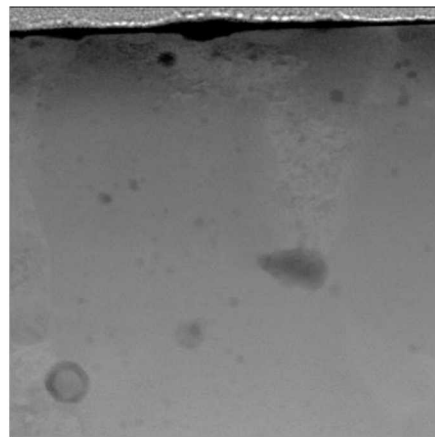
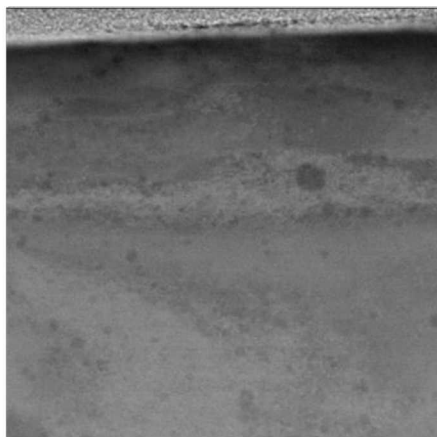
# Effect of irradiation on BM (low dose)

0 dpa

1 dpa

25 dpa

HAADF

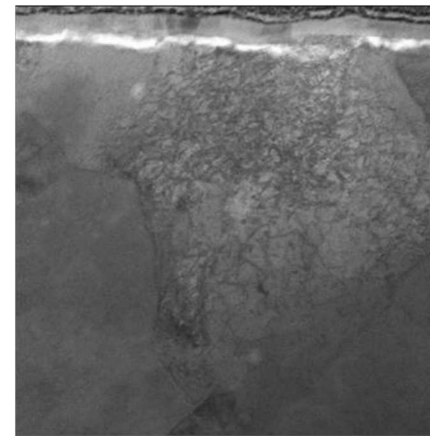
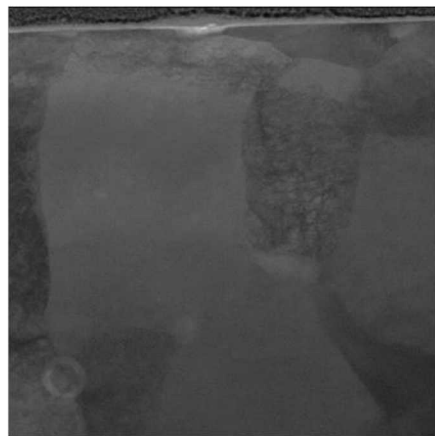
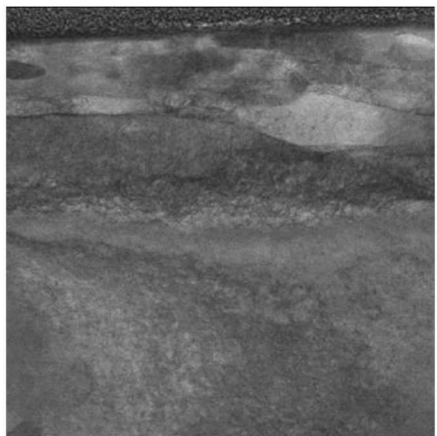


(a)

(b)

(c)

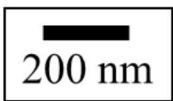
BF



(d)

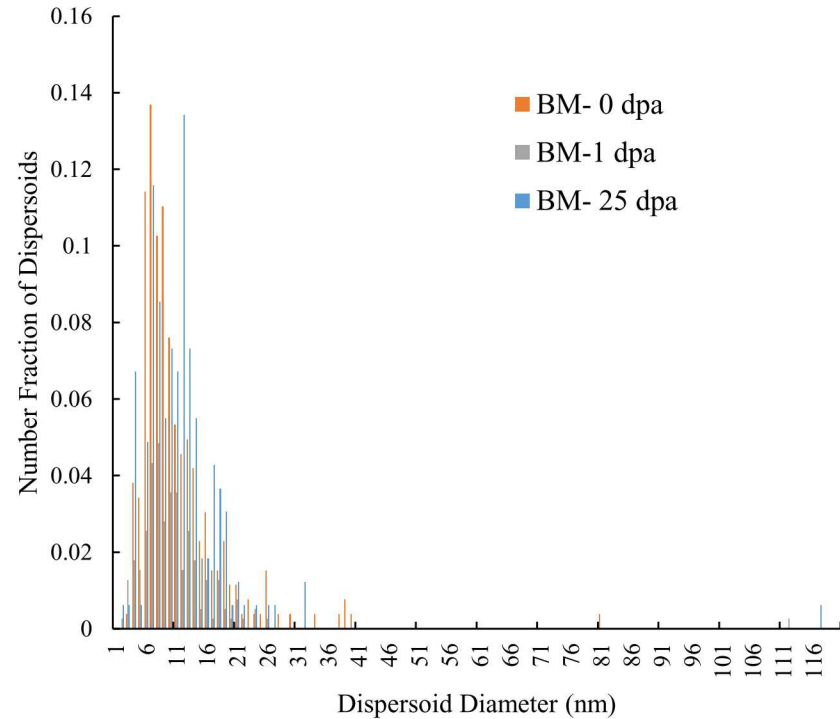
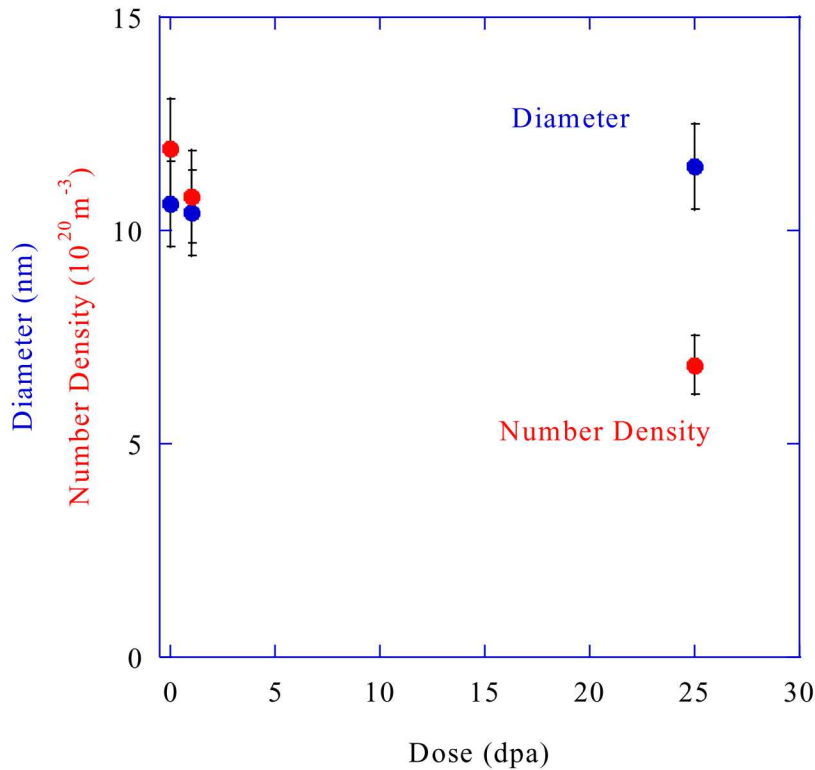
(e)

(f)





# Dispersoid Evolution with Increased dpa (BM)



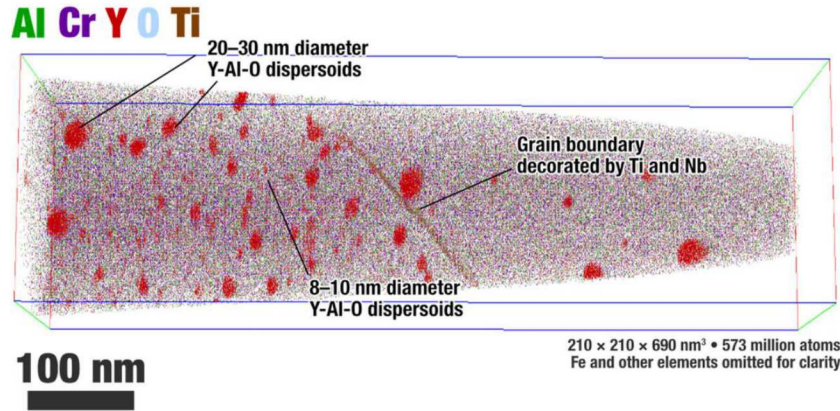
- Evidence of small amount of coarsening with irradiation
  - Increased diameter, decreased number density
  - Attributed to Ostwald coarsening



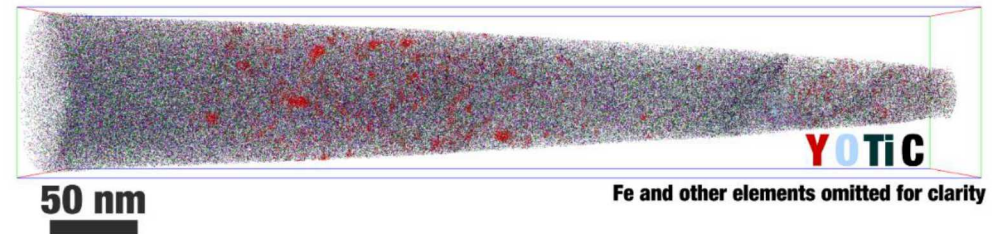


# APT: Evidence of heterogeneous dispersoid distribution

0 dpa



25 dpa



B.W. Baker et al., *Materials Science and Engineering A* (2014) 217-227.

- Heterogeneous dispersoid distribution at both 0 and 25 dpa
- Grain boundaries enriched with Ti/C at 25 dpa

\*APT data provided by K. Knipling at Naval Research Laboratory





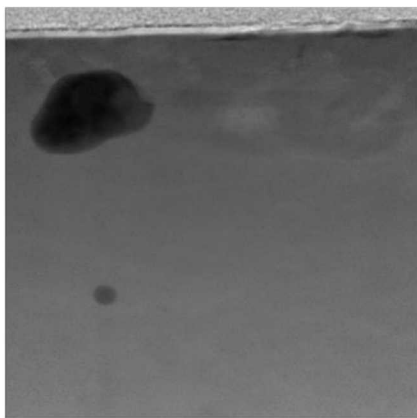
# Effect of irradiation on SZ (up to 25 dpa)

0 dpa

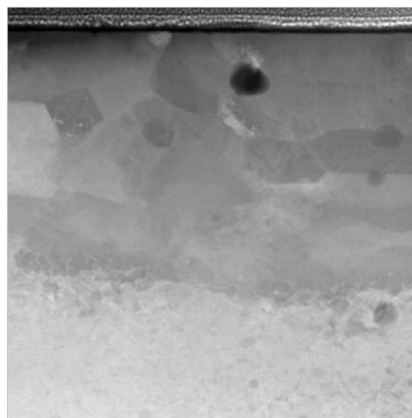
1 dpa

25 dpa

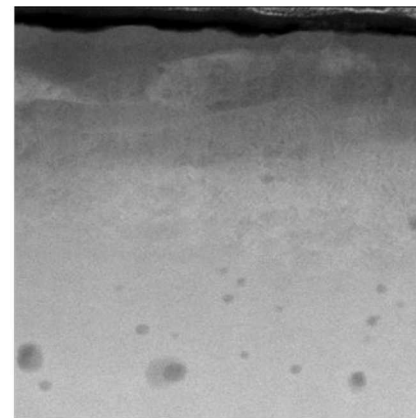
HAADF



(a)

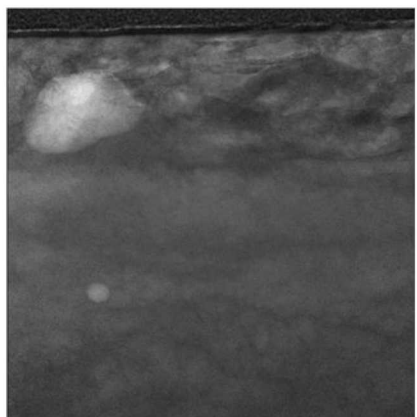


(b)

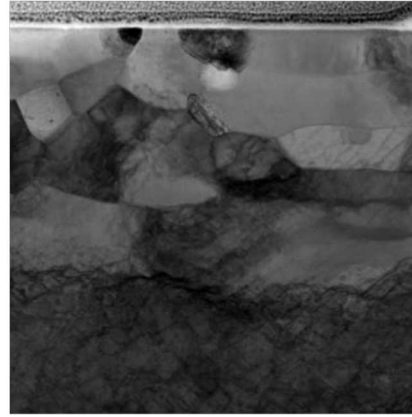


(c)

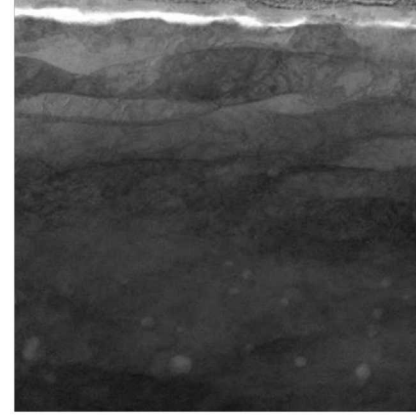
BF



(d)

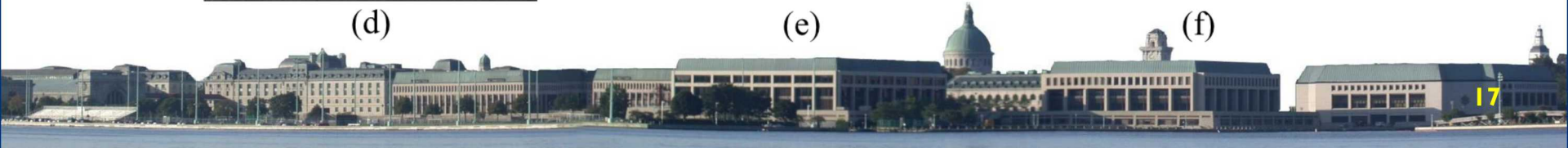


(e)



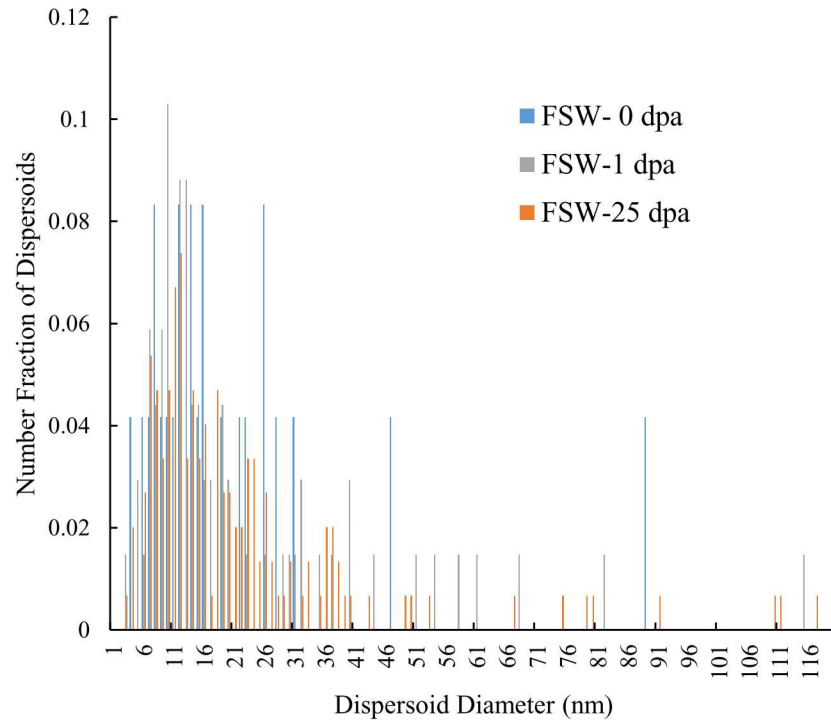
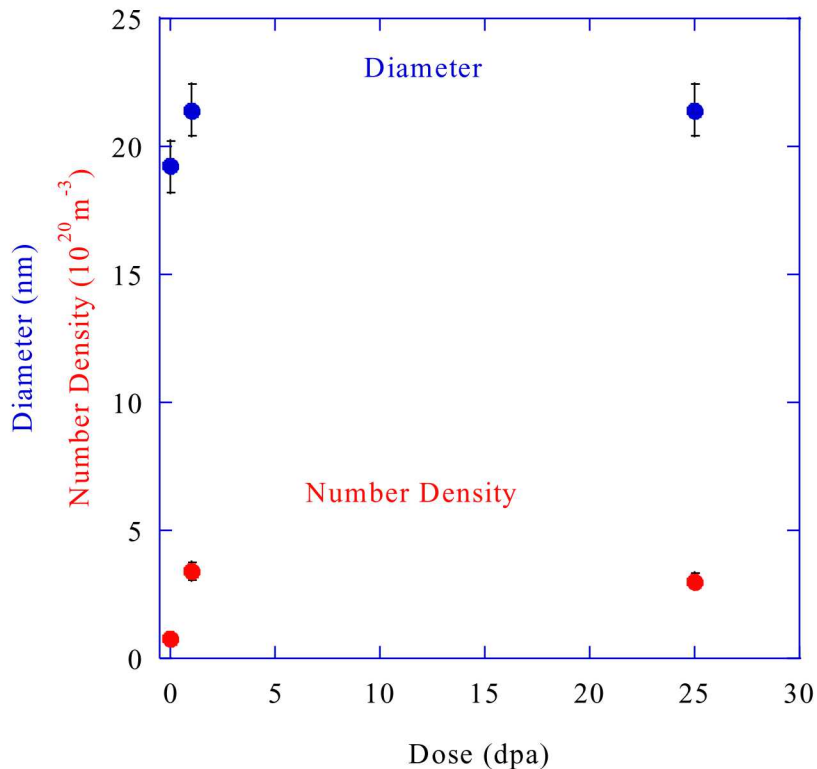
(f)

200 nm





# Dispersoid Evolution with Increased dpa (SZ)

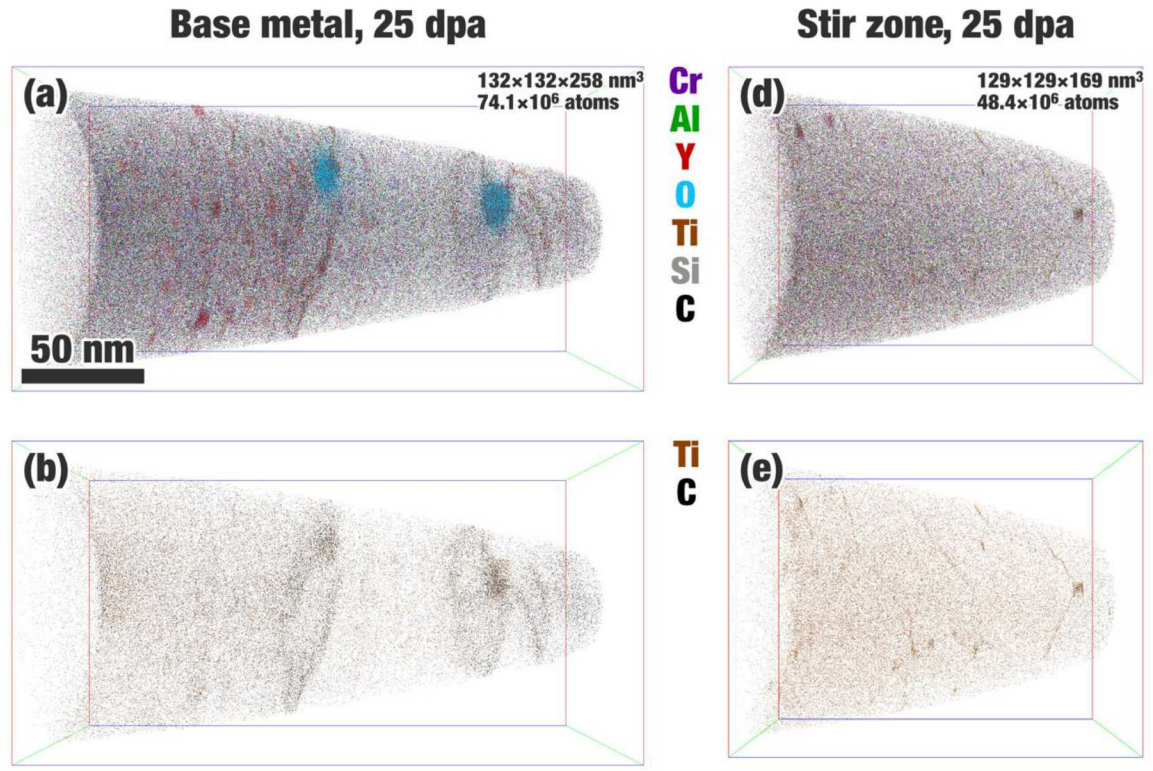


- Increase in number density due to small dispersoids re-precipitating post welding observed by 1 dpa
- Attributed to radiation enhanced diffusion
  - Increased diameter and increased number density





# APT: Evidence of Re-precipitation?

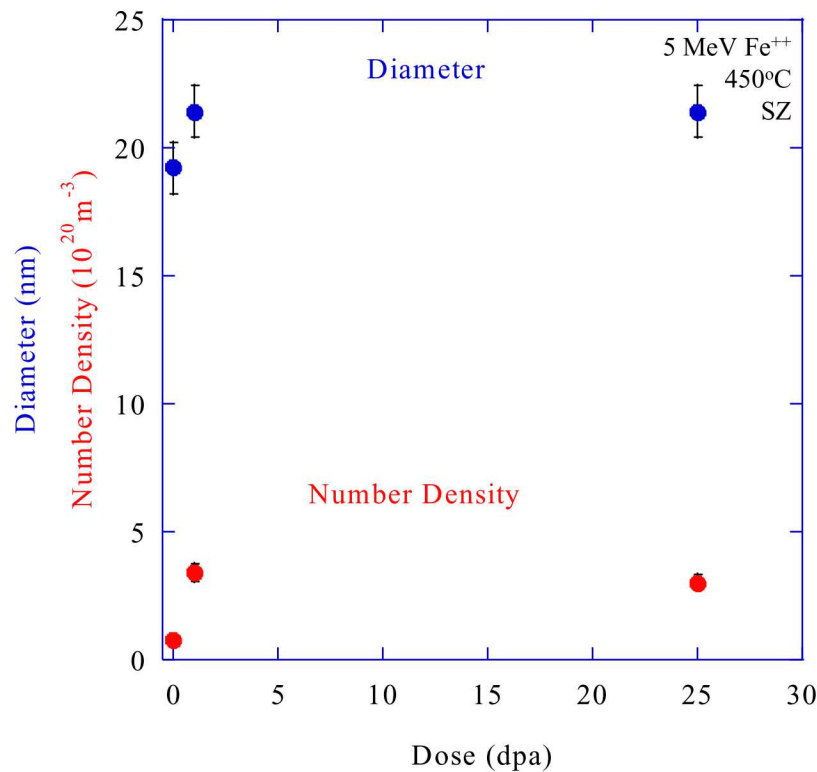
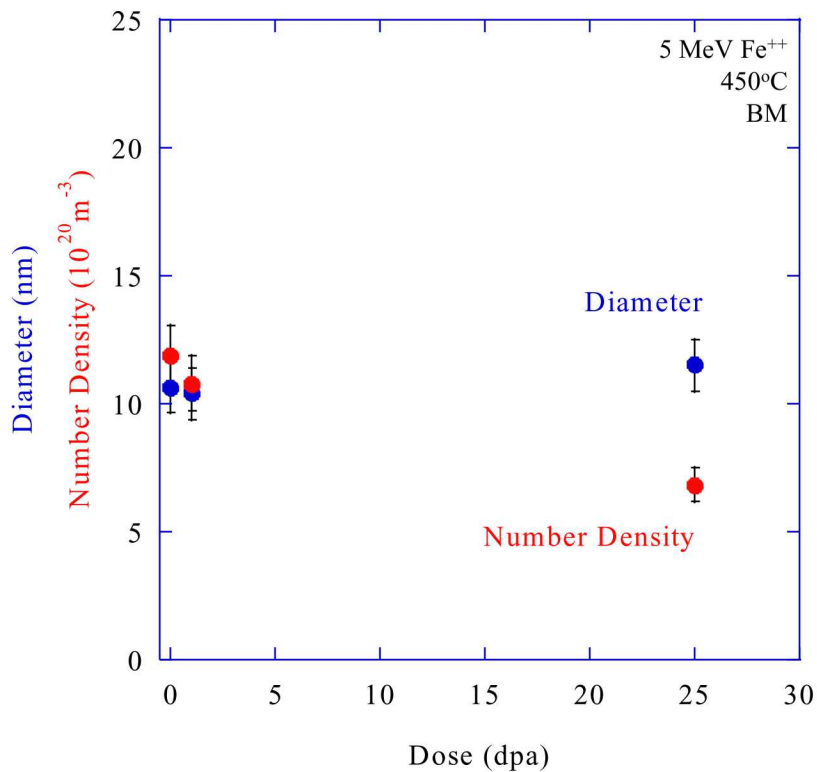


- Small precipitates seen in all tips of irradiated SZ
- No larger coarsened precipitates observed at 25 dpa
  - Likely due to the low density of very large precipitates





# Differences between BM and SZ



- Interesting implications- re-precipitation reverses softening of SZ
- Two different mechanisms potentially explain different evolution





# Low Dose Conclusions

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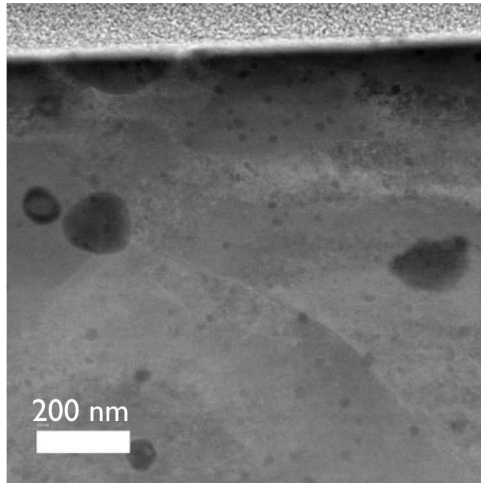
- Dispersoids in the base material increased in diameter and decreased in number density
  - Attributed to Ostwald Coarsening
- Dispersoids in stir zone increased in both diameter and number density due to re-precipitation of dispersoids
  - Attributed to Radiation enhanced diffusion



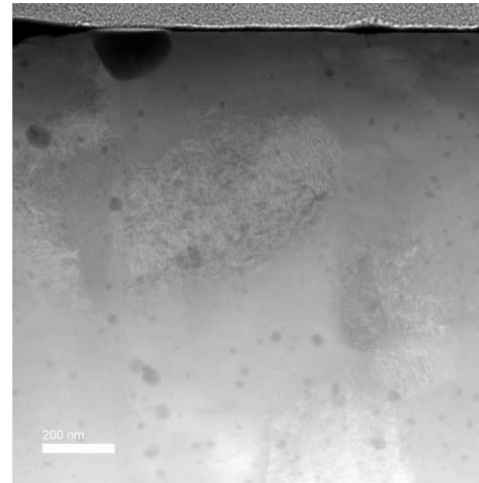


# Base Material: 450°C up to 200 dpa

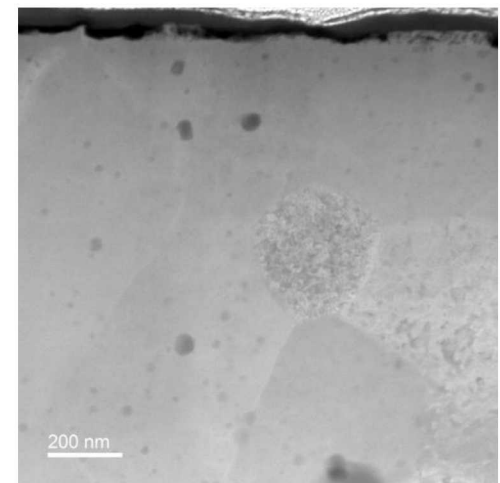
0 dpa



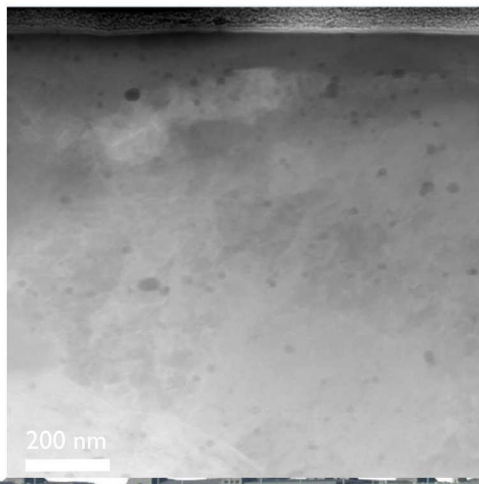
1 dpa



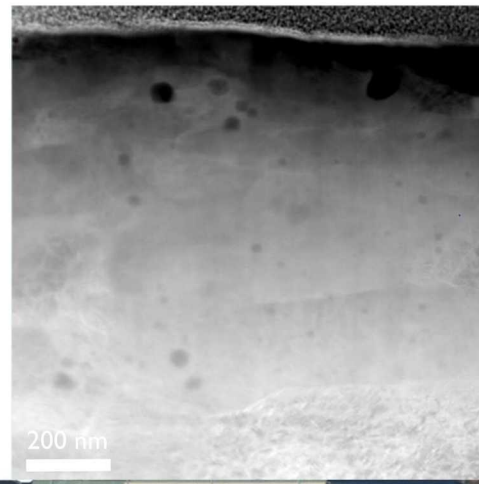
25 dpa



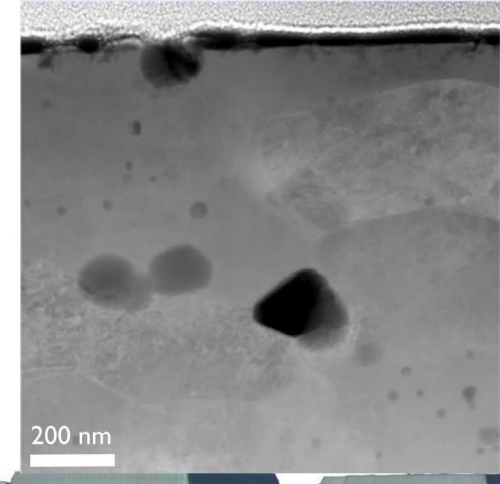
50 dpa



100 dpa

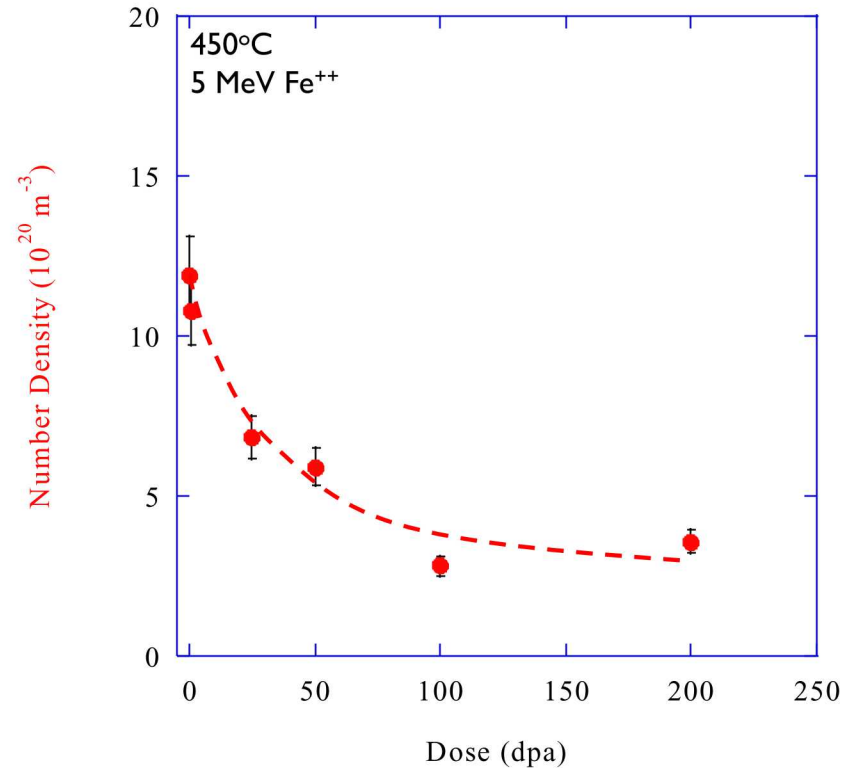
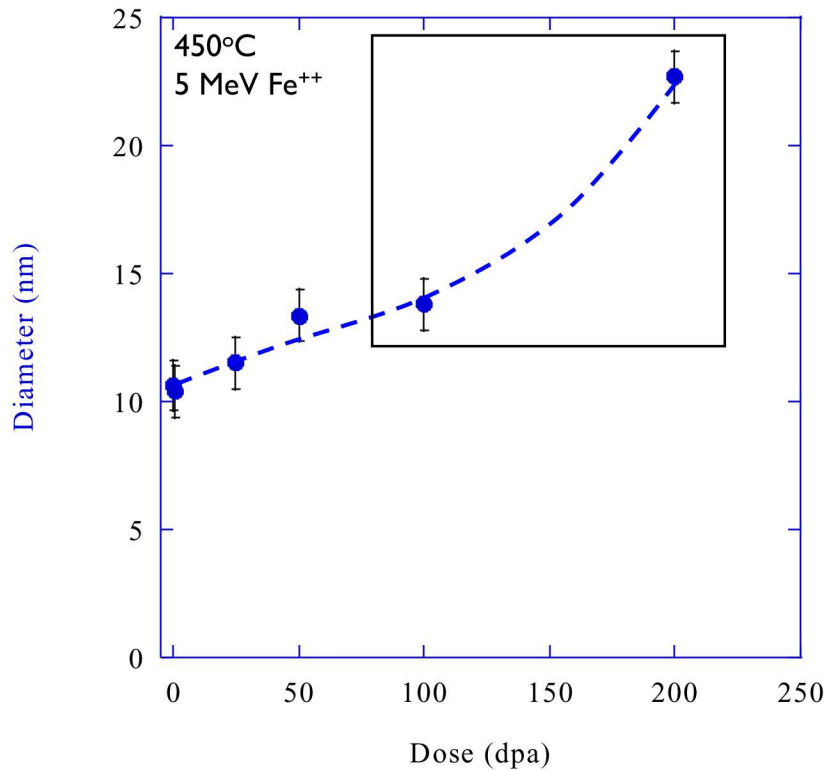


200 dpa





# Dispersoid Evolution with Increased dpa (BM)

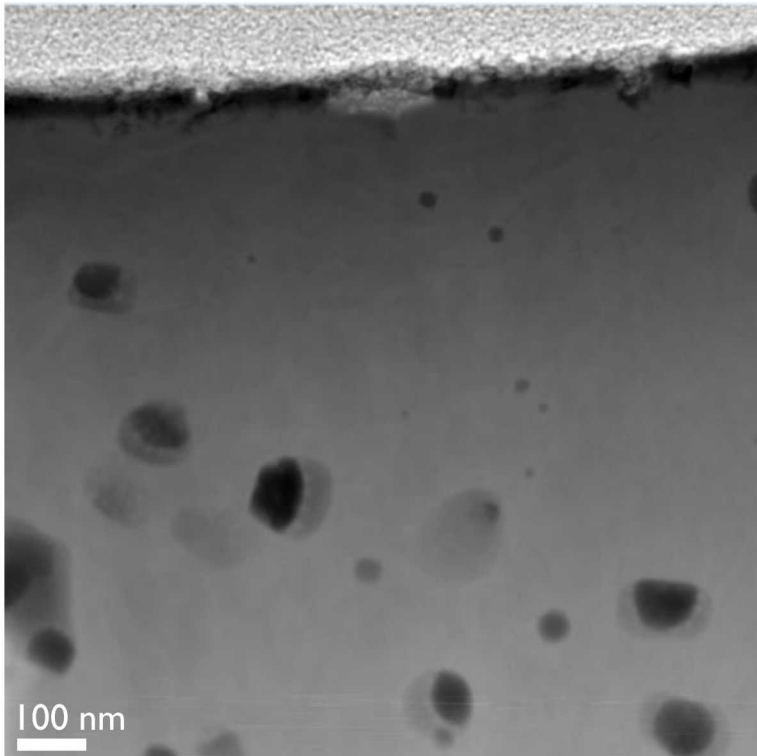


- Evidence of Ostwald coarsening with irradiation
  - Continues low dose behavior of increased diameter, decreased number density
- Large jump in diameter at 200 dpa- formation of voids?





# Formation of voids or coarsening of Y-Al-O?

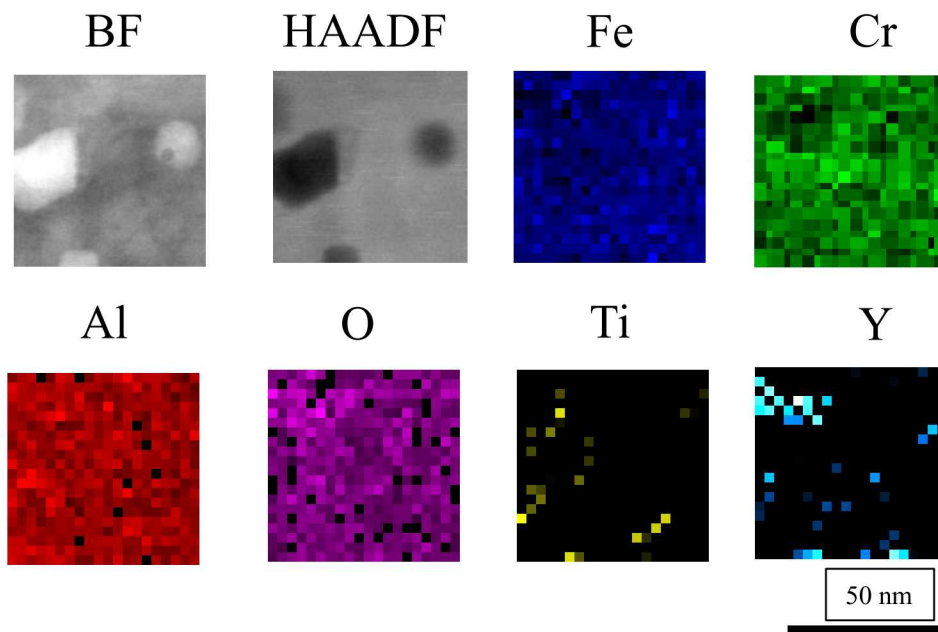
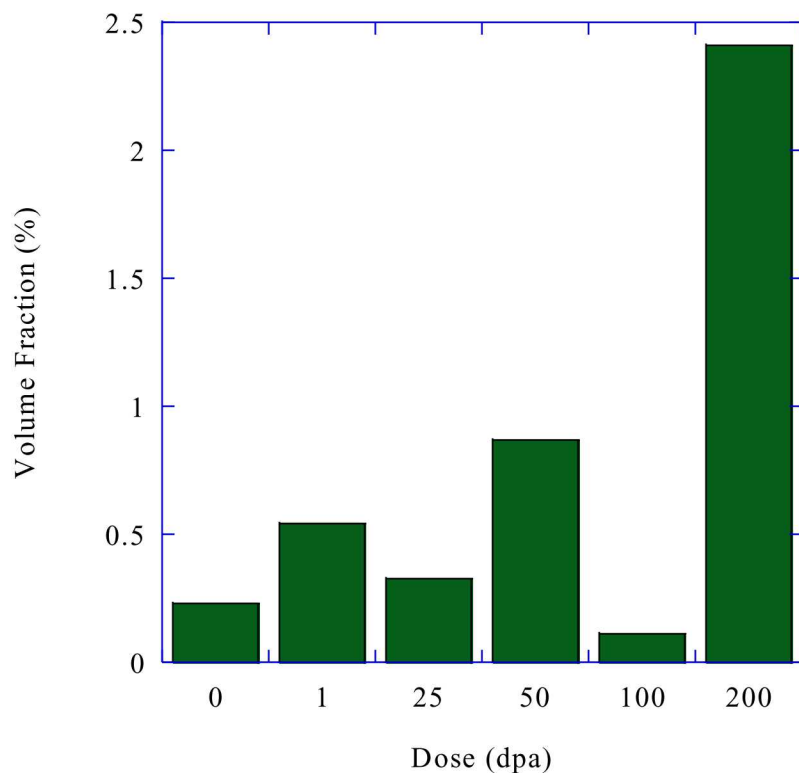


- STEM HAADF- Z contrast so Y-Al-O could look like a void
- CTEM BF can be used to identify by focusing series for voids, but time consuming and difficult to get accurate sizes
- Best approach suggested by Parish et al with a combination STEM HAADF, STEM through focus imaging coupled with high fidelity EDS (ChemiStem)
  - Will be attempting in next RTE to approximate precipitate coarsening versus void swelling

Parish, C. M., Field, K. G., Certain, A. G., & Wharry, J. P. (2015). *Journal of Materials Research*, 30(9), 1275–1289.  
<https://doi.org/10.1557/jmr.2015.32>



# Evolution of Volume Fraction



- Volume fraction suggest new large phase forming (suspected voids) by 200 dpa
- EDS Scans inconclusive
  - Plan on using ChemiStem to get better idea of what these features are





# APT confirms large Al-O and Y-Al-O



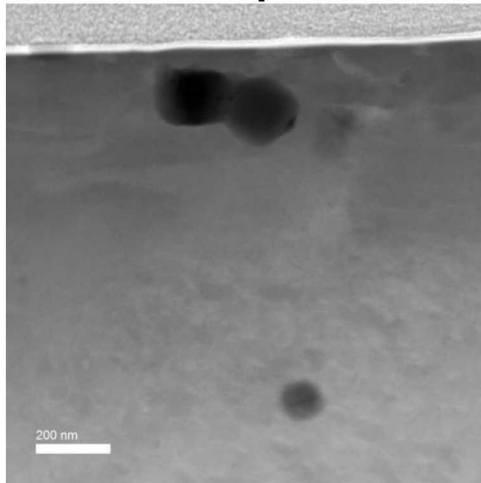
- CrC precipitates were not observed at 1 or 25 dpa
- Ti co-located with Cr-C as well



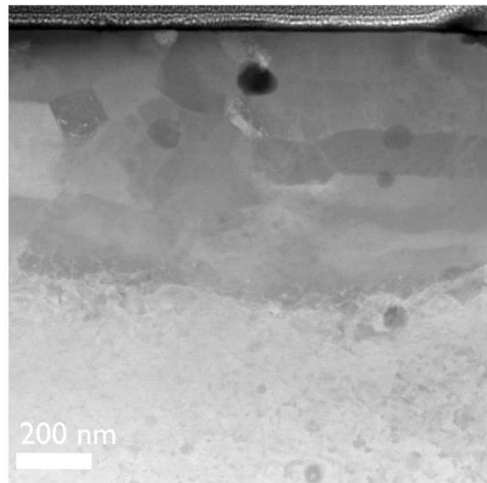


# Stir Zone: 450°C up to 200 dpa

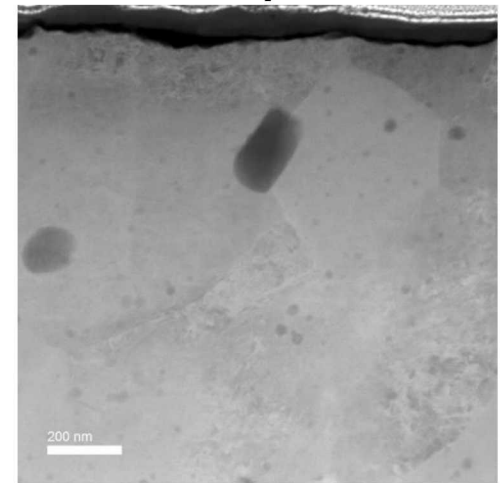
0 dpa



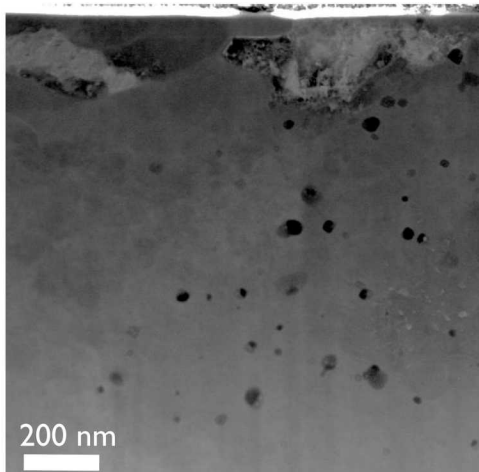
1 dpa



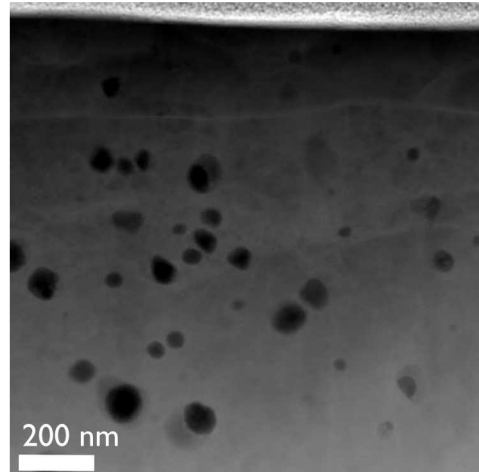
25 dpa



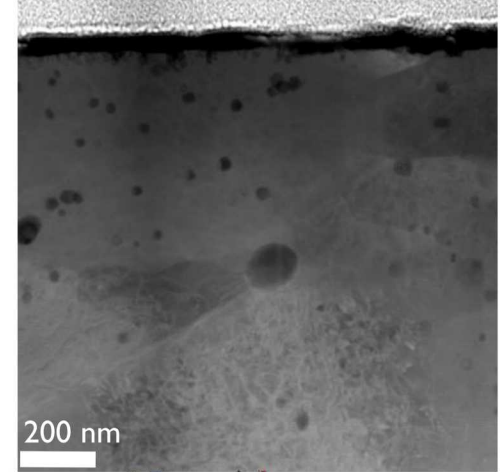
50 dpa



100 dpa

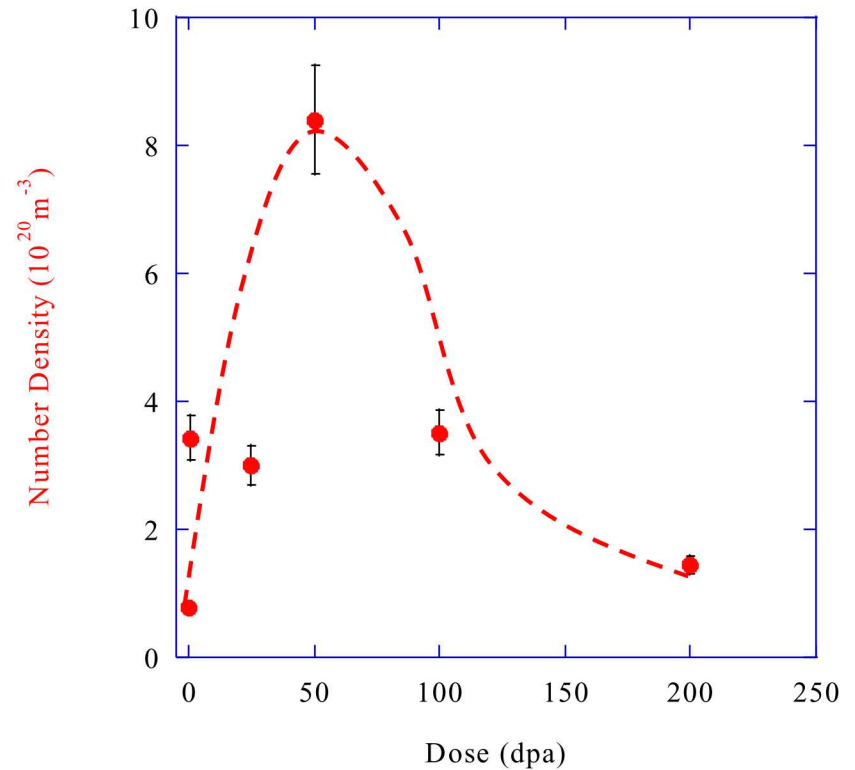
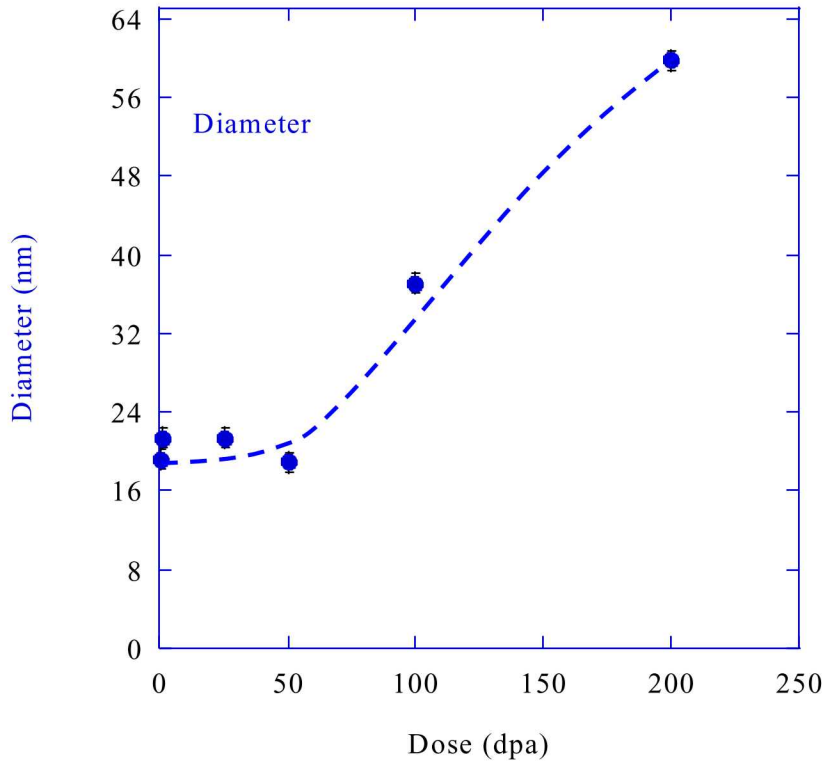


200 dpa





# Dispersoid Evolution with Increased dpa (SZ)

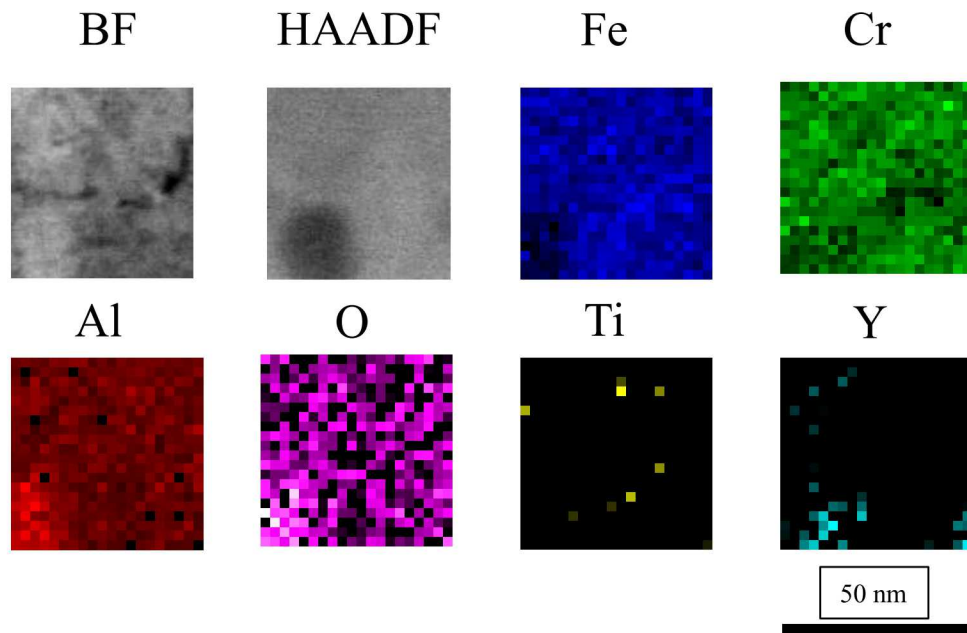
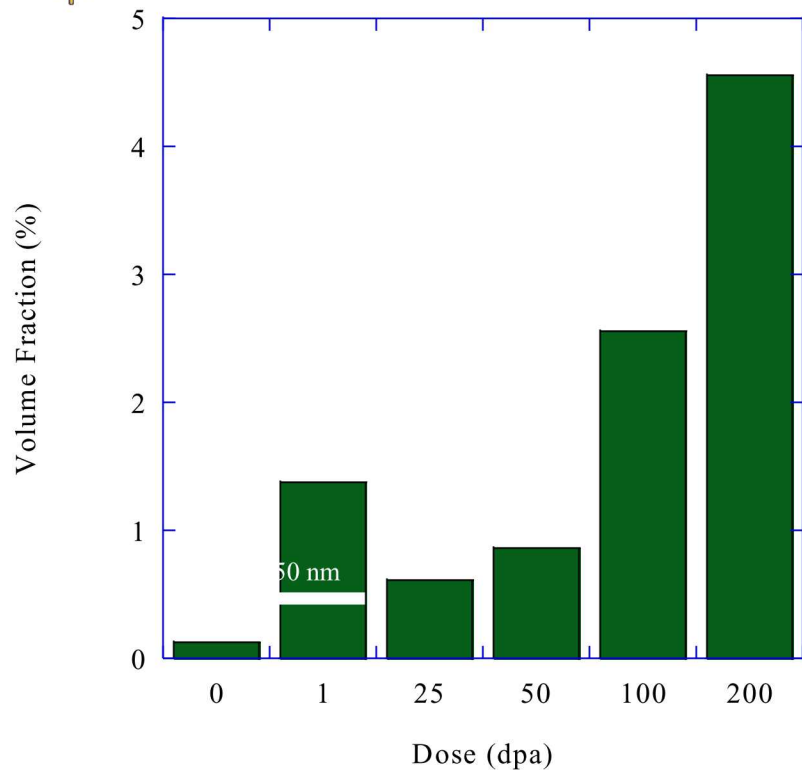


- Rapid growth at 100 dpa and beyond likely due to some void swelling similar to jump at 200 dpa for BM





# Evolution of Volume Fraction?



- Similar to BM-volume fraction suggest new large phase forming by 100 dpa
- EDS Scans inconclusive
  - Plan on using ChemiStem to get better idea of what these features are





# Qualitative coarsening observed with APT in SZ

Al

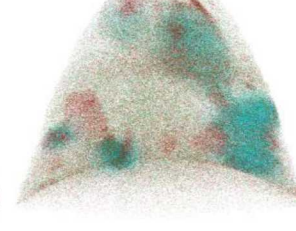
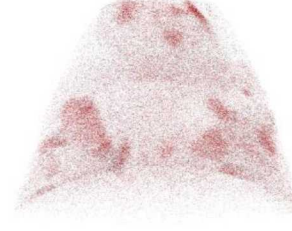
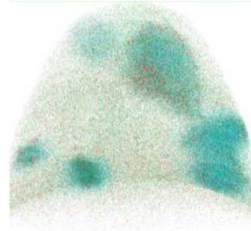
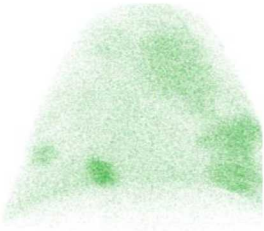
Y

Y-Al-O

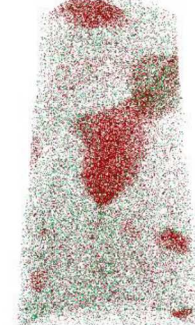
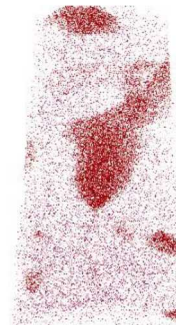
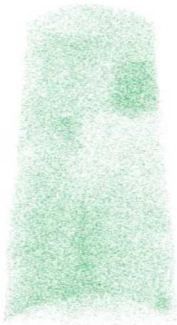
Cr-C

Y-Al-CrC

20 nm

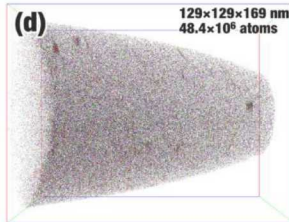


20 nm



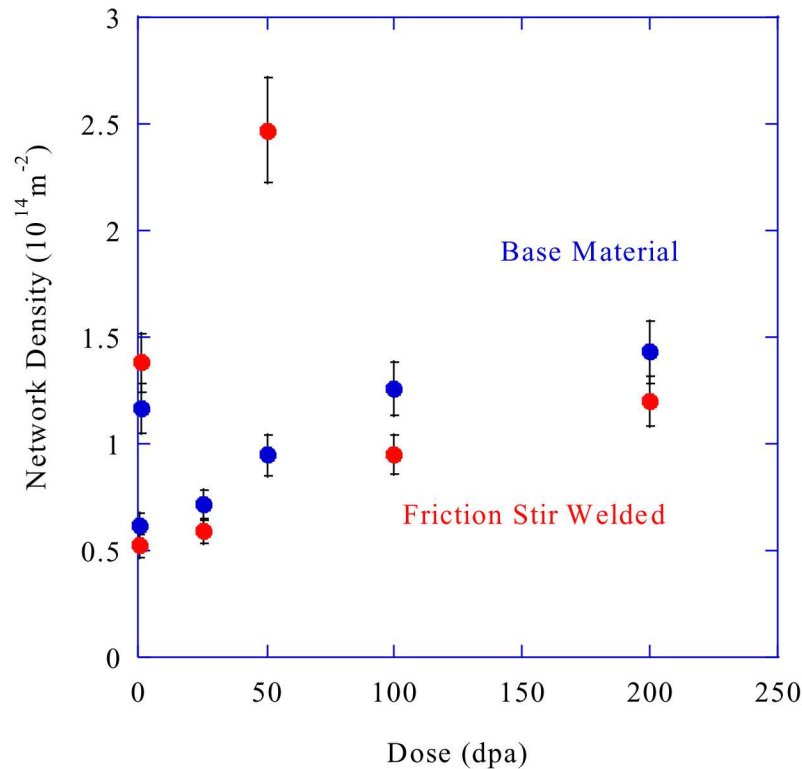
Stir zone, 25 dpa

Cr  
Al  
Y  
O  
Ti  
Si  
C

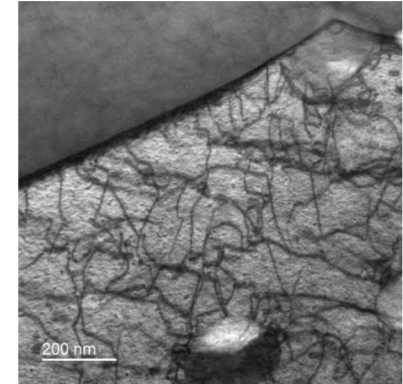




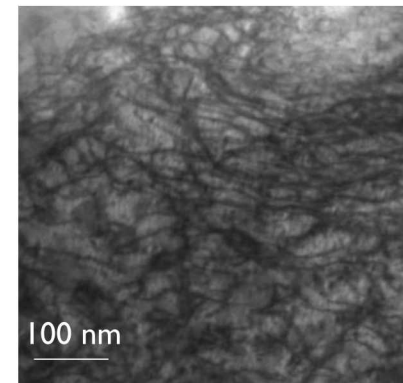
# Evolution of Network Density



SZ, as received



SZ, 50 dpa



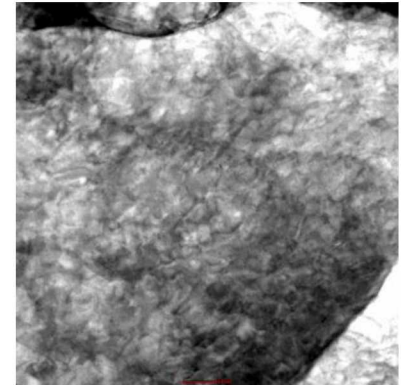
- Network density correlated to strength and typically increases with irradiation according to literature
  - Small decrease in network line density with welding likely due to annealing



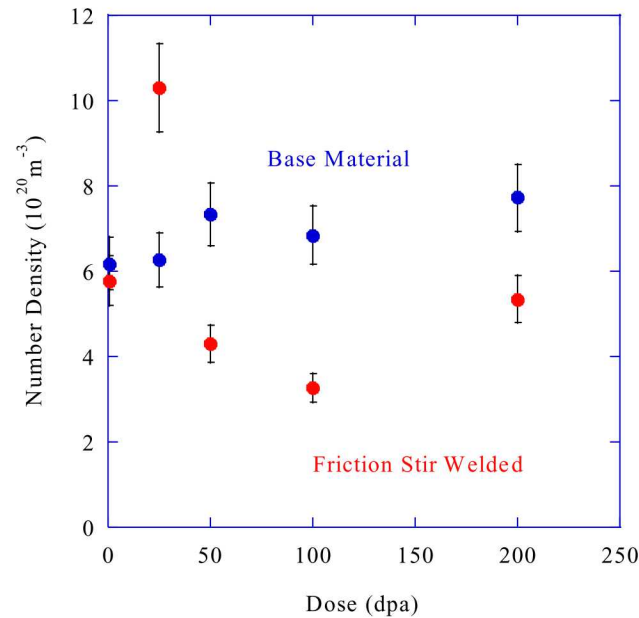
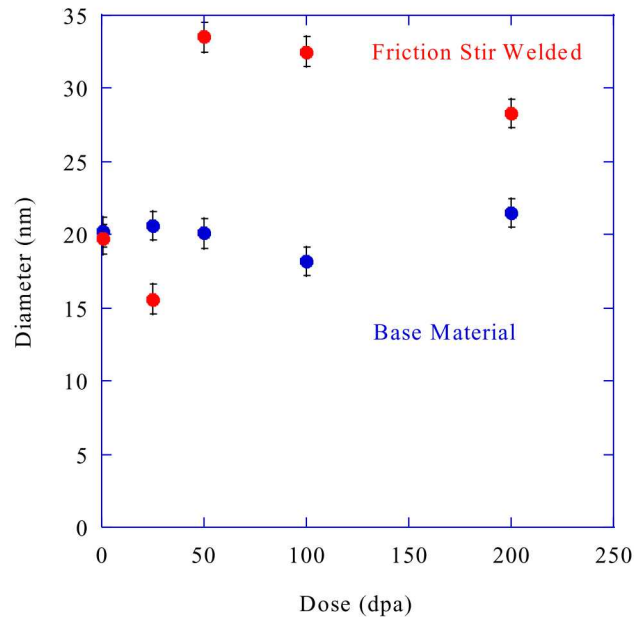
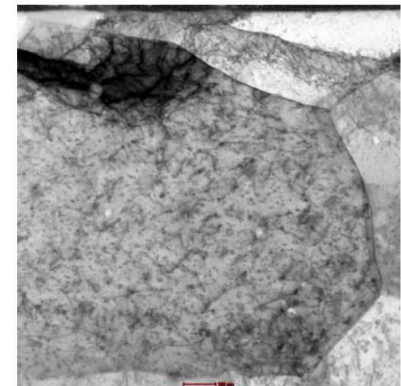


# Evolution of Dislocation Loops

BM, 450°C, 50 dpa



SZ, 450°C, 50 dpa



- Relatively large loops formed by 1 dpa also recovers lost strength from welding



# Significant Findings

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- With irradiation, coarsening was observed in the dispersoids in the base material up to 200 dpa.
- The dispersoids in the stir zone exhibited both coarsening as well as potential re-precipitation of dispersoids lost during the welding process suggesting that strength lost due to welding process may be recovered via irradiation.
- At high dose (200 dpa for BM and 100 dpa for SZ), suspected irradiation induced voids may have formed.
- Preliminary APT results suggest formation of CrCs by 50 dpa as well as coarsening of Al-O and Y-Al-O
- Dislocation microstructure stable in BM but continues to evolve slowly in SZ





# Future Work

- Temperature dependence of BM, SZ
  - Irradiations funded by RTE 17-1032
  - Characterization funded by RTE 18-1396 (in progress)
- Effect of welding conditions
  - Irradiations funded by RTE 17-1032
  - Characterization funded by RTE 18-1396 (in progress)

	400°C	450°C	500°C
<b>1</b>	MA956-BM MA956-FSW-H	MA956-BM MA956-FSW-H	
<b>25</b>	MA956-BM MA956-FSW-H	MA956-BM MA956-FSW-H	
<b>50</b>	MA956-BM, MA956-FSW-H MA956-FSW-M	MA956-BM, MA956-FSW-H	MA956-BM MA956-FSW-H MA-956-M
<b>100</b>	MA956-BM, MA956-FSW-H MA956-FSW-M	MA956-BM MA956-FSW-H MA-956-M	MA956-BM, MA956-FSW-H MA956-FSW-M
<b>200</b>		MA956-BM MA956-FSW-H MA956-FSW-M	

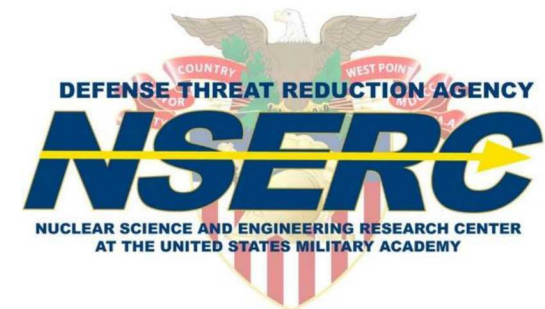
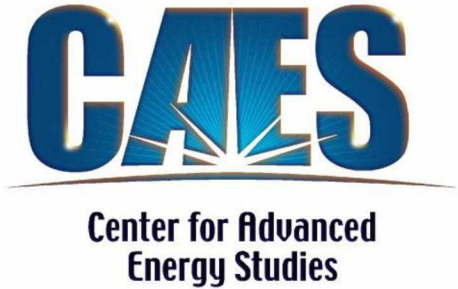
Sample Condition Matrix  
MA956-FSW-H: high heat input  
MA956-FSW-M: medium heat input





# Additional Acknowledgments

- Many thanks to NSUF RTE program and CAES
  - J. Taylor, Y. Wu and J. Burns
- Military Academic Collaboration program at Sandia National Laboratories
- Defense Threat Reduction Agency
  - Consequence Analysis Research for the Defense Threat Reduction Agency Nuclear Science and Engineering Research Center (DTRA/NSERC).





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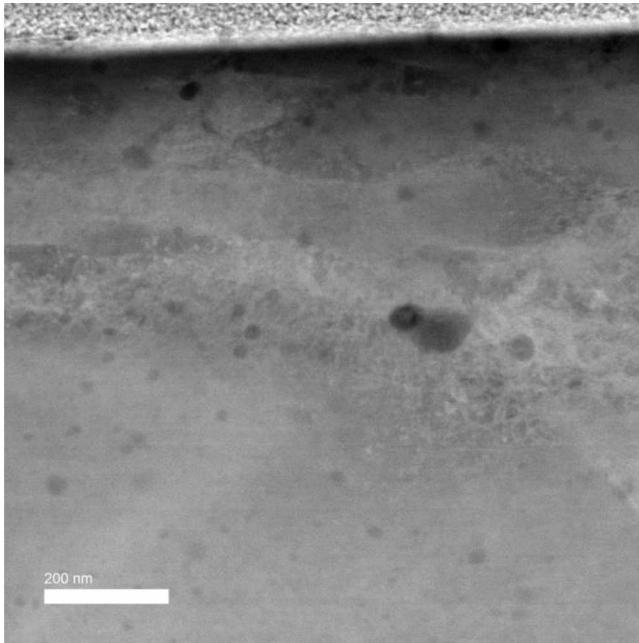
Questions?



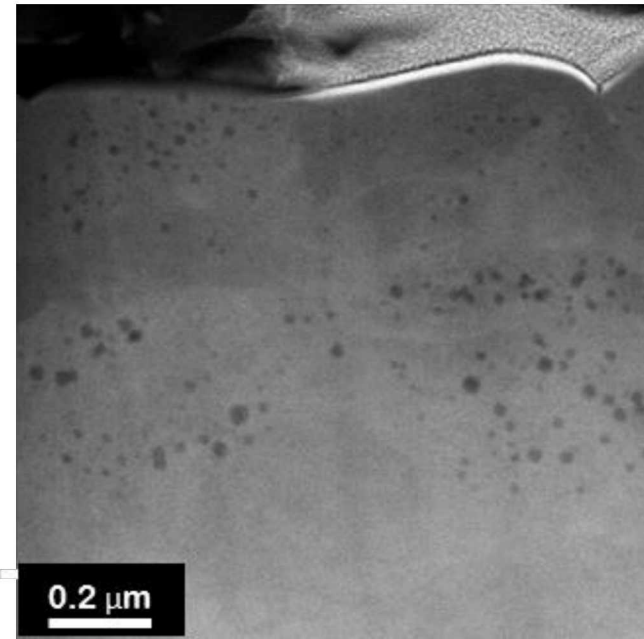


# Dispersoids or voids?

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MA956, unirradiated  
Black features are dispersoids



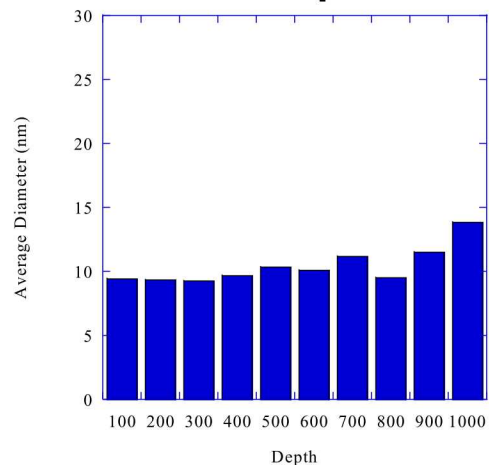
HT9, 440C, 188 dpa  
Black features are voids



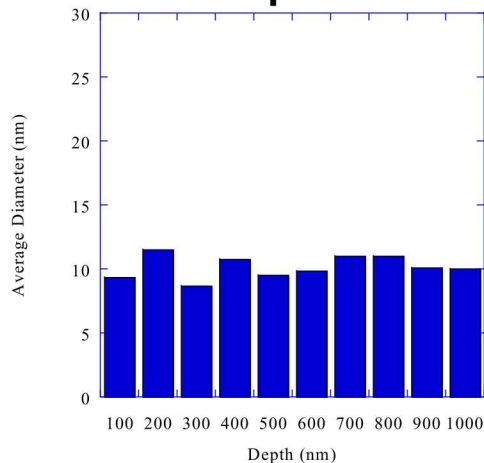


# Depth vs Diameter (BM)

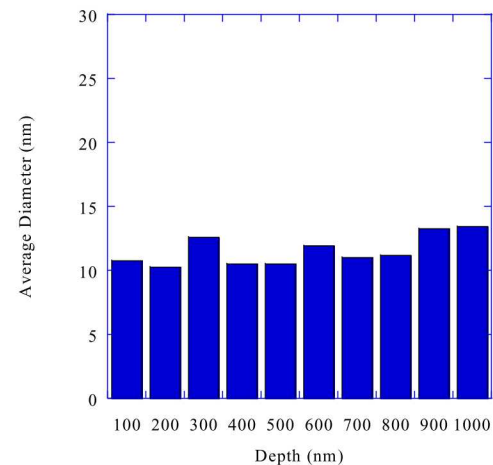
### 0 dpa



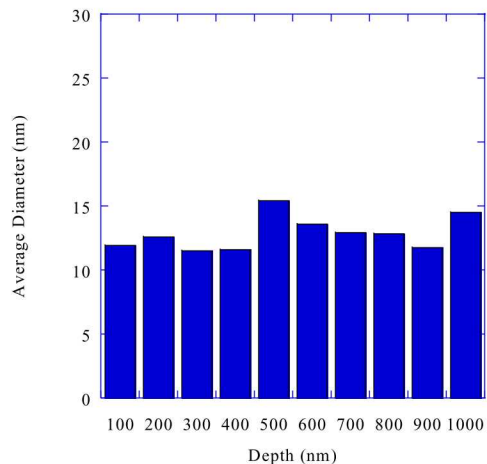
### 1 dpa



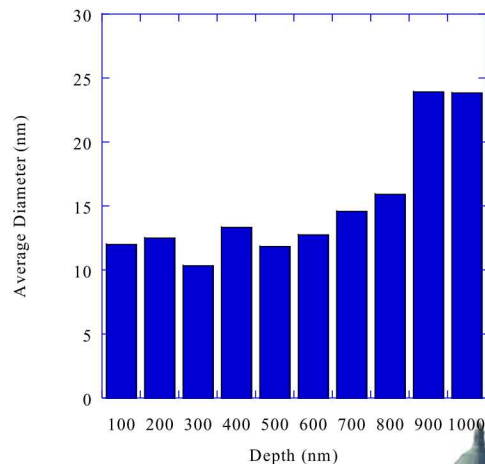
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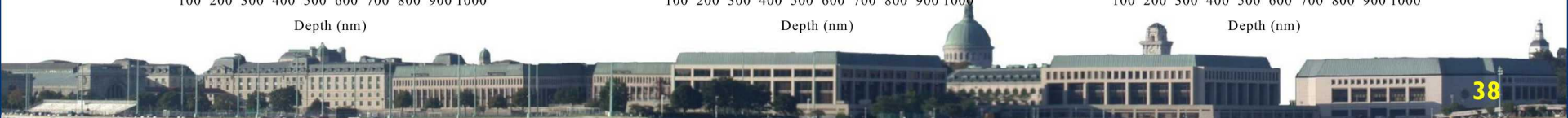
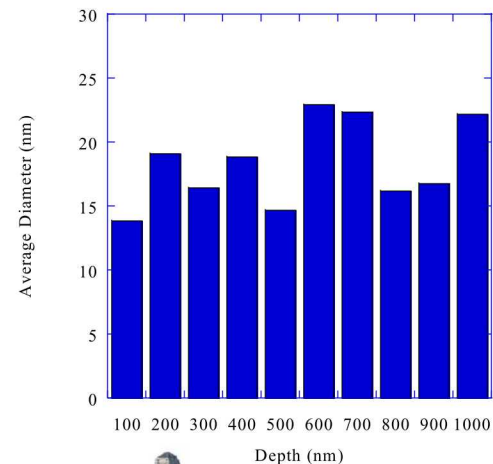
### 50 dpa



### 100 dpa



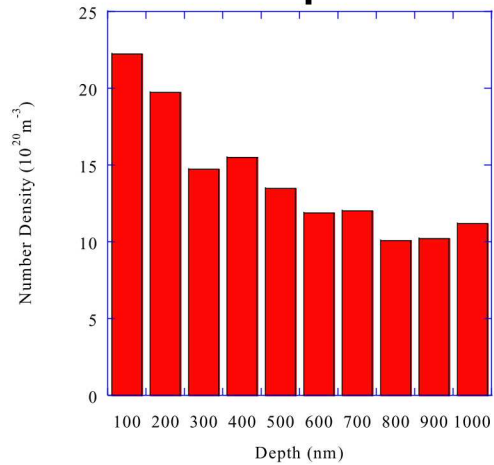
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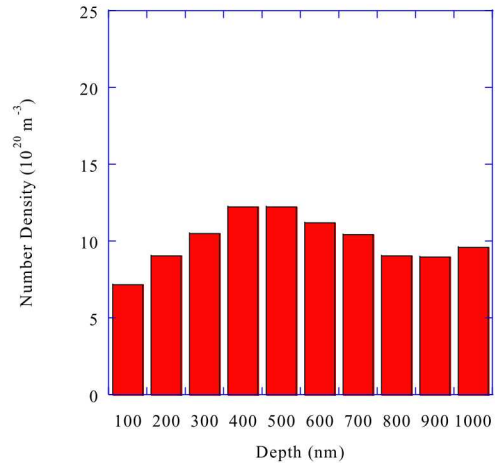


# Depth vs Number Density (BM)

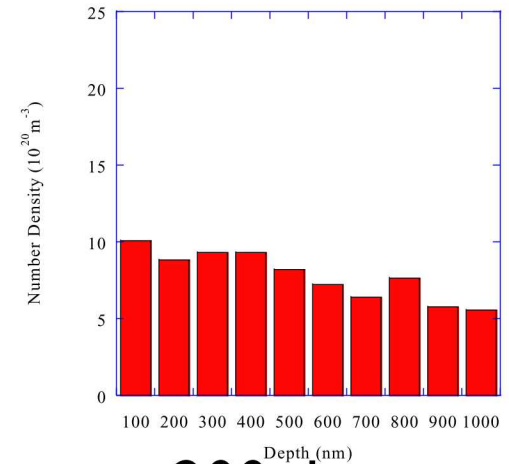
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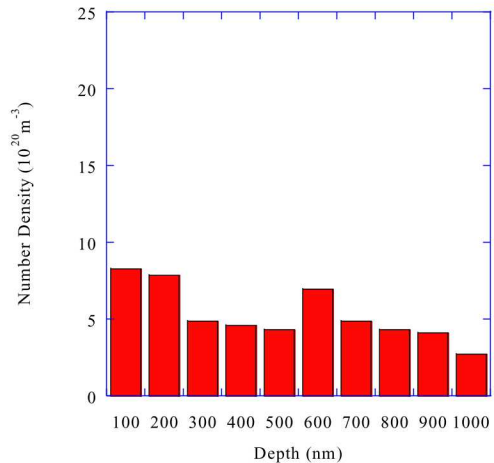
1 dpa



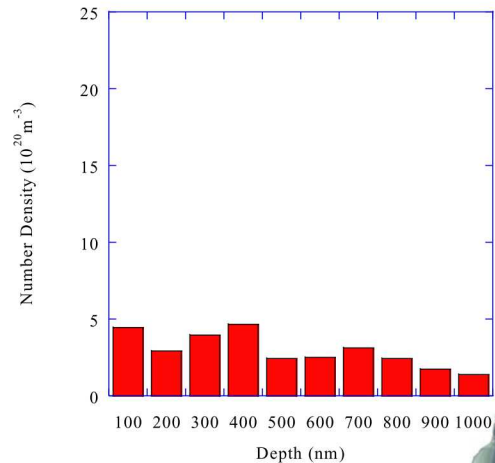
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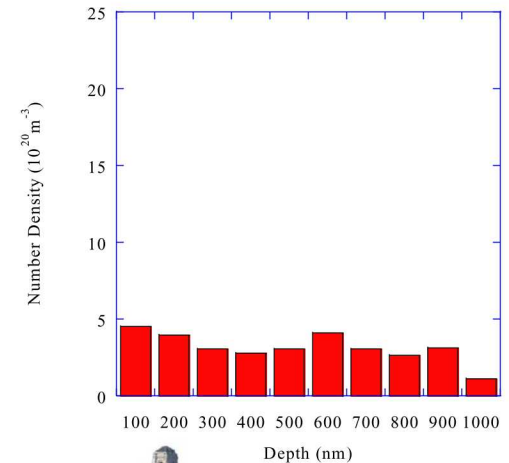
50 dpa



100 dpa



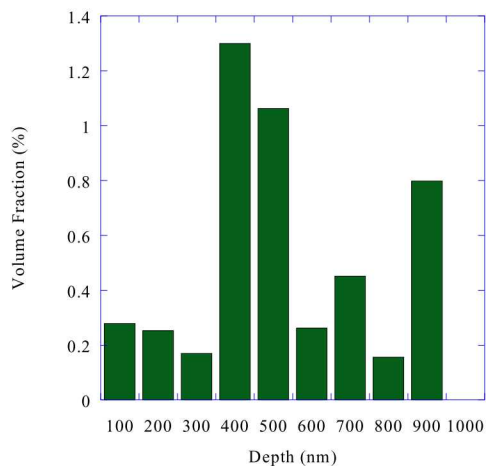
200 dpa



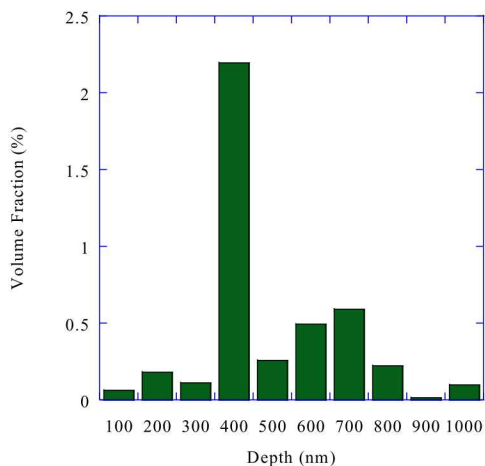


# Depth vs Number Density BM

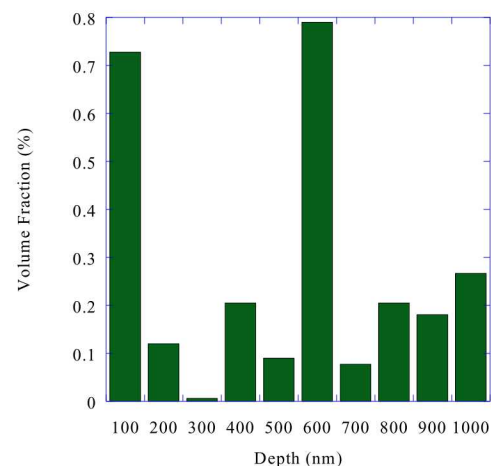
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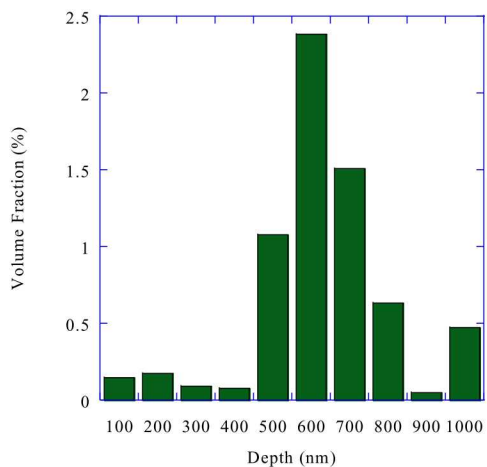
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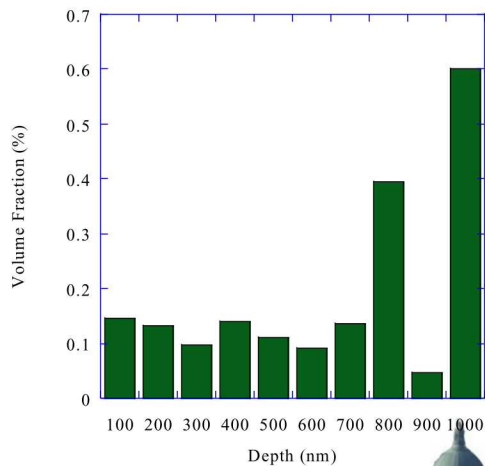
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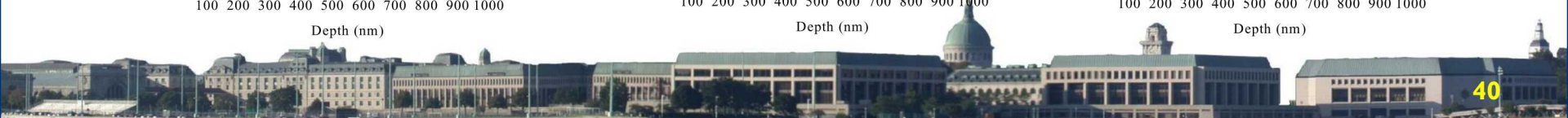
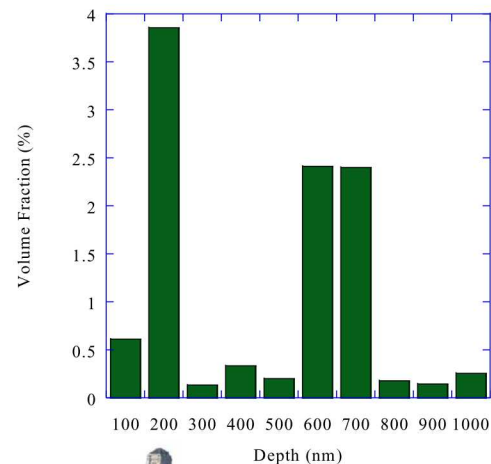
### 50 dpa



### 100 dpa



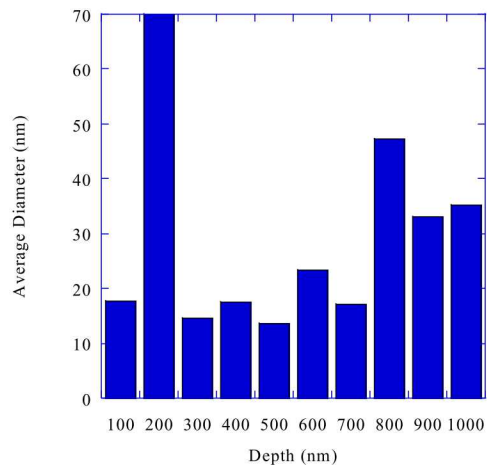
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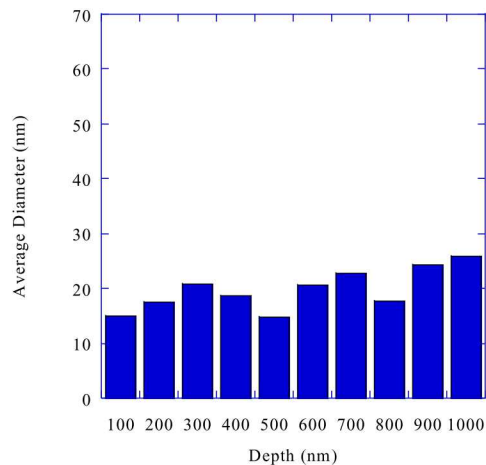


# Depth vs Diameter (SZ)

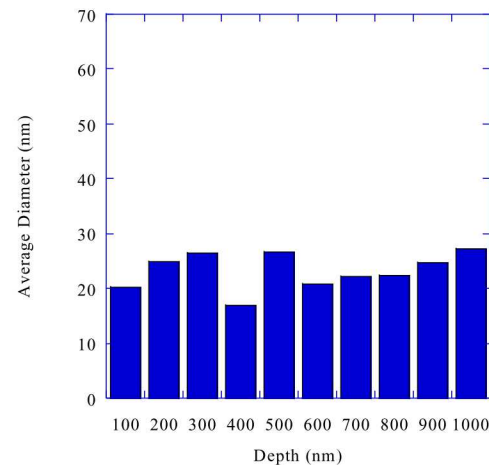
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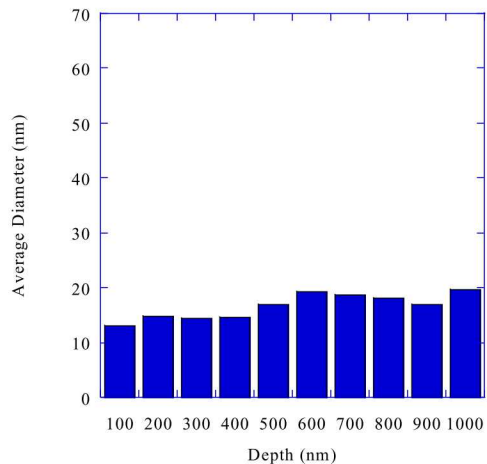
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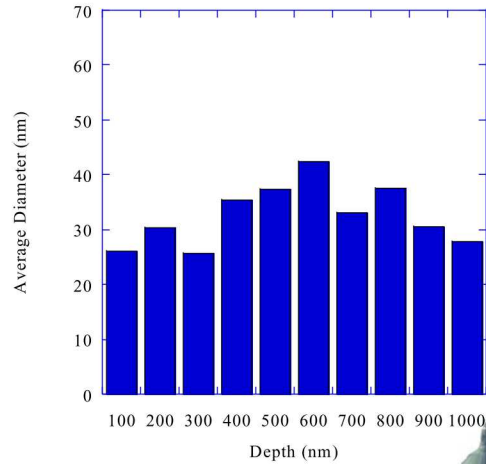
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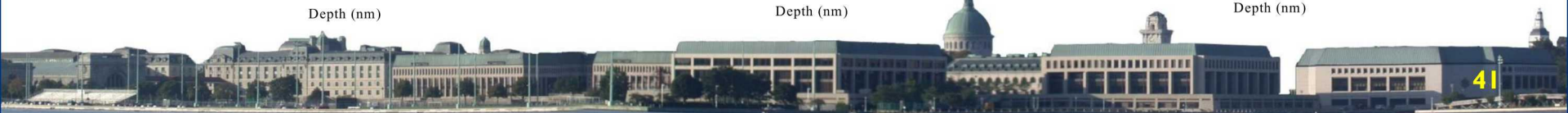
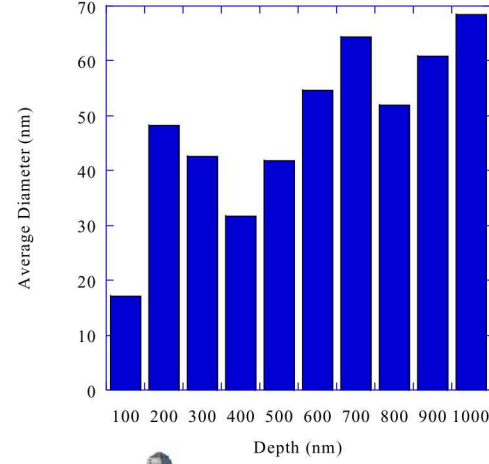
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## 100 dpa



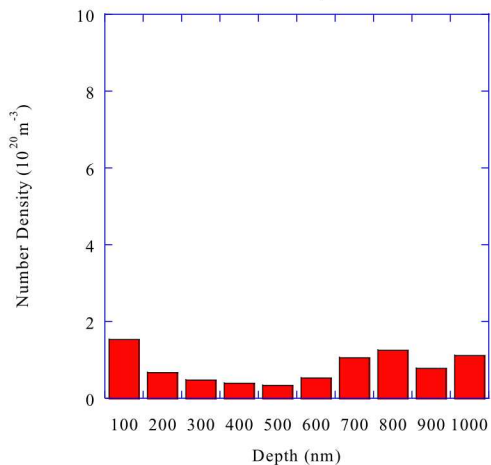
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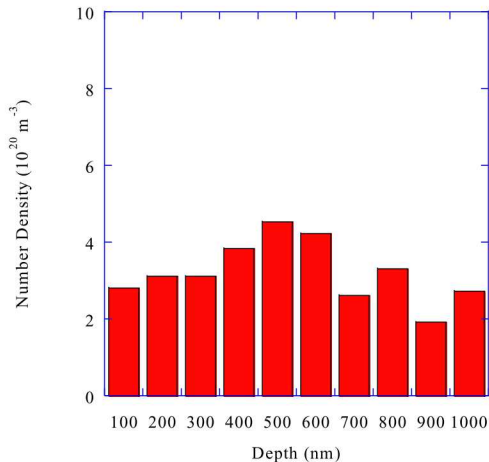


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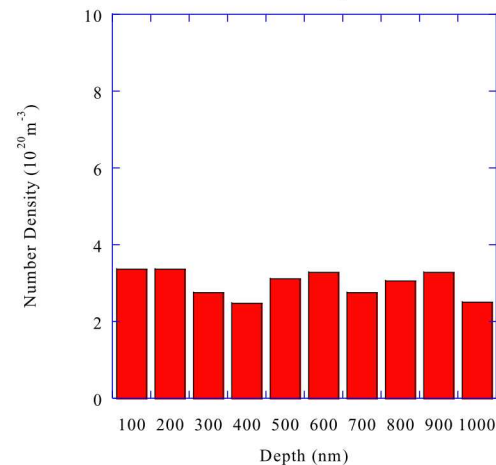
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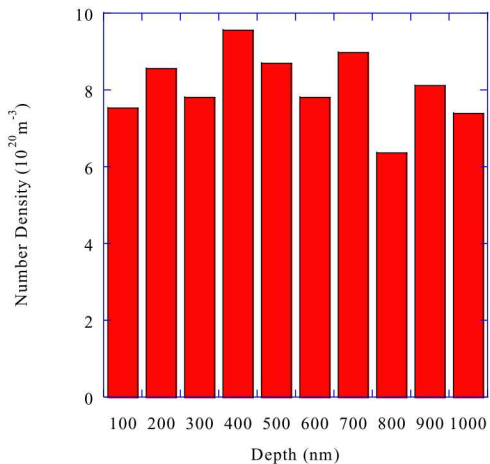
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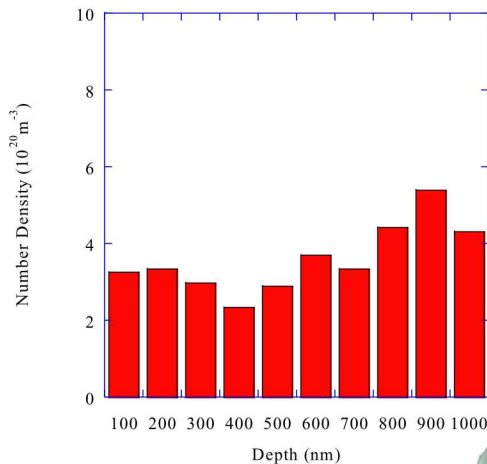
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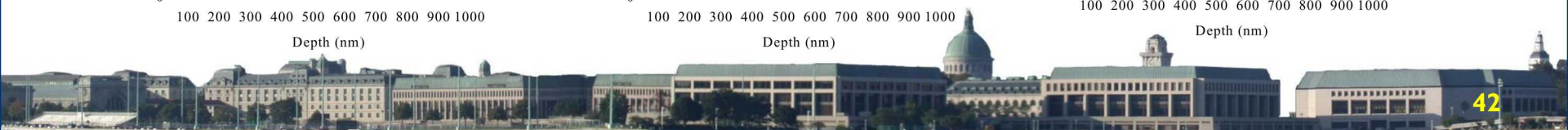
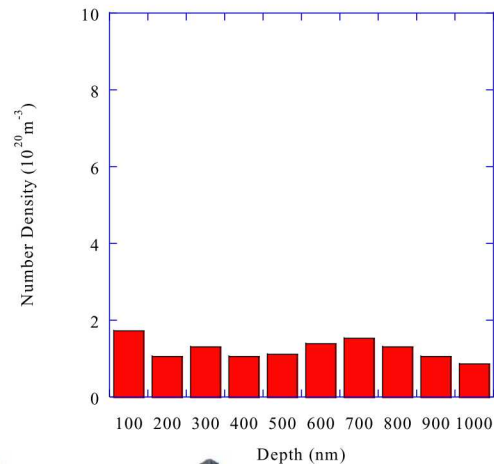
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### 100 dpa



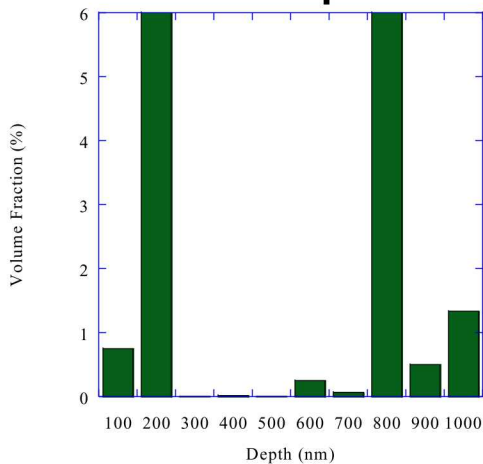
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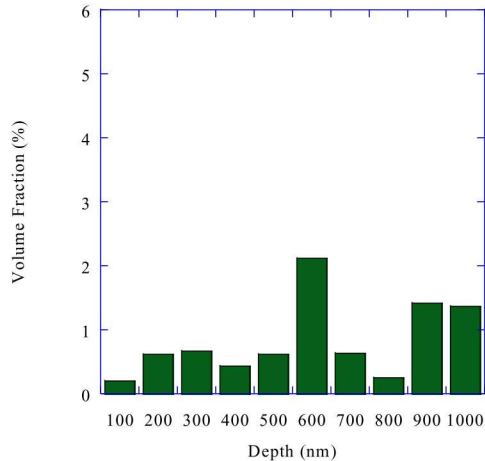


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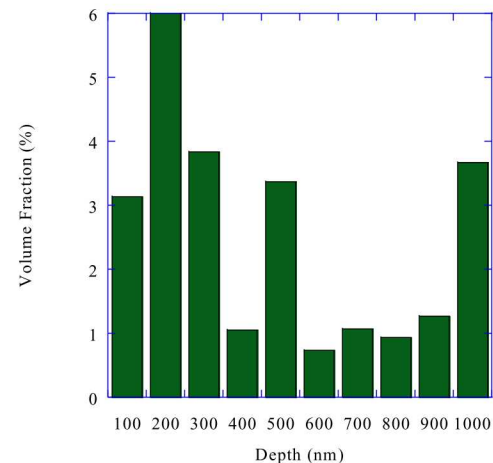
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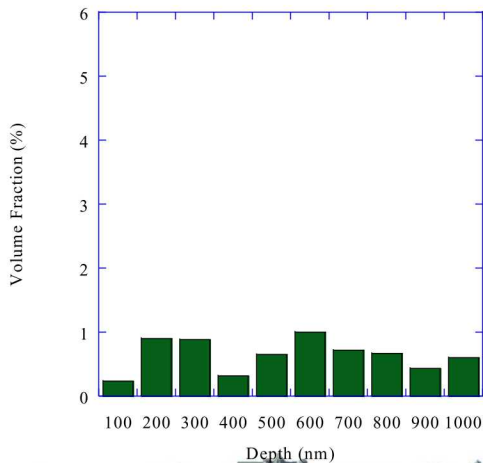
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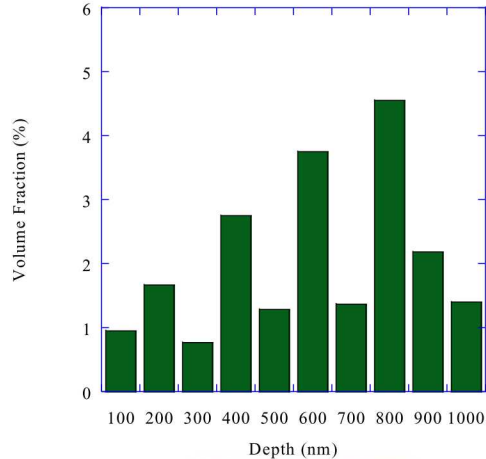
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### 50 dpa



### 100 dpa



### 200 dpa

