SAND2019-2555C

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

Elizabeth Getto[1], B. Tobie[1], S. Briggs[2], K. Hattar[2], B. Baker[1], Keith Knipling[3], J.P. Wharry[4]
[1]United States Naval Academy, Annapolis, MD
[2]Sandia National Laboratory, Albuquerque, NM
[3]Naval Research Laboratory, Washington, D.C.
[4]Purdue University, West Lafayette, IN

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TMS 2019

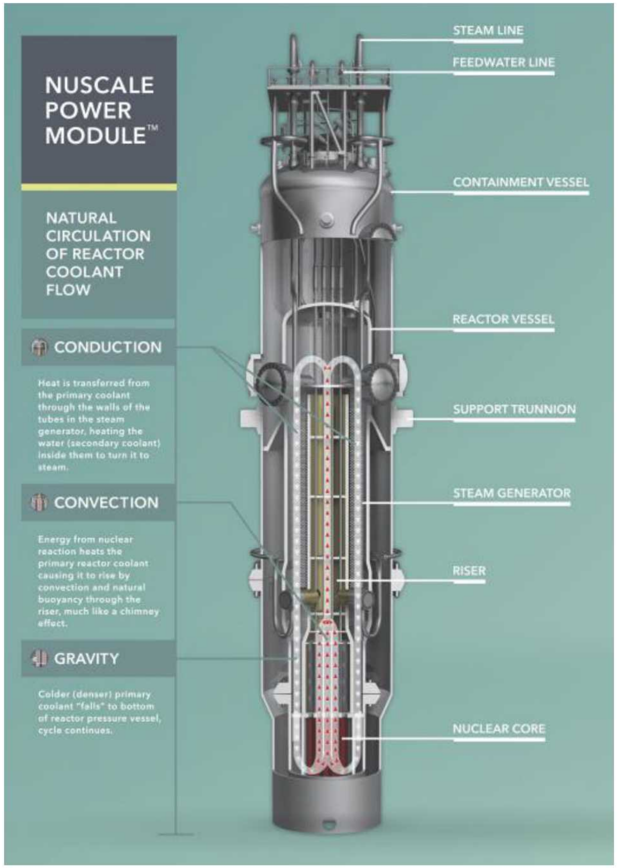
Motivation- Materials Challenges for DOE and DOD

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



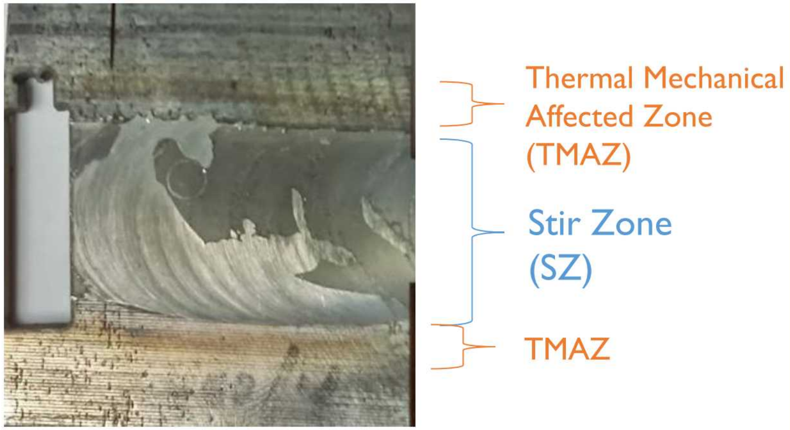
50 MWe modular reactor design

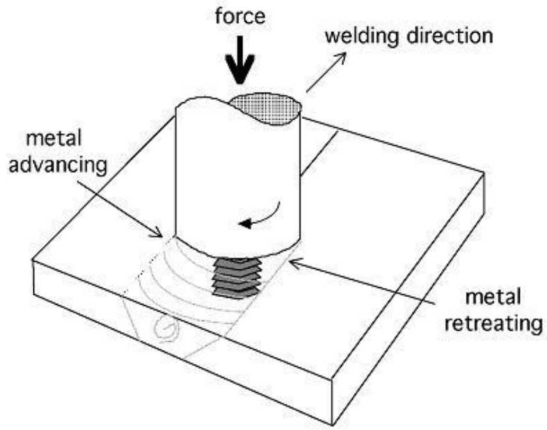
Courtesy of NuScale Power

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



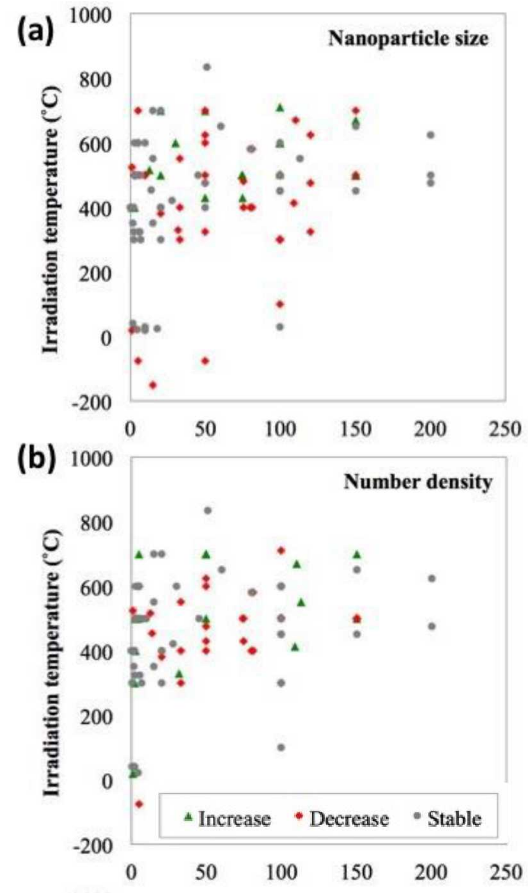


H. Bhadeshia, "Friction Stir Welding," University of Cambridge

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 and nanohardness of ODS steel MA956 at 450°C up to 25 dpa

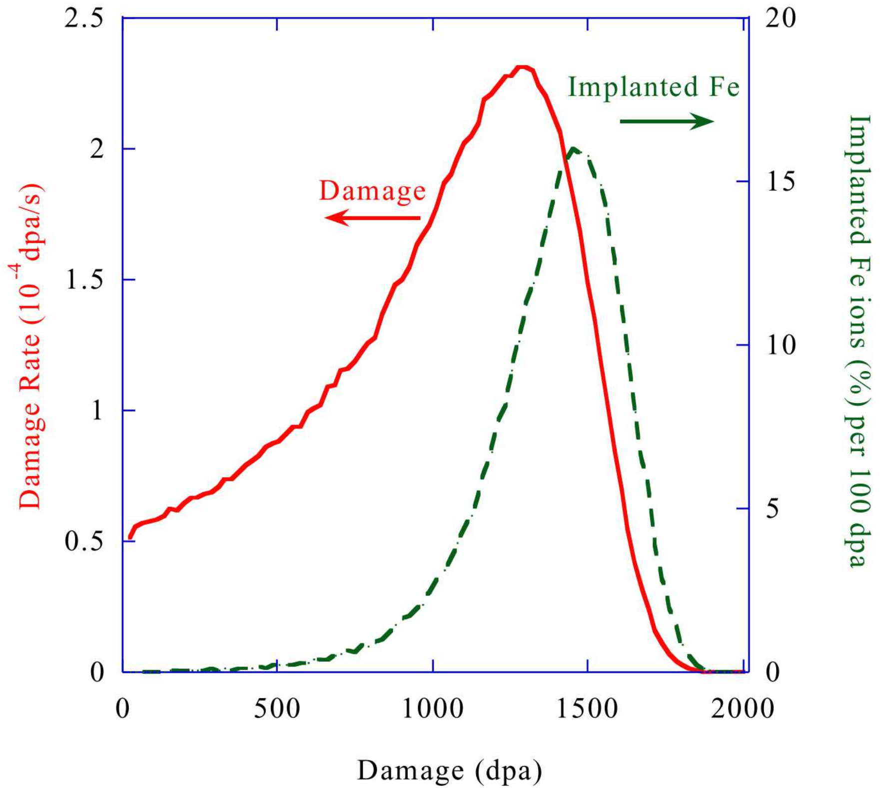
Approach

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

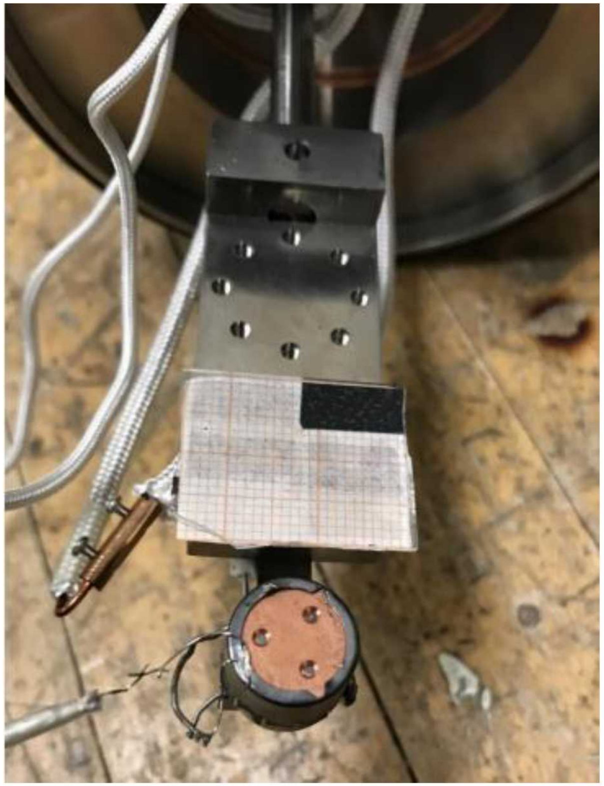
Ion Experiment Design

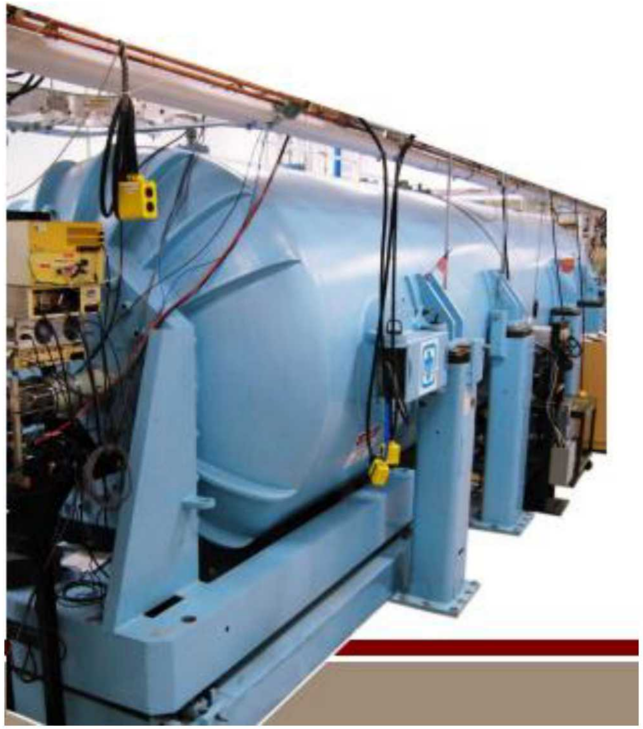
Alloy	Fe	Cr	Al	Y_2O_3	Ti	Mn	Si	Ni	C	Mo	S	P
MA956 (wt%)	Bal	19.93	4.75	0.51	0.39	0.09	0.08	0.04	0.023	0.02	0.008	0.006

- MA956
- Irradiation of tem bar samples with 5 MeV Fe^{2+} 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



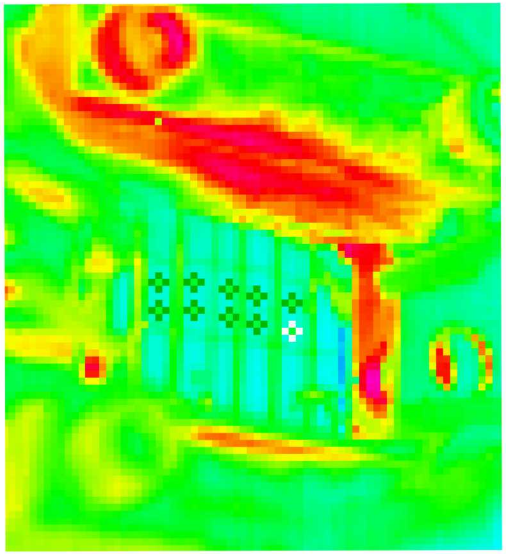
Ion Experiment Design (SNL)

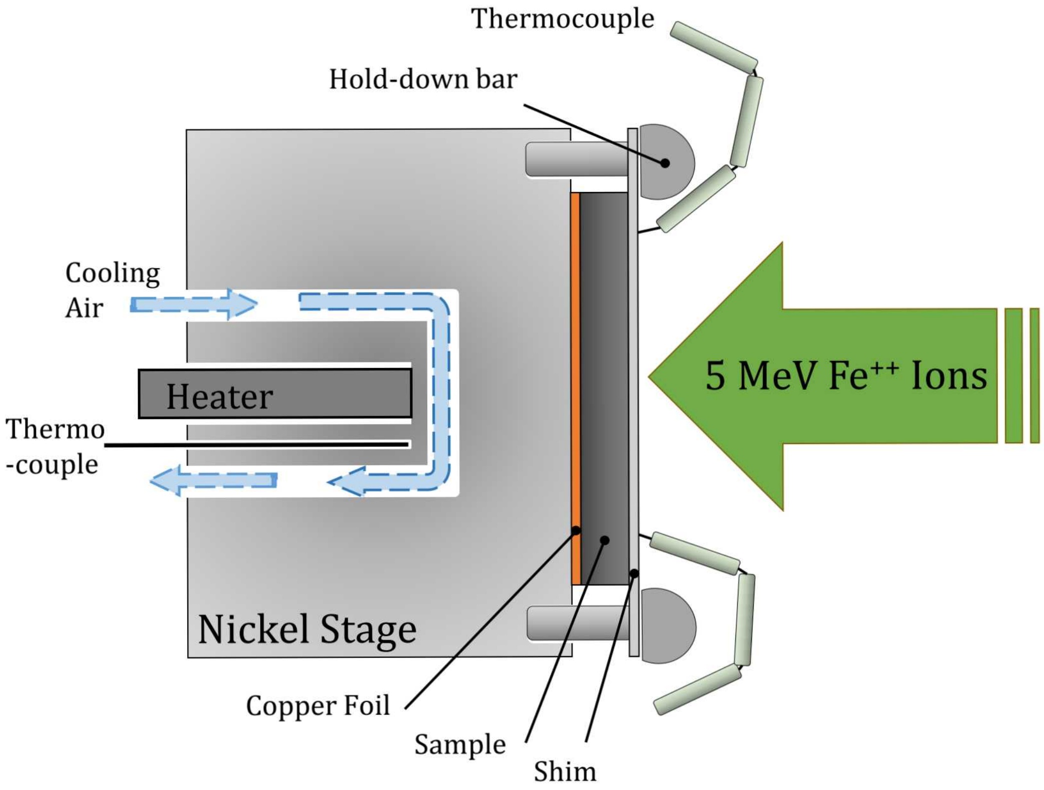




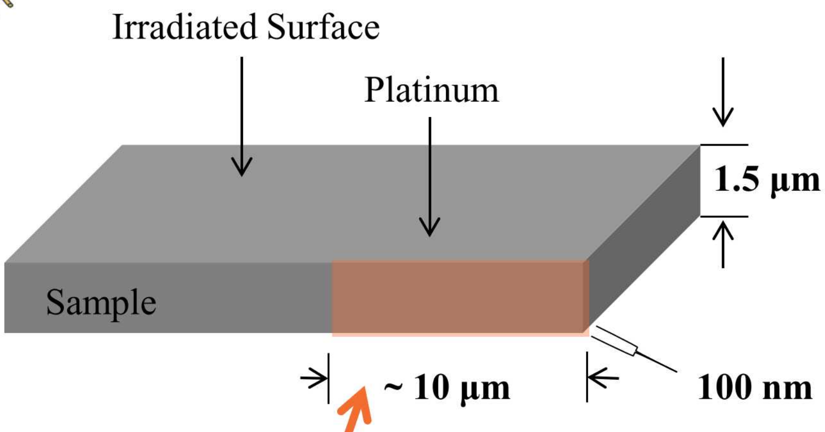
- Irradiations performed at SNL on the 6 MV Tandem
- LabView controlled button heater used to maintain temperature

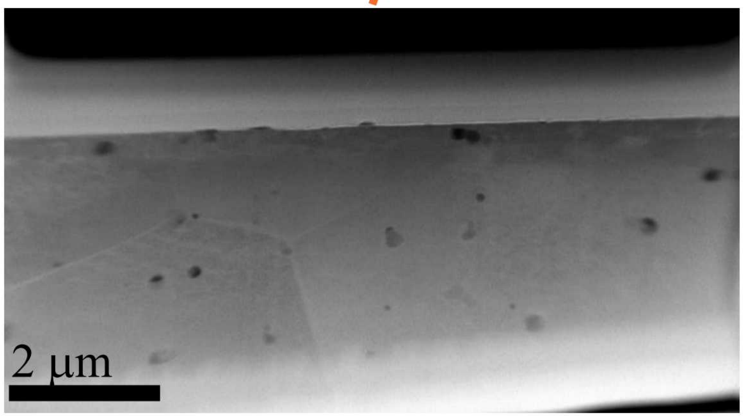
Ion Experiment Design (MIBL)





Microstructure Analysis

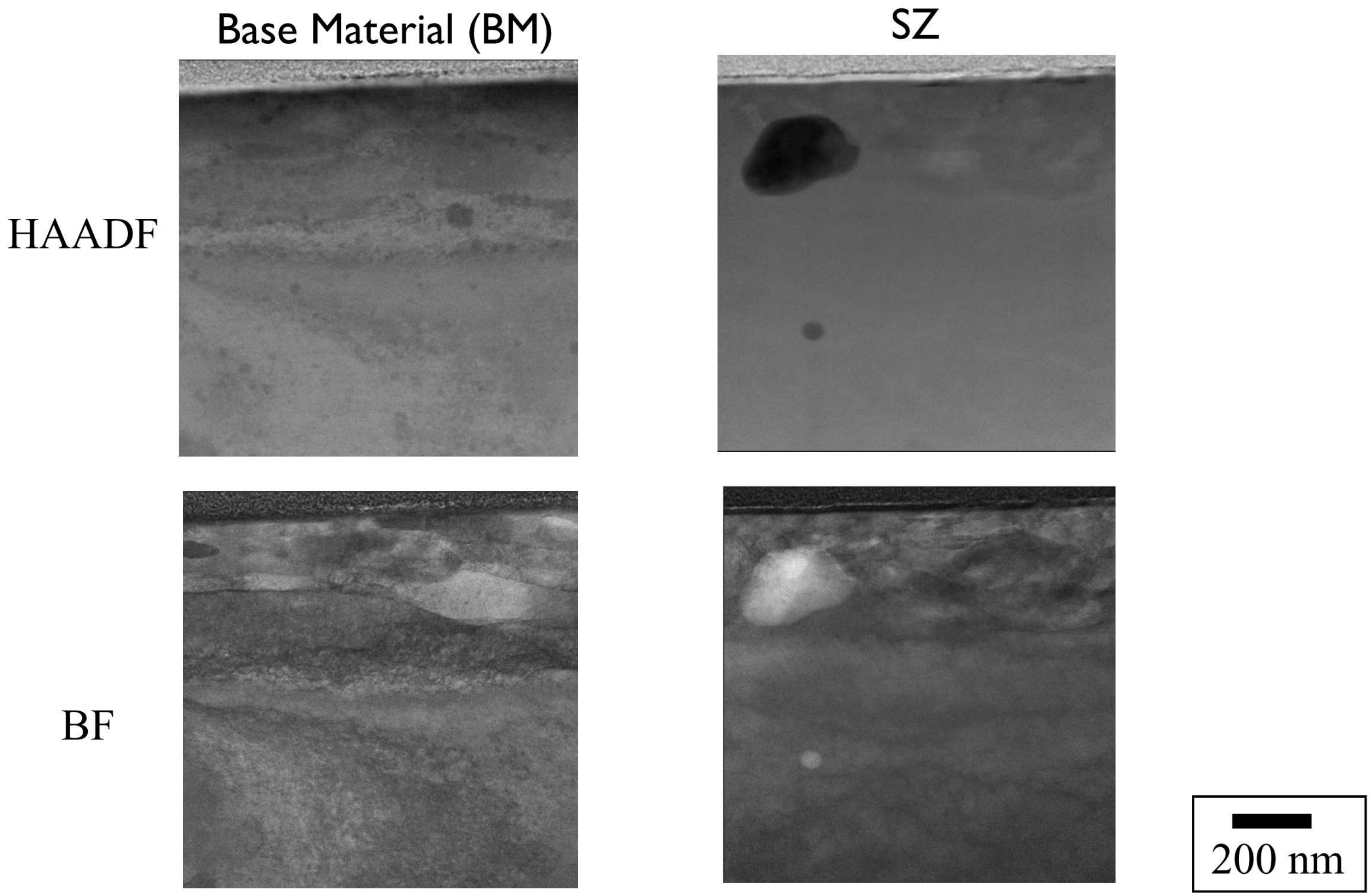




MA956-Friction Stir Welded

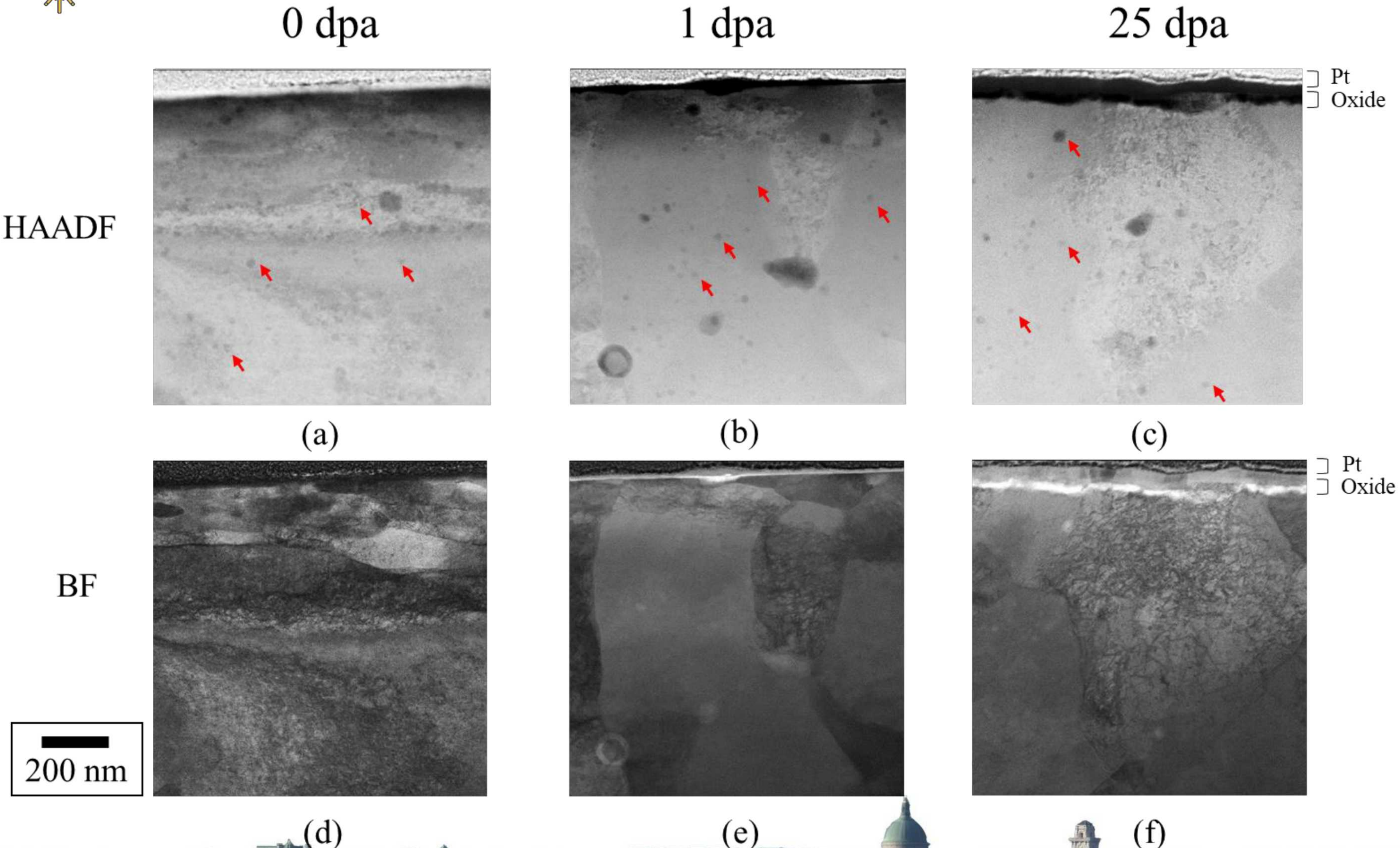
- Samples prepared via liftout method using Focused Ion Beam (FIB) at MaCS at CAES and at Naval Research Laboratory (NRL)
- Cross section liftout maintains integrity of surface while allowing depth profiling of dispersoid distribution
- Dispersoids imaged in high angular annular dark field in STEM mode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

MA956: As received microstructure

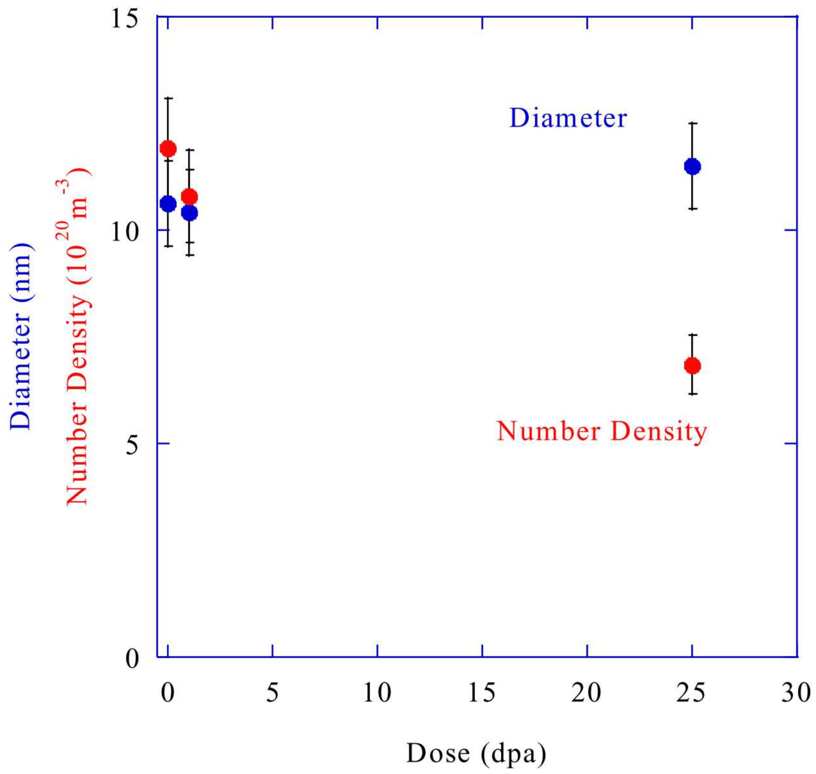


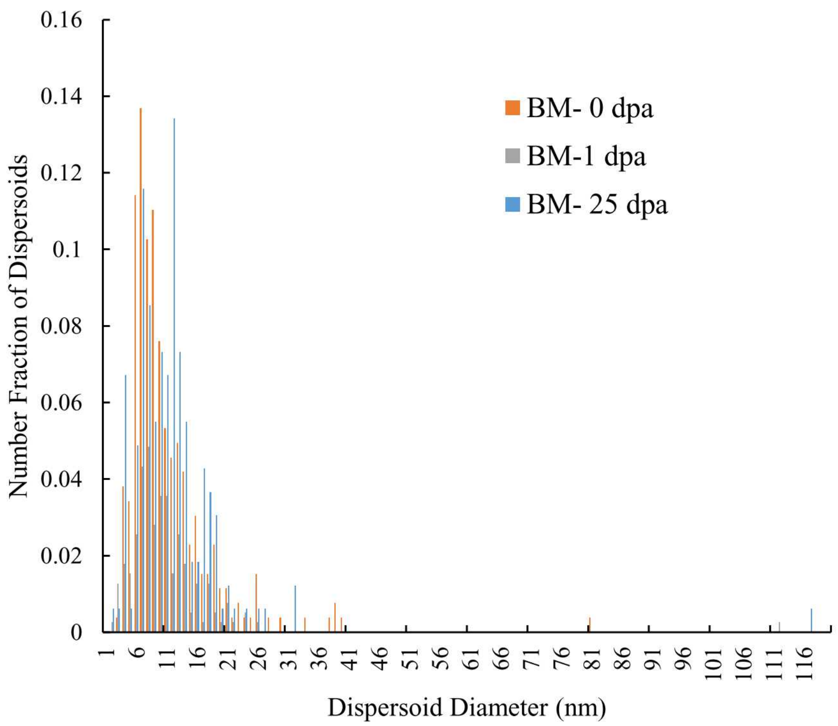
- Dispersoids much more visible with z contrast in HAADF images
- Qualitatively, fewer and larger dispersoids

Effect of irradiation on BM (low dose)



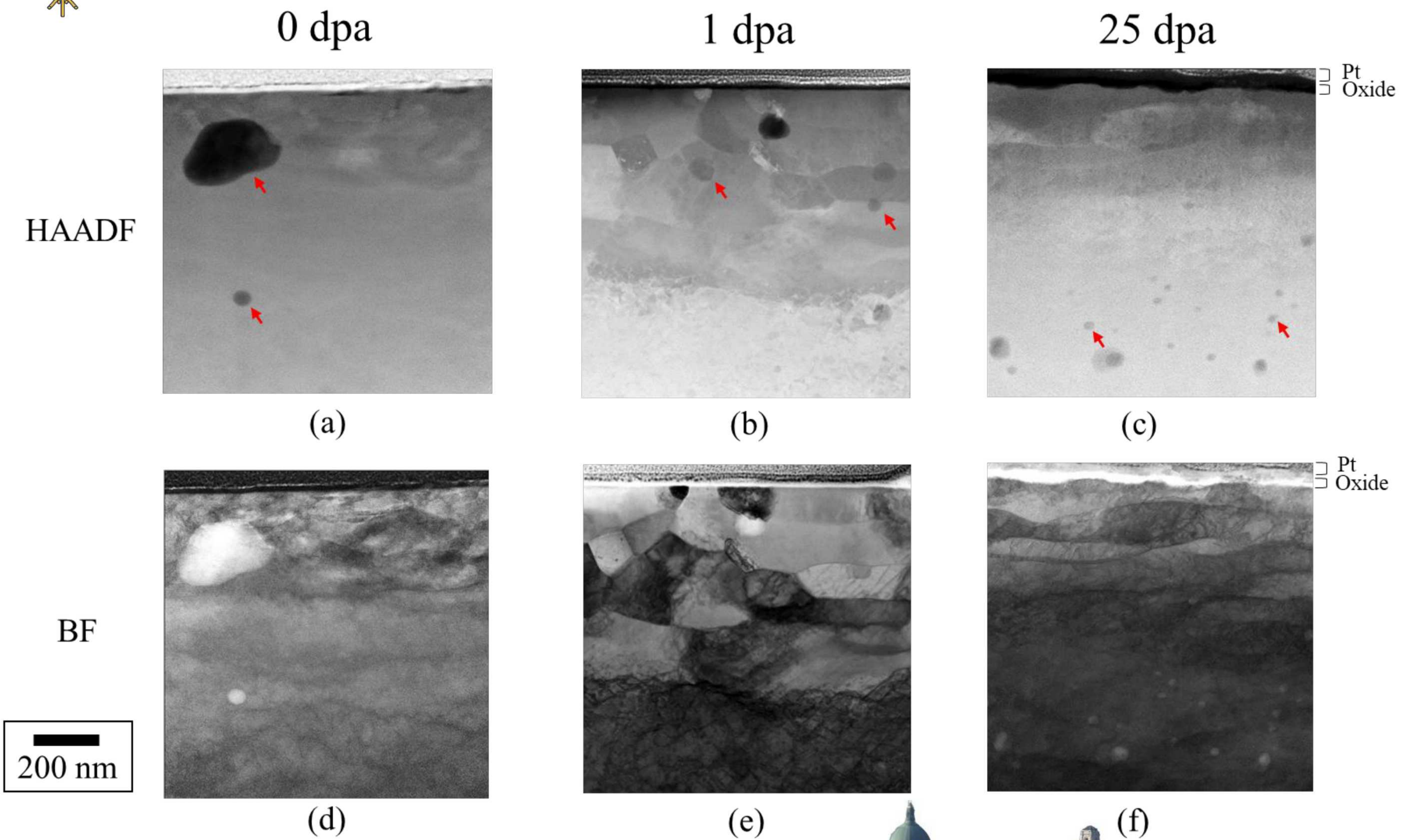
Dispersoid Evolution with Increased dpa (BM)



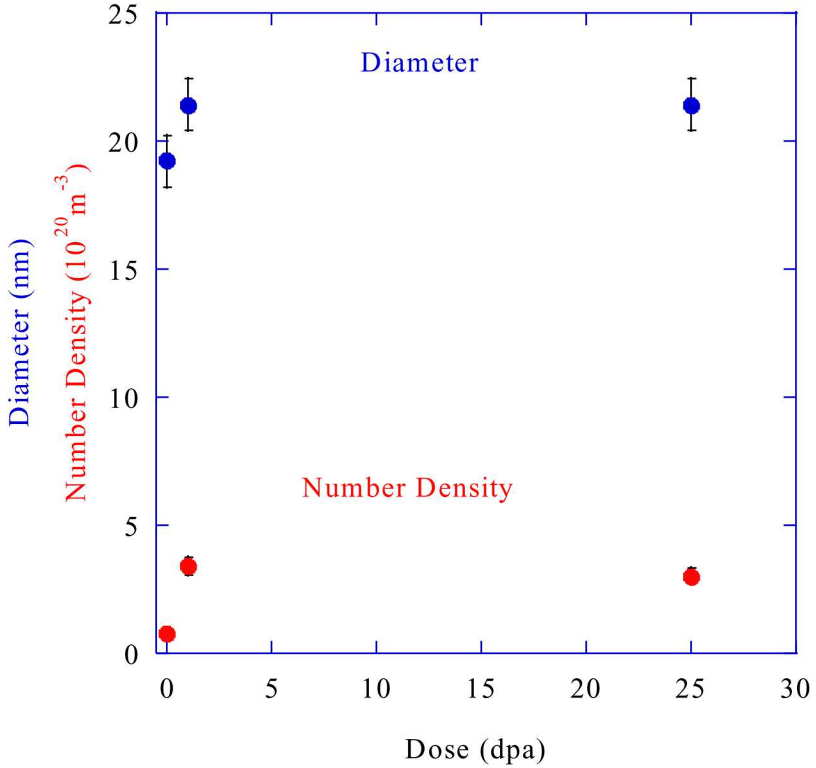


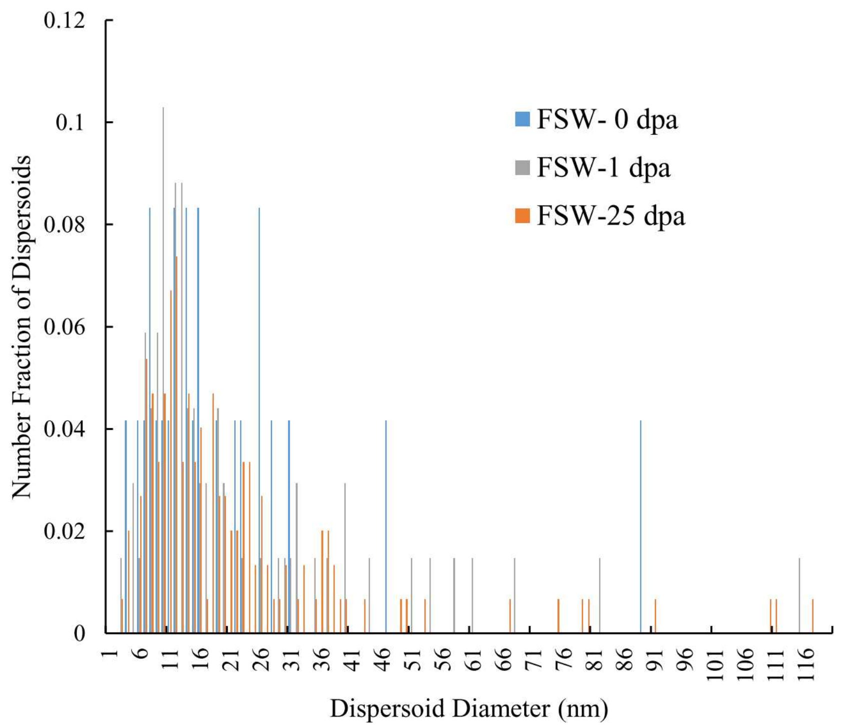
- Evidence of small amount of coarsening with irradiation
  - Increased diameter, decreased number density
  - Attributed to Ostwald coarsening

Effect of irradiation on SZ (up to 25 dpa)



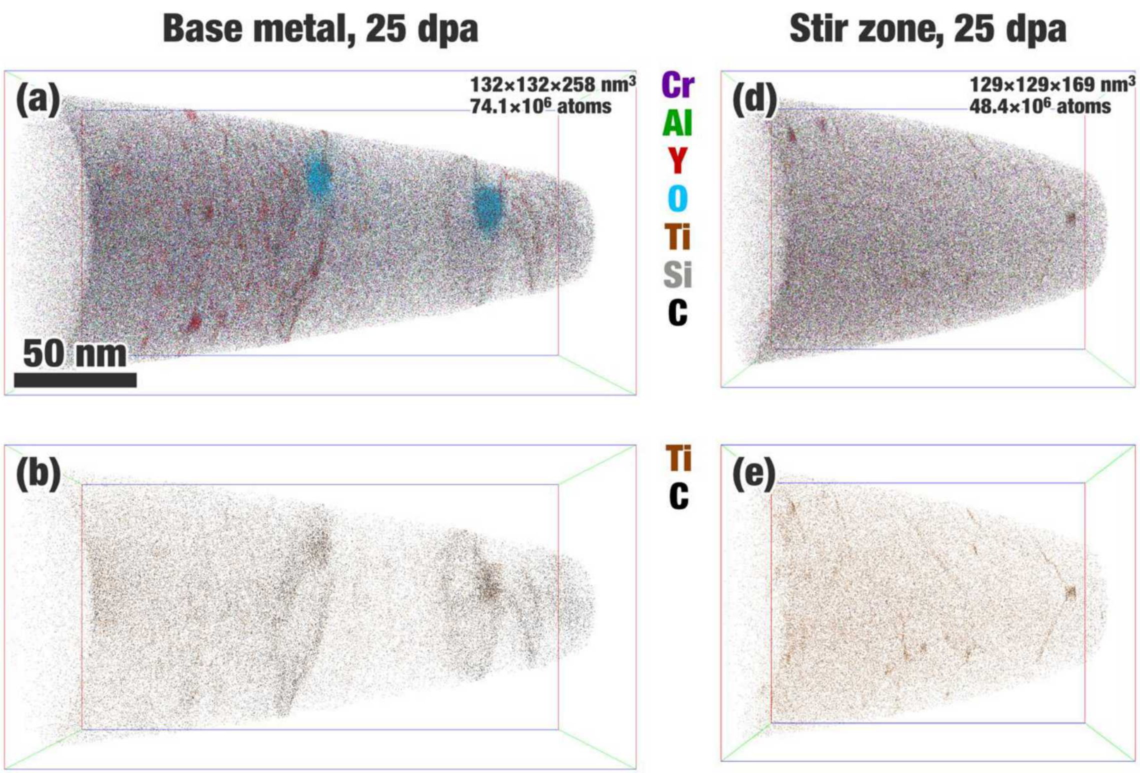
Dispersoid Evolution with Increased dpa (SZ)





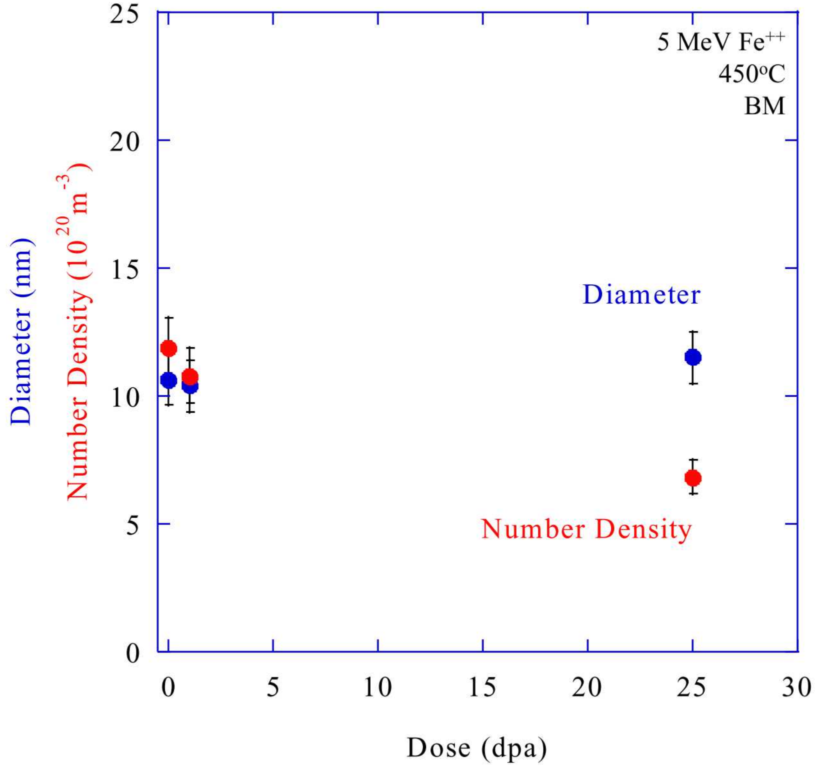
- Increase in number density due to small dispersoids re-precipitating post welding observed by 1 dpa
- Attributed to radiation enhanced diffusion
  - Increased diameter and increased number density

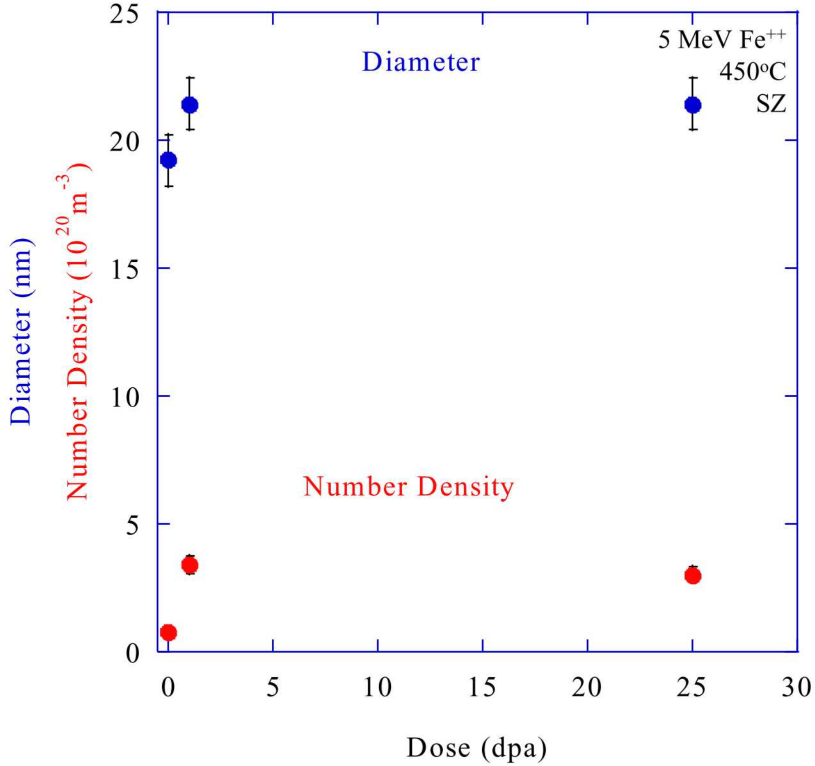
APT: Evidence of Re-precipitation?



- 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

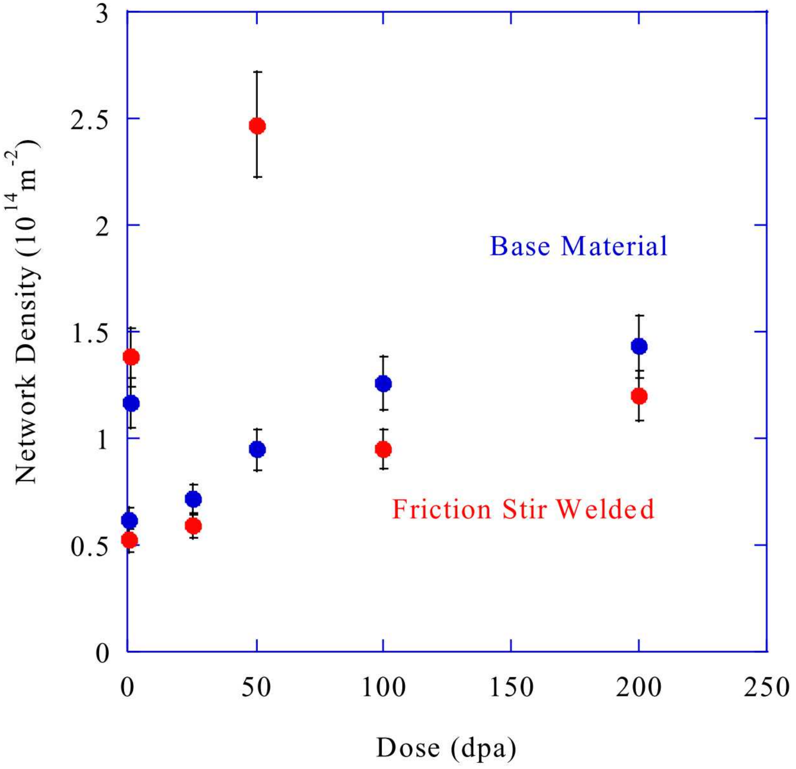
Differences between BM and SZ



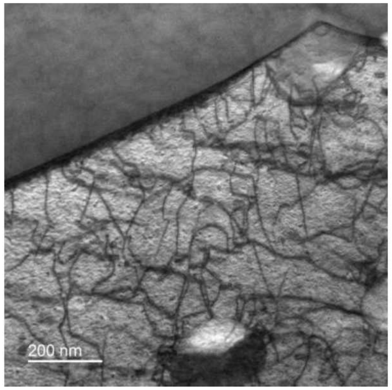


- Interesting implications- re-precipitation reverses softening of SZ
- Two different mechanisms potentially explain different evolution

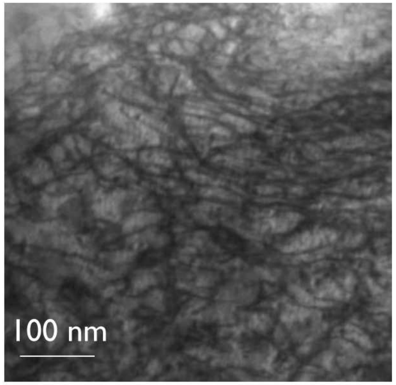
Evolution of Network Density



SZ, as received

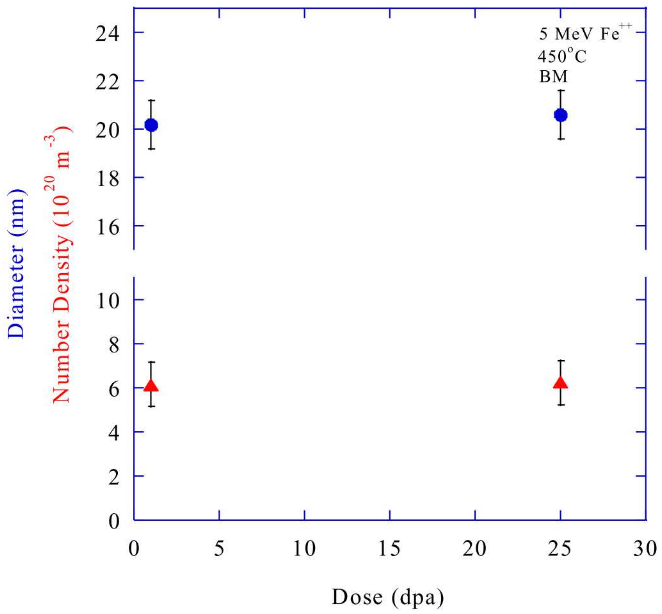


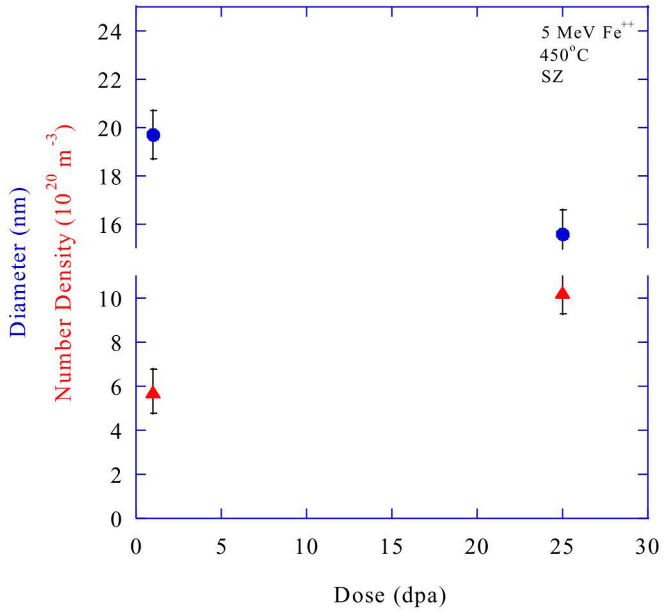
SZ, 50 dpa



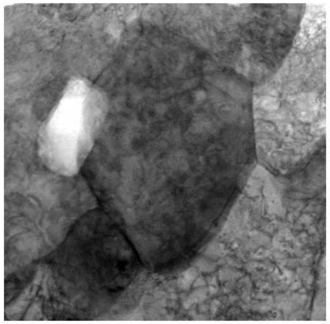
- Network density correlated to strength and typically increases with irradiation according to literature
  - Small decrease in network line density with welding likely due to annealing

Evolution of Dislocation Loops

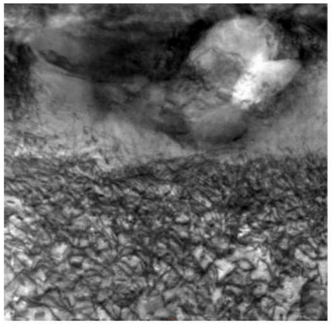




BM, 450°C, 25 dpa



SZ, 450°C, 25 dpa



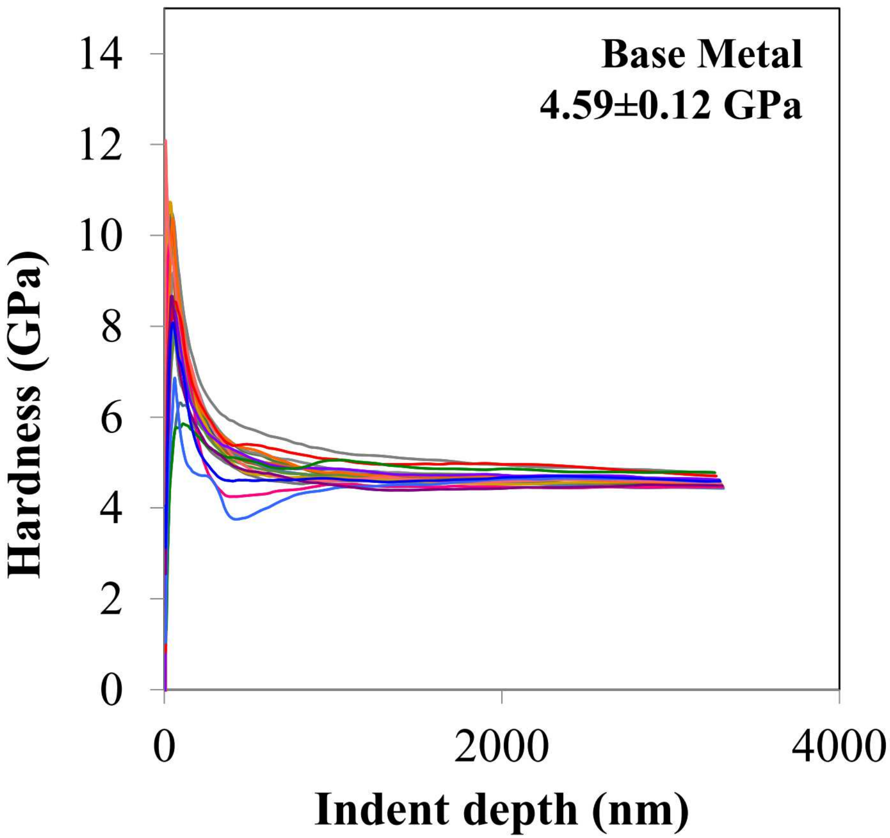
100 nm

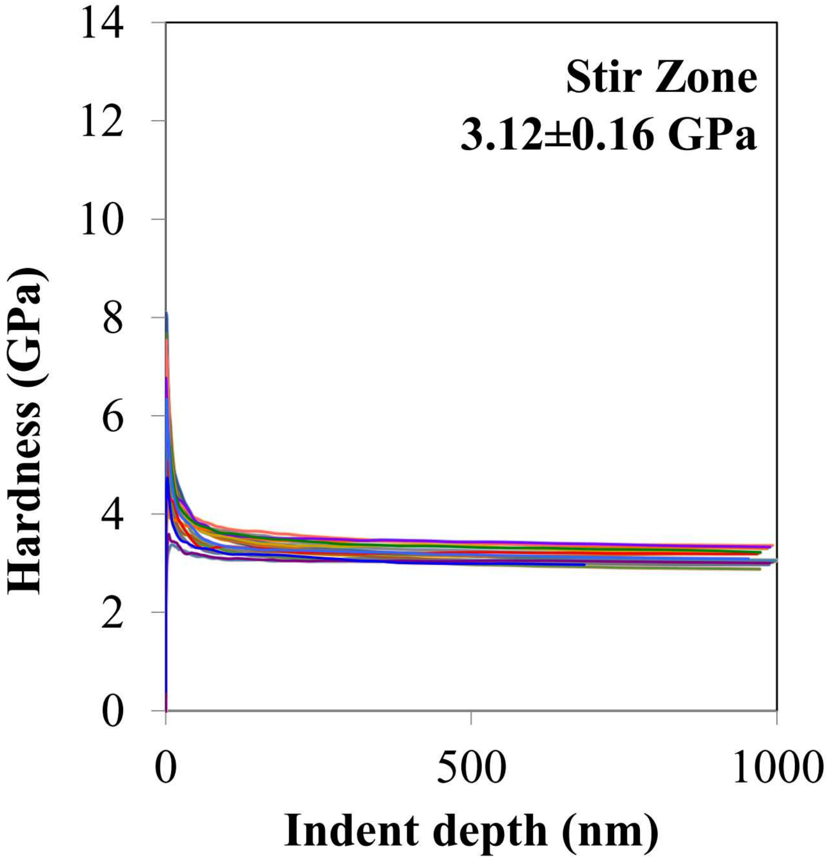
- Relatively large loops formed by 1 dpa suggest recovery of lost strength from welding

Overview of nanoindentation experiments

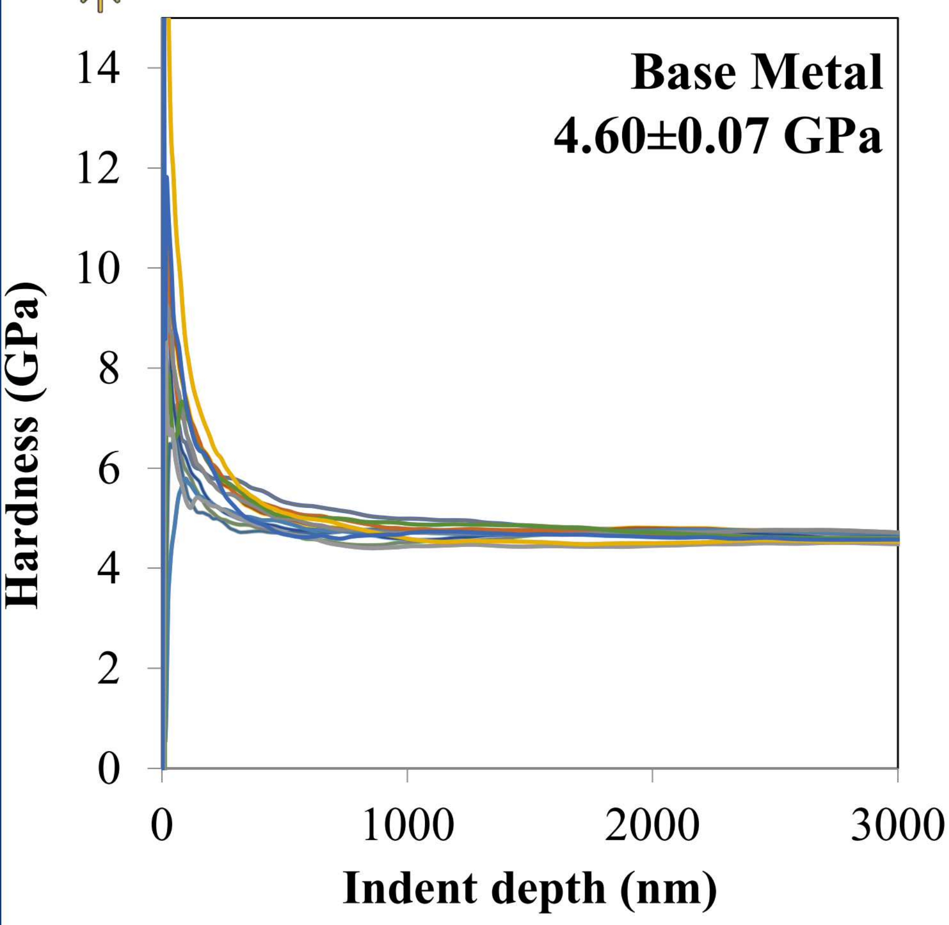
- Nanomechanics iMicro operated in dynamic indentation mode (continuous stiffness mode) at Purdue University
- Tests run in depth-controlled mode
  - Max depth was set to ~3000 nm for base metal and ~1000 nm for stir zone (20 nanoindents made in base metal and in stir zone
- Results obtained are hardness and modulus as a function of depth

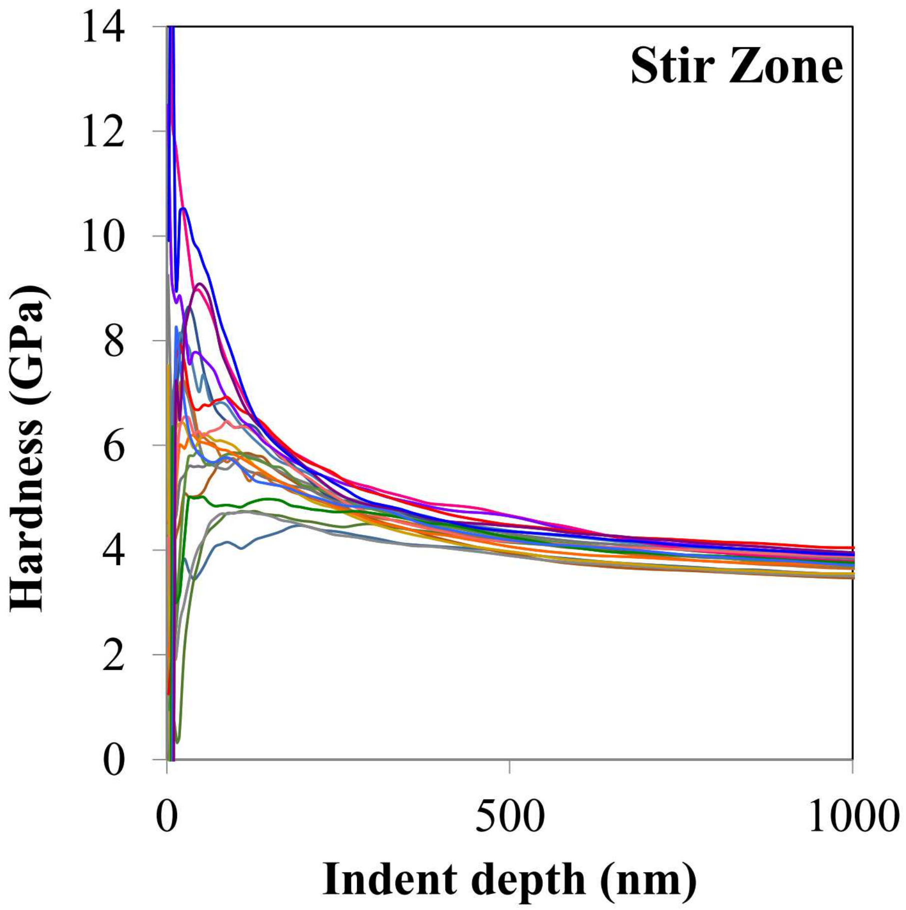
As received and As welded (0 dpa)





BM and SZ at 1 dpa





- Hardness bump from ~300-600 nm due to irradiation

Summary of Nanoindentation

Region	Nanohardness [GPa]		Yield strength [MPa]
	Average value	St dev	Average value
Base metal, as-welded	4.59	0.09	1326
Stir zone, as-welded	3.12	0.16	901
Base metal, 1 dpa	4.49	0.22	1330
Stir zone, 1 dpa	3.35	0.07	969
Vickers: Base metal, as-welded	N/A		1059
Vickers: Stir zone, as-welded	N/A		667

- Nanoindentation values consistently higher than previous Vickers results

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
- Suspected hardness increase in SZ confirmed via nanoindentation. Nanoindentation values not consistent with previous measurements.

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).

CAES
Center for Advanced Energy Studies

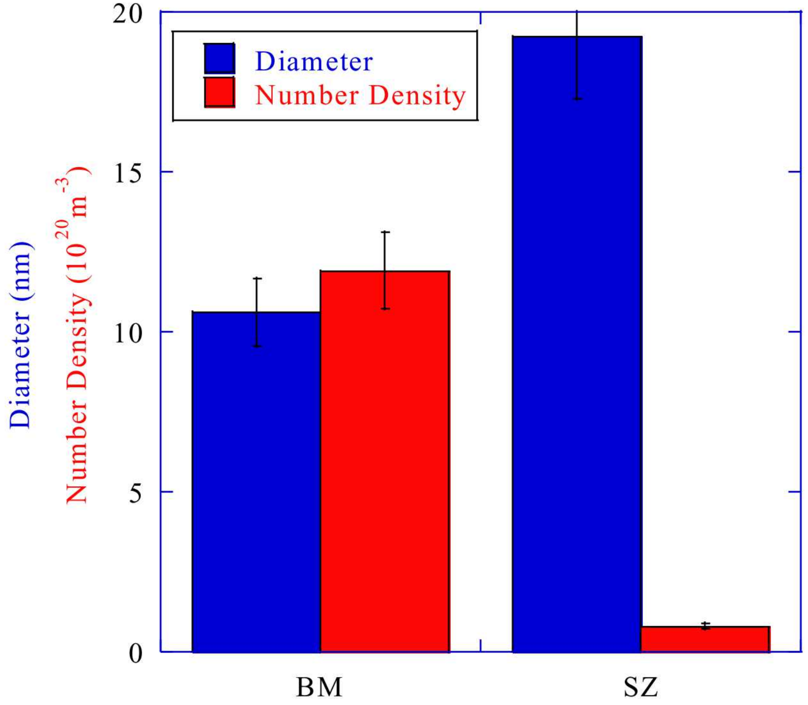
Sandia National Laboratories

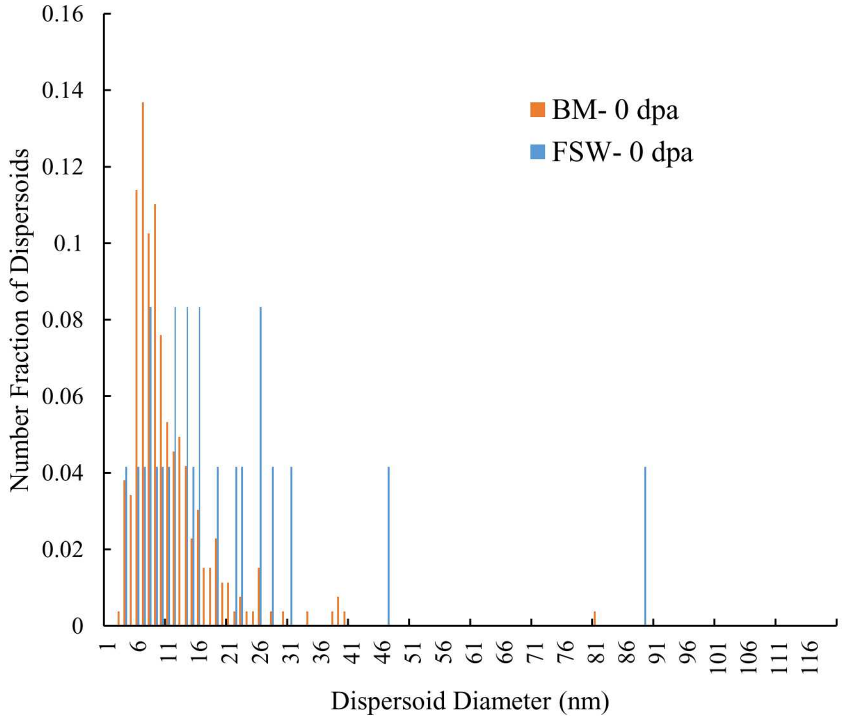
DEFENSE THREAT REDUCTION AGENCY
NSERC
NUCLEAR SCIENCE AND ENGINEERING RESEARCH CENTER
AT THE UNITED STATES MILITARY ACADEMY

Questions?

Extra slides

Quantitative Comparison of Dispersoids, 0 dpa

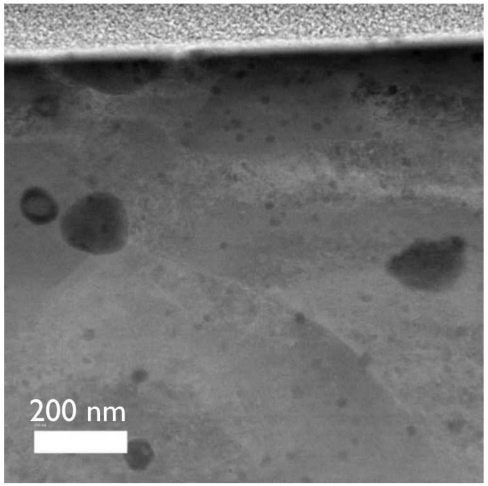




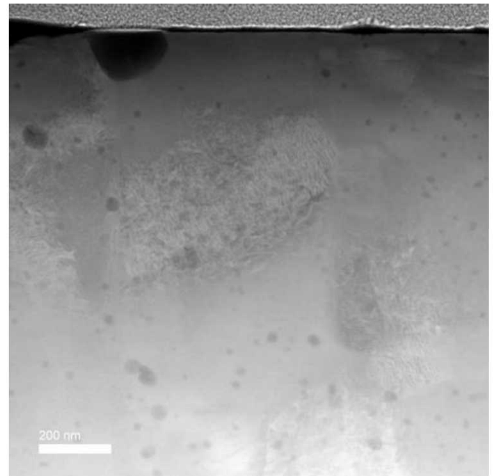
- Coarsening mechanisms (increased diameter, decreased number density and flatter distribution) consistent with previous results
- Mechanical testing reported softening with friction stir welding
  - Larger grains, fewer dispersoids

Base Material: 450°C up to 200 dpa

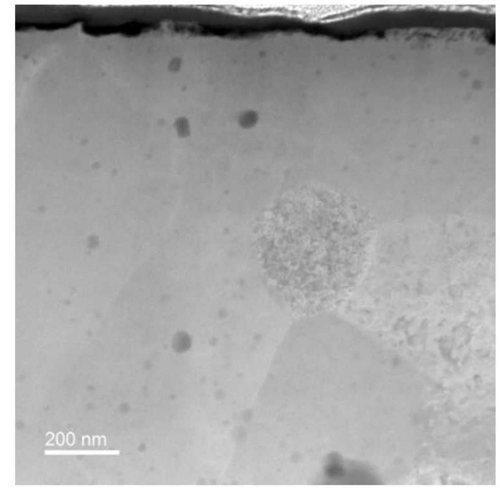
0 dpa



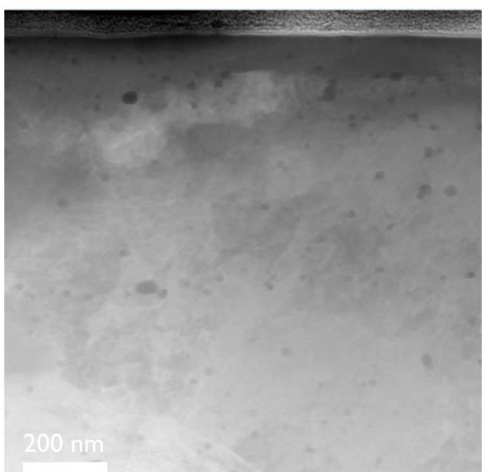
1 dpa



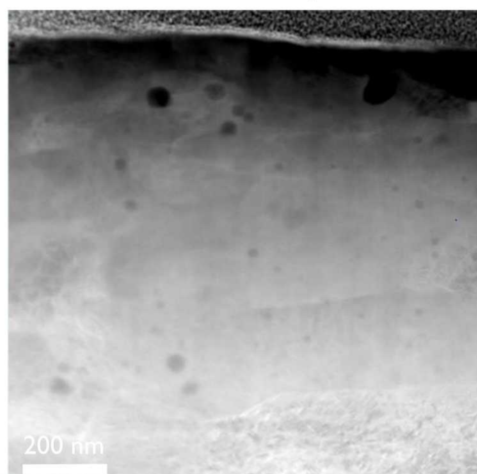
25 dpa



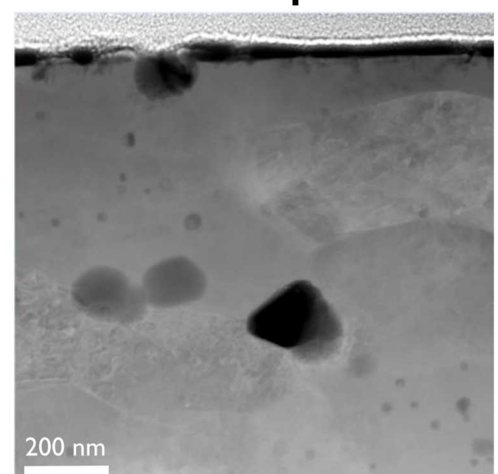
50 dpa



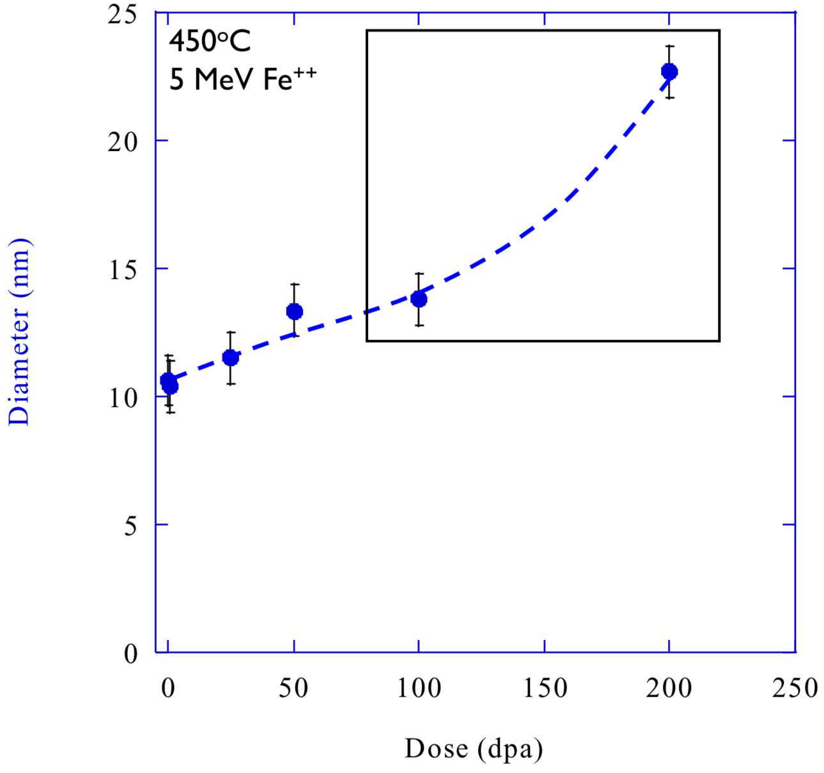
100 dpa

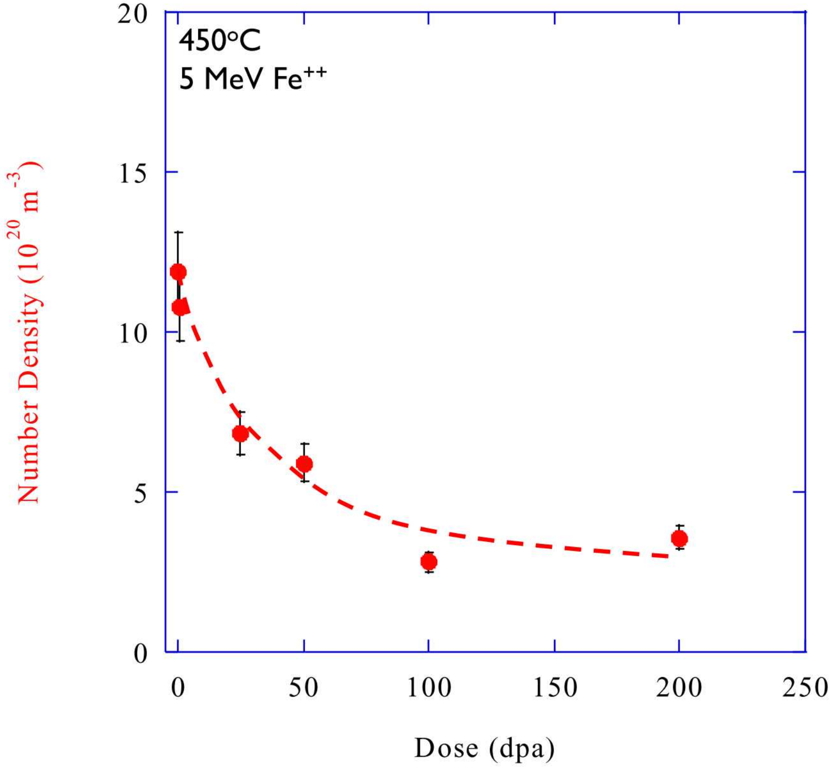


200 dpa



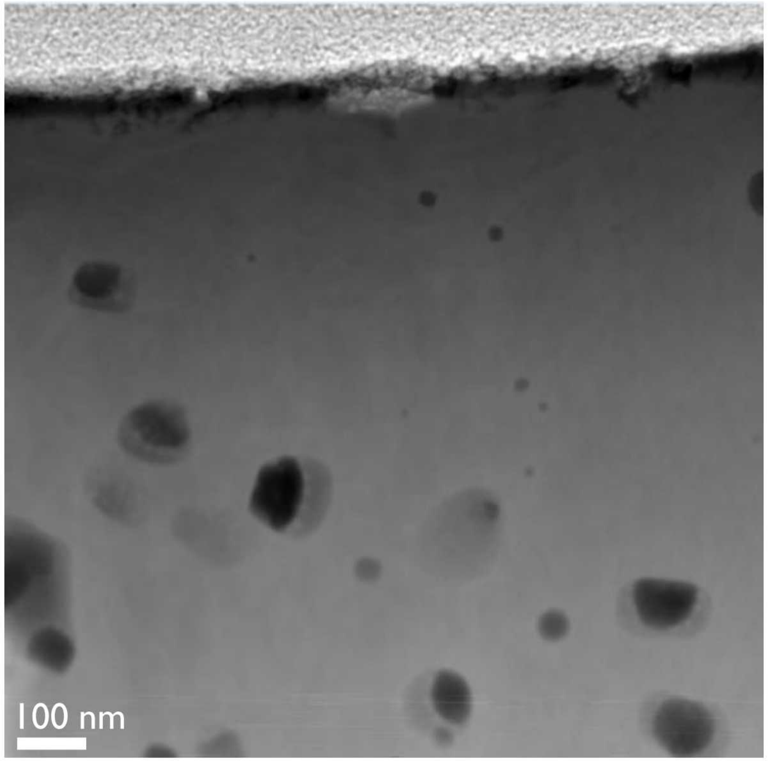
Dispersoid Evolution with Increased dpa (BM)





- 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

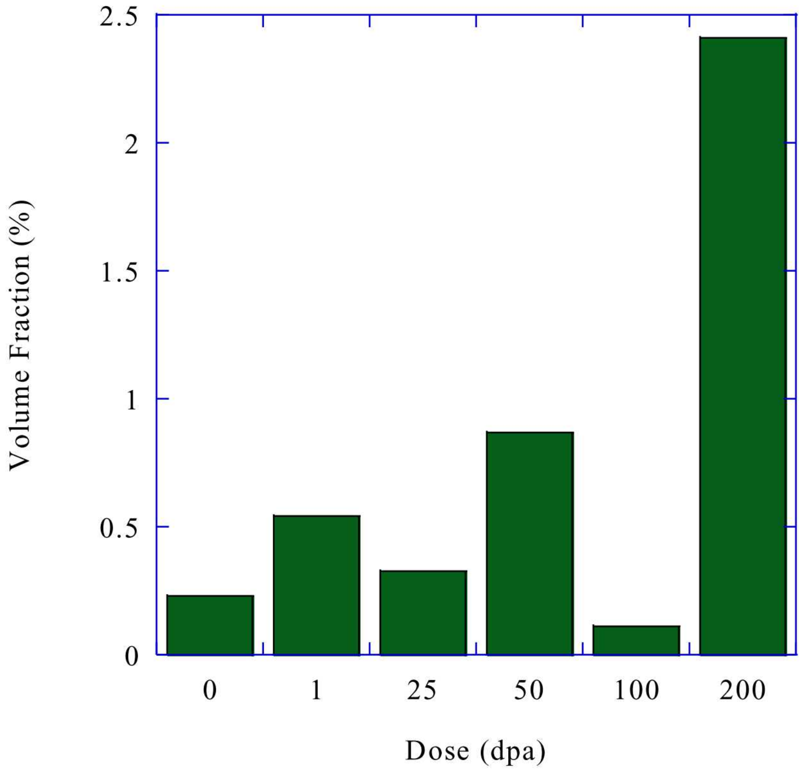
Formation of voids or coarsening of Y-Al-O?

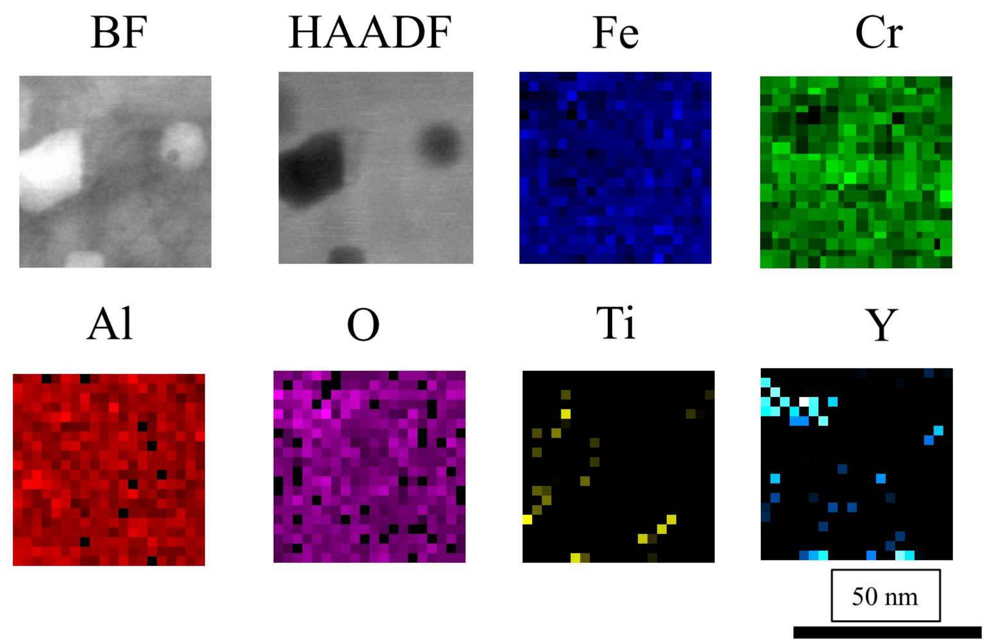


- STEM HAADF- Z contrast so Y-Al-O could look like a void
- CTEM BF can be used to identify by focusing series for voids, but time consuming and difficult to get accurate sizes
- Best approach suggested by Parish et al with a combination STEM HAADF, STEM through focus imaging coupled with high fidelity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

Evolution of Volume Fraction





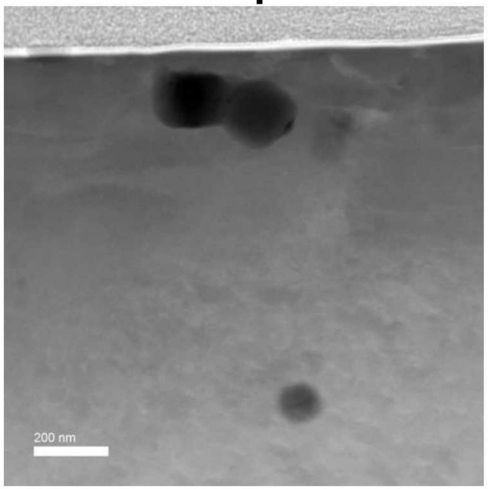
- Volume fraction suggest new large phase forming (suspected voids) by 200 dpa
- EDS Scans inconclusive
  - Plan on using ChemiStem to get better idea of what these features are

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

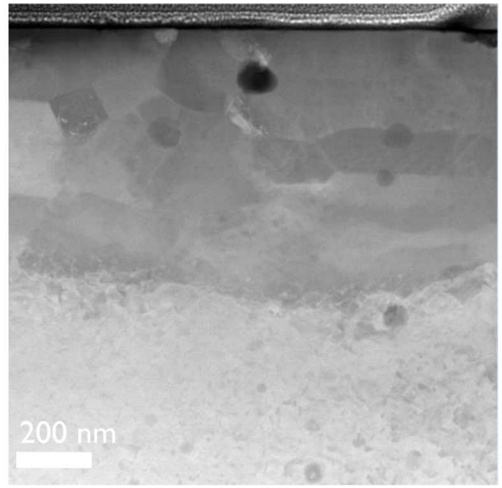
- CrC precipitates were not observed at 1 or 25 dpa
- Ti co-located with Cr-C as well

Stir Zone: 450°C up to 200 dpa

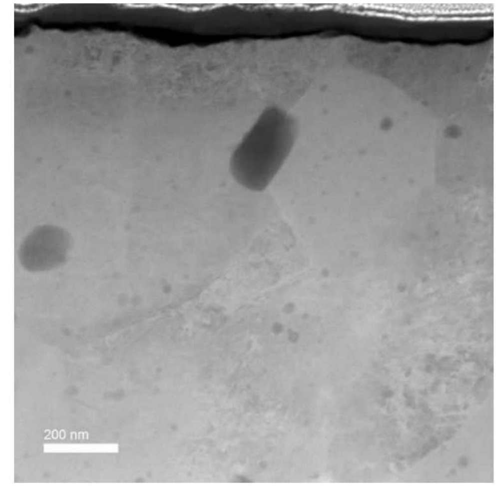
0 dpa



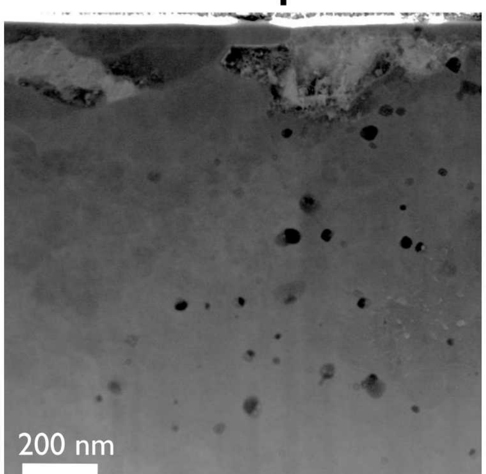
1 dpa



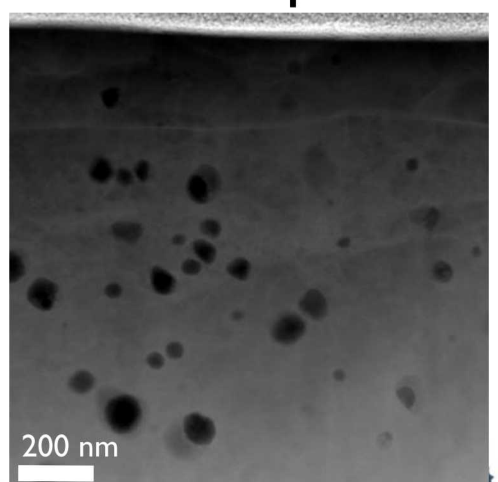
25 dpa



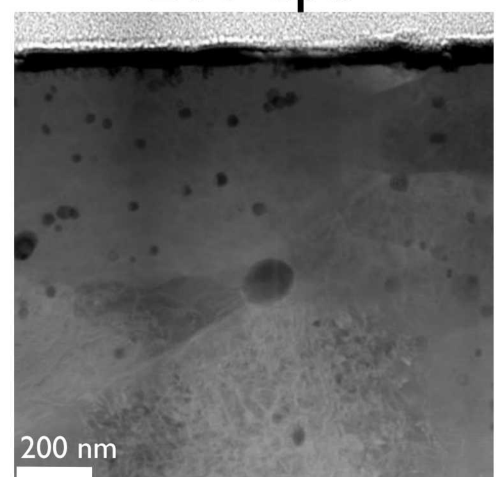
50 dpa



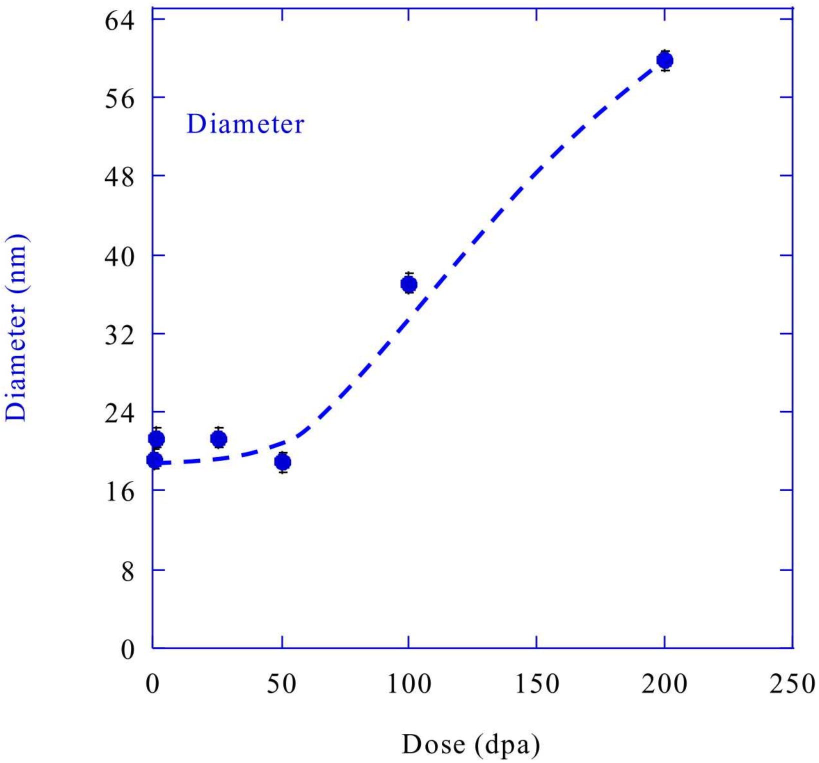
100 dpa

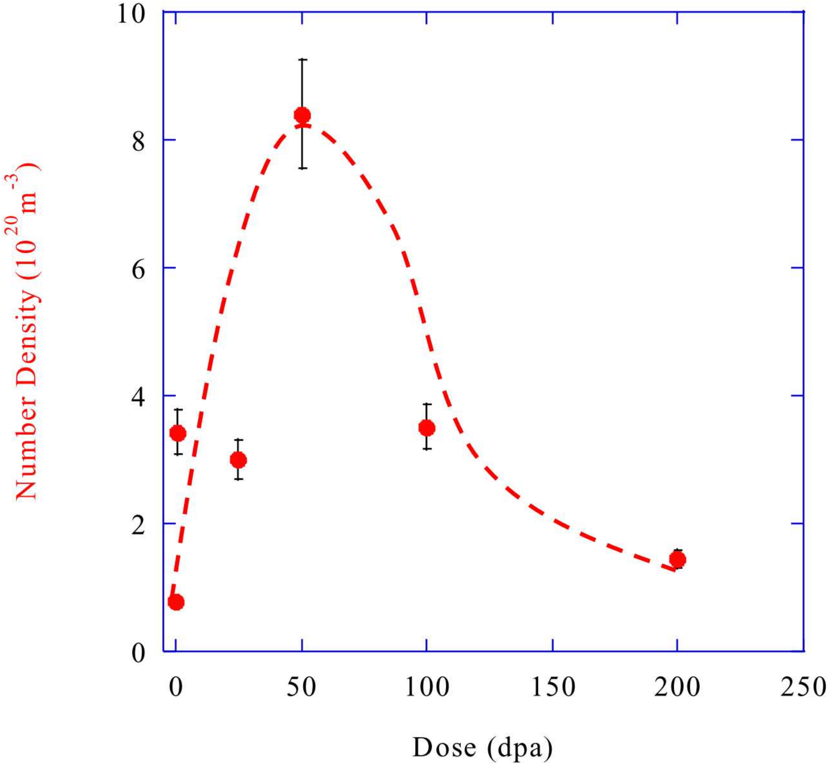


200 dpa



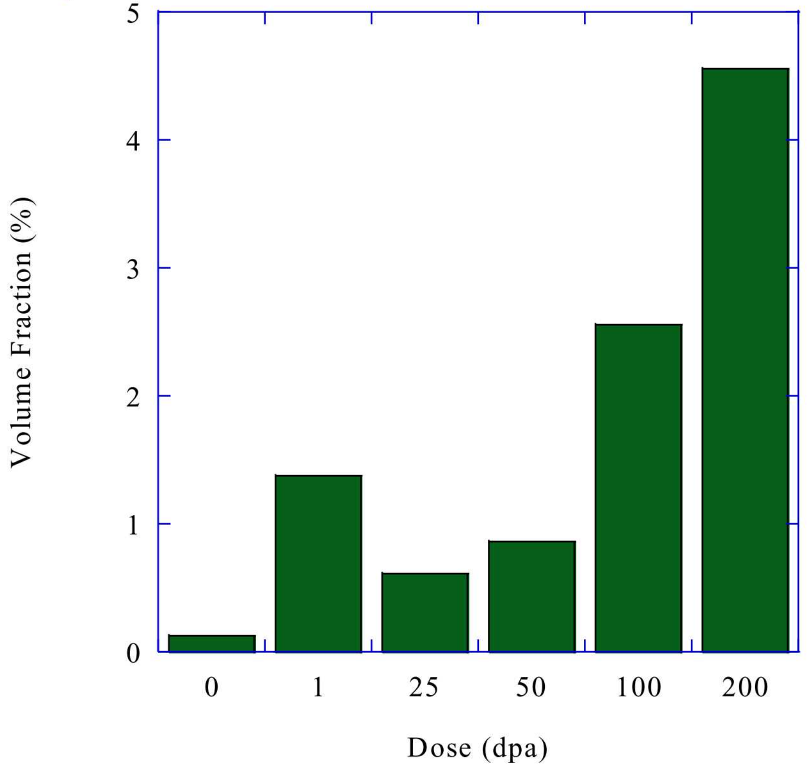
Dispersoid Evolution with Increased dpa (SZ)

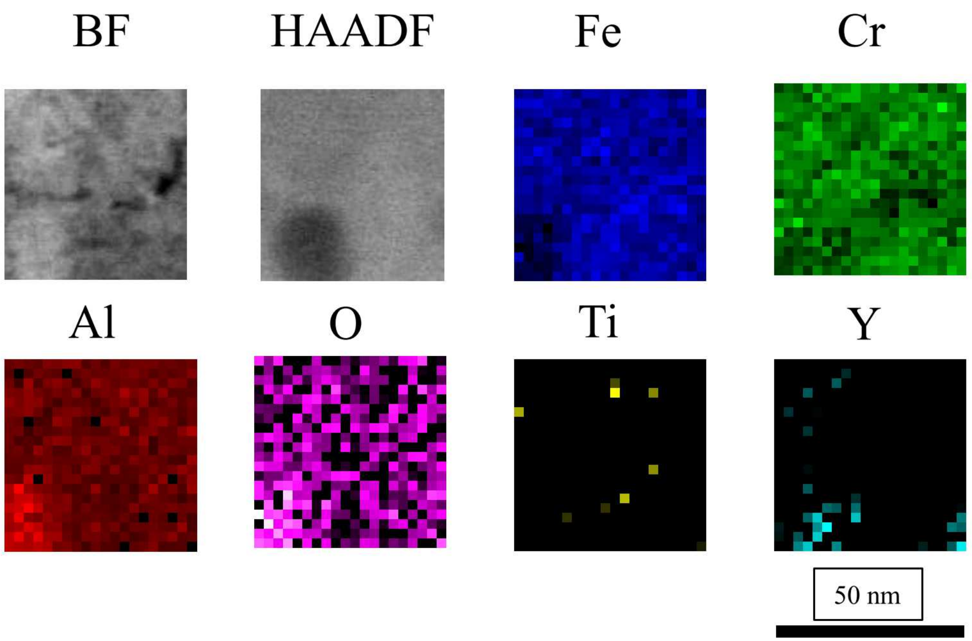




- Rapid growth at 100 dpa and beyond likely due to some void swelling similar to jump at 200 dpa for BM

Evolution of Volume Fraction?

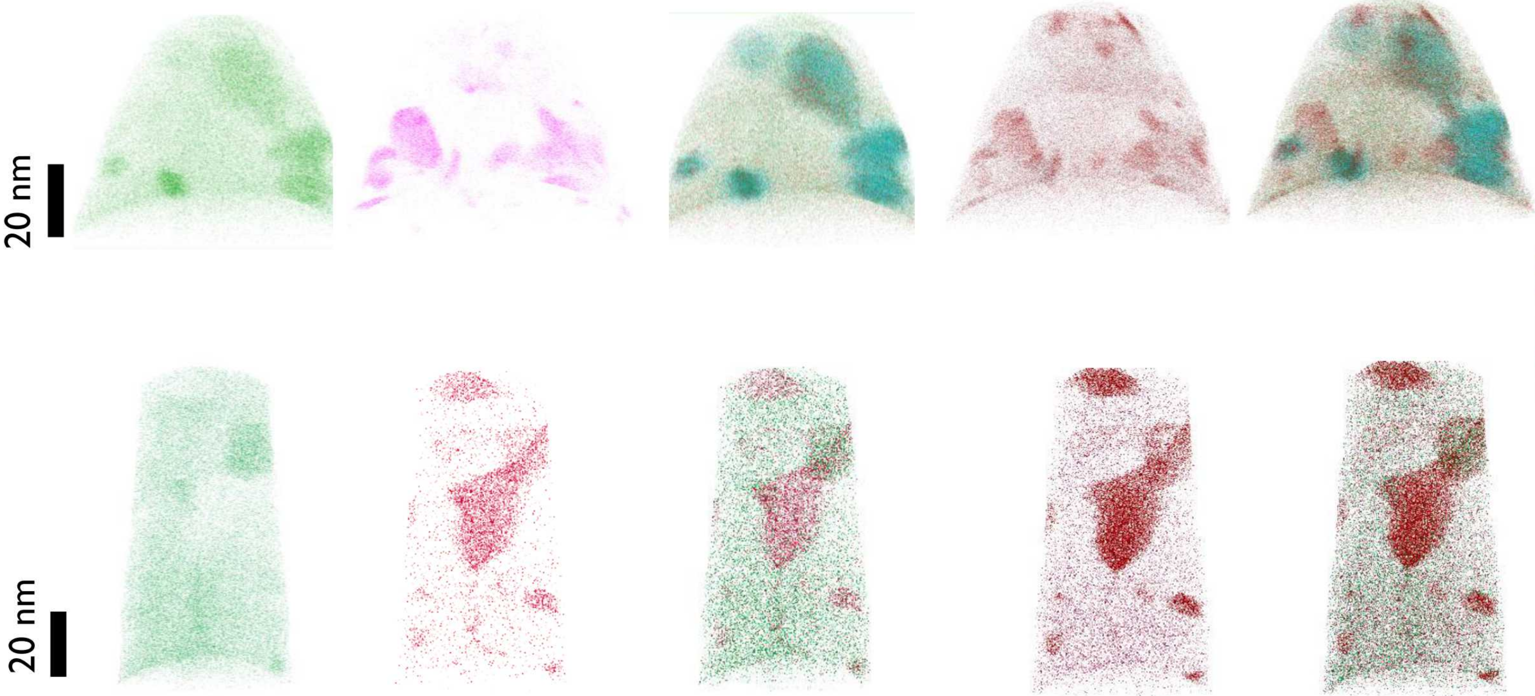


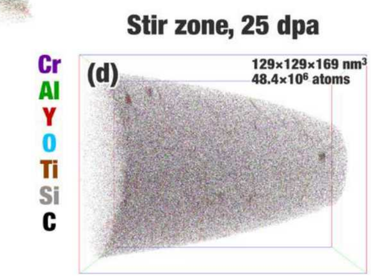


- Similar to BM-volume fraction suggest new large phase forming by 100 dpa
- EDS Scans inconclusive
  - Plan on using ChemiStem to get better idea of what these features are

Qualitative coarsening observed with APT in SZ

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





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

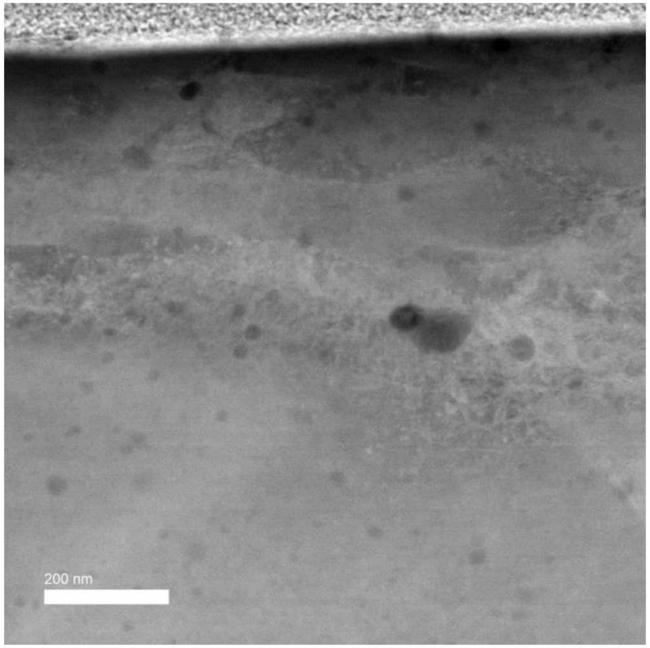
Future Work (in progress)

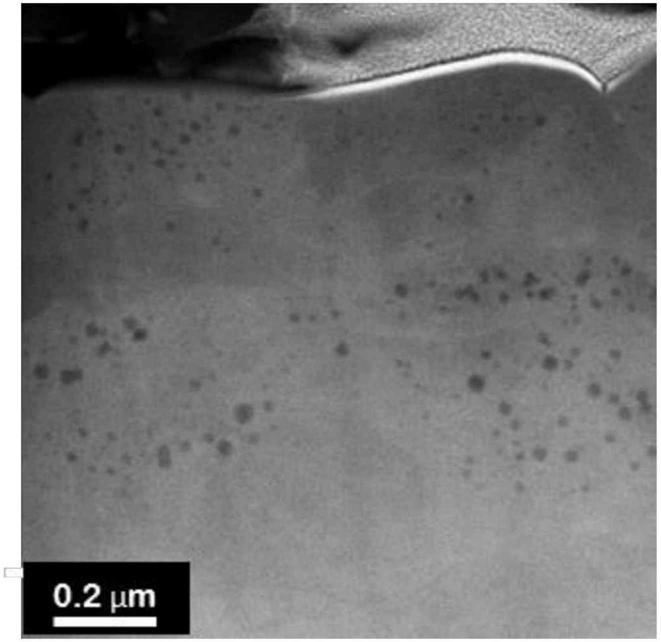
- Temperature dependence of BM, SZ
  - Irradiations funded by RTE 17-1032
  - Characterization funded by RTE 18-1396 (in progress)
- Effect of welding conditions
  - Irradiations funded by RTE 17-1032
  - Characterization funded by RTE 18-1396 (in progress)

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

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

Dispersoids or voids?





MA956, unirradiated
Black features are dispersoids

HT9, 440C, 188 dpa
Black features are voids