Microstructural evolution of neutron irradiated T91 ferritic-martensitic steel in the advanced test reactor

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Acknowledgements

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Objectives of my PhD Project

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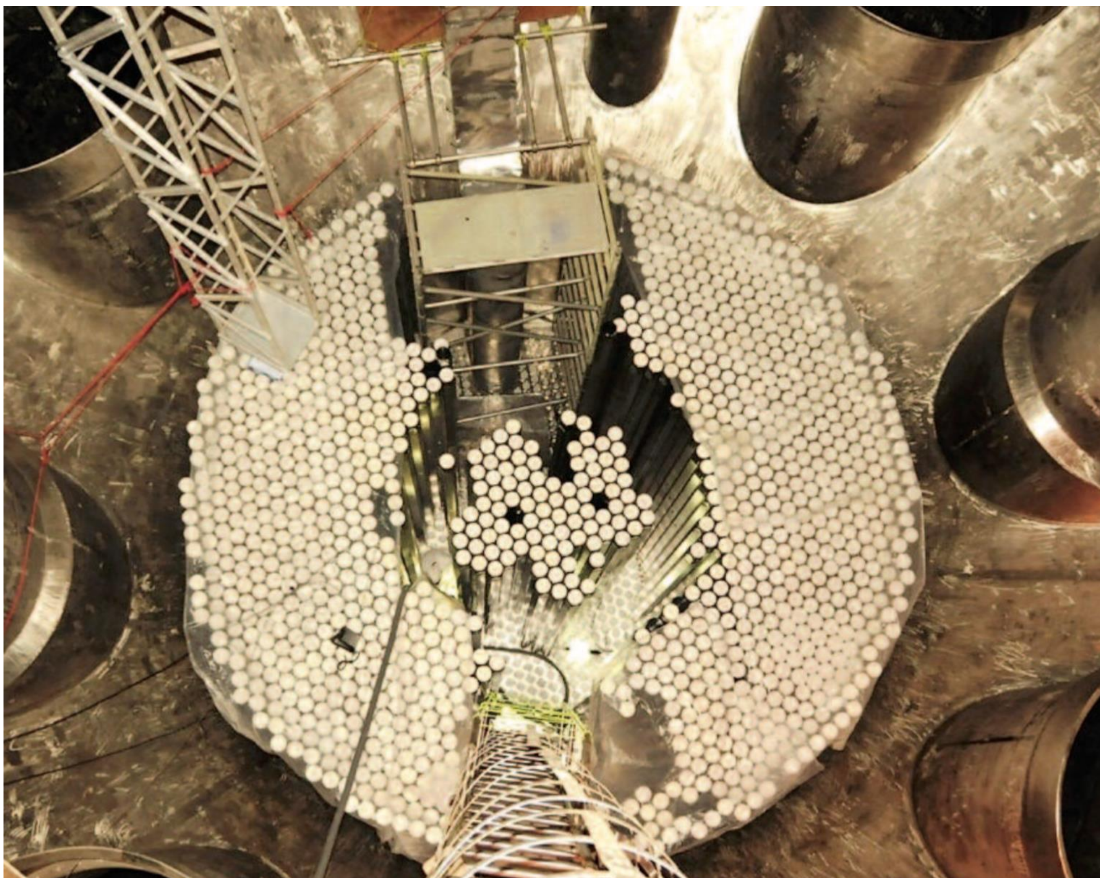
1. Utilise atom probe tomography and nanoindentation to study the microstructure of T91 steel in both unirradiated and irradiated conditions.

2. To understand the nucleation, evolution, and effect of Mn-Ni-Si ppts that are formed by neutron irradiation between 326-377 oC of T91 steel.

1. ASTM Grade 91 steel A213 (Tube 91; T91)

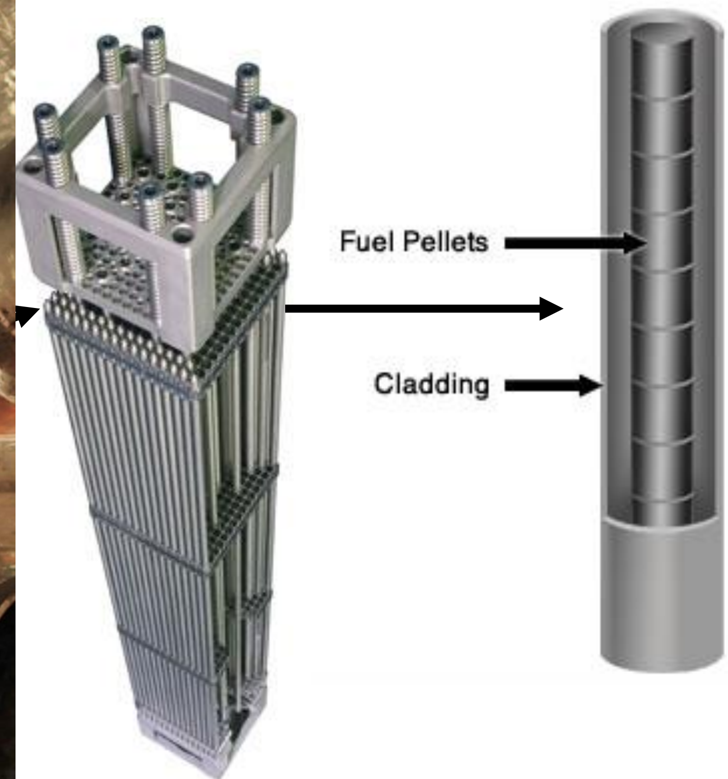
Application: Nuclear Fuel Cladding

T91 is a candidate for fuel cladding, duct/wrapper and structural material for sodium-cooled and lead-cooled fast fission reactors



Fuel Assembly

Fuel Rod



T.P. Davis, ONR-RRR-088, 2018, Office for Nuclear Regulation http://www.onr.org.uk/documents/2018/onr-rrr-088.pdf

ASTM A213 T91

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INTERNATIONAL

Designation: A 213/A 213M – 06a

Used in USDOE-NE standards

Standard Specification for Seamless Ferritic and Austenitic Alloy-Steel Boiler, Superheater, and Heat-Exchanger Tubes[1]

This standard is issued under the fixed designation A 213/A 213M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

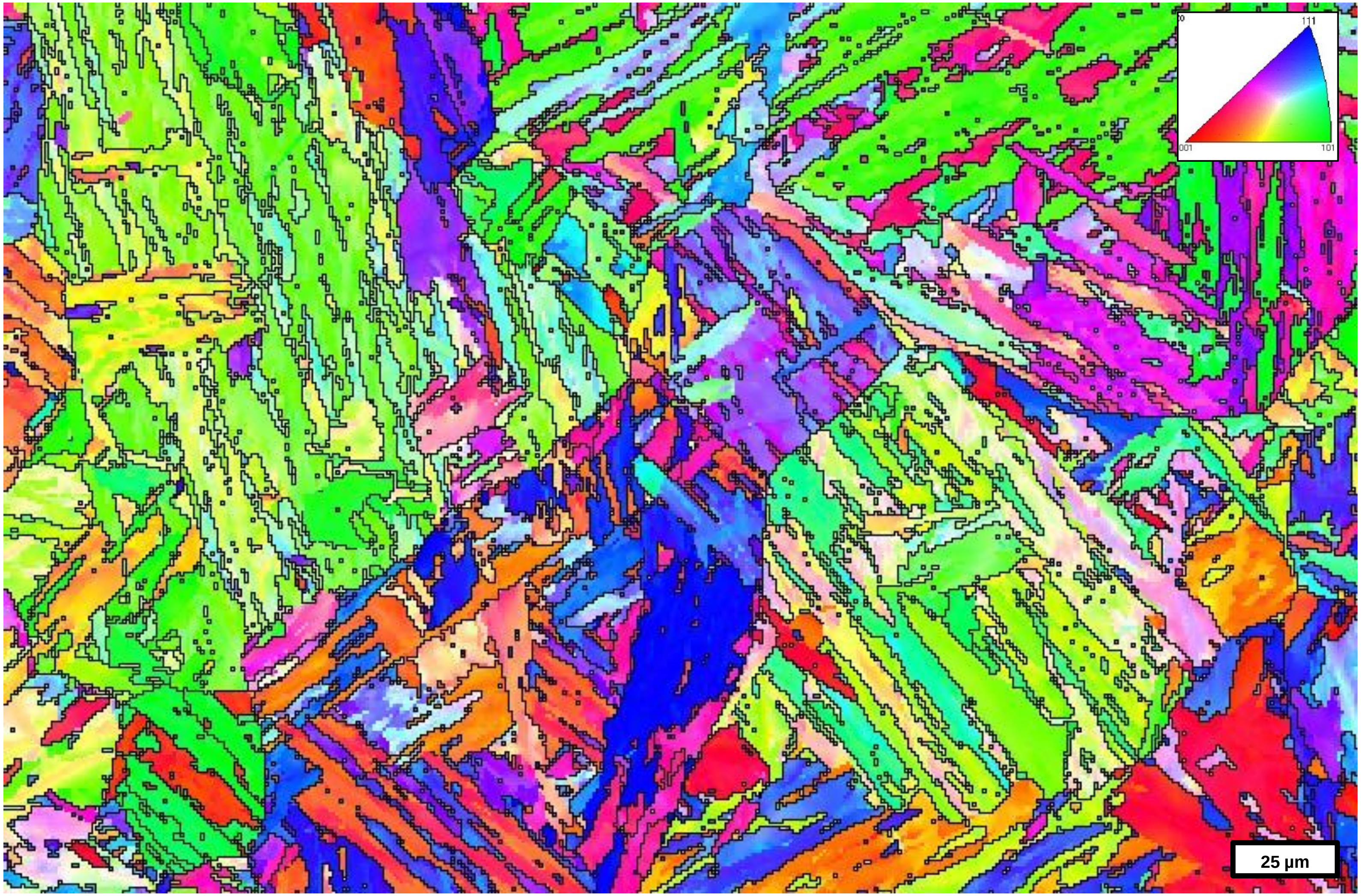
This standard has been approved for use by agencies of the Department of Defense.

Tube = diameter $3.2 \text{ mm} < d < 127 \text{ mm}$

thickness $0.4 \text{ mm} < t < 12.7 \text{ mm}$

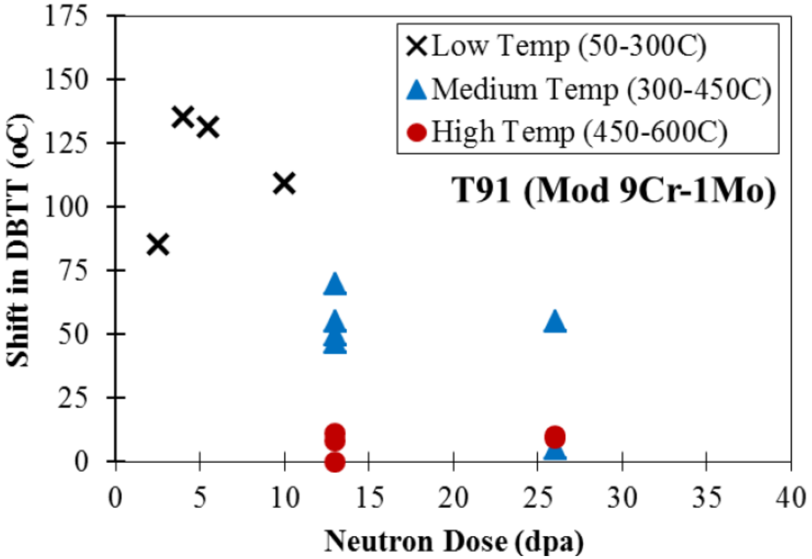
Fuel cladding tubes ~ 10 mm diameter; 0.5 mm thick

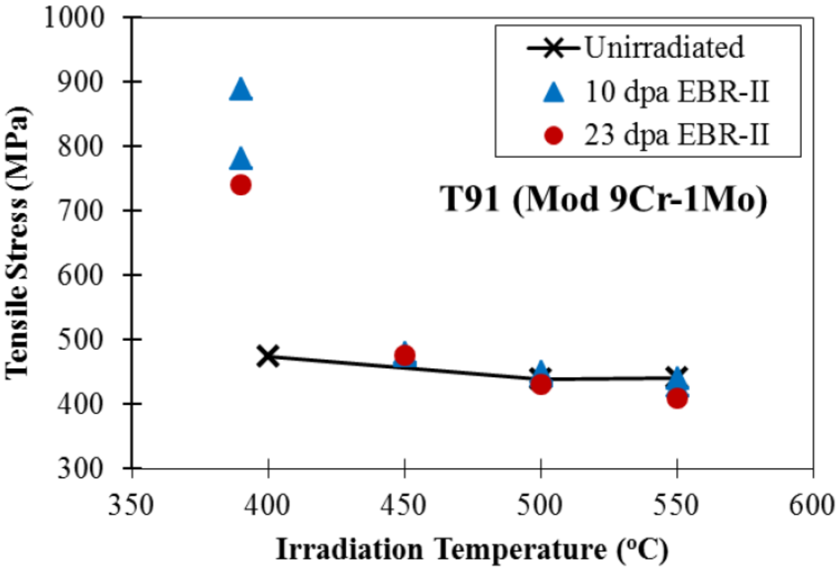
T91 Ferritic-Martensitic Steel

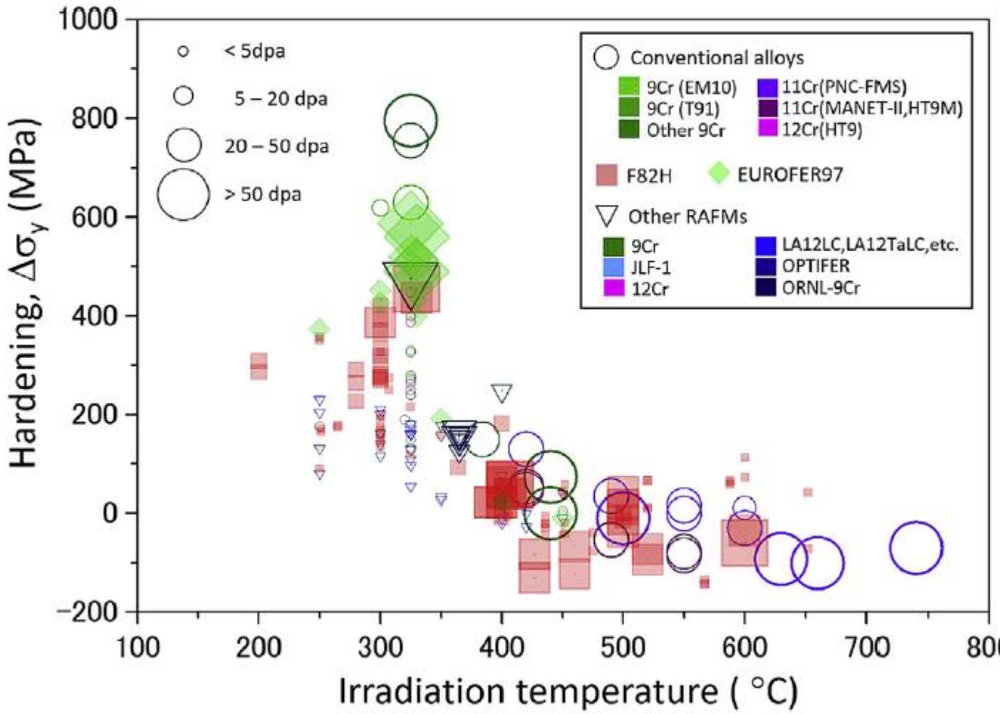


Steel	Composition (wt %)													
	C	Cr	Mn	Si	Mo	Nb	N	P	S	Al	V	Ti	Zr	Ni	Fe
T91	0.07	9.24	**0.47**	**0.28**	0.96	t	t	**0.02**	0.02	t	0.21	t	t	**0.16**	Bal.

Embrittlement of T91 Ferritic-Martensitic Steel







Cabet et al. JNM 523 2019 510

T.P. Davis, 'ONR-RRR-088: Review of the Iron-Based Materials Applicable for the Fuel and Core of Future Sodium Fast Reactors (SFR)'. Office for Nuclear Regulation, 2018

Neutron Irradiation of T91 in ATR

Nuclear Science User Facilities

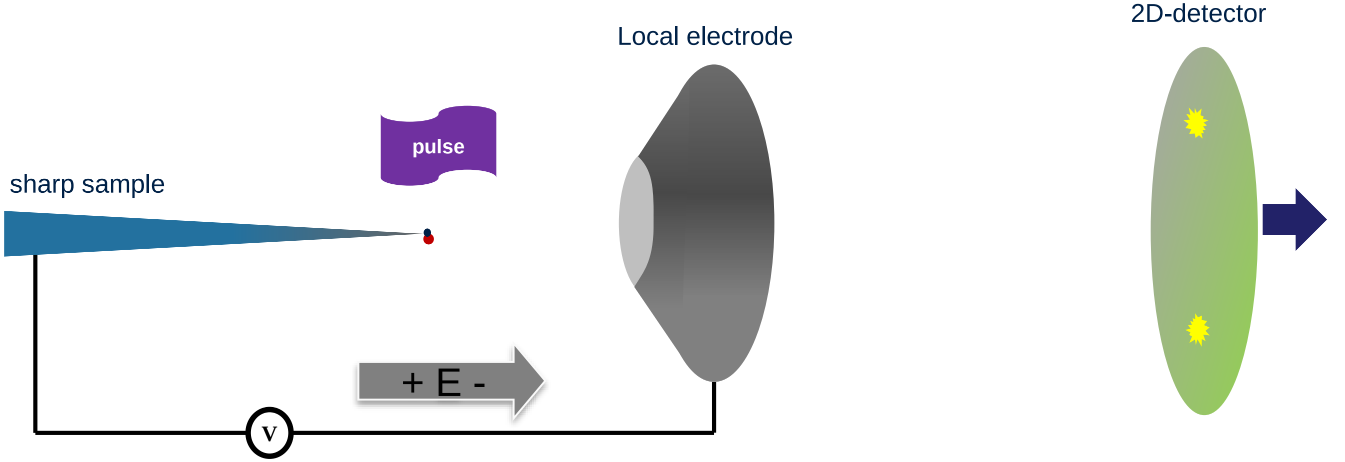
Project: Characterization of the Microstructures and Mechanical Properties of Advanced Structural Alloys for Radiation Service: A Comprehensive Library of ATR Irradiated Alloys and Specimen

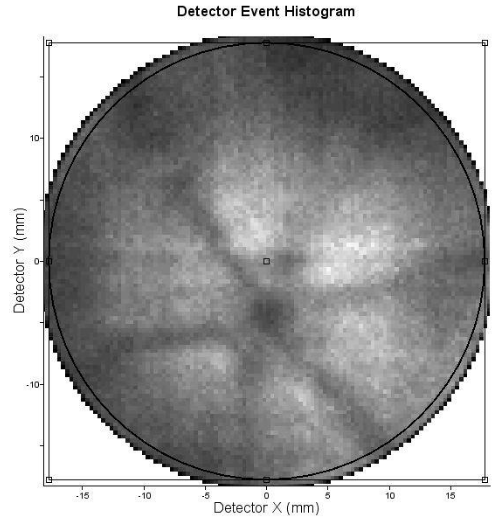
Steel	Temp (^oC)	Dose (dpa)	Flux ($n \cdot cm^{-2} \cdot s^{-1}$)
T91	326	2.14	2.3E+14
	372	8.82	

First: How does Atom probe work?

- Sharp needle sample; tip radius 50-100nm; 50K temp; high vacuum
- Apply an electric field (2000-10,000V); the field is concentrated at the sharp tip to ~40 V/nm.
- Produces field evaporation at the tip of the needle when a short additional **voltage or laser pulse** is applied.
- If we know when the ions leave the tip then their time-of-flight can be related to their mass-to-charge ratio:

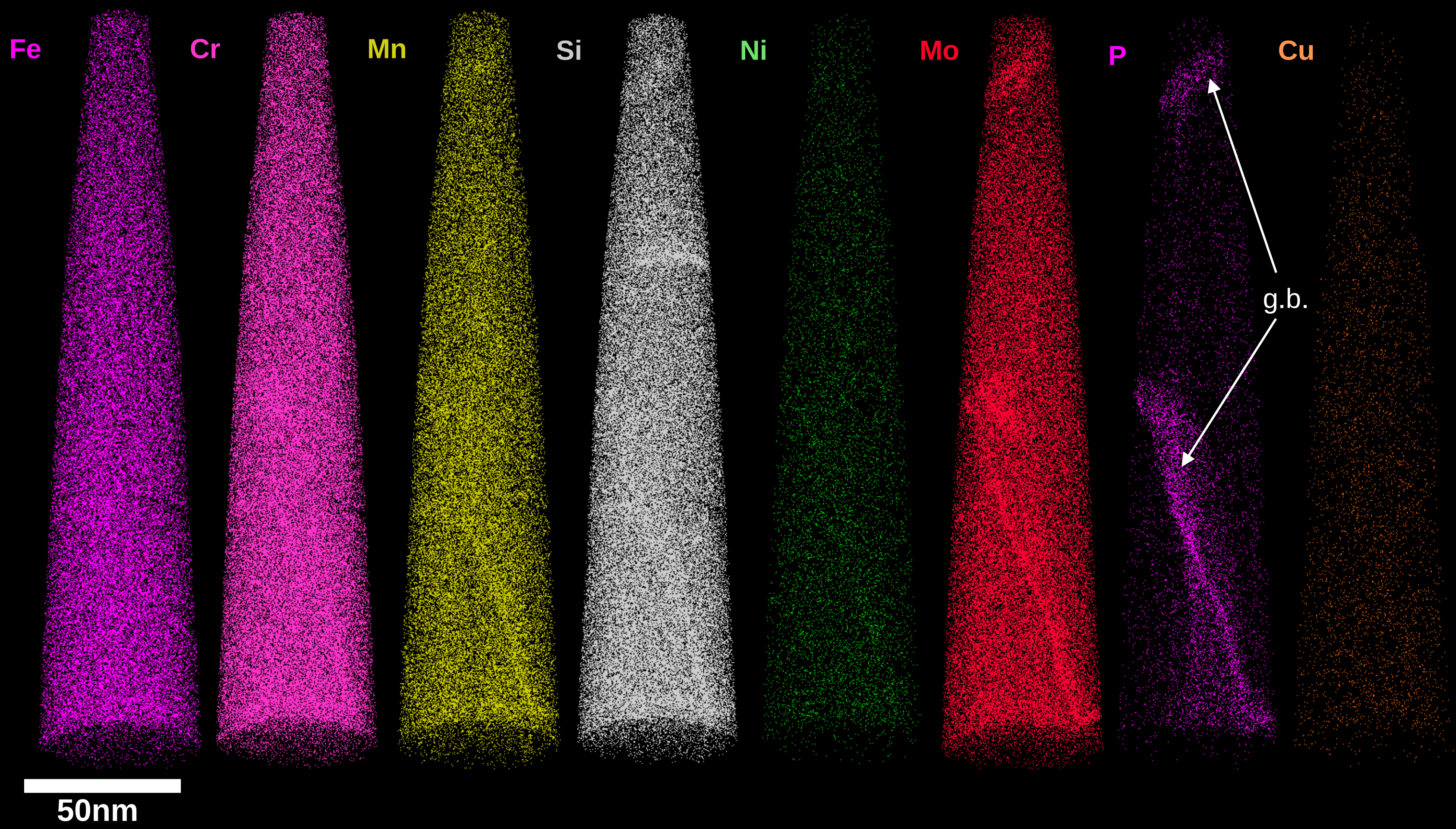
$$\frac{\text{mass}}{\text{charge}} = 2eV\left(\frac{\text{time}}{\text{flight distance}}\right)^2$$



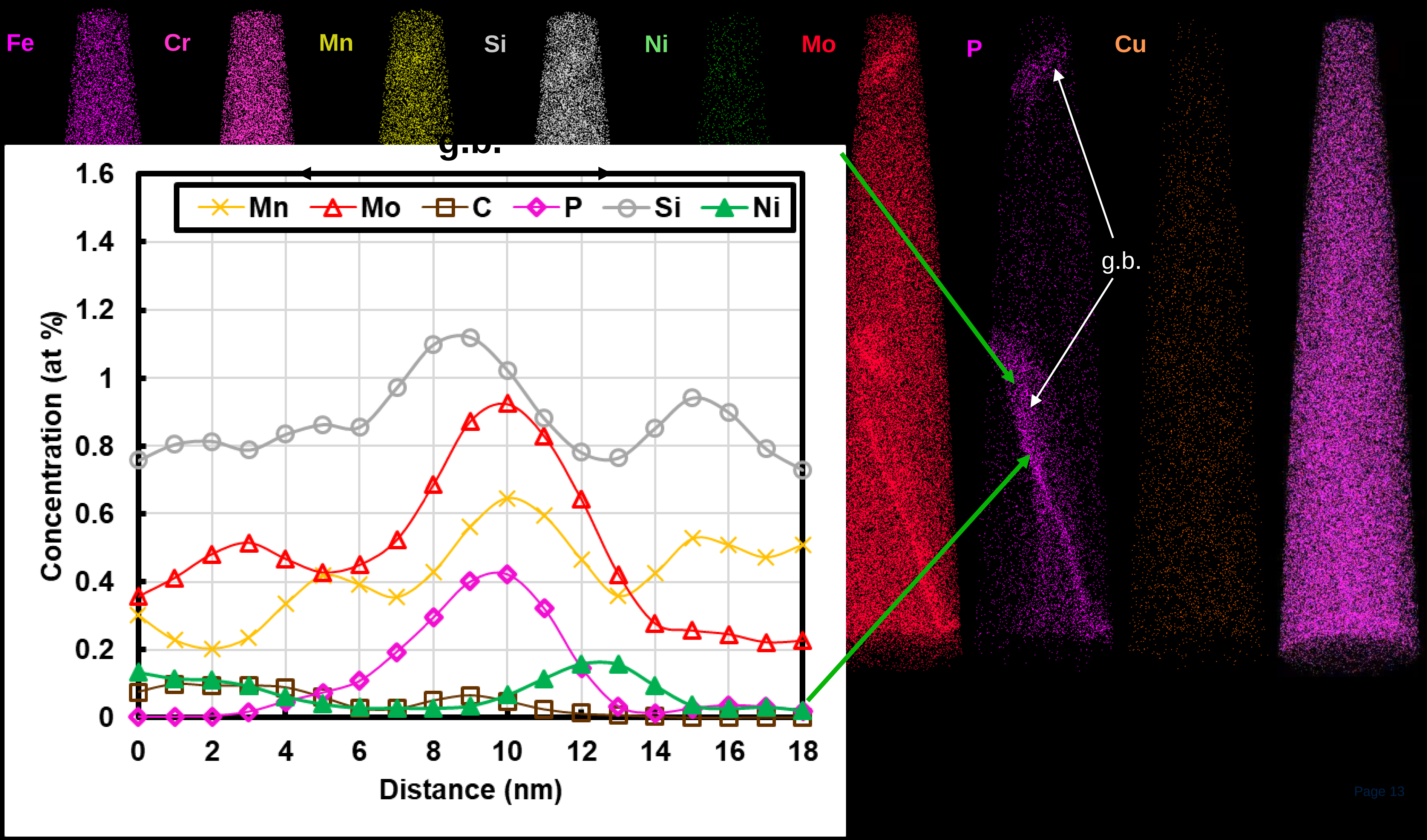


As Received T91 Steel

T91 Steel: As-Received Characterisation



T91 Steel: As-Received Characterisation



2.14 dpa 326 ^{o}C T91 Steel

T91: 2.14 dpa 320C: Mn-Ni-Si cluster formation

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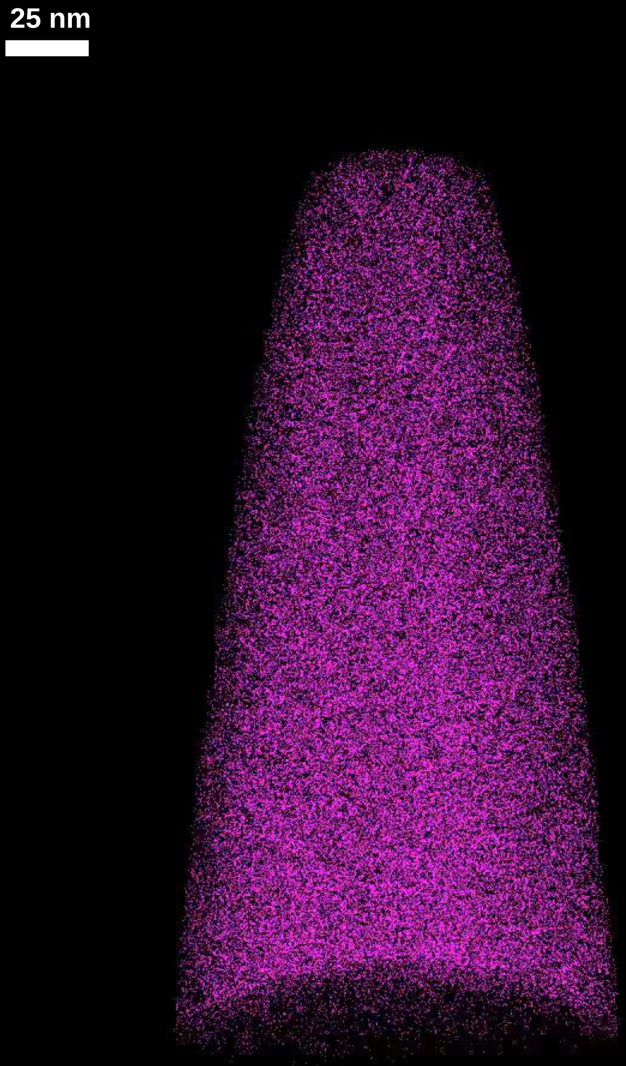


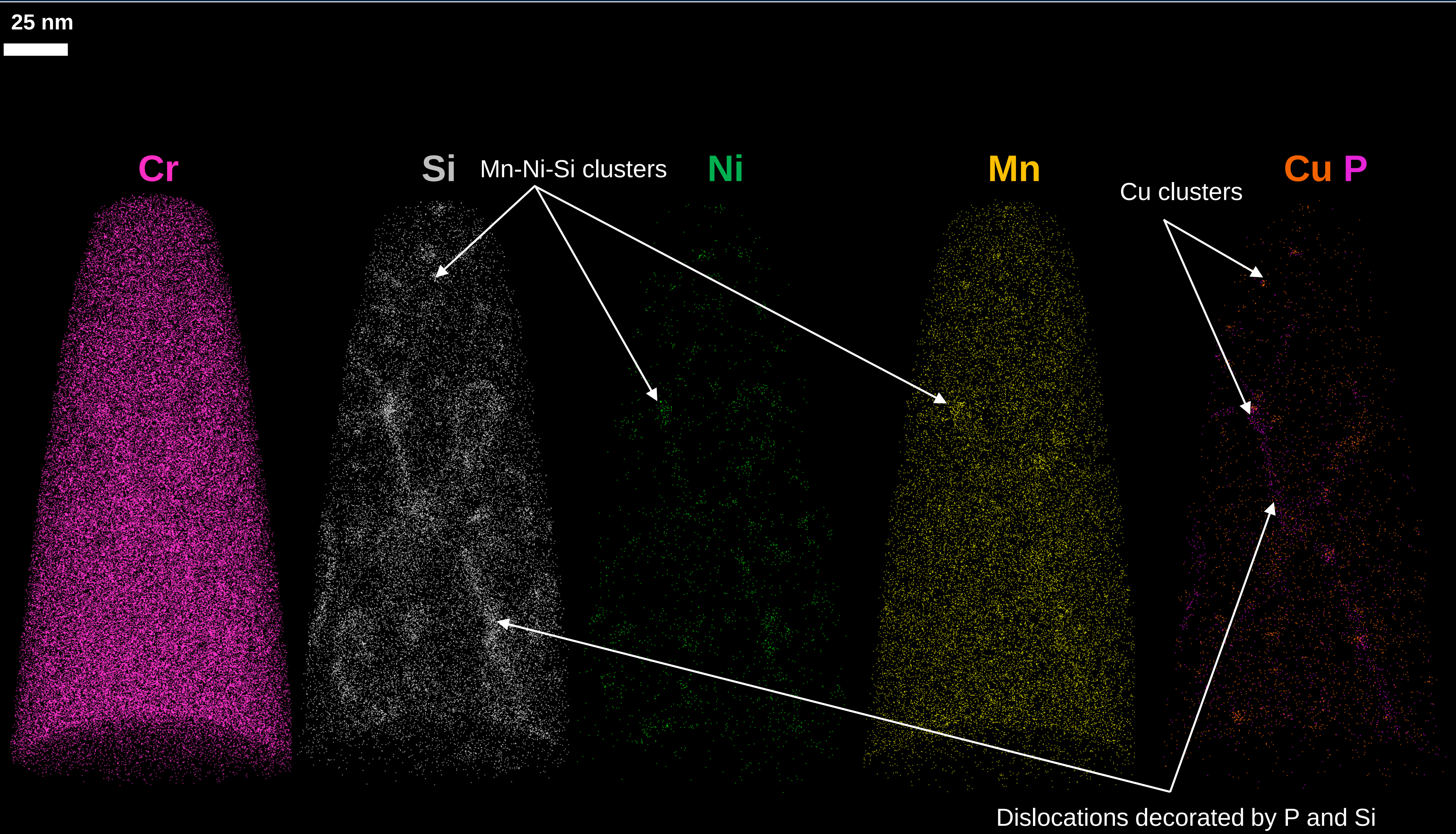
Cr Si Ni Mn Cu P

Segregation of Si, Mn, Ni, P and Cu

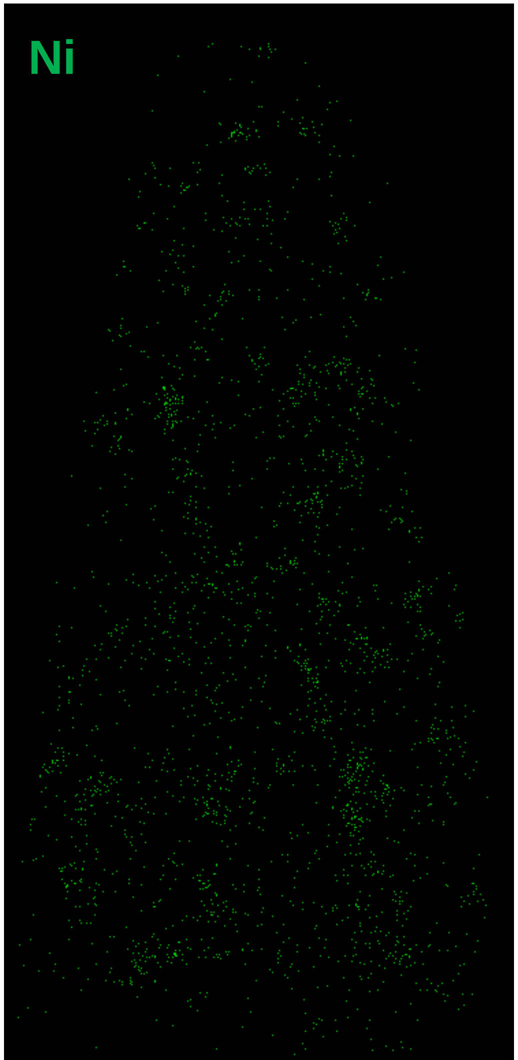
1) Cu forms clusters
2) P forms clusters
3) Si segregates to P and Cu clusters
4) Ni segregates to Si and Cu clusters
5) Mn segregates to Si, Ni and Cu clusters
6) Mn-Ni-Si clusters appear to form adjacent to Cu clusters
7) P and Si segregation to dislocations

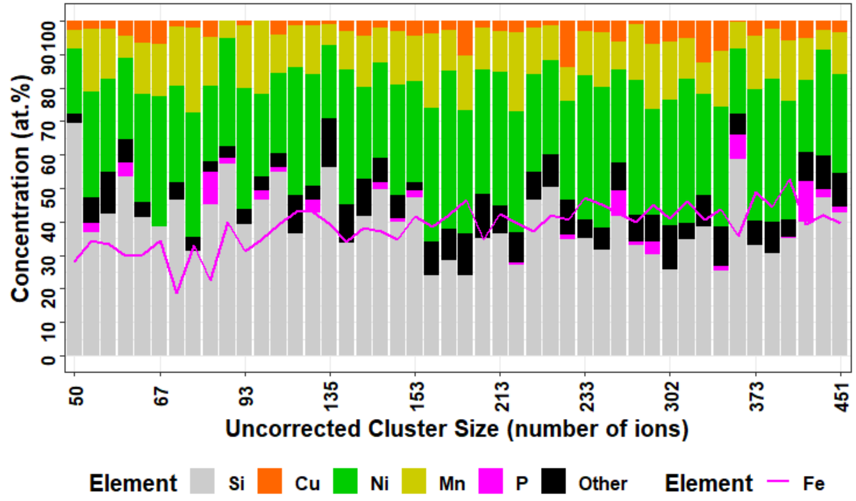
Partially forming ‘G-Phase’ Mn-Ni-Si ppts

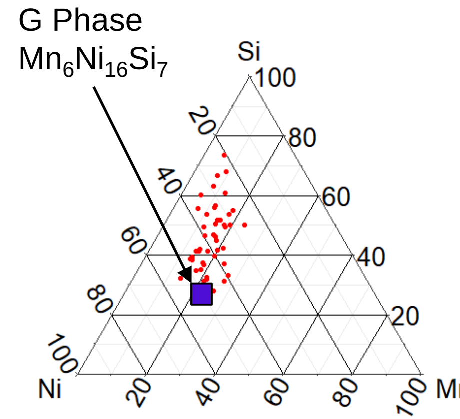
T91: 2.14 dpa 320C: Mn-Ni-Si cluster

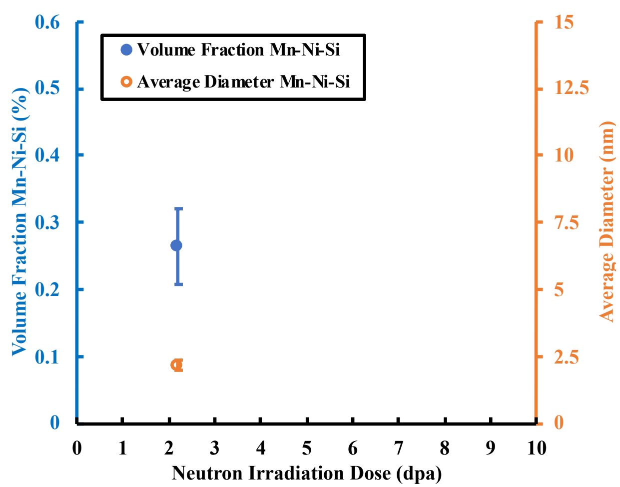


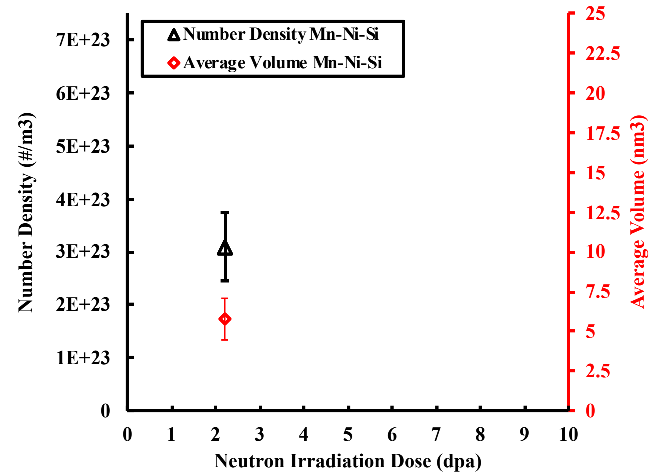
T91: 2.2 dpa 320C Mn-Ni-Si clusters



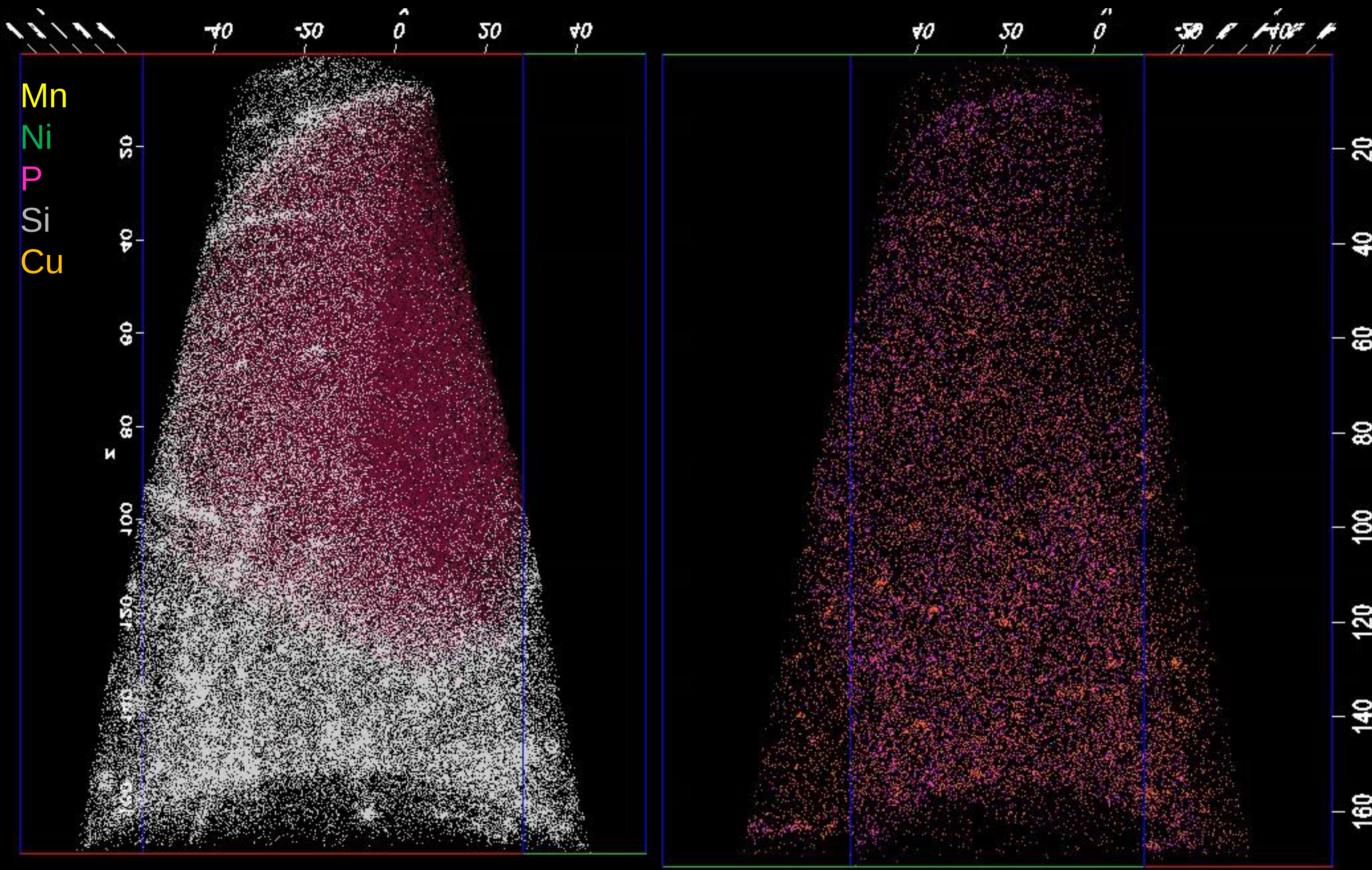






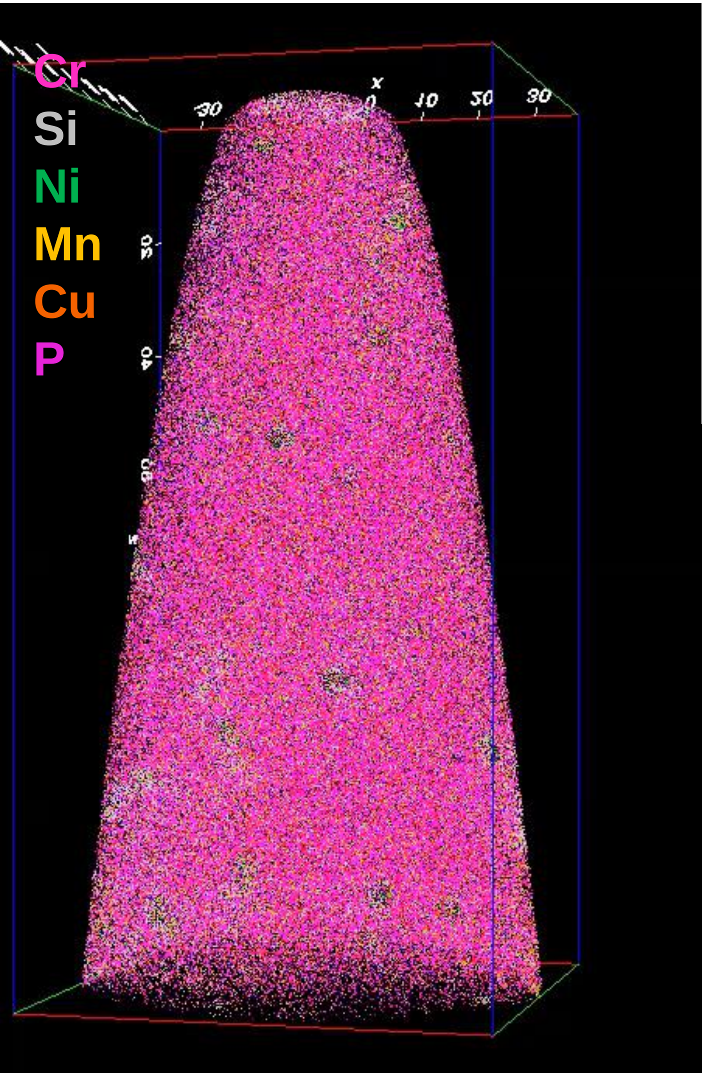


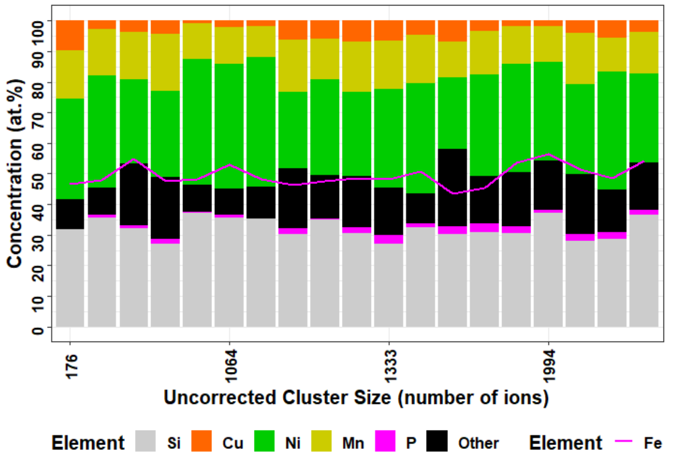
T91: 2.2 dpa 320C Bonus

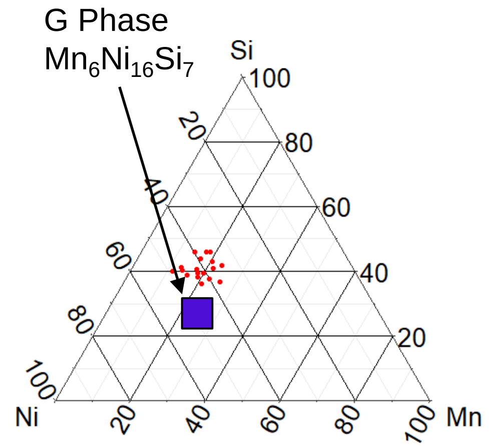


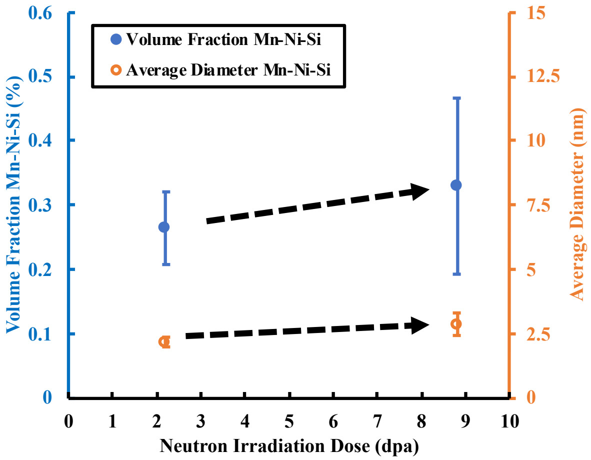
T91 8.8 dpa 370 ^{o}C

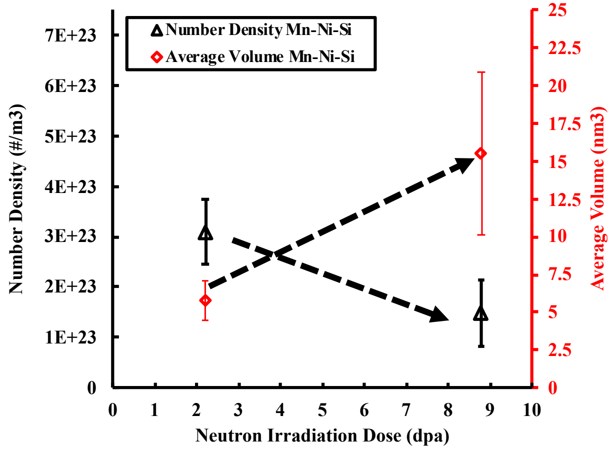
T91: 8.8 dpa; 370C – Mn-Ni-Si clusters





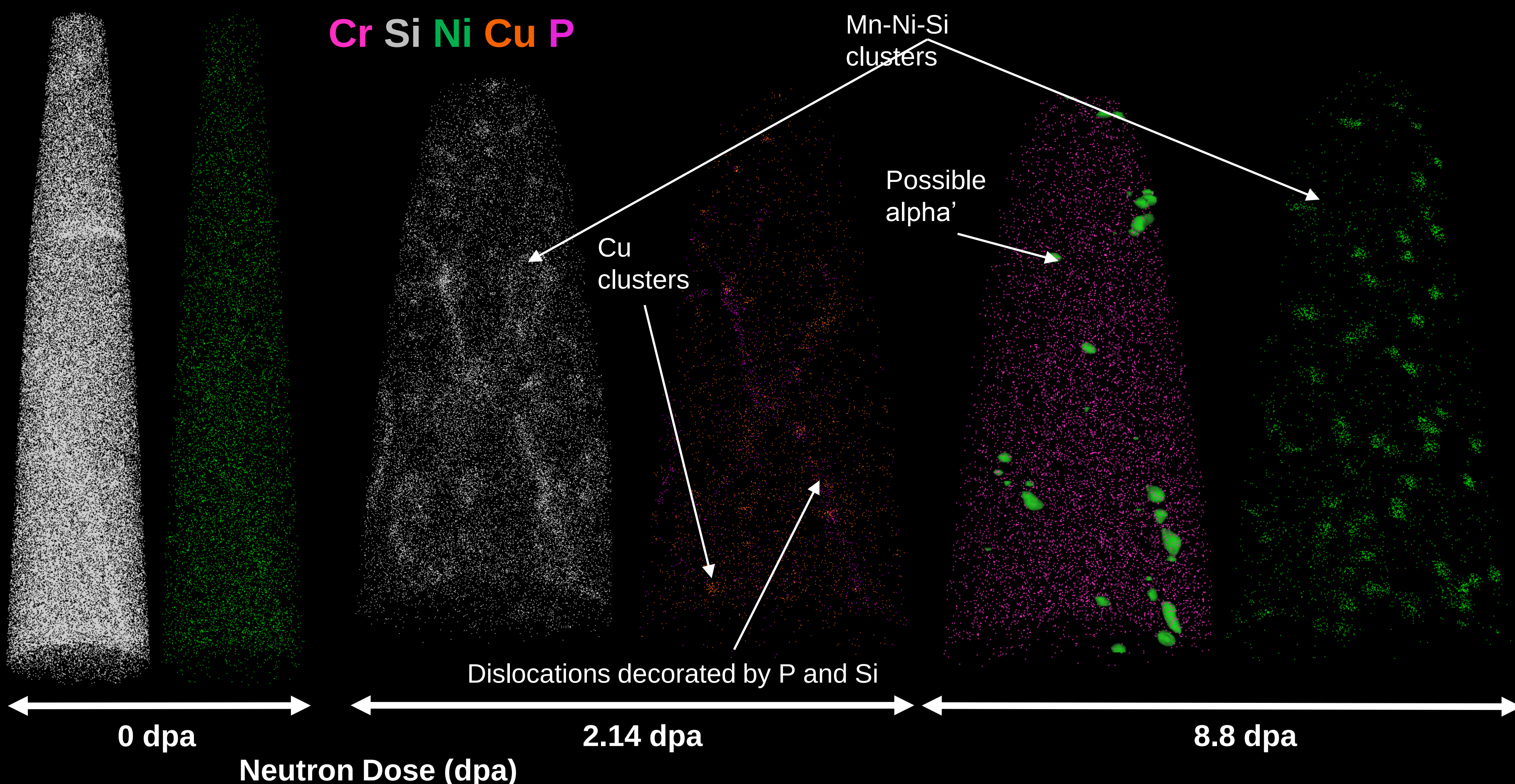






Evolution of the microstructure

Microstructure Evolution of Irradiated T91 Steel



What does the atom probe data tell us about irradiation of T91 steel?

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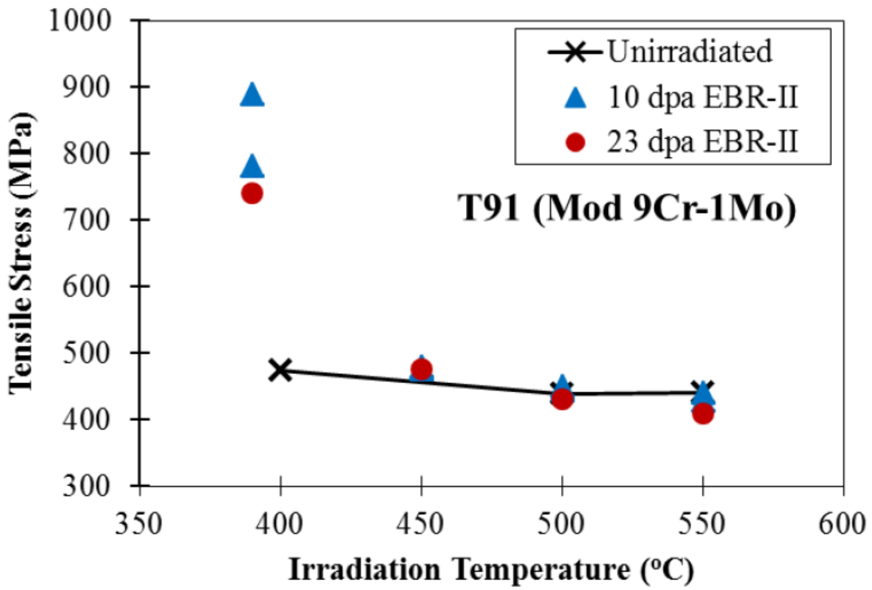
- Embrittlement (increase in yield stress) is shown below 400 °C. G-Phase ppts inhibit dislocation motion, which contributes majorly to the embrittlement of T91 steel below 400 °C (as shown in figure to the left [3]).

- Observed Mn-Ni-Si clusters at 2.14 dpa and grew in size, volume fraction, average diameter and average volume. However, decreased in number density but this measurement could be affected by localised features in atom probe datasets. No alpha prime observed.



T.P. Davis, 'ONR-RRR-088: Review of the Iron-Based Materials Applicable for the Fuel and Core of Future Sodium Fast Reactors (SFR)'. Office for Nuclear Regulation, 2018

- Future work: nanoidentation and prior austenite grain boundary analysis of neutron irradiated T91 steel

- Overall this research provides an insight into the microstructural evolution of neutron irradiated T91 steel and provides a better understanding of the degradation of nuclear reactor core materials.

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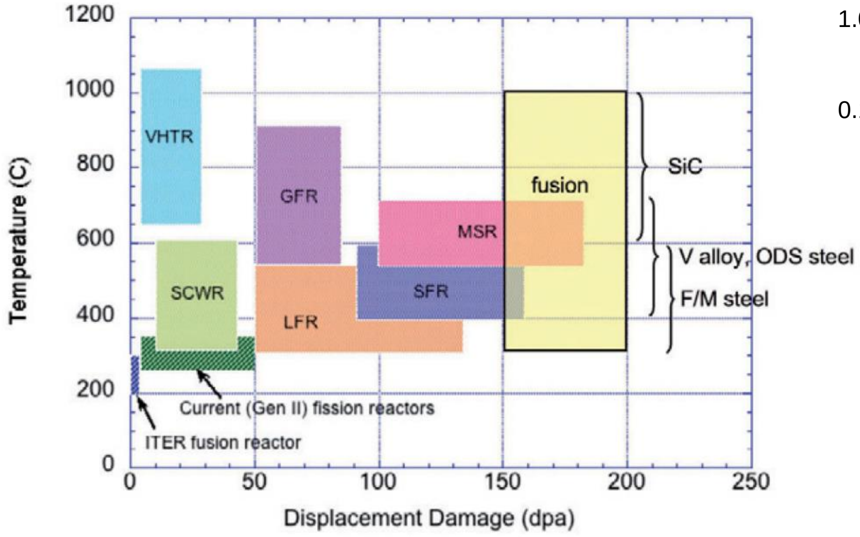
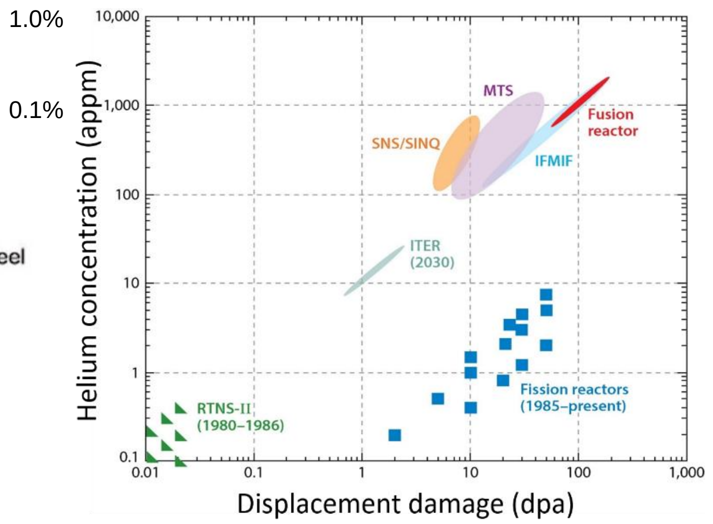
Extra Slides

Challenge for structural materials



Overview of the temperature regimes against expected displacement damage of various current and future reactors [1].

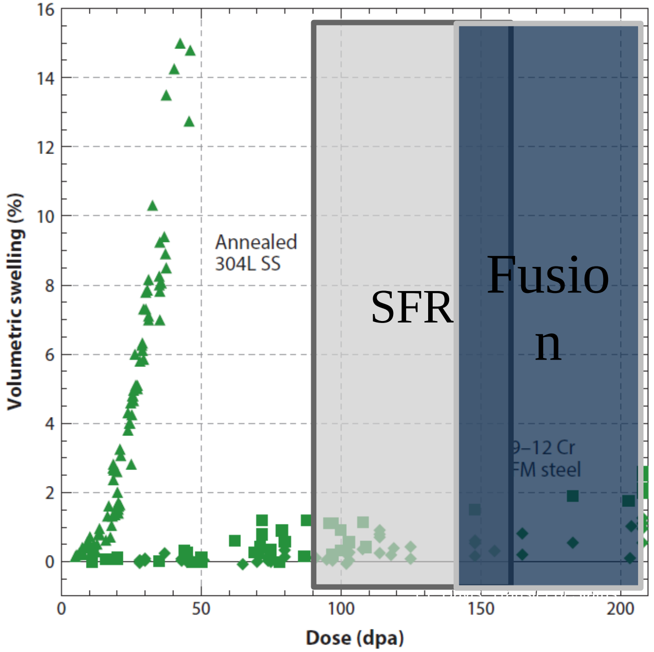


Helium production in advanced steels in fission, fusion and neutron experiments [2].

[1] Zinkle, S. J., & Busby, J. T. (2009) *Mater. Today*, 12(11), 12–19.
[2] Zinkle, S. J., & Snead, L. L. (2014) *Annu. Rev. Mater. Res*, *44*, 241–67.

Austenitic Stainless Steels

304 and 316 austenitic stainless steel grades are the work horse material in the nuclear industry however…



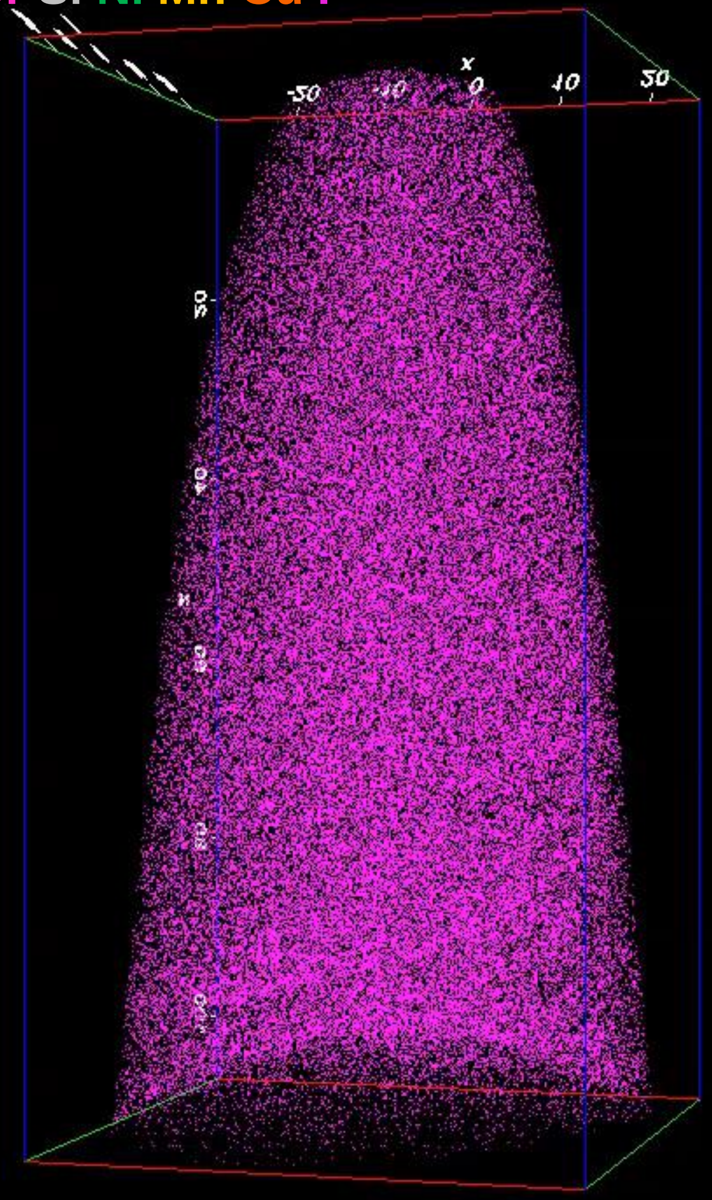
Comparison between volumetric swelling of 304L and 9-12Cr ferritic/martensitic steels [3].

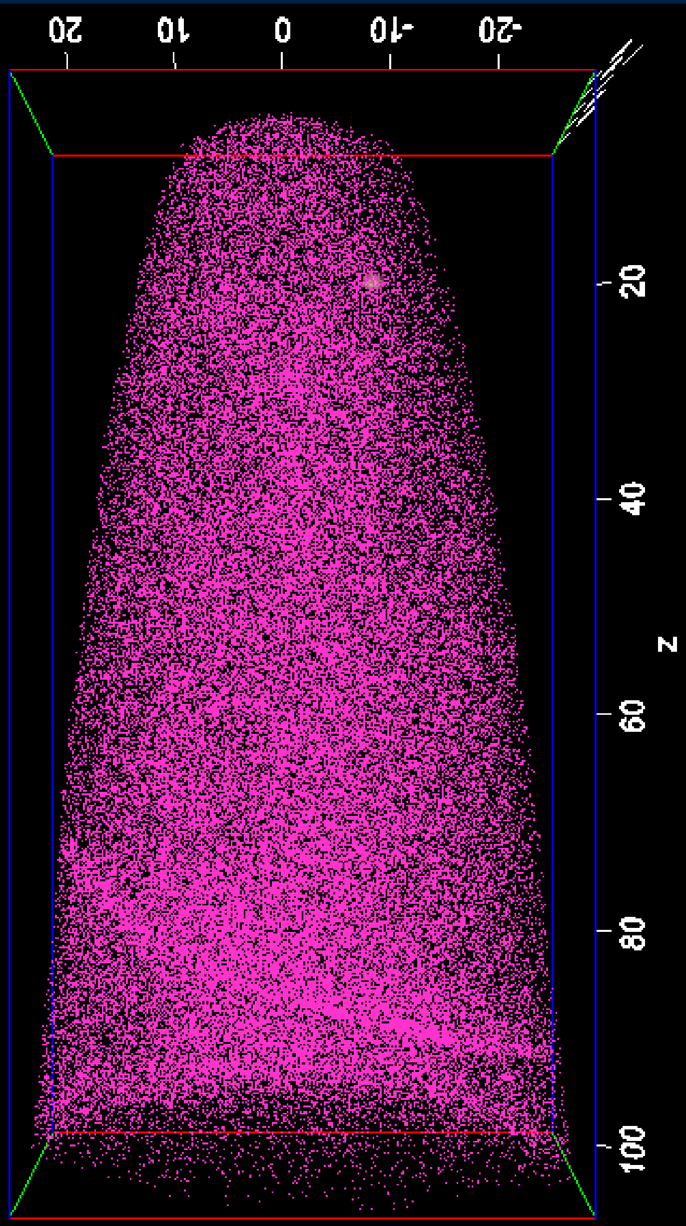
Radiation swelling of 316 stainless steel [4]

[3] S.J. Zinkle, L.L. Snead, Annu. Rev. Mater. Res. 44 (2014) 241–67.
[4] Mansur, L. K., (1994). *J. Nucl. Mater., 216, 97–123*

T91: 8.8 dpa; 370C – Alpha prime? R33_09813

Cr Si Ni Mn Cu P





28/02/2019

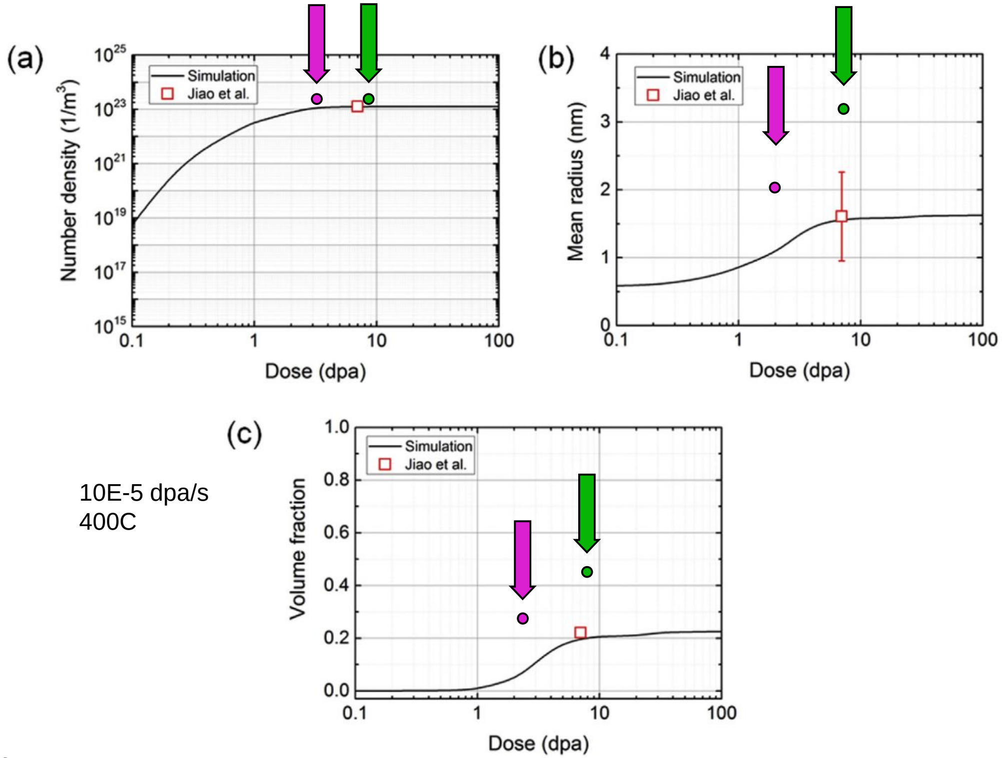
Mn-Ni-Si cluster evolution vs dose

Mn-Si-Ni ppts in T91 controlled by G-phase thermodynamics, RIS, dislocation enhanced heterogeneous nucleation sites. Model produced by using CALPHAD, Lattice monte carlo and cluster dynamics.

2.2 dpa 320C neutron

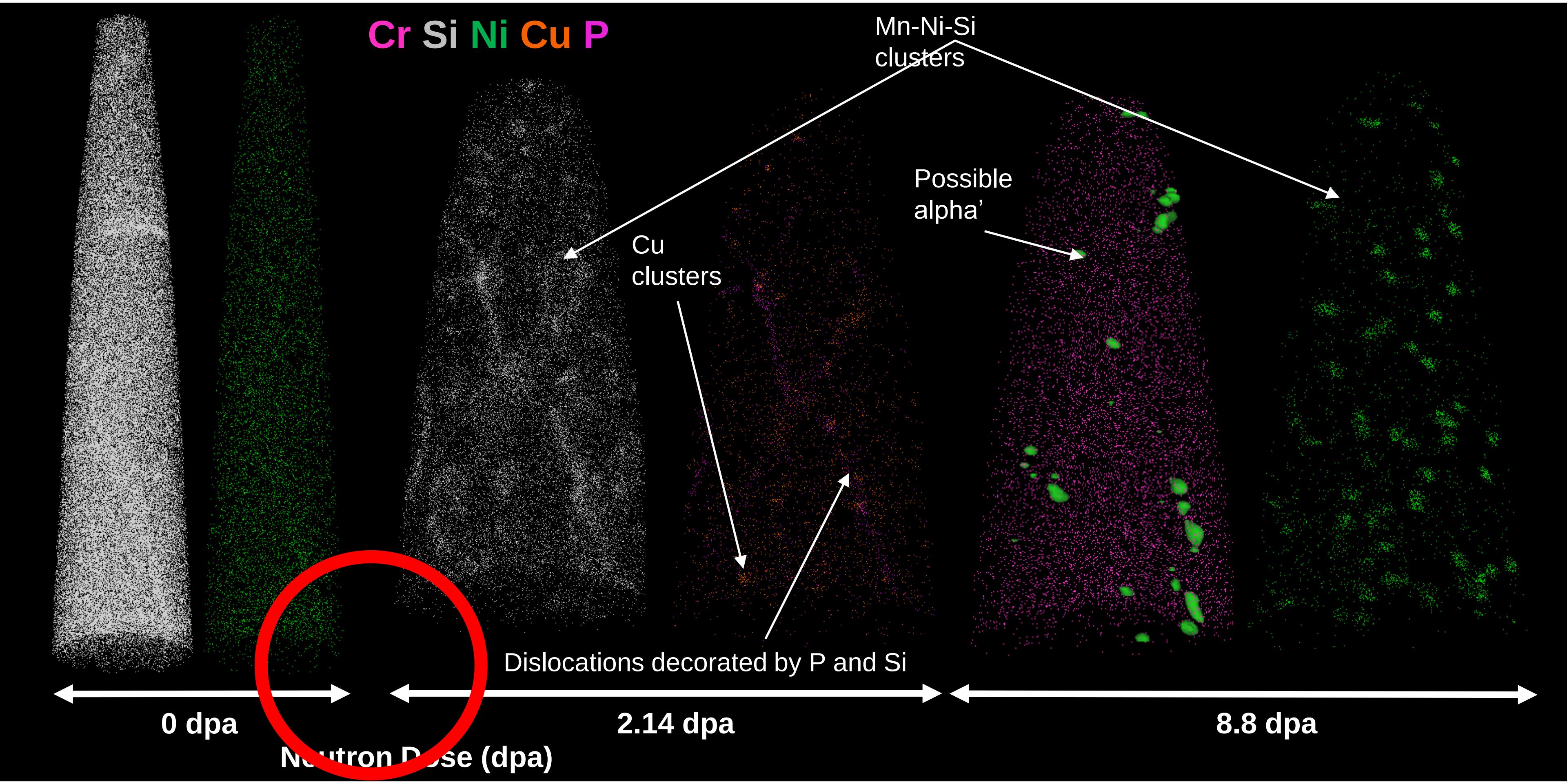
8.8 dpa 370C neutron

10E-5 dpa/s
400C



J.-H. Ke, H. Ke, G. R. Odette, and D. Morgan, ‘Cluster dynamics modeling of Mn-Ni-Si precipitates in ferritic-martensitic steel under irradiation’, *Journal of Nuclear Materials*, vol. 498, pp. 83–88, Jan. 2018, doi: 10.1016/j.jnucmat.2017.10.008.

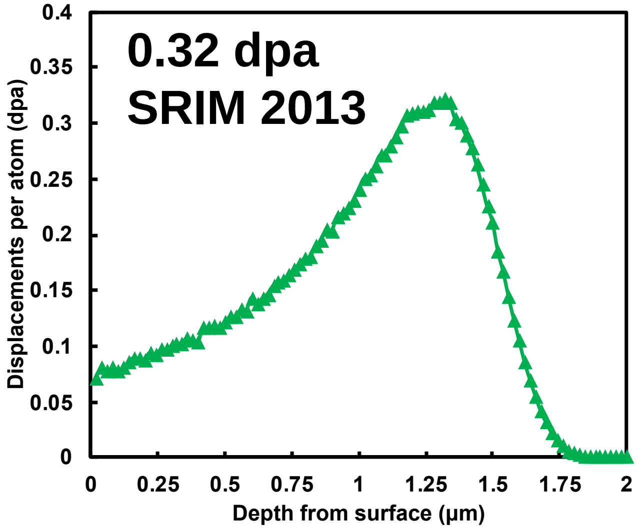
Microstructure Evolution of Irradiated T91 Steel

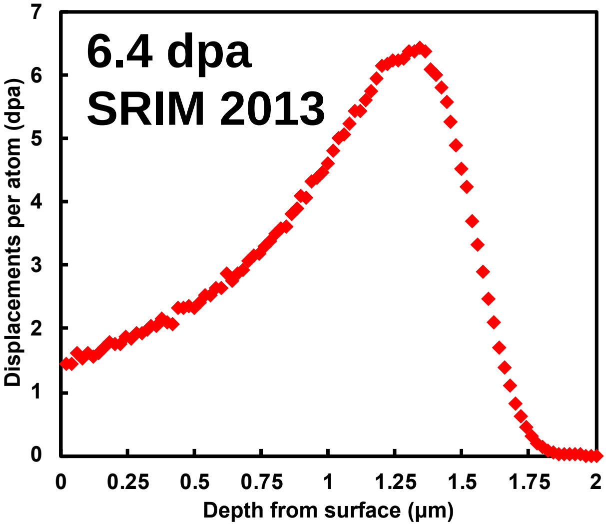


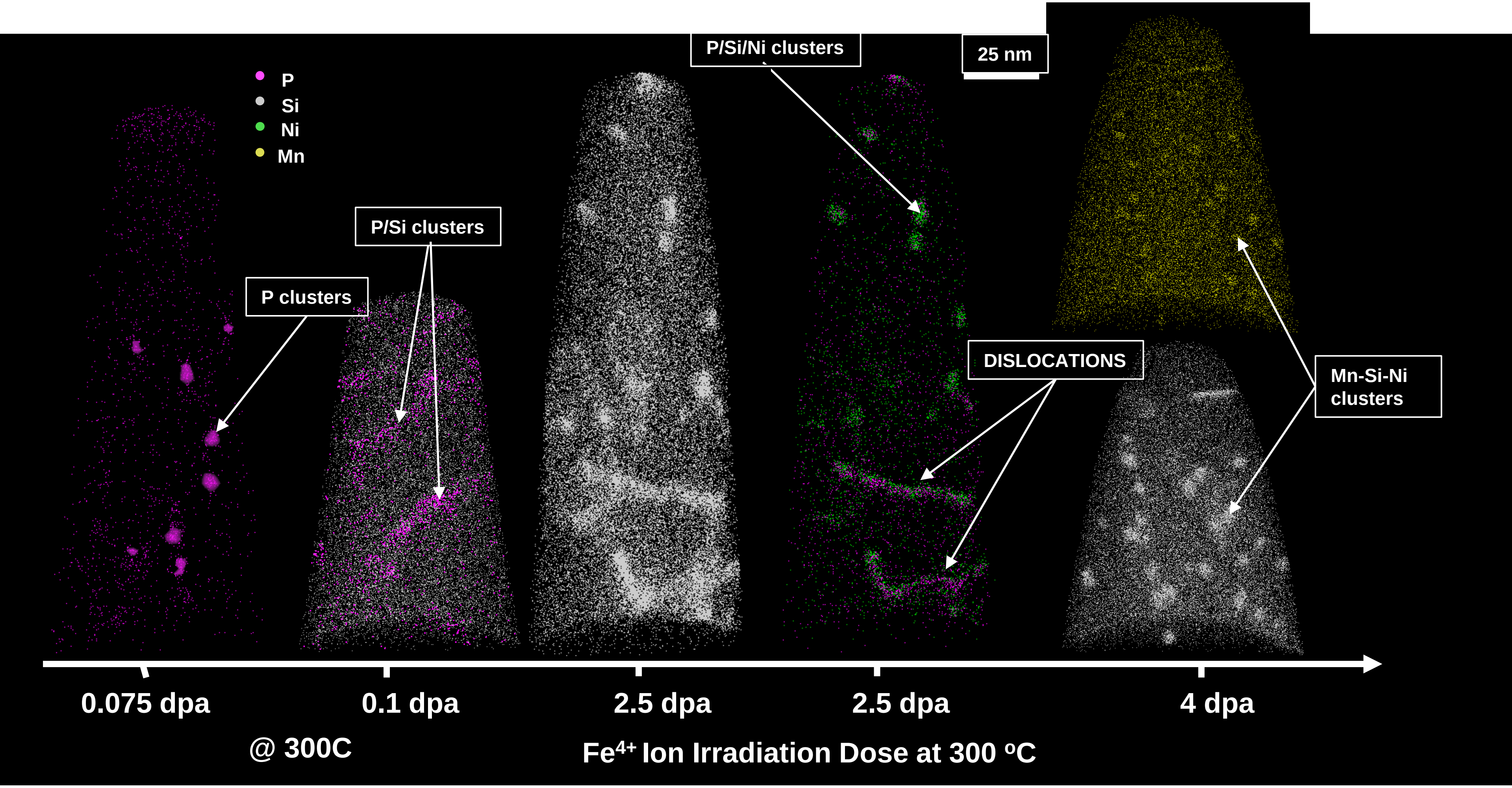
Ion Irradiation between 0.1 to 4.0 dpa

The University of Manchester
Dalton Nuclear Institute

Steel	Temp (°C)	Fe^{4+} Dose at Bragg Peak (dpa)	Flux (ions·cm^{-2}·s^{-1})	Fluence (ions·cm^{-2}·s^{-1})
T91	300	0.32	4.8×10^{11}	3.5×10^{14}
		6.4	4.4×10^{11}	6.5×10^{15}

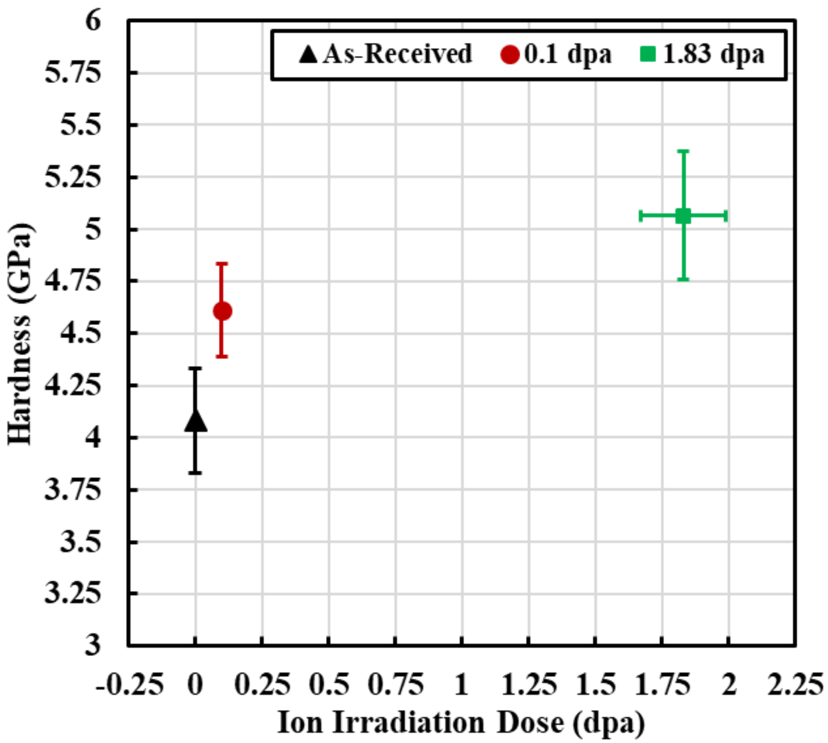






P
Si
Ni
Mn
P clusters
P/Si clusters
P/Si/Ni clusters
25 nm
DISLOCATIONS
Mn-Si-Ni clusters
0.075 dpa
0.1 dpa
2.5 dpa
2.5 dpa
4 dpa
@ 300C
Fe4+ Ion Irradiation Dose at 300 ºC

Nanohardness and Mn-Ni-Si clusters



- Mn-Ni-Si clusters
- To do