



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

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NSUF Users Meeting



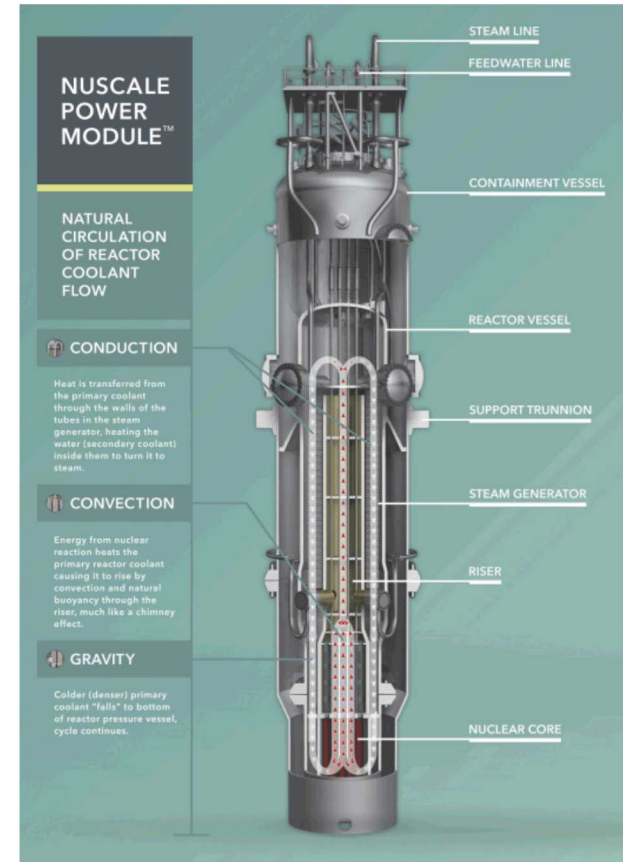
Motivation- Materials Challenges for DOE and DOD

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



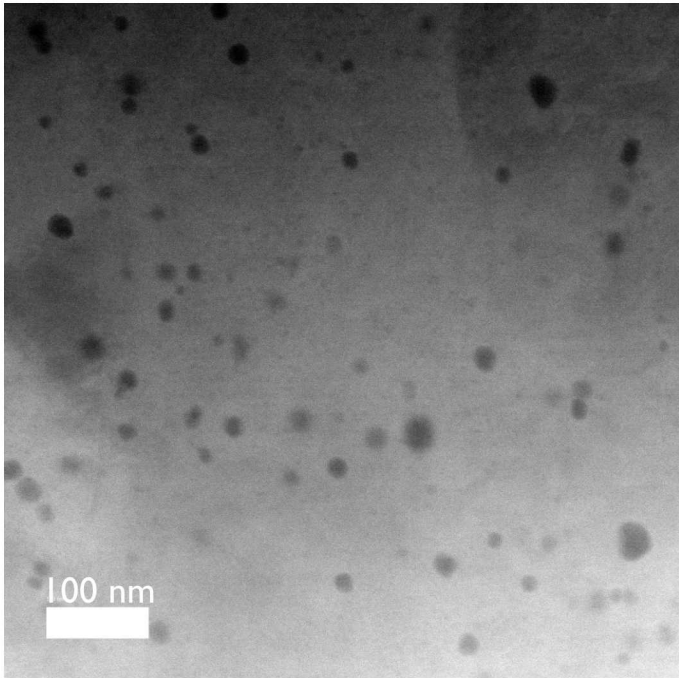
50 MWe modular reactor design

Courtesy of NuScale Power





ODS Steel- A possible reactor material?



MA956 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

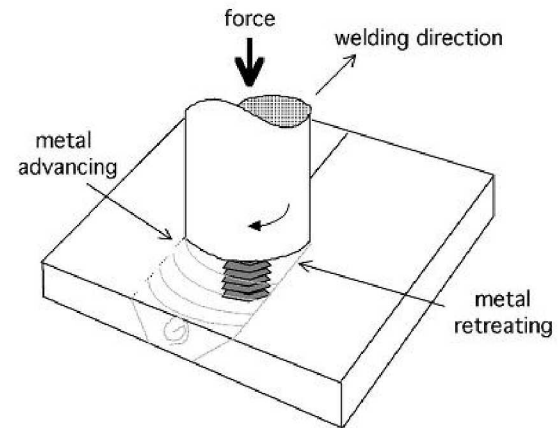
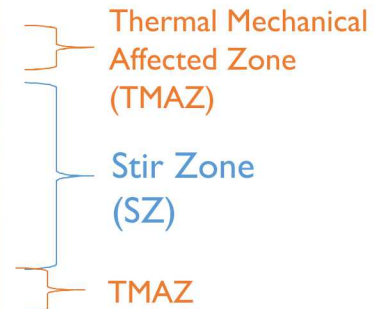




Friction Stir Welding (FSW)

- 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

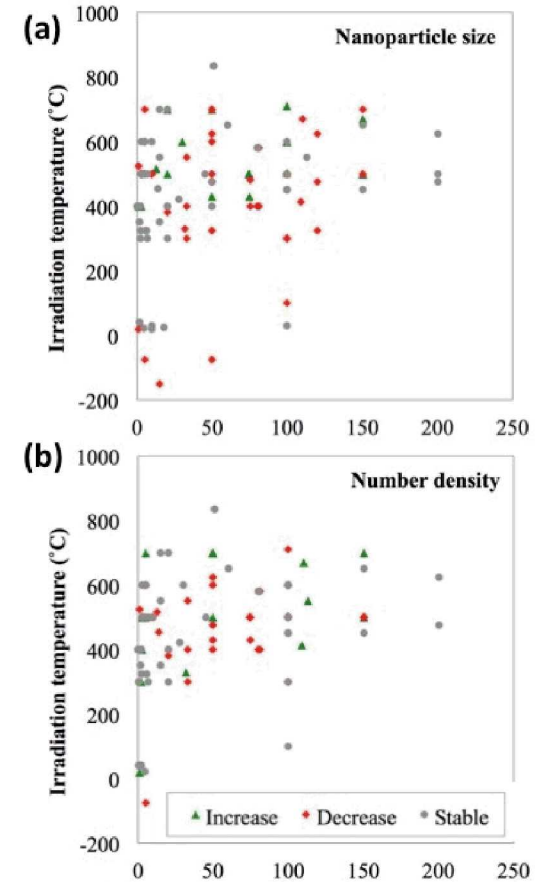




Dispersoid Behavior Inconsistent throughout Literature

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

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





Research Objective

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





Approach

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)

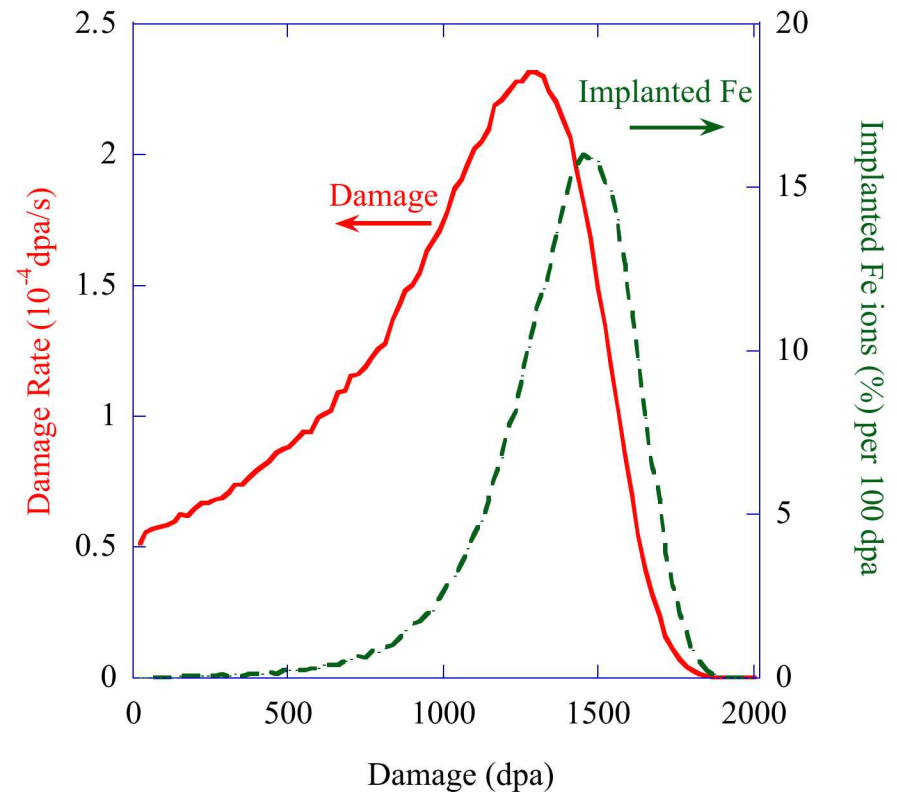




Ion Experiment Design

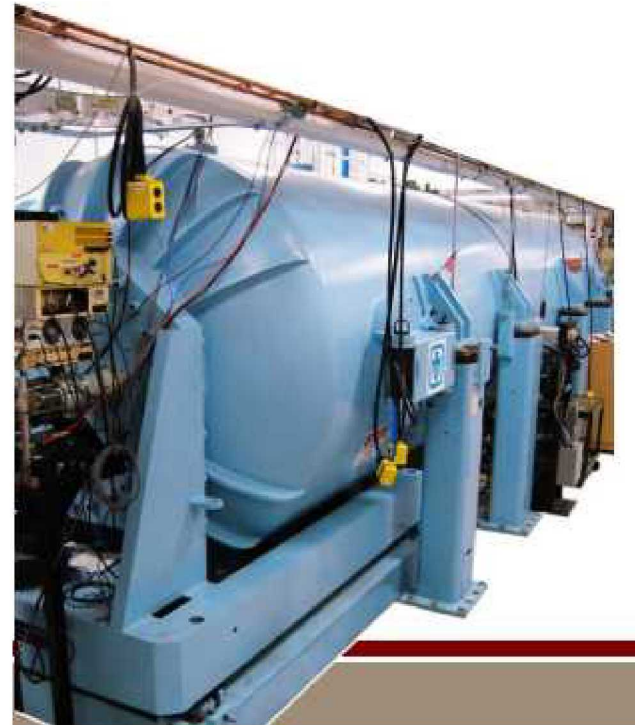
Alloy	Fe	Cr	Al	Y ₂ O ₃	Ti	Mn	Si	Ni	C	Mo	S	P
MA956 (wt%)	Bal	19.93	4.75	0.51	0.39	0.09	0.08	0.04	0.023	0.02	0.008	0.006

- MA956
- Irradiation of tem bar samples with 5 MeV Fe²⁺ 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_d=40 eV
 - Raster Scanning





Ion Experiment Design (SNL)

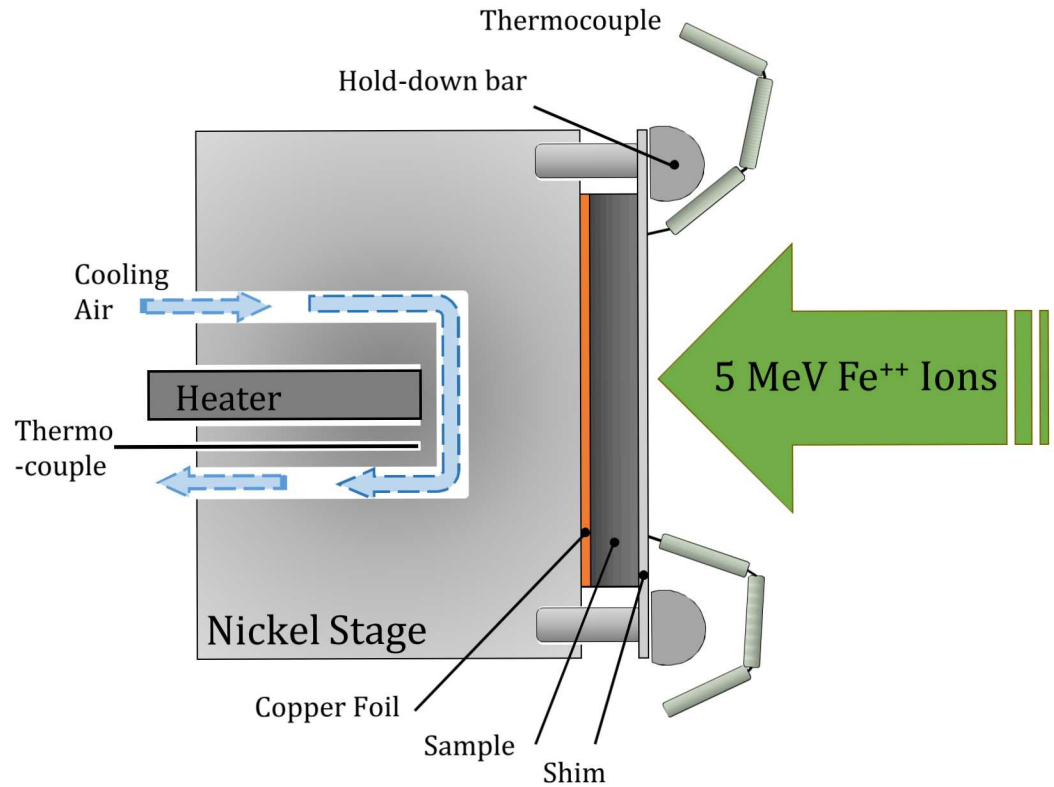
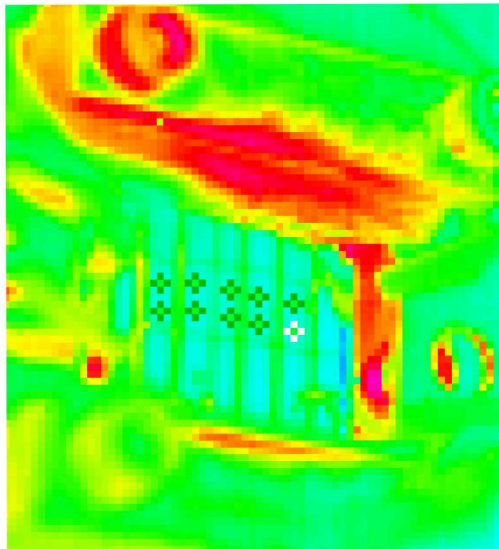


- Irradiations performed at SNL on the 6 MV Tandem
- LabView controlled button heater used to maintain temperature



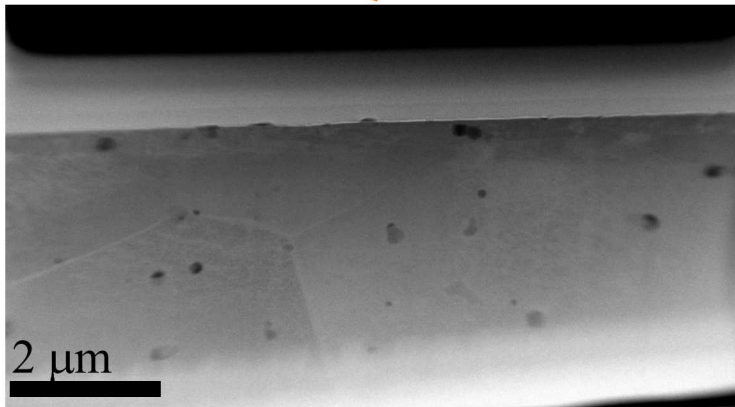
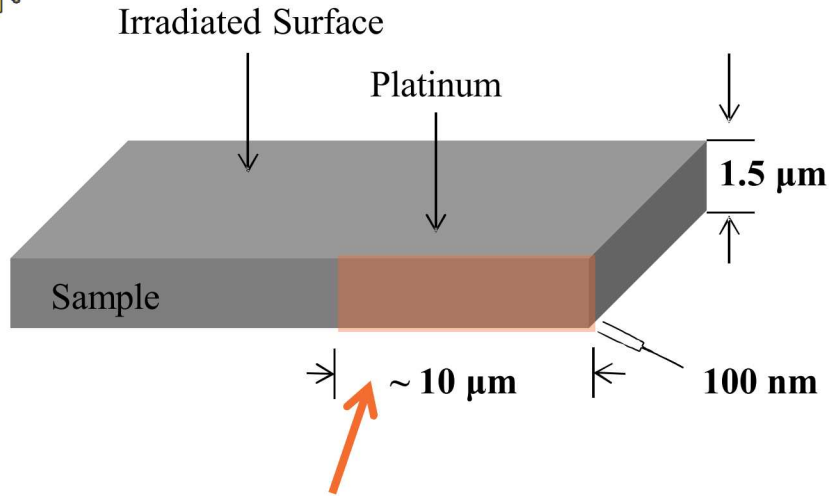


Ion Experiment Design (MIBL)





Microstructure Analysis



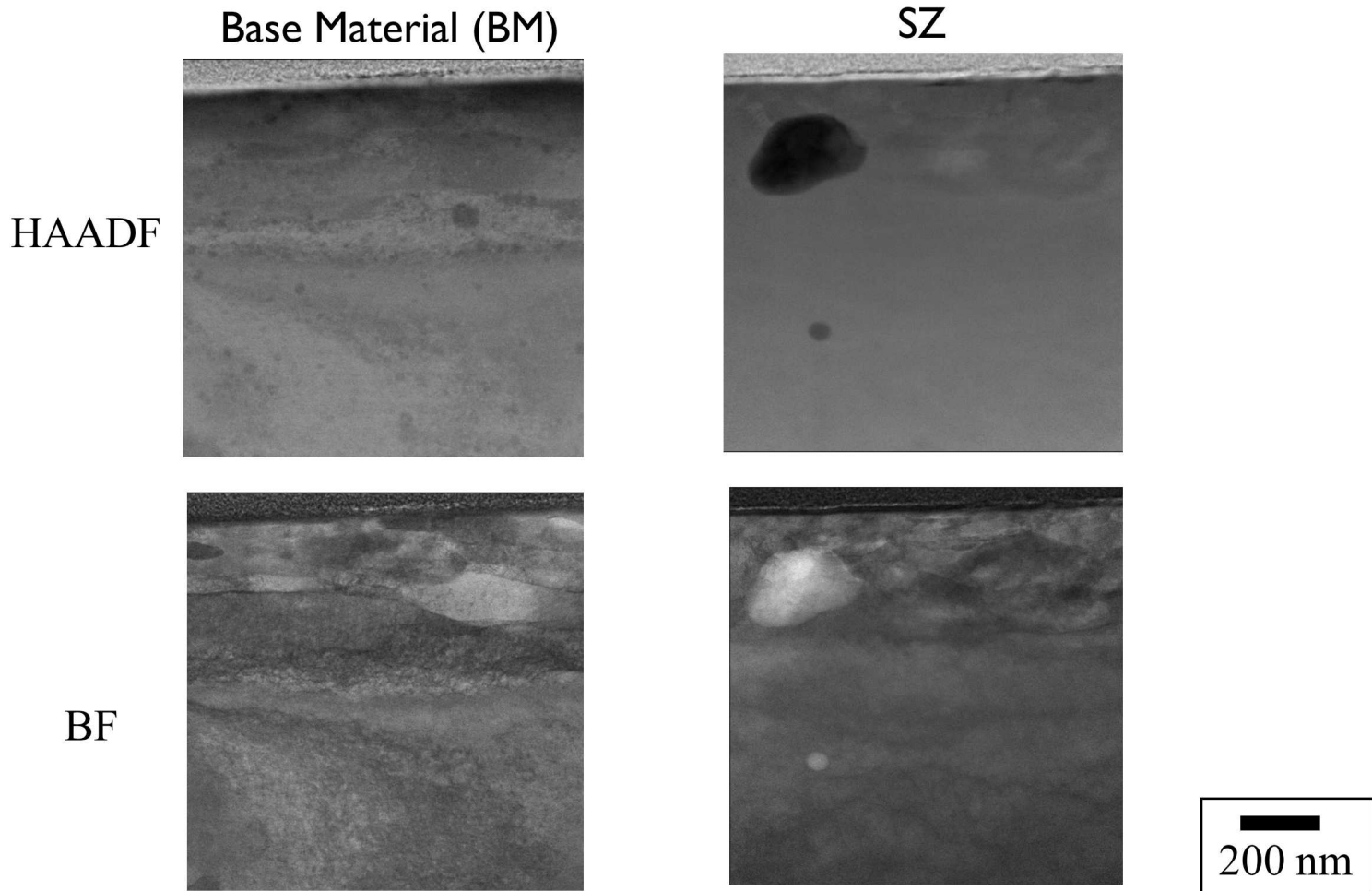
MA956-Friction Stir Welded

- 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



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



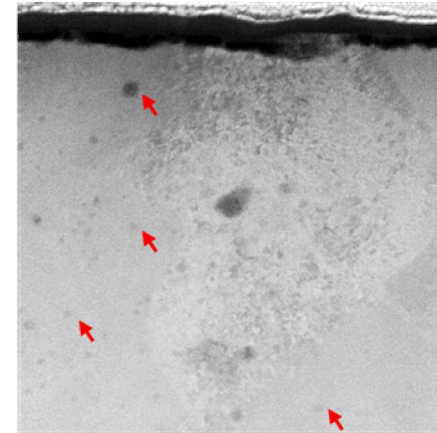
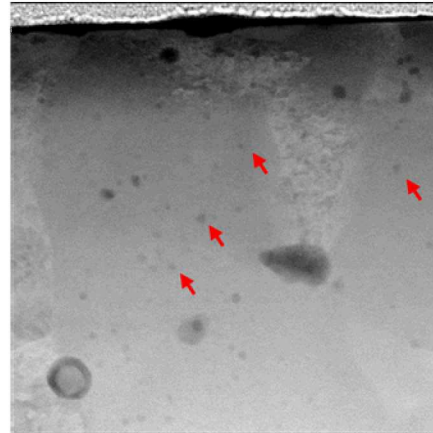
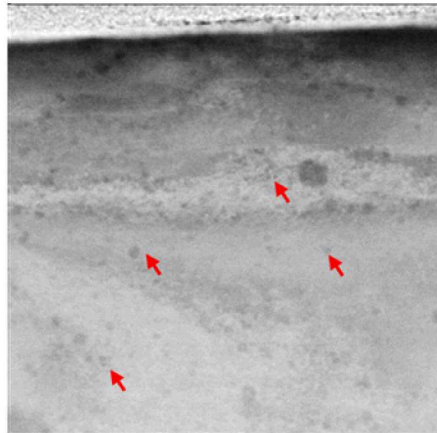


Effect of irradiation on BM (low dose)

0 dpa

1 dpa

25 dpa



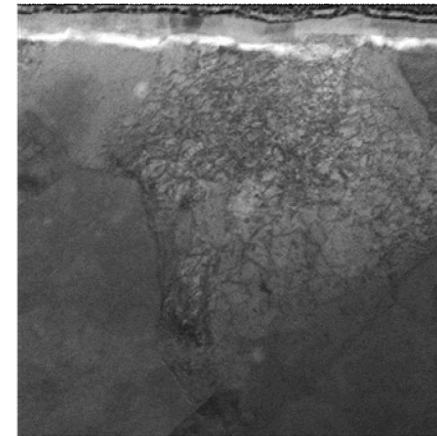
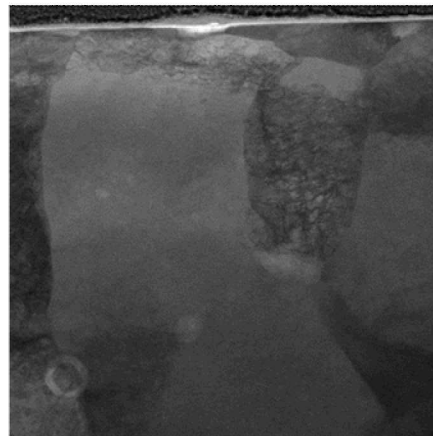
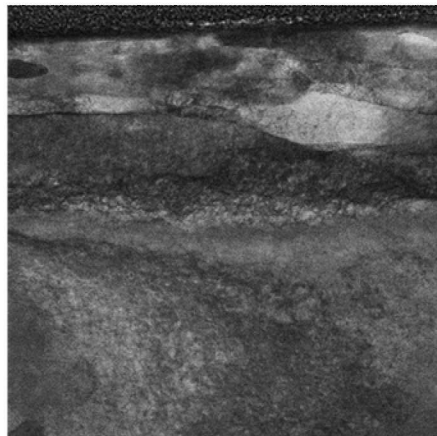
Pt
 Oxide

HAADF

(a)

(b)

(c)



Pt
 Oxide

BF

(d)

(e)

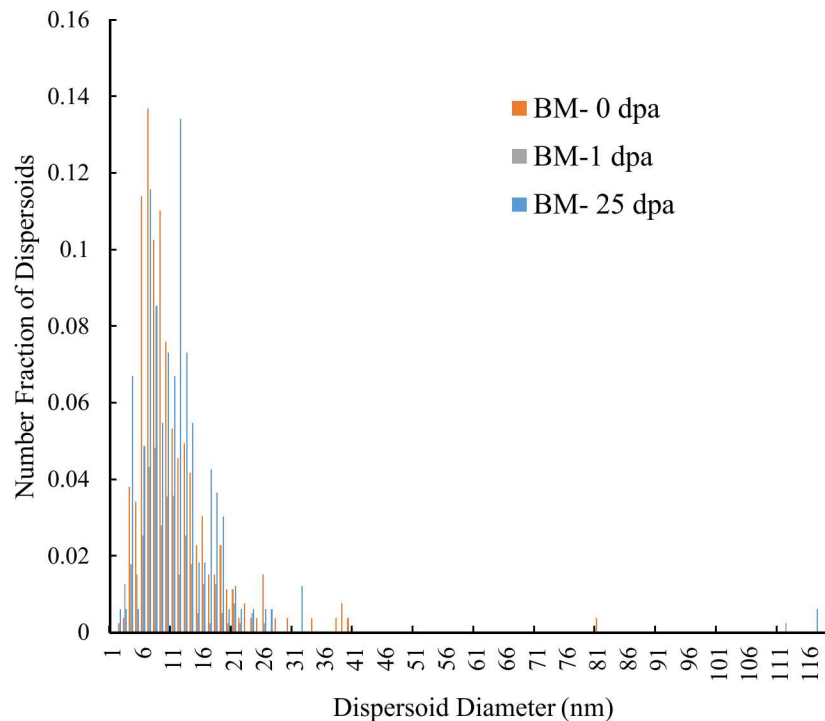
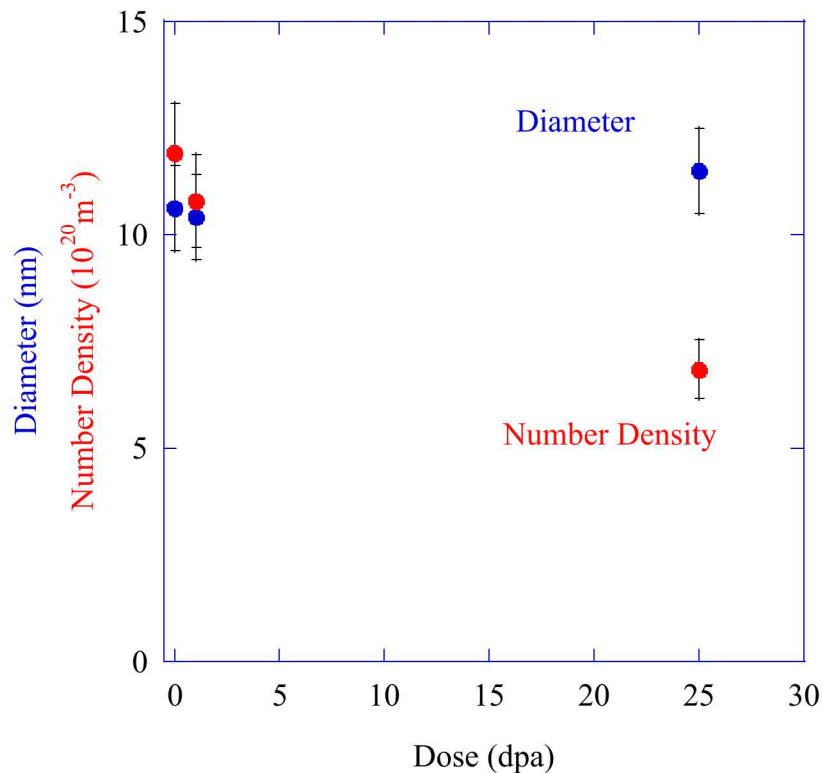
(f)

200 nm





Dispersoid Evolution with Increased dpa (BM)



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





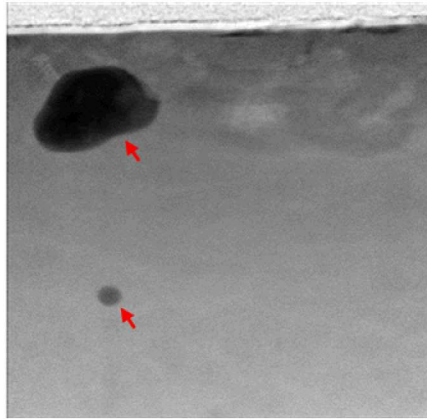
Effect of irradiation on SZ (up to 25 dpa)

0 dpa

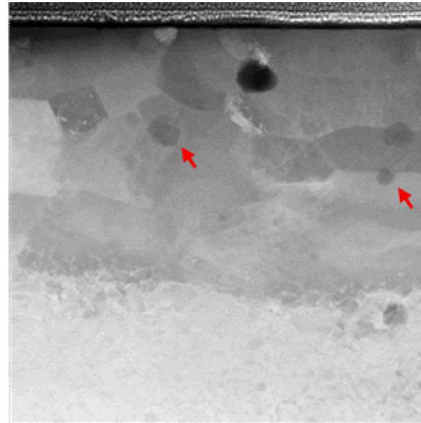
1 dpa

25 dpa

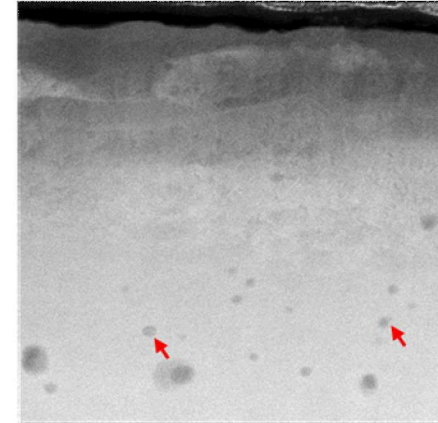
HAADF



(a)



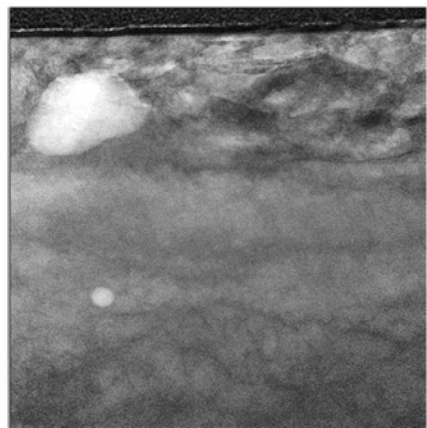
(b)



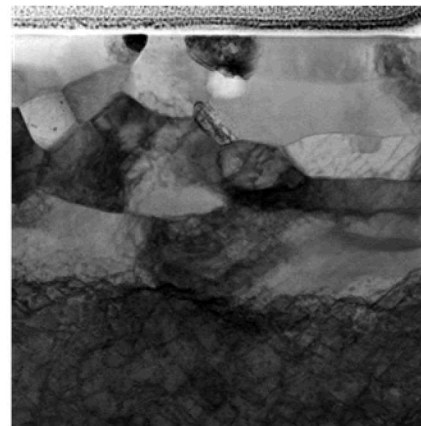
(c)

Pt
Oxide

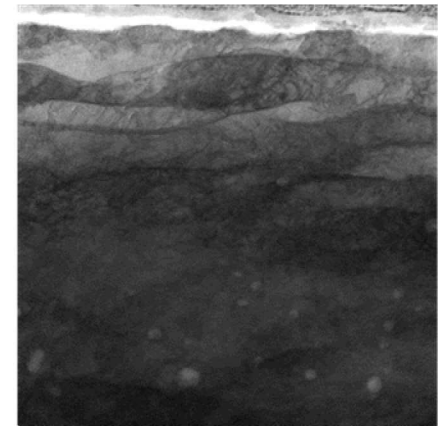
BF



(d)



(e)



(f)

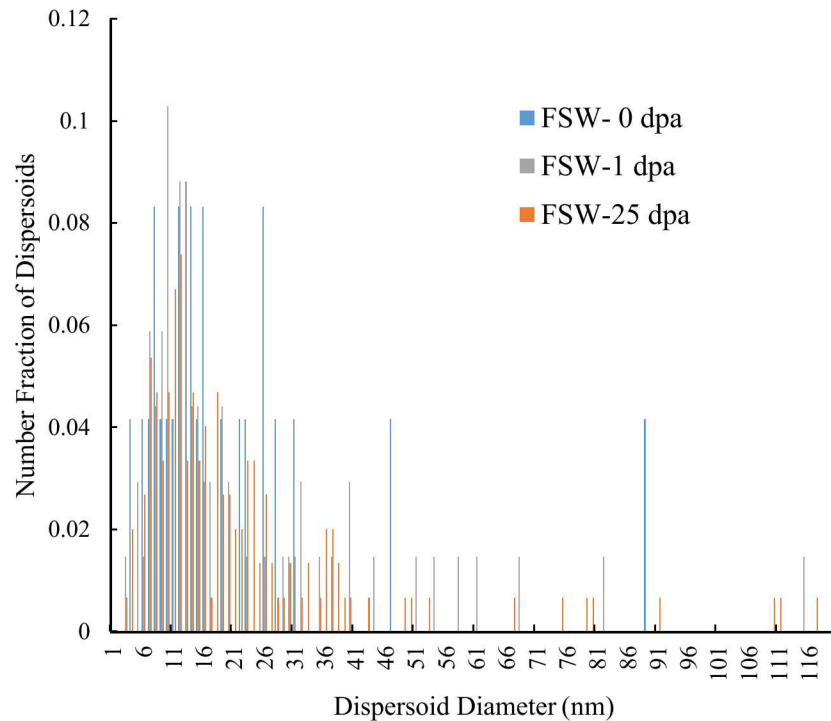
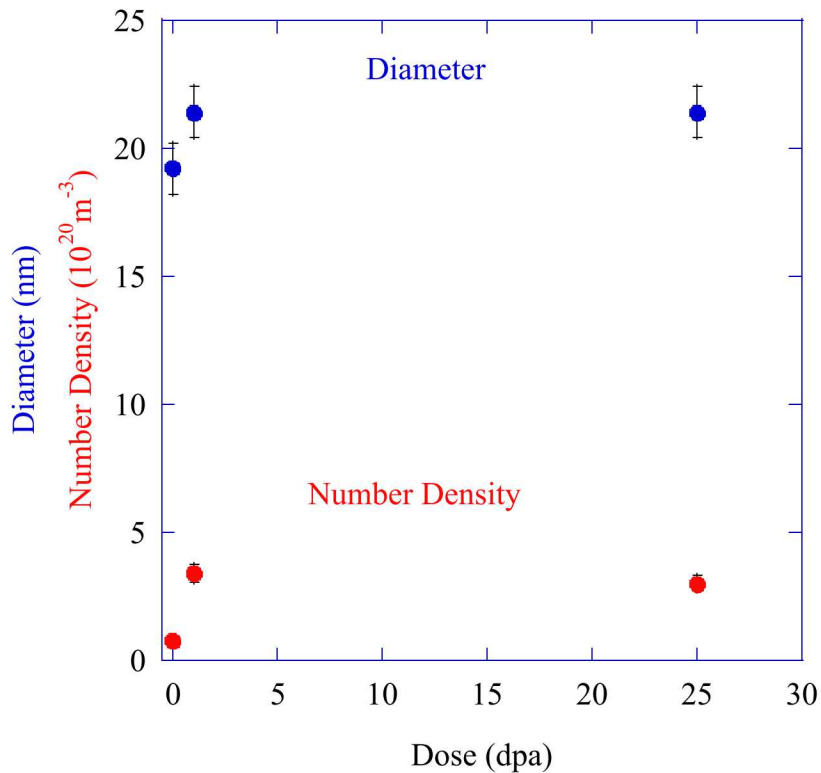
Pt
Oxide

200 nm





Dispersoid Evolution with Increased dpa (SZ)

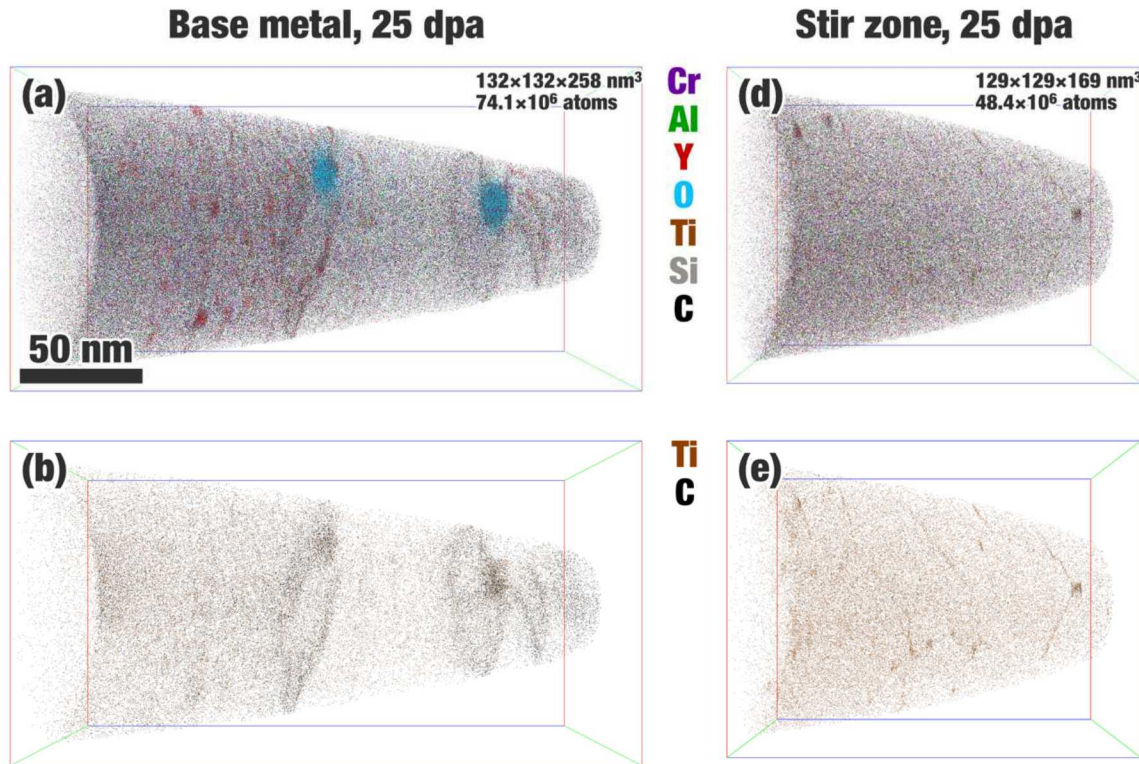


- Increase in number density due to small dispersoids re-precipitating post welding observed by 1 dpa
- Attributed to radiation enhanced diffusion
 - Increased diameter and increased number density





APT: Evidence of Re-precipitation?

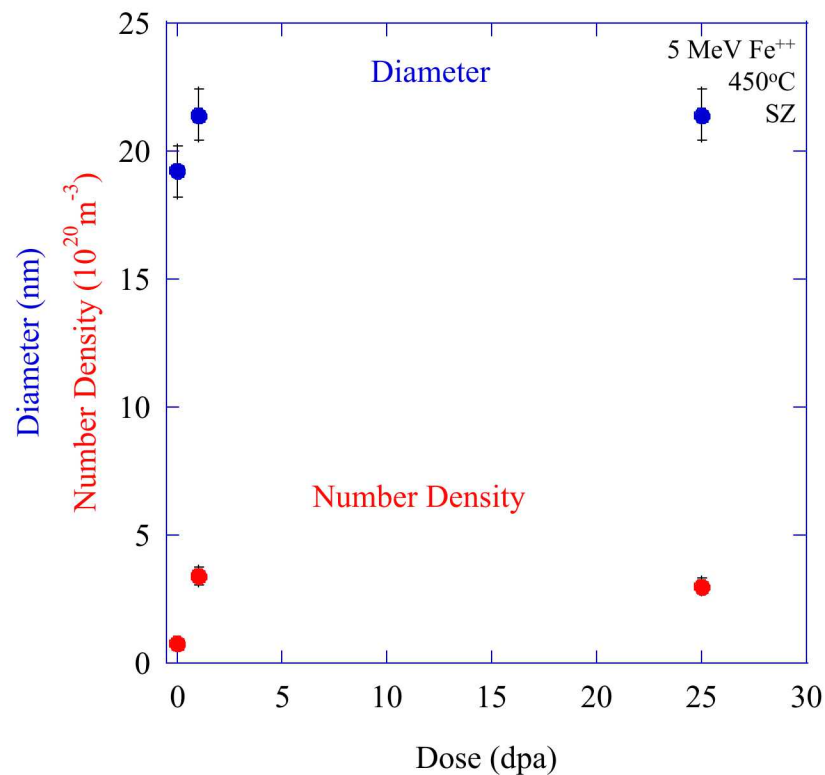
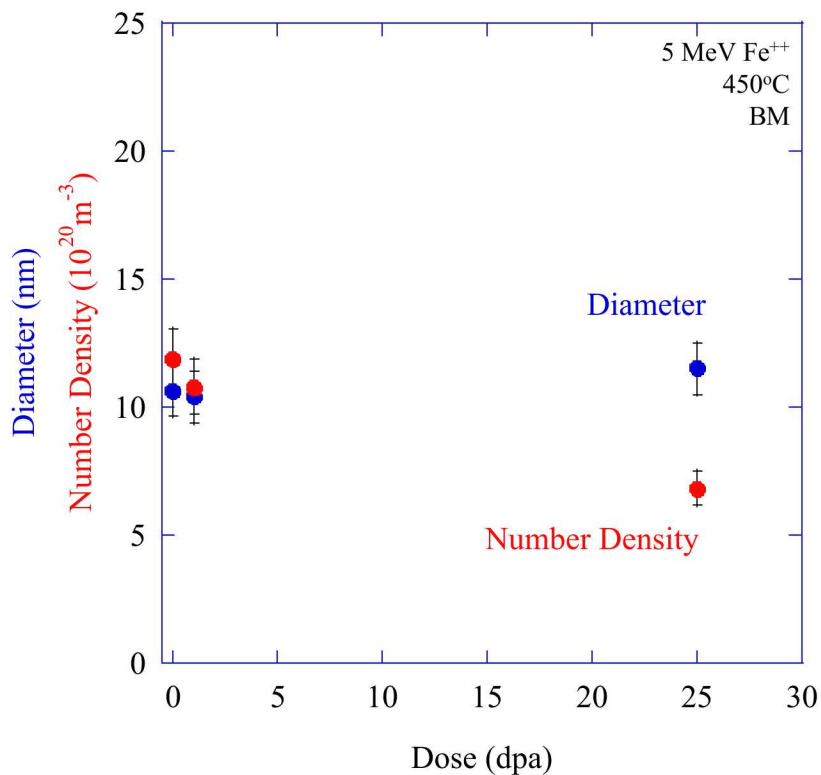


- Small precipitates seen in all tips of irradiated SZ
- No larger coarsened precipitates observed at 25 dpa
 - Likely due to the low density of very large precipitates





Differences between BM and SZ



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





Low Dose Conclusions

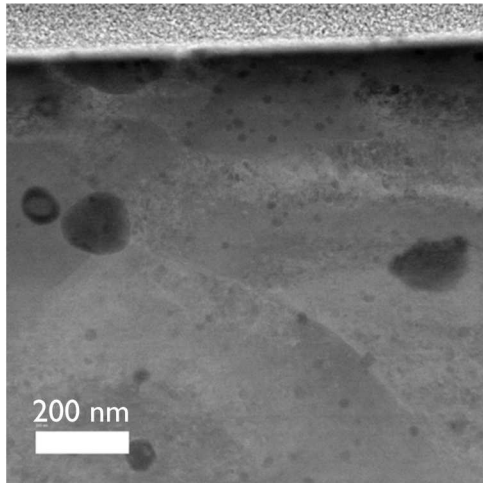
- Dispersoids in the base material increased in diameter and decreased in number density
 - Attributed to Ostwald Coarsening
- Dispersoids in stir zone increased in both diameter and number density due to re-precipitation of dispersoids
 - Attributed to Radiation enhanced diffusion



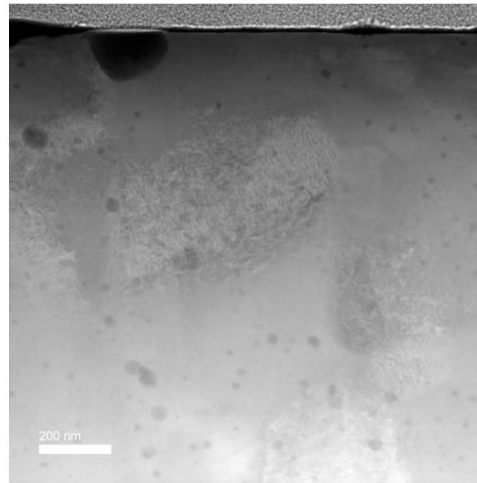


Base Material: 450°C up to 200 dpa

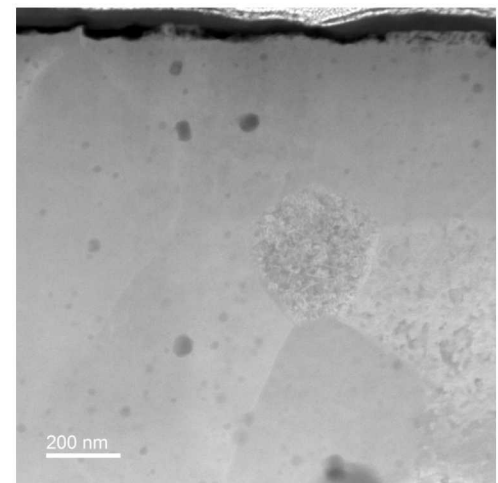
0 dpa



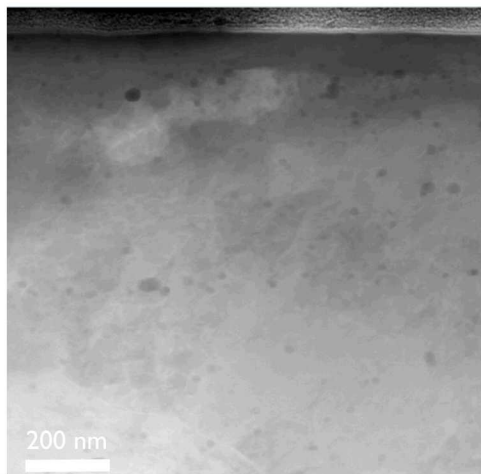
1 dpa



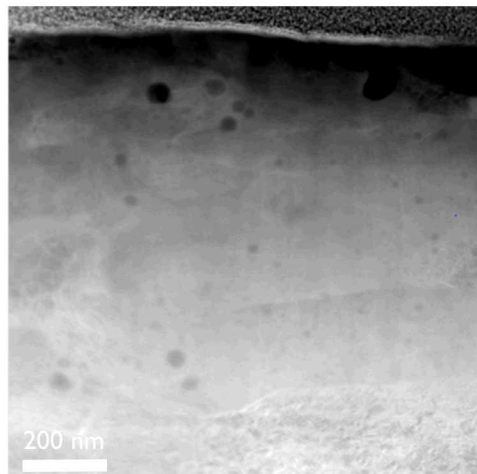
25 dpa



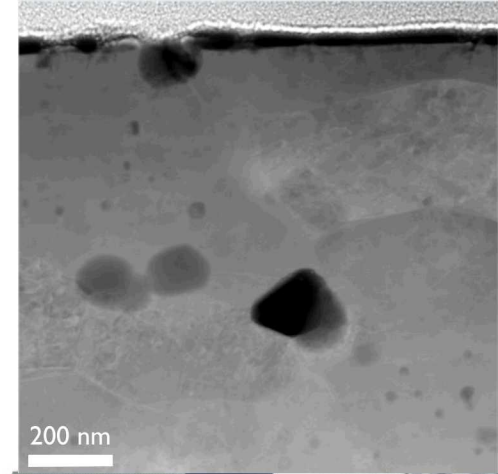
50 dpa



100 dpa

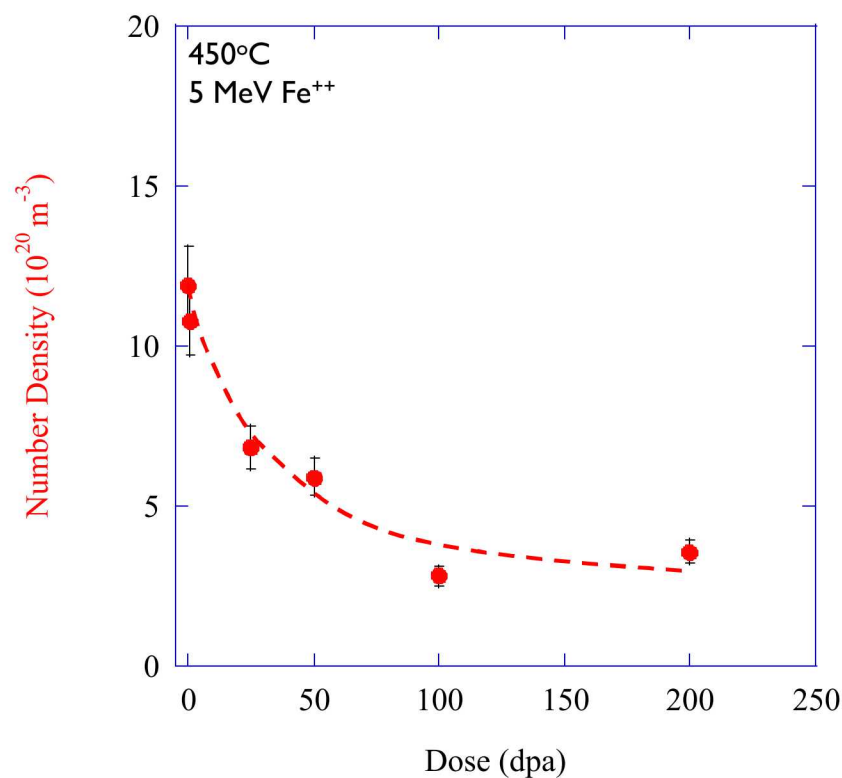
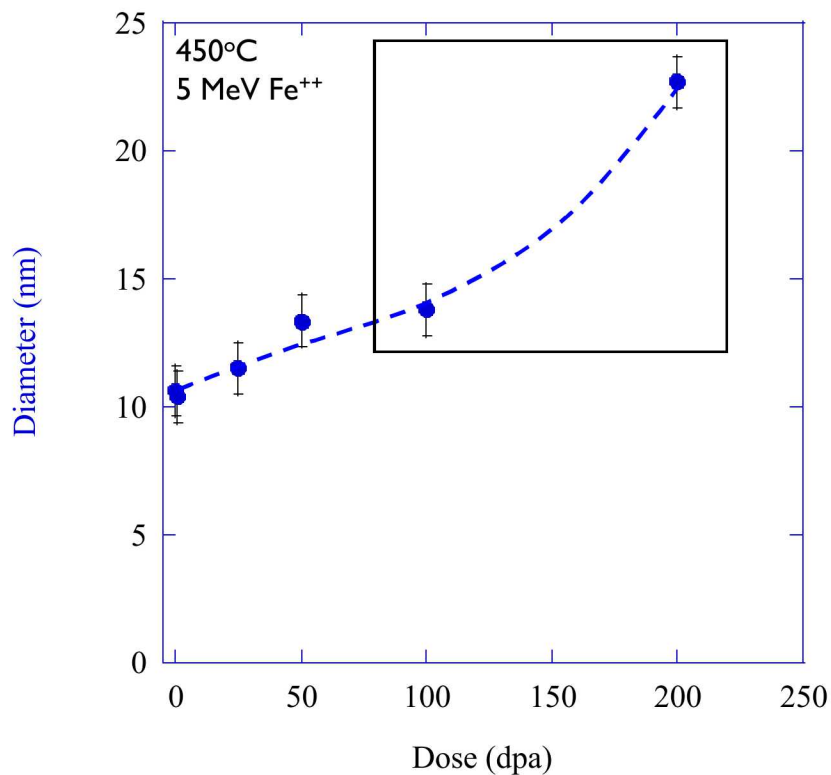


200 dpa





Dispersoid Evolution with Increased dpa (BM)

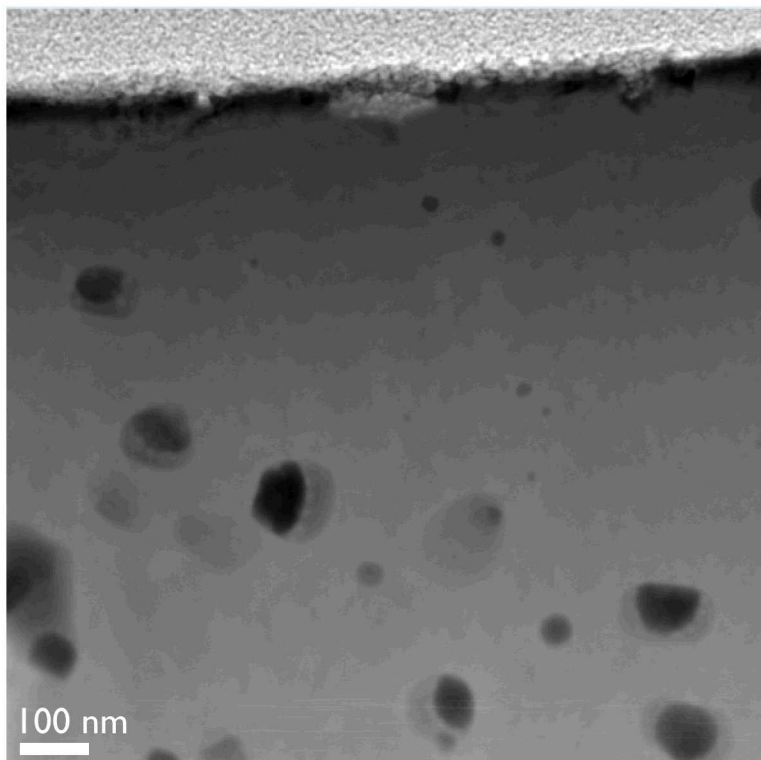


- Evidence of Ostwald coarsening with irradiation
 - Continues low dose behavior of increased diameter, decreased number density
- Large jump in diameter at 200 dpa- formation of voids?
 - TEM characterization at MFC scheduled for this summer





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



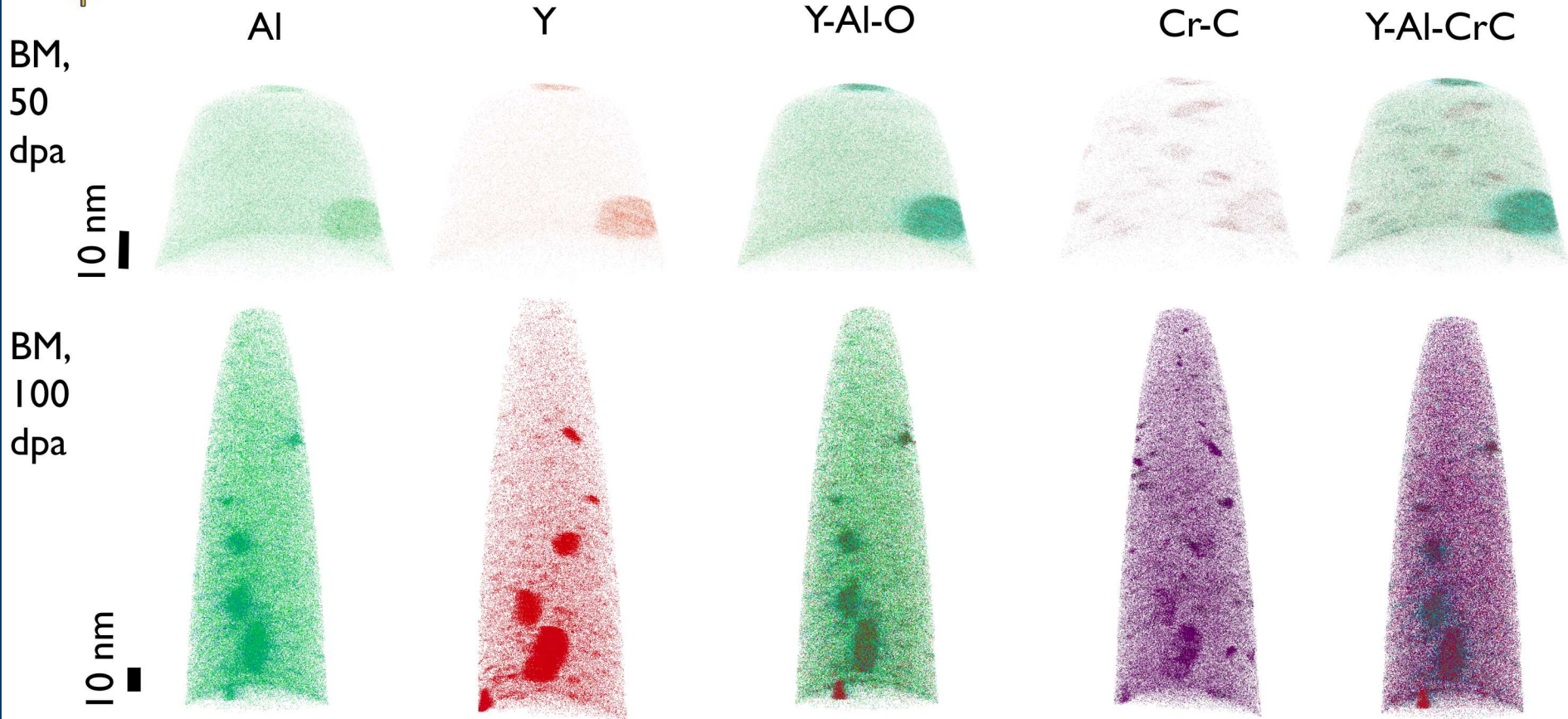
- STEM HAADF- Z contrast so Y-Al-O could look like a void
- CTEM BF can be used to identify by focusing series for voids, but time consuming and difficult to get accurate sizes
- Best approach suggested by Parish et al with a combination STEM HAADF, STEM through focus imaging coupled with high fidelity 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>





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

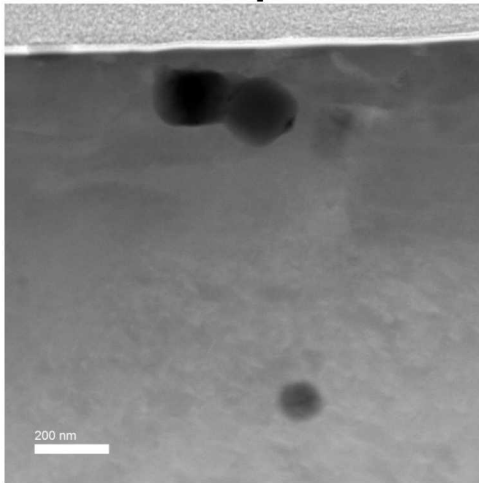


- CrC precipitates were not observed at 1 or 25 dpa
- Ti co-located with Cr-C as well

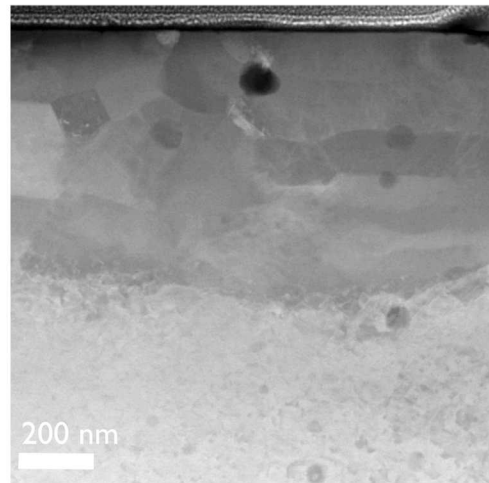


Stir Zone: 450°C up to 200 dpa

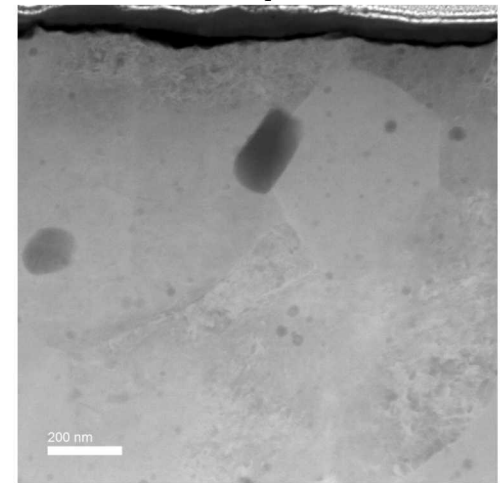
0 dpa



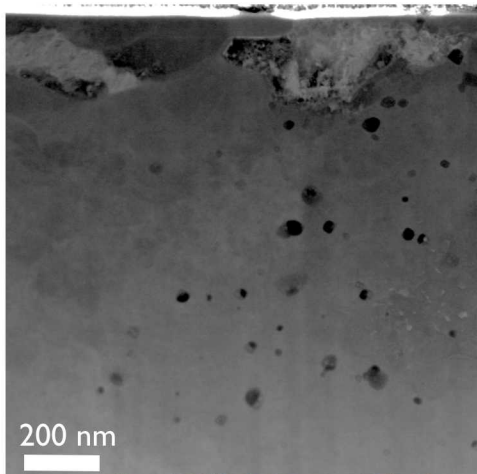
1 dpa



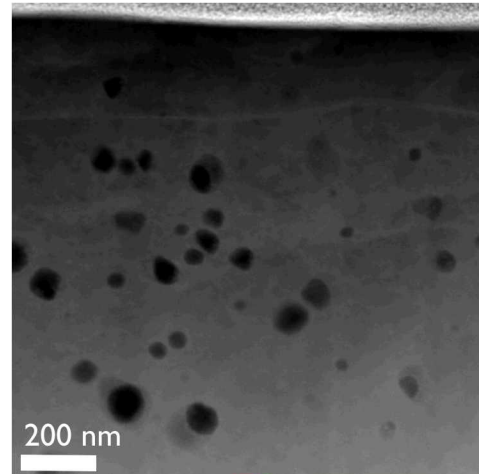
25 dpa



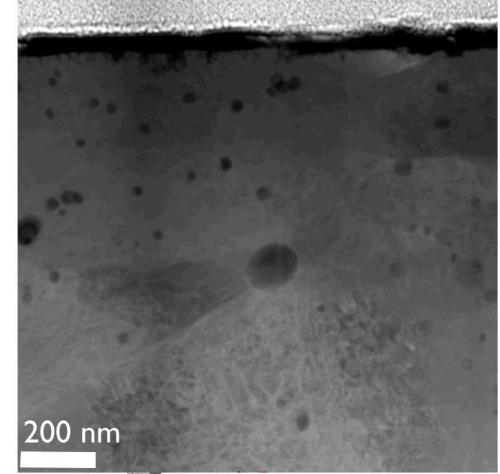
50 dpa



100 dpa

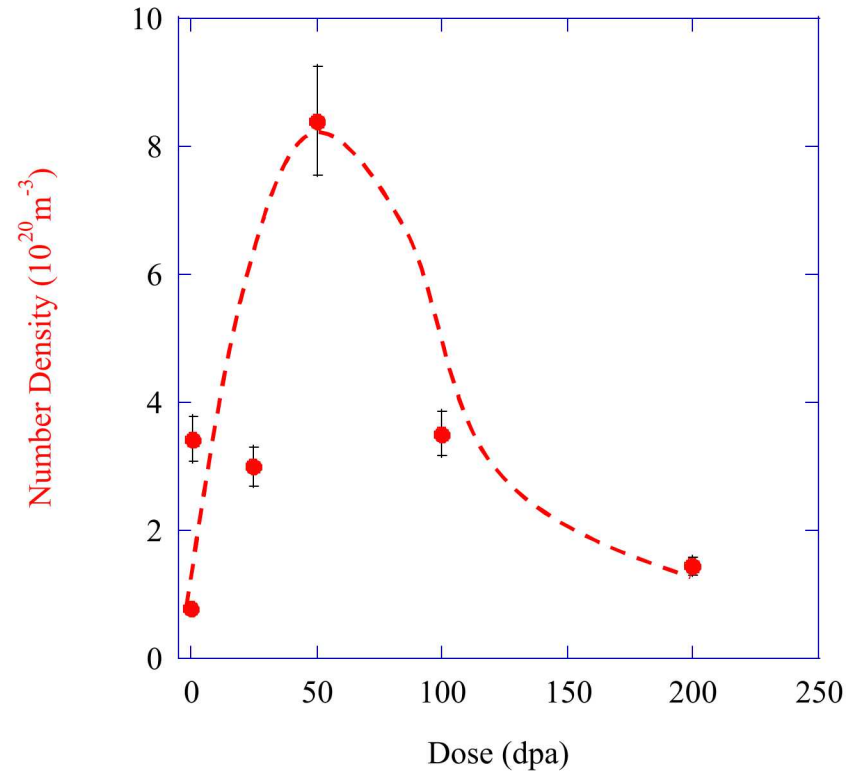
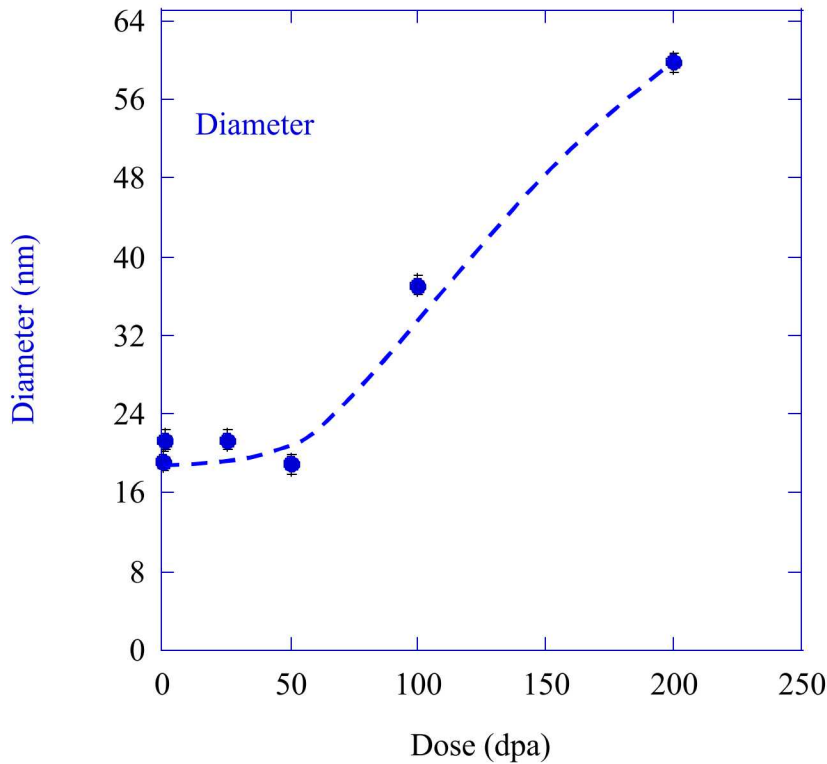


200 dpa





Dispersoid Evolution with Increased dpa (SZ)



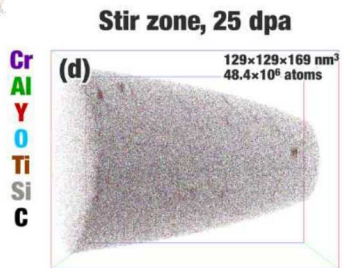
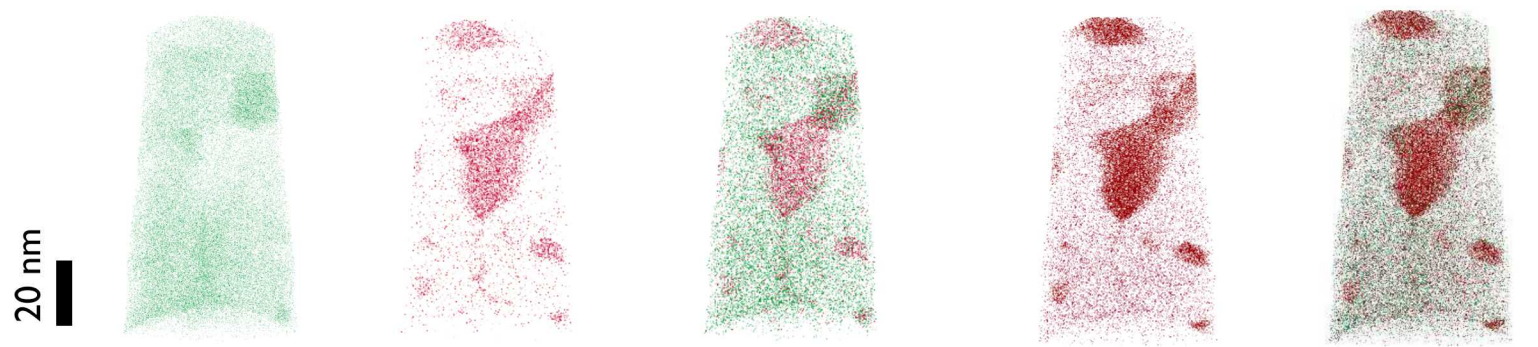
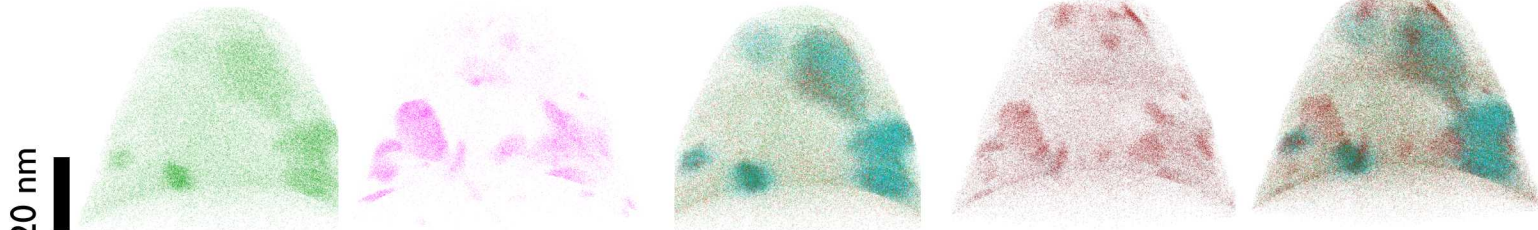
- Rapid growth at 100 dpa and beyond likely due to some void swelling similar to jump at 200 dpa for BM





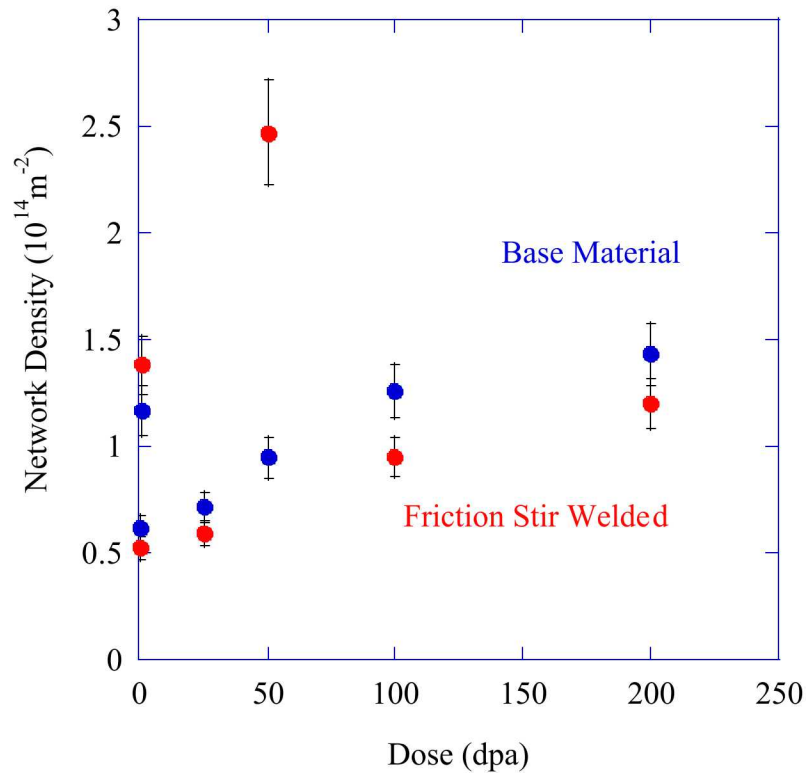
Qualitative coarsening observed with APT in SZ

Al Y Y-Al-O Cr-C Y-Al-CrC

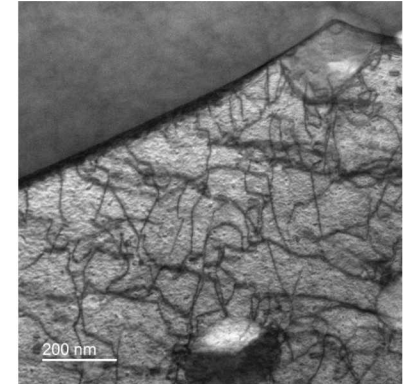




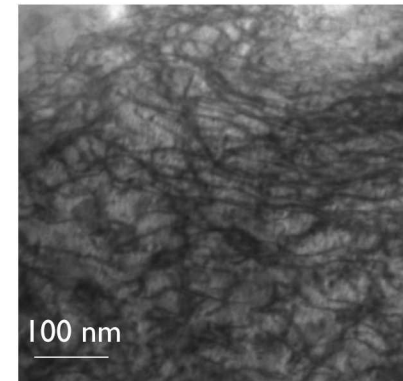
Evolution of Network Density



SZ, as received



SZ, 50 dpa



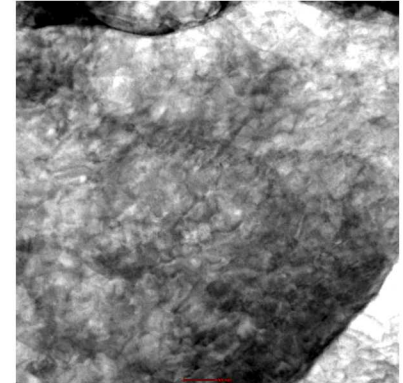
- Network density correlated to strength and typically increases with irradiation according to literature
 - Small decrease in network line density with welding likely due to annealing



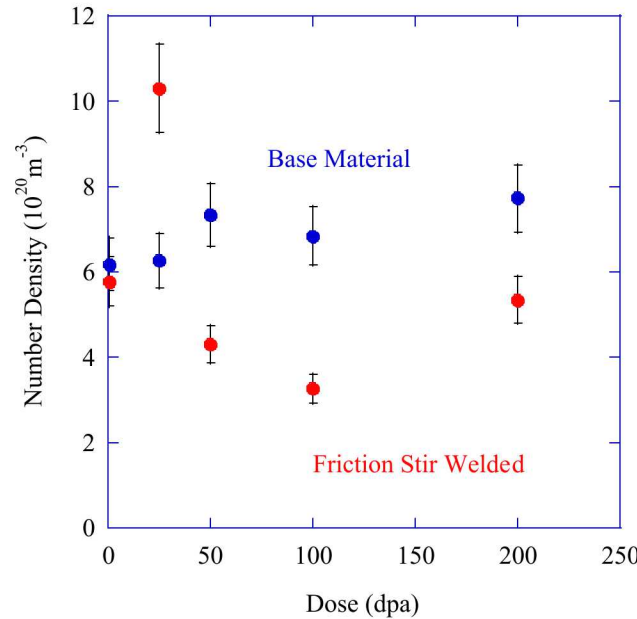
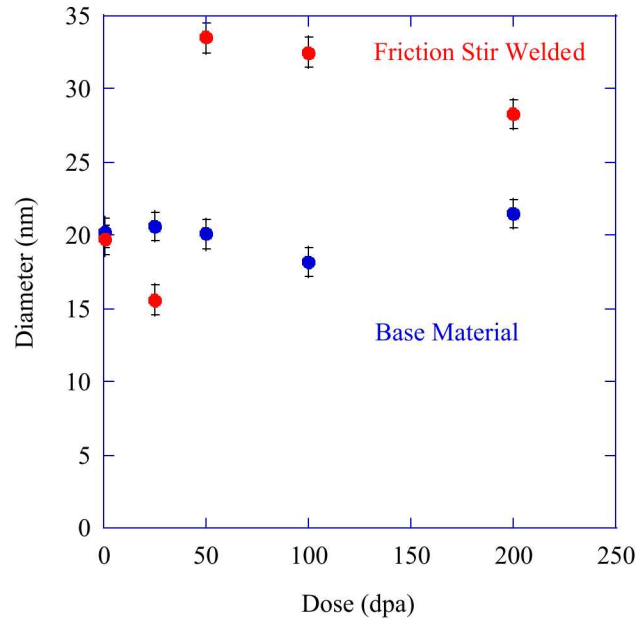
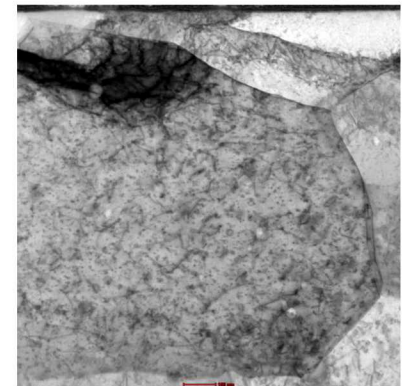


Evolution of Dislocation Loops

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

- 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 (in progress)

- Temperature dependence of BM, SZ
 - Irradiations complete
 - Characterization at MFC
- Effect of welding conditions
 - Irradiations complete
 - Characterization at MFC

	400°C	450°C	500°C
1	MA956-BM MA956-SZ-H	MA956-BM MA956-SZ-H	
25	MA956-BM MA956-SZ-H	MA956-BM MA956-SZ-H	
50	MA956-BM, MA956-SZ-H MA956-SZ-M	MA956-BM, MA956-SZ-H	MA956-BM MA956-SZ-H MA-956-M
100	MA956-BM, MA956-SZ-H MA956-SZ-M	MA956-BM MA956-SZ-H MA-956-SZ-M	MA956-BM, MA956-SZ-H MA956-SZ-M
200		MA956-BM MA956-SZ-H MA956-SZ-M	

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





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- 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).

