Investigating the nanoscale segregation and characterisation of generation IV fission reactor core structural steels

Thomas P. Davis

PhD Researcher (2016-2020)
Clarendon Scholar
Department of Materials
University of Oxford

Supervisors: Prof. D.E.J. Armstrong, Prof. M.P. Moody,
Dr P.A.J. Bagot, Dr M.A. Auger

Email: thomas.davis@materials.ox.ac.uk

26/02/2019 – 2nd Postgraduate Research Symposium of Ferrous Metallurgy

Outline of Talk

1. T91 Ferritic-martensitic and Oxide Dispersion Strengthened Steel
2. Characterisation of ODS Steels
3. Irradiation Techniques
4. Analysis of irradiated T91
5. Conclusions

Objectives of my PhD Project

1. Investigate the nano-oxide particle nature of oxide dispersion strengthened steel and how these particles influence the radiation resistance and effect the mechanical properties.

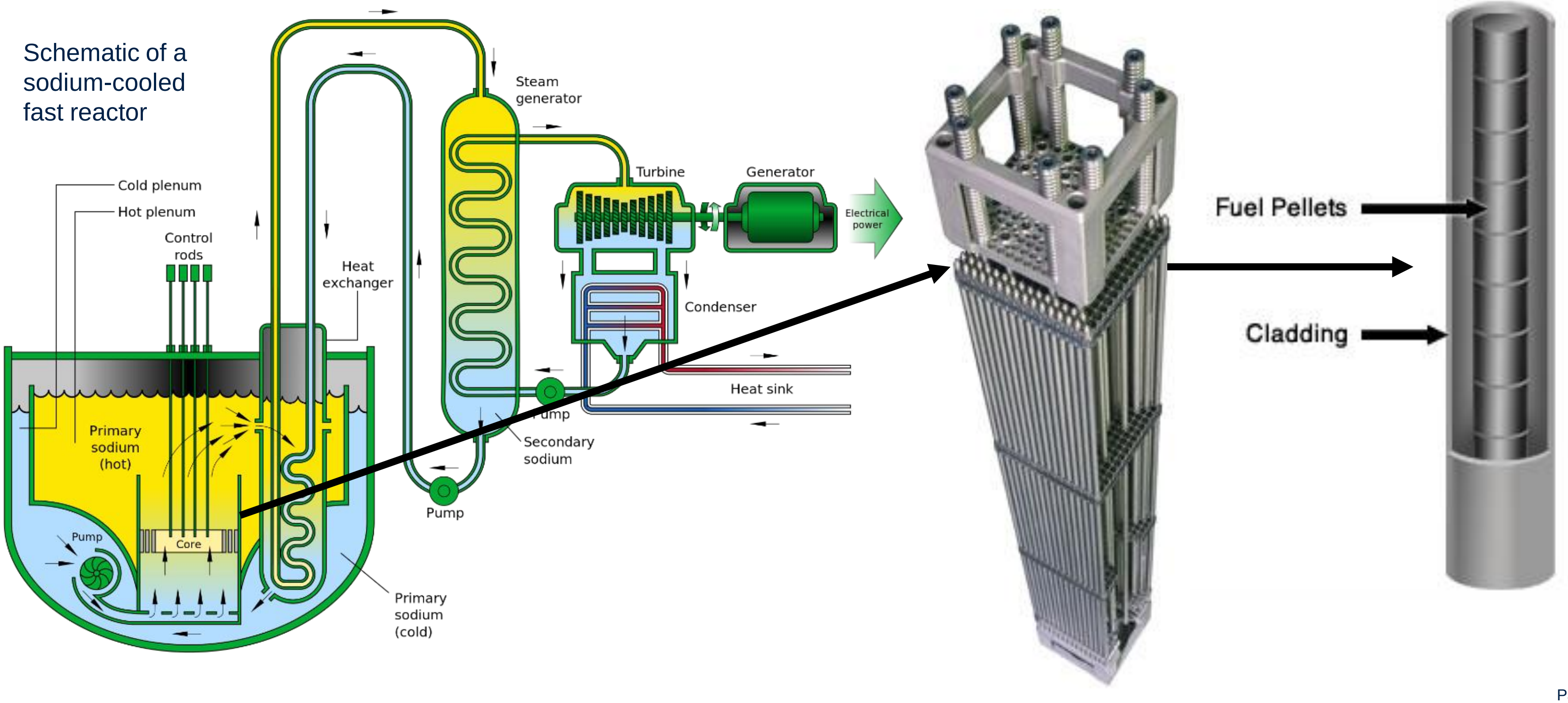
2. Investigate the induced chemical segregation from neutron and ion irradiation of Si, Ni, Mn, and Cr in T91 steel at low temperatures (100 to 300C)

1. Ferritic-Martensitic T91 and Oxide Dispersion Strengthened (ODS) Steel

What are these materials and why are they important to the nuclear industry?

Advanced nuclear fuel cladding

- T91 is the fuel cladding and duct structural material for sodium-cooled fast fission reactors
- ODS steels are the potential future fuel cladding material for these reactors
    - Withstand greater radiation damage > fuel can stay in the reactor for longer
    - Increases the structural integrity safety margin



Schematic of a sodium-cooled fast reactor

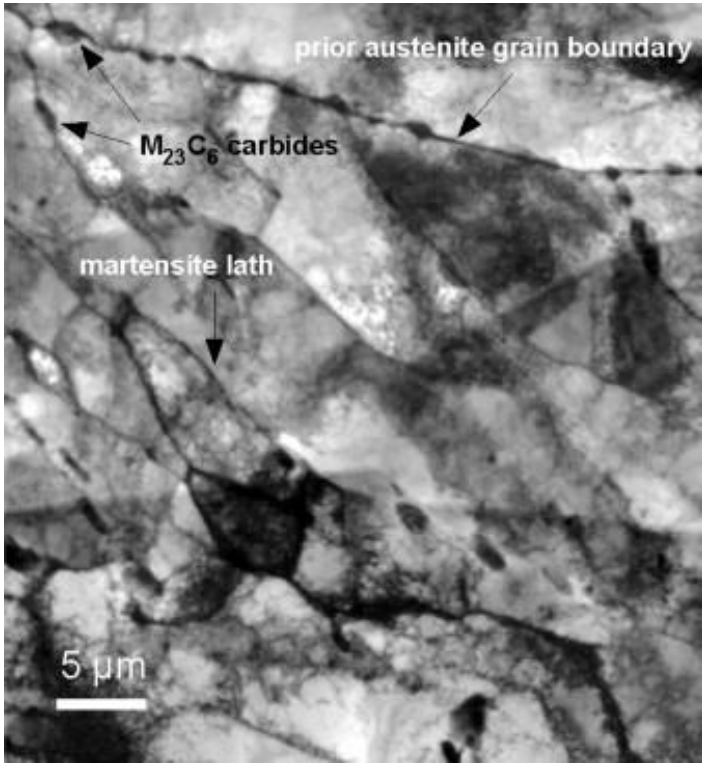
Fuel Assembly

Fuel Rod

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

T91 Steel

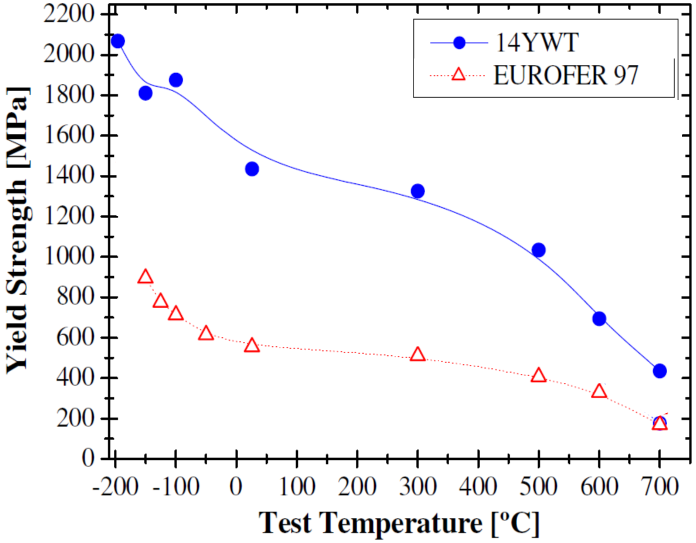
- 9% Cr - 1Mo% steel, tempered, ferritic martensitic microstructure
- Used in Fast Flux Test Reactor (USA) as test cladding and proposed in multiple future sodium-cooled fast reactors as the cladding and duct material.



C. Keller et al., Materials Science and Engineering A, 527, 24-25, p 6758-6764

ODS Steel

- Ferritic 9-14Cr, 3W, 0.25Ti steels with dispersed Y_2O_3 before consolidation, ferritic microstructure
- Reaction between Ti and Y_2O_3 and forms stable nano-sized (2-10nm) $Y_2Ti_2O_7$ oxide particles (generally). Typical features of these nano-oxides are:
  - Radiation recombination zones
  - Thermodynamically very stable
  - Dislocation motion barrier



D.A. McClintock et al. Journal of Nuclear Materials, Volume 392, Issue 2, 2009, 353-359

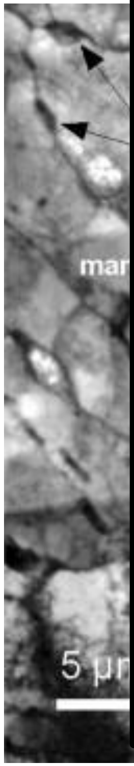
T9

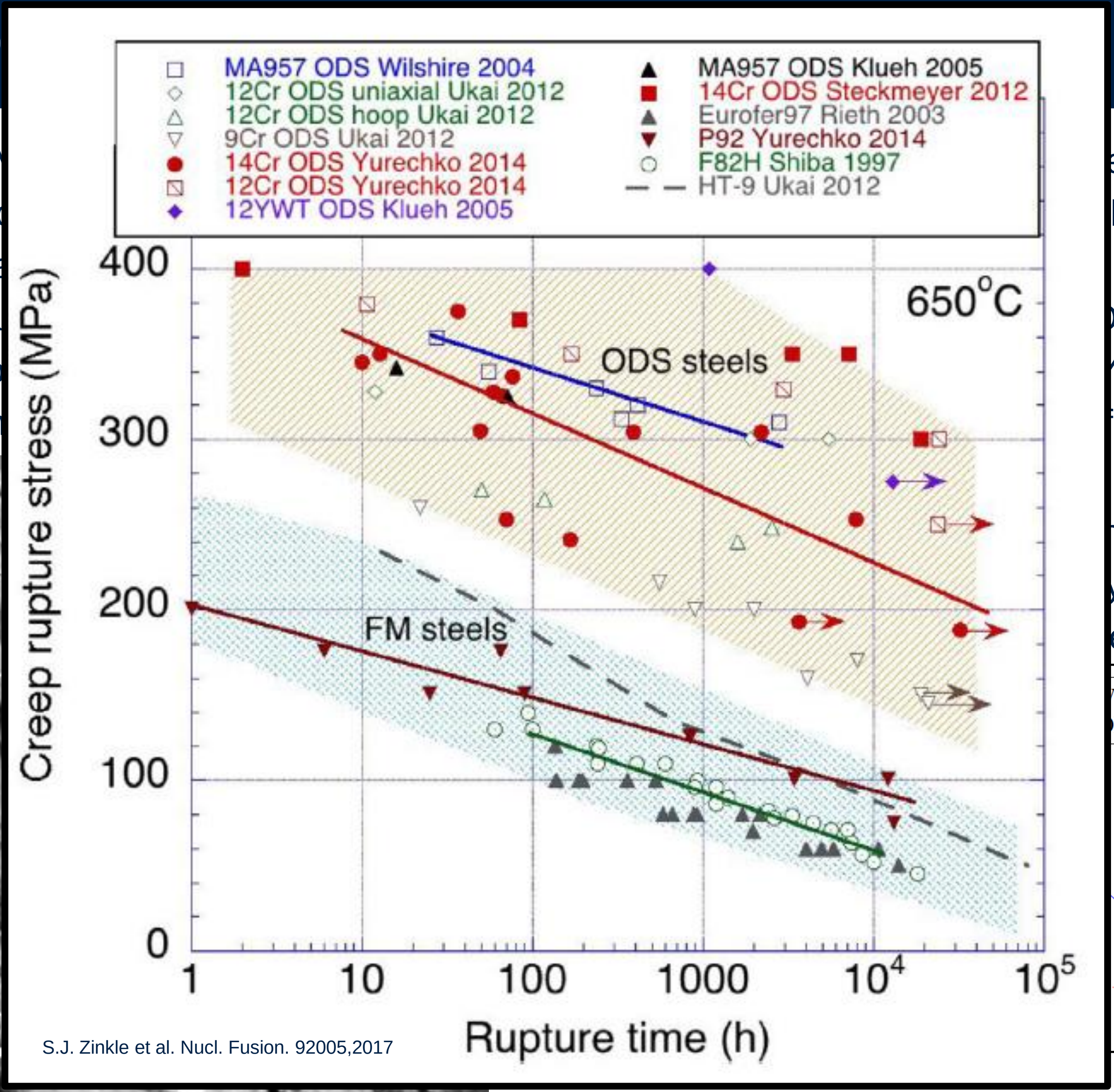
- 9% Cr - 1M eels with martensitic lidation, ferritic
- Used in Fa cladding a O_3 and forms sodium-co $Y_2Ti_2O_7$ oxide and duct m features of these

n zones

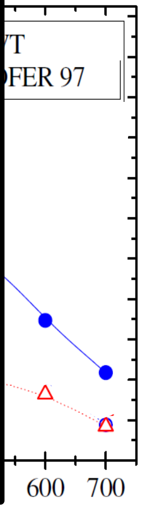
y stable

er





S.J. Zinkle et al. Nucl. Fusion. 92005,2017



C. Keller et al., Materials Science and Engineering A, 527, 24-25, p 6758-6764

D.A. McClintock et al. Journal of Nuclear Materials, Volume 392, Issue 2, 2009, 353-359

2. Characterisation of ODS Steel

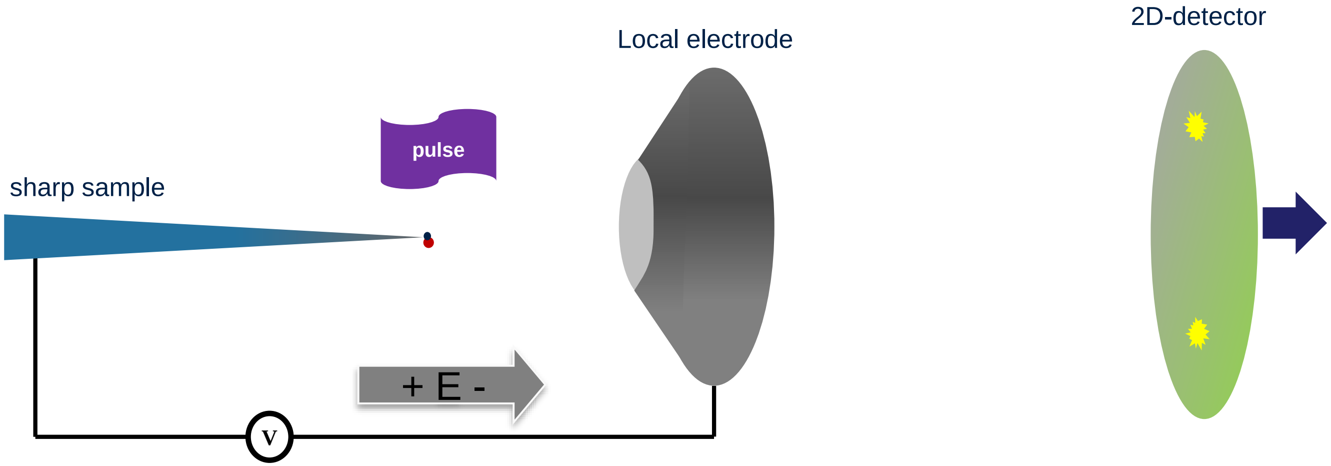
T91 is well characterised in the literature

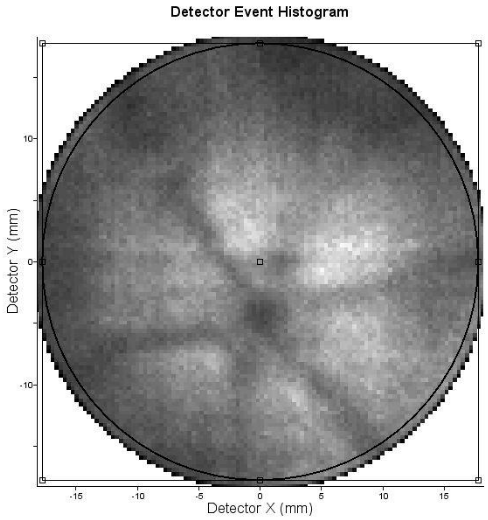
ODS steel's characterisation: nano-oxide particle distribution, grain structure and nano-hardness

First: How does Atom probe work?

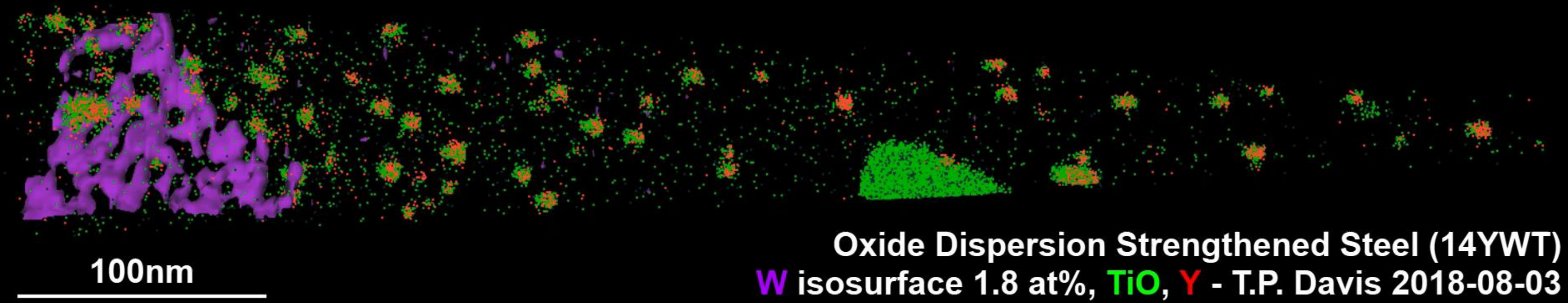
- Sharp needle sample; tip radius 50-100nm; 50K temp; high vacuum
- Apply an electric field (2000-10,000V); the field is concentrated at the sharp tip to ~40 V/nm.
- Produces field evaporation at the tip of the needle when a short additional **voltage or laser pulse** is applied.
- If we know when the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

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





Atom Probe of As-Received ODS Steel 14YWT

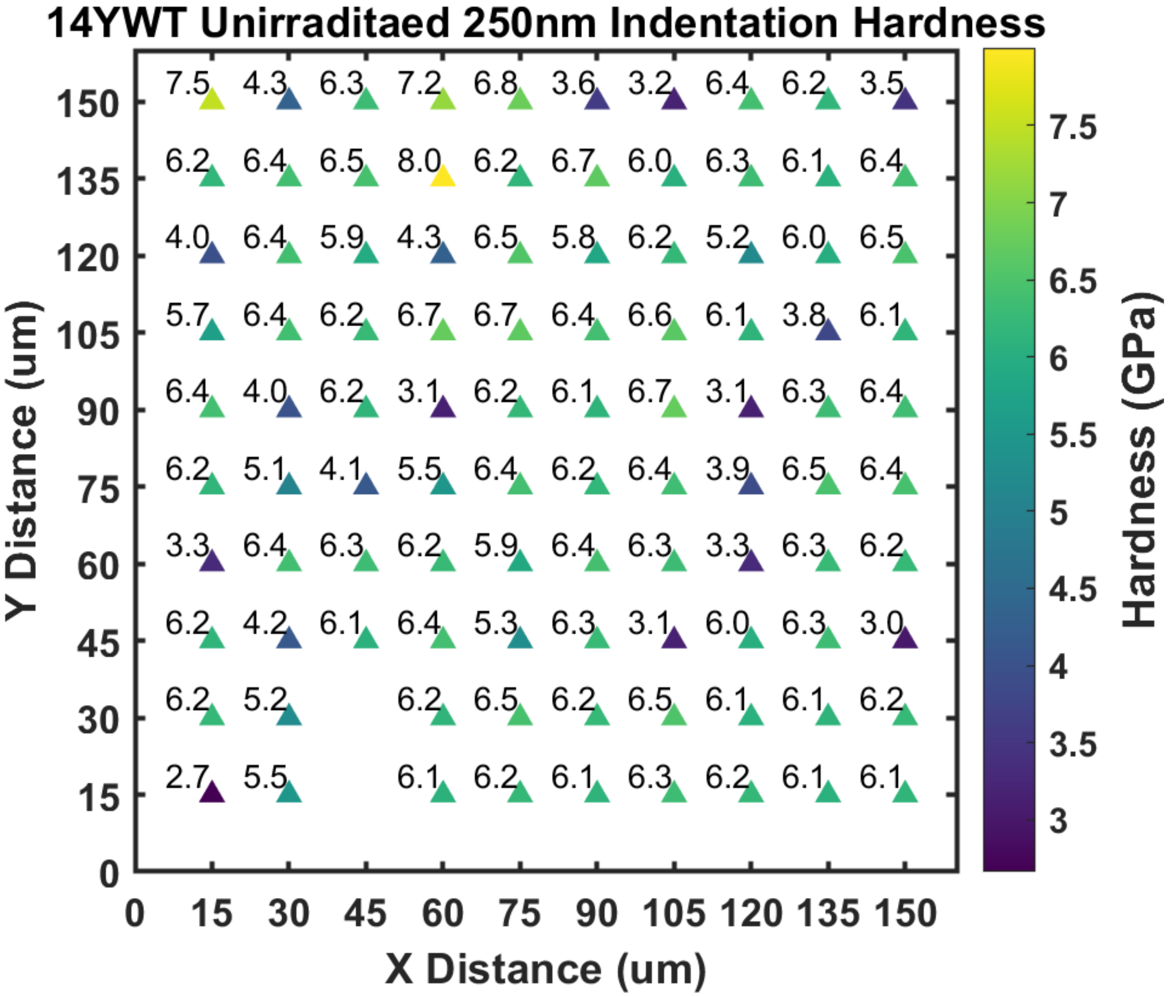


Oxide Dispersion Strengthened Steel (14YWT)
W isosurface 1.8 at%, TiO, Y - T.P. Davis 2018-08-03

Unirradiated ODS 14YWT nano-hardness

What are the controlling factors to the mechanical properties?
- Grain structure
- Y-Ti-O particle distribution
- Carbides

Nanoindentation
- 250nm indents
- 10 by 10 grid, 15 µm spacing
- Berkovich Indenter
- G200 nanoindentor used

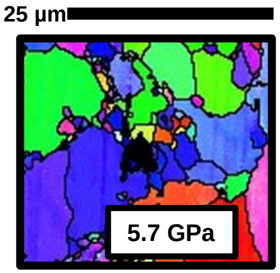


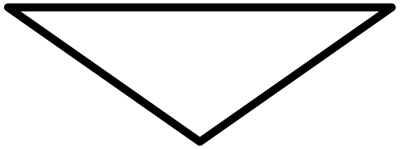
	15	30	45	60	75	90	105	120	135	150
150	7.5	4.3	6.3	7.2	6.8	3.6	3.2	6.4	6.2	3.5
135	6.2	6.4	6.5	8.0	6.2	6.7	6.0	6.3	6.1	6.4
120	4.0	6.4	5.9	4.3	6.5	5.8	6.2	5.2	6.0	6.5
105	5.7	6.4	6.2	6.7	6.7	6.4	6.6	6.1	3.8	6.1
90	6.4	4.0	6.2	3.1	6.2	6.1	6.7	3.1	6.3	6.4
75	6.2	5.1	4.1	5.5	6.4	6.2	6.4	3.9	6.5	6.4
60	3.3	6.4	6.3	6.2	5.9	6.4	6.3	3.3	6.3	6.2
45	6.2	4.2	6.1	6.4	5.3	6.3	3.1	6.0	6.3	3.0
30	6.2	5.2		6.2	6.5	6.2	6.5	6.1	6.1	6.2
15	2.7	5.5		6.1	6.2	6.1	6.3	6.2	6.1	6.1

Y-Ti-O particle distribution effect on hardness

High hardness (5.7 GPa) indent

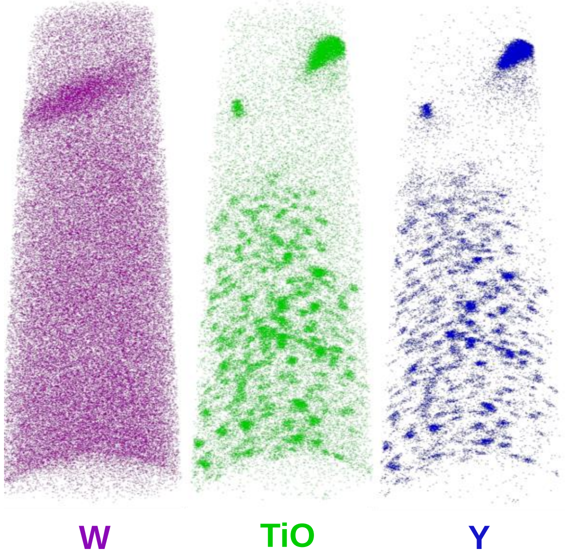
Y-Ti-O distribution: 2.77nm, 3.6×10^{24} clusters per m^3





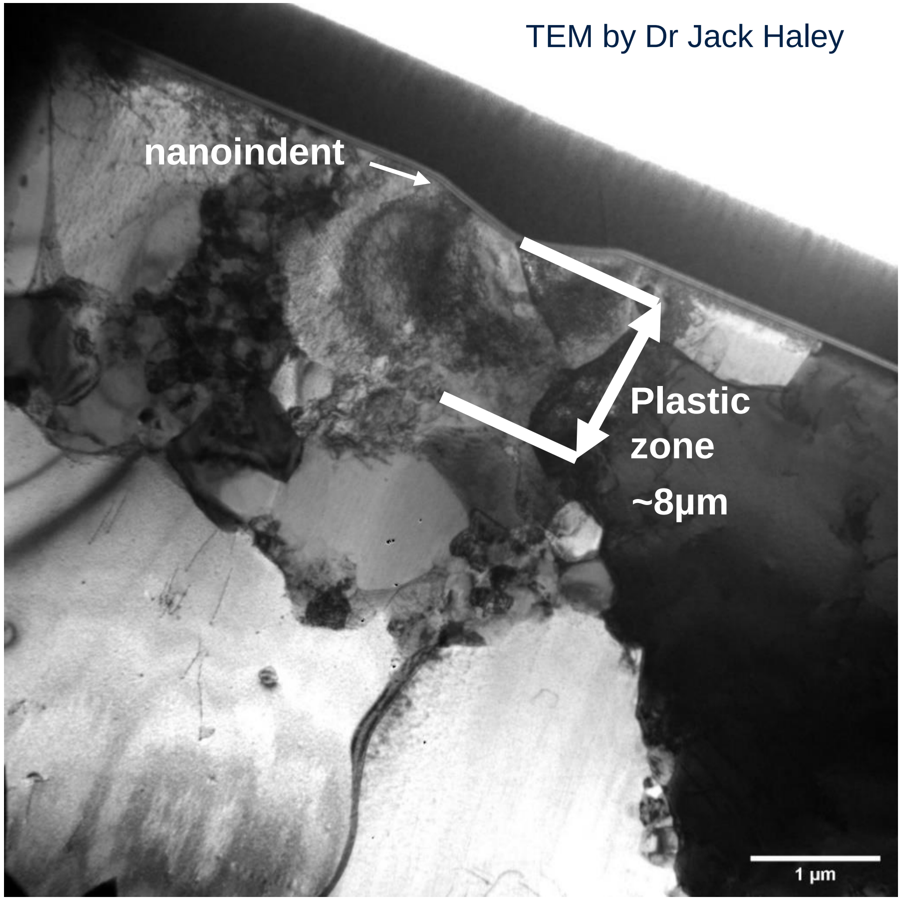
250nm indent

Lost material due to FIB milling ~50-100nm



500nm

Plastic zone to 1µm depth for 50-100 nm depth

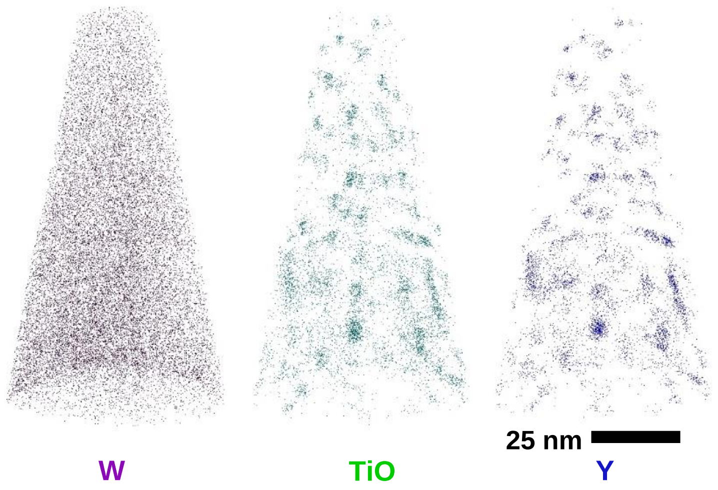


TEM by Dr Jack Haley

Similar hardness, different indent

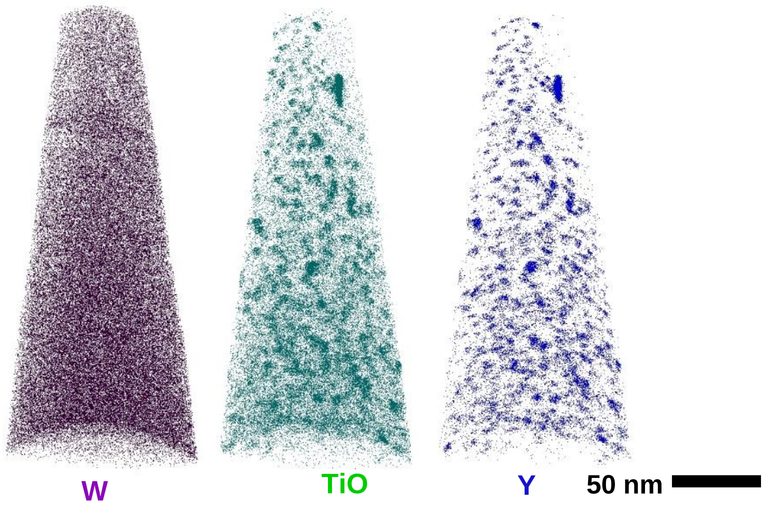
Y-Ti-O particle distribution effect on hardness

- Another high hardness (6.4 GPa) indent shows little Y-Ti-O particle distribution



Y-Ti-O distribution: 2.61nm, $1.9X10^{24}$ clusters per m^3

- Low hardness indent (4.0 GPa) has significant amount of Y-Ti-O particle distribution



Y-Ti-O distribution: 2.47nm, $5.62X10^{24}$ clusters per m^3

Conclusion: Y-Ti-O nano-oxide appears might not <u>directly</u> impact the hardness of ODS materials, as previously believed. Further examination is ongoing (TEM).

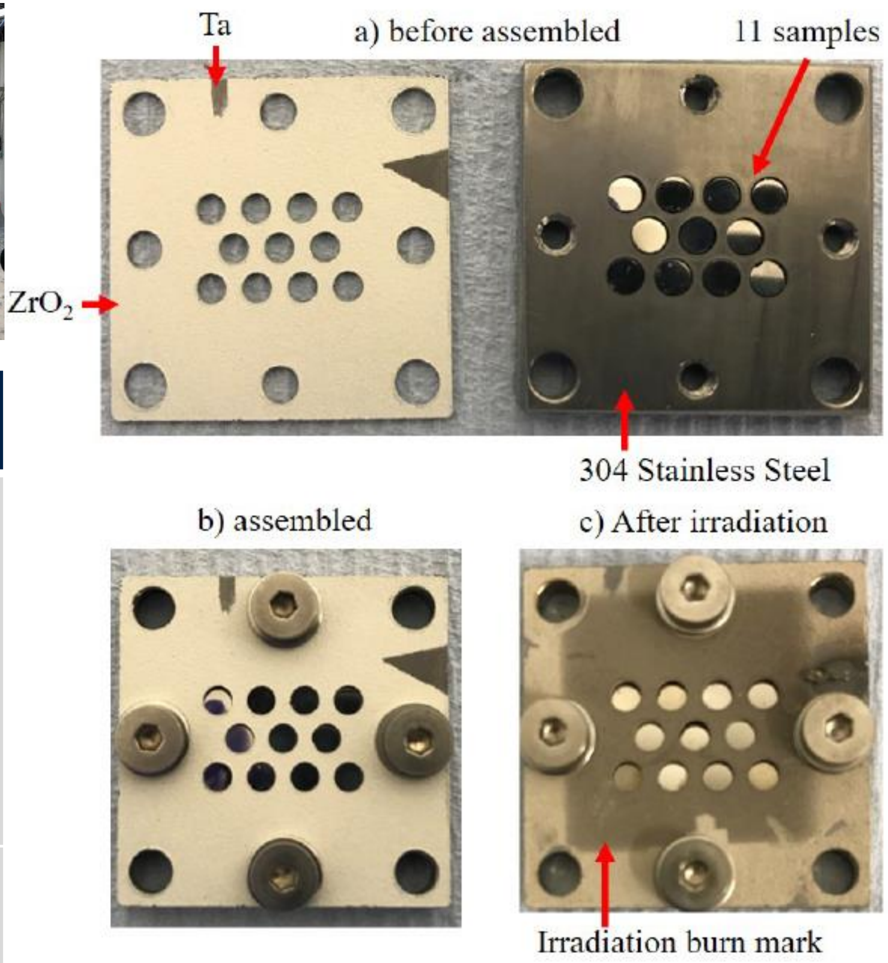
UNIVERSITY OF
OXFORD

3. Neutron and Ion Irradiation of both T91 and ODS steel

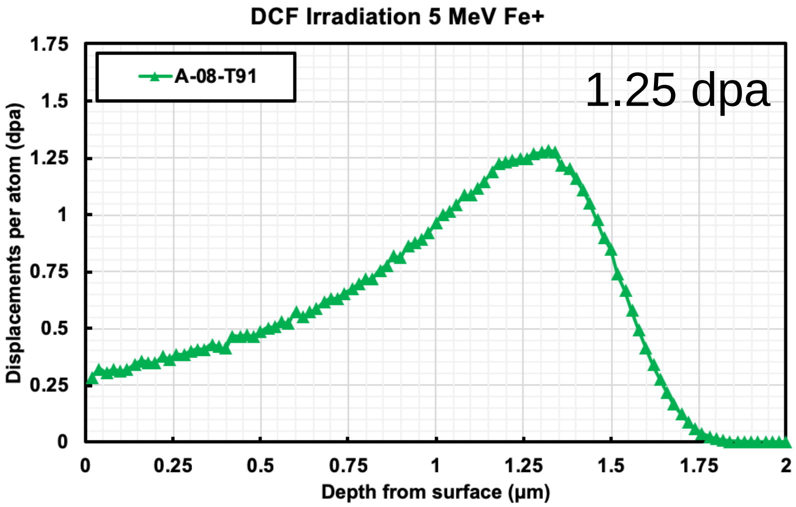
Ion Irradiation: Dalton Cumbrian Facility

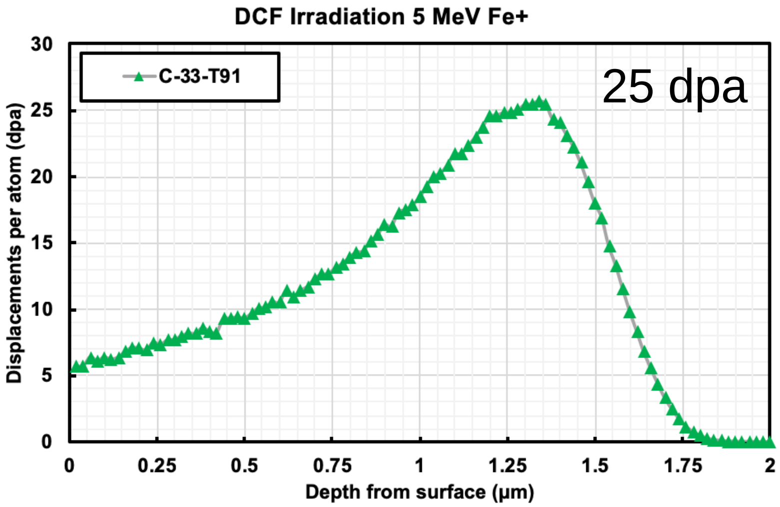
- Fe 4+ ions are used to simulate neutron damage
- Experiments conducted over 1 week in April 2018

Material	Dose (dpa)	Dose rate (dpa/s)	Temp C
T91	0.41 (x2)	1.0E-5	301
	0.39 (x4)	5.6E-4	311
	7.33 (x4)	5.0E-4	300
	0.39 (x4)	4.4E-4	111
	7.33 (x4)	5.4E-4	105
	0.2 (x1)(surrey)	9.0E-6	290
	0.2 (x1)(surrey)	1.0E-5	290
	1.6 (x1)(surrey)	1.0E-6	290
14YWT (HIP)	0.42 (x1)	1.0E-5	301
	0.41 (x2)	5.8E-4	311
	7.56 (x2)	5.1E-4	300
	0.40 (x2)	4.6E-4	111
	7.56 (x2)	5.6E-4	105



Damage Profiles in T91 DCF Irradiation 5 MeV





Conditions: 100C and 300C
Flux slow: 1x10^{-5} dpa/s (11.2 hours)
Flux fast: 5.8X10^{-4} dpa/s (11.6 mins)

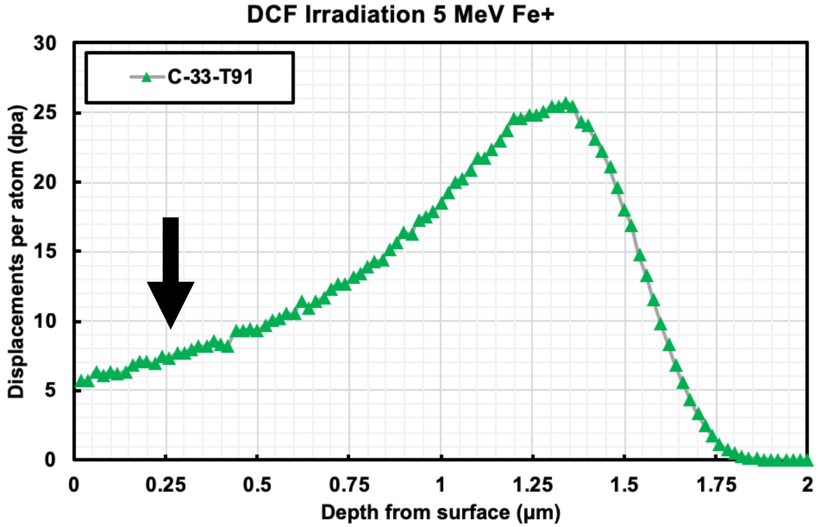
Conditions: 100C and 300C
~~Flux slow:~~
Flux fast: 5.0X10^{-4} dpa/s (4.1 hours)

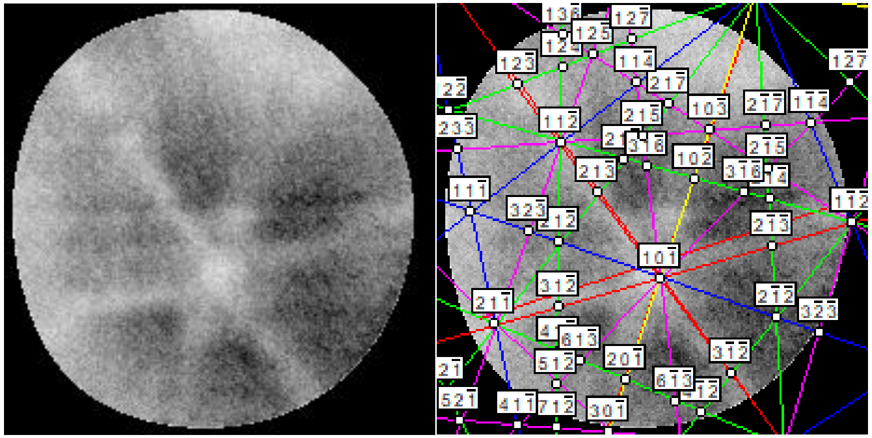
4. Analysis of ion irradiated T91 steel

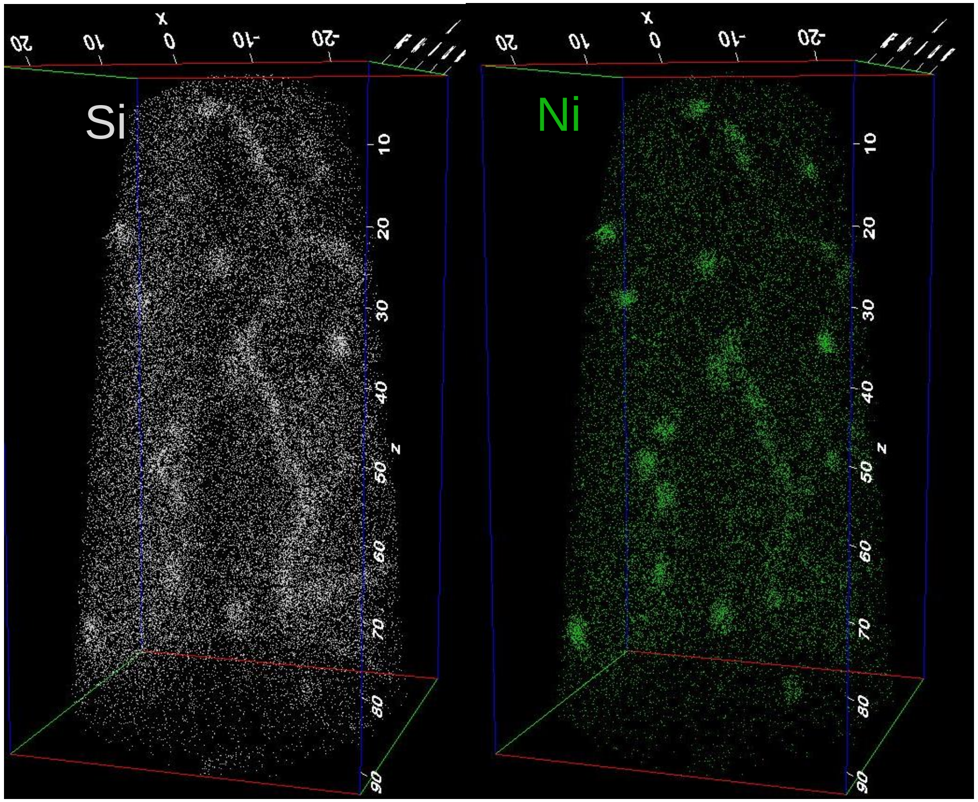
T91: Analysis of the segregation of Mn, Si, Ni G-phases and Cr at 300C

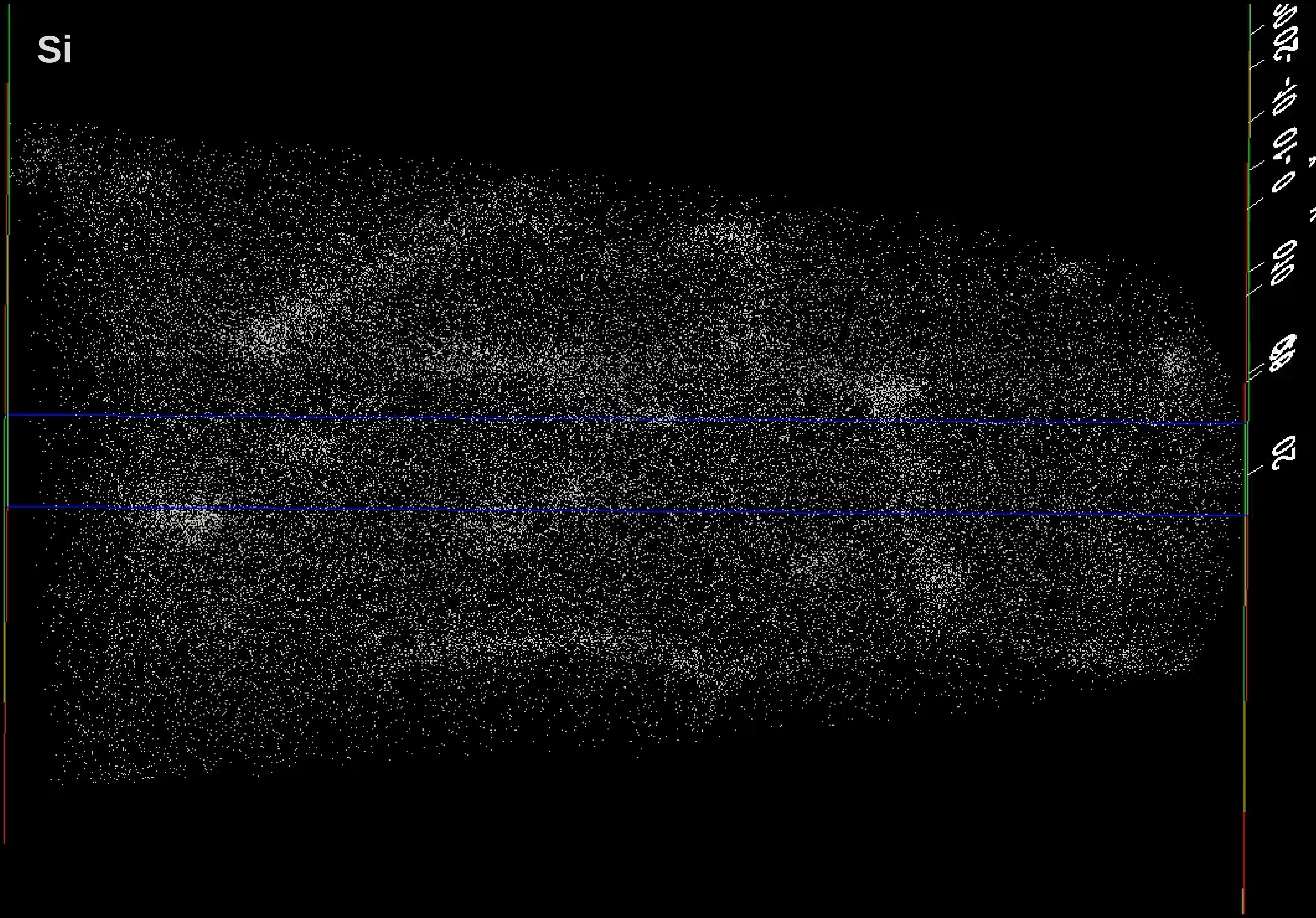
T91: 5-6 dpa Fe 4+, 300C, 6 hours irradiation

- 200 nm below surface

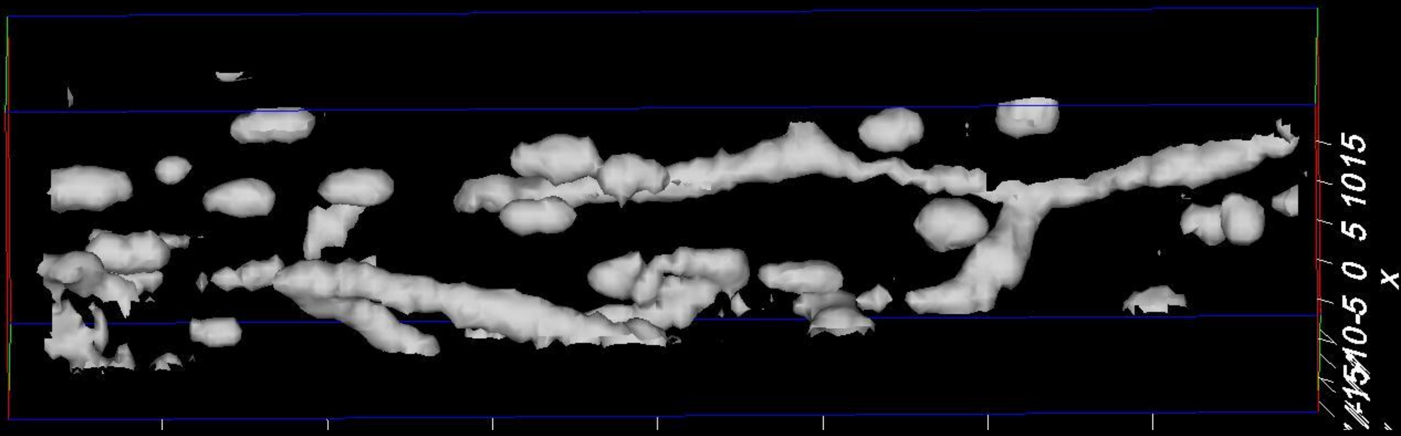


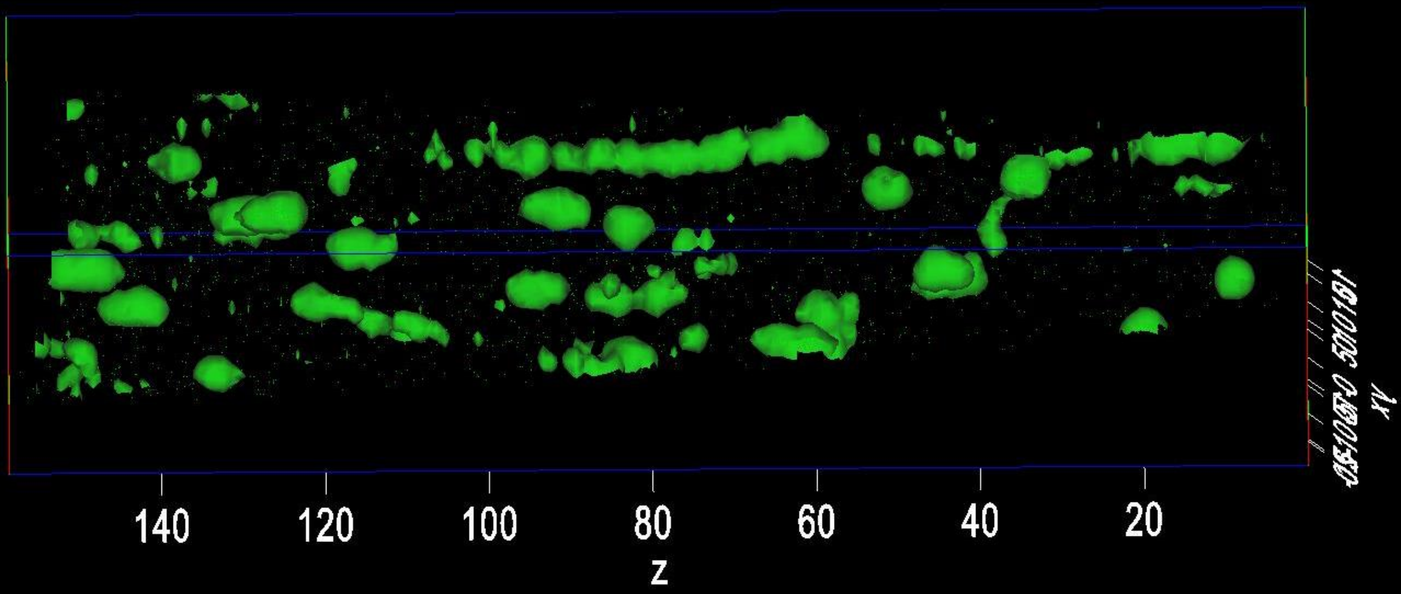




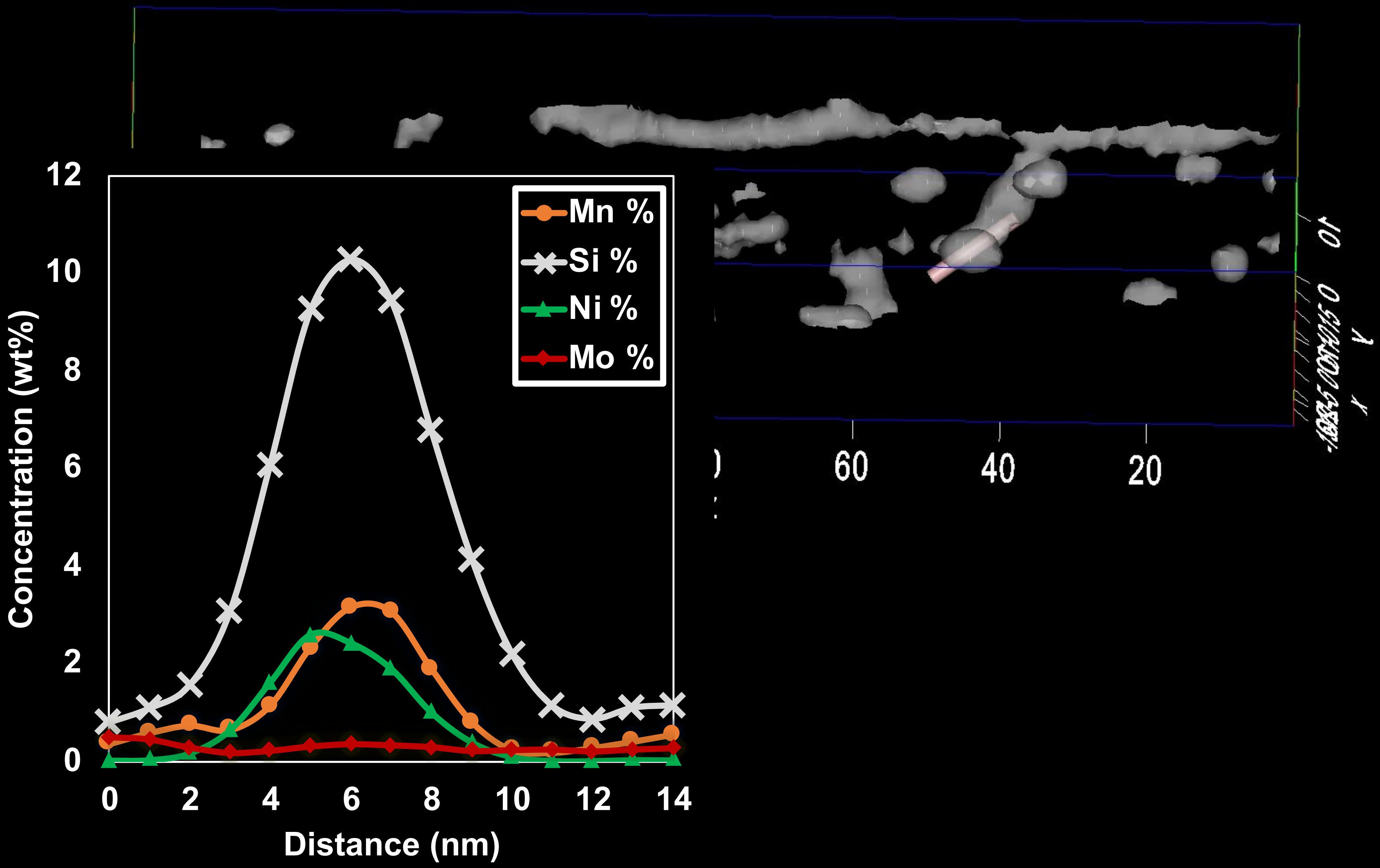

Si

Dislocations and G-Phase like diffusion


2.35 % Si isosurface

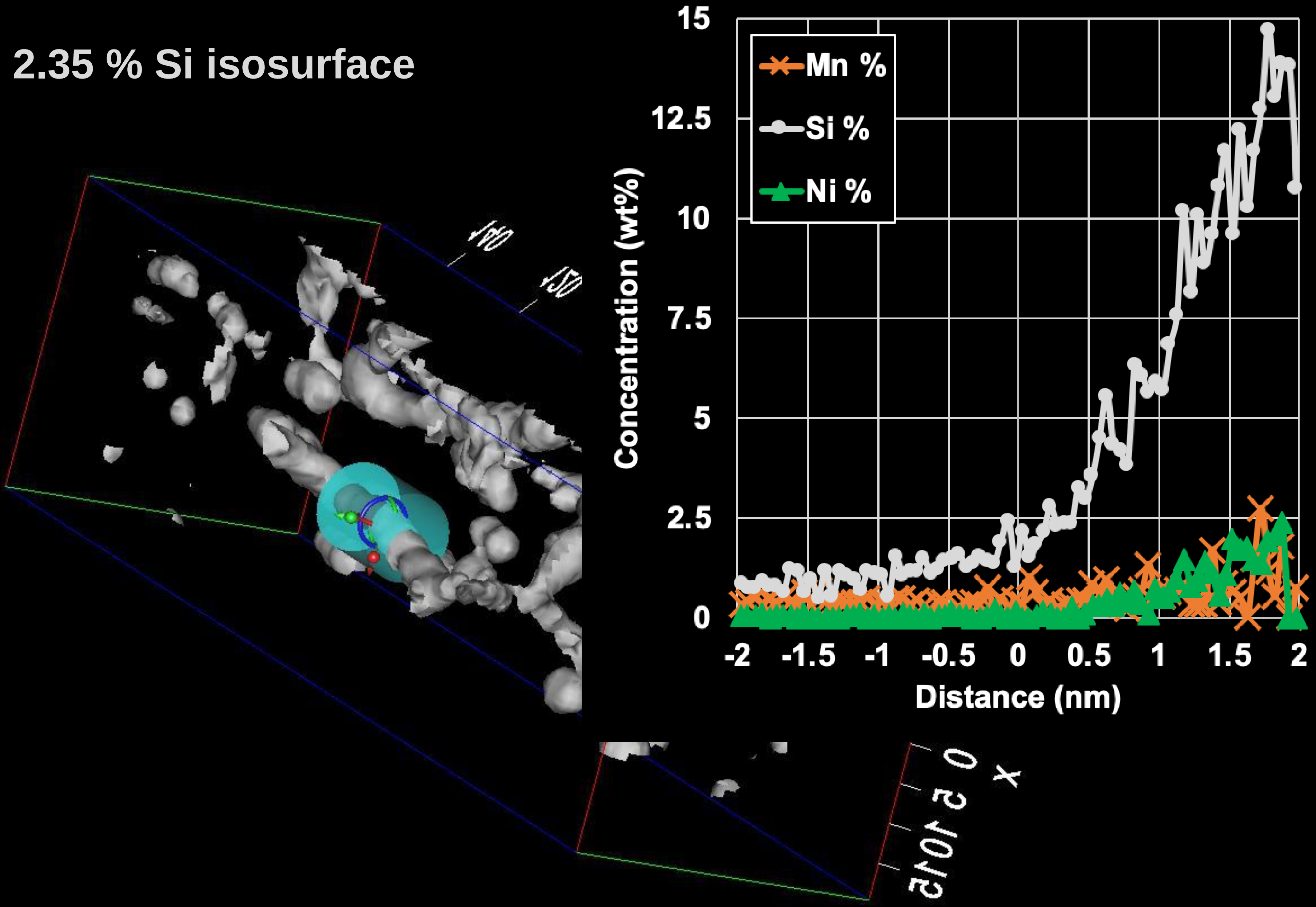

0.5% Ni isosurface

G-phase like segregation in T91 from Irradiation



Segregation of Si, Mn and Ni to Dislocations

2.35 % Si isosurface



UNIVERSITY OF
OXFORD

5. Conclusions & Future Work

Take home messages & Future Work

- Objectives are:

  1. Investigate the nano-oxide particle nature of oxide dispersion strengthened steel and how these particles affect the mechanical properties and radiation resistance.
  2. Investigate the chemical segregation of Si, Ni, Mn and Cr in T91 steel as an approach to validating ions as a surrogate for neutron irradiation at low temperatures.

- Atom probe tomography has provided an insight into the nature of these oxide-particles in ODS steels and chemical segregation in T91 (at low temperatures 100-300)

- Future:

  - Neutron irradiation is on going with MARIA reactor and visiting Idaho National Laboratory in summer
  - Analysis of more T91 ion irradiated (100C and dpa) before heading to Idaho for neutron comparisons
  - Analysis of ion irradiated ODS steel – how resistance are these alloys to radiation damage?

Investigating the nanoscale segregation and characterisation of generation IV fission reactor core structural steels

Thomas P. Davis

PhD Researcher (2016-2020)
Clarendon Scholar
Department of Materials
University of Oxford

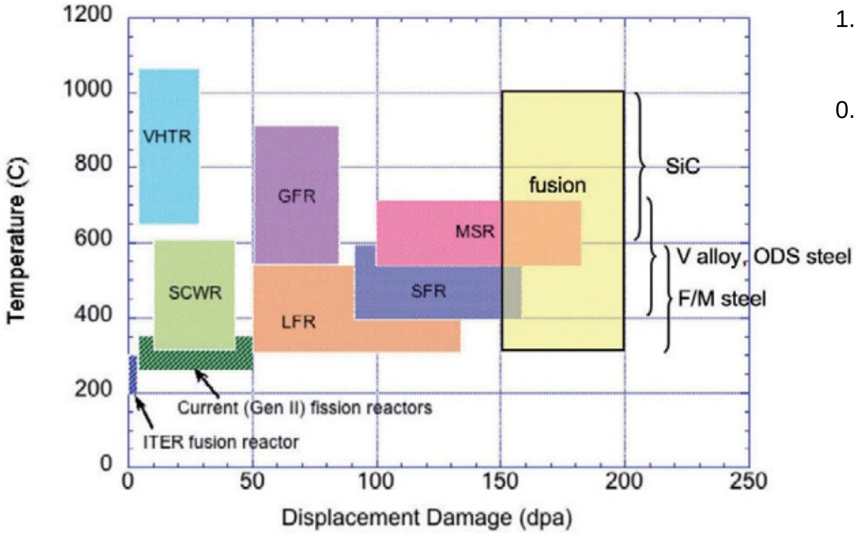
Supervisors: Prof. D.E.J. Armstrong, Prof. M.P. Moody,
Dr P.A.J. Bagot, Dr M.A. Auger

Email: thomas.davis@materials.ox.ac.uk

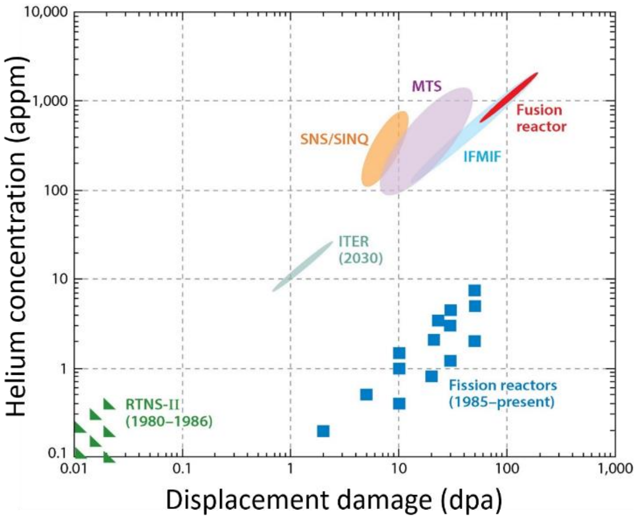
26/02/2019 – 2nd Postgraduate Research Symposium of Ferrous Metallurgy

Extra Slides

Challenge for structural materials



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

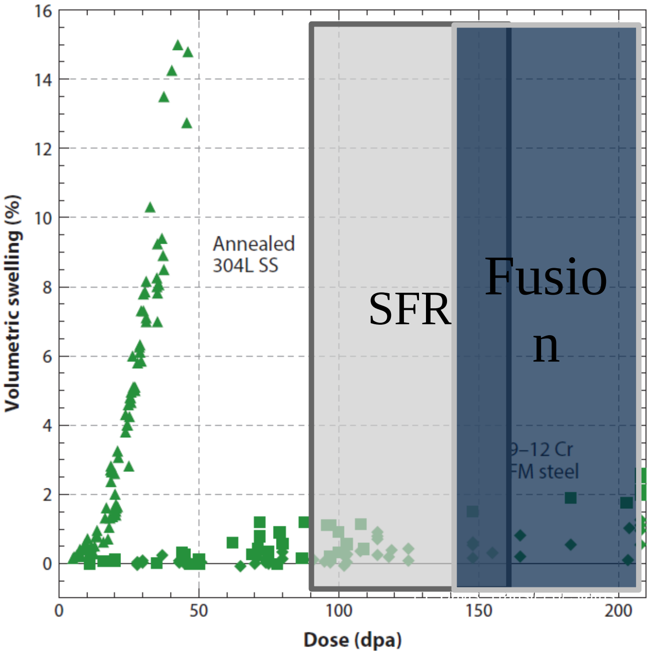


Helium production in advanced steels in fission, fusion and neutron experiments [2].

[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*

ODS Steel’s Heterogeneity Properties

ODS Steel manufactured at the University of Oxford

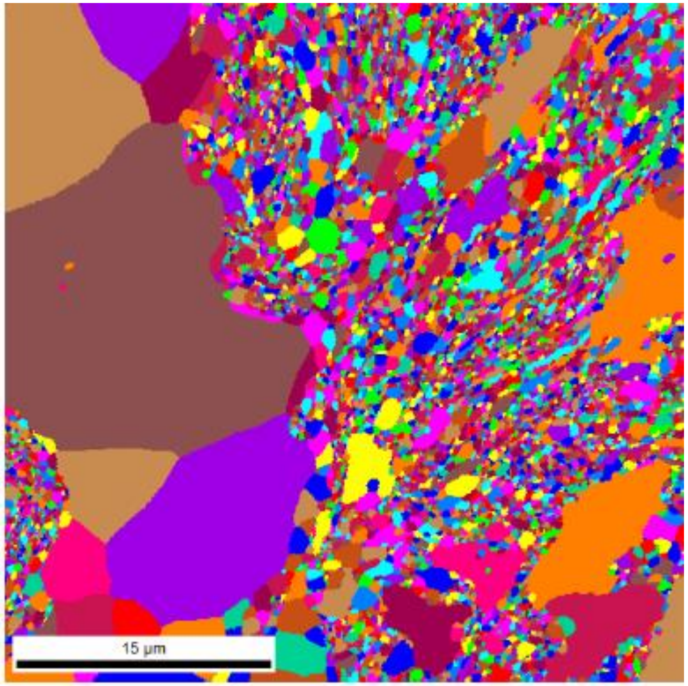
  - HIPed 1150ºC at 150 MPa for 4 hours
  - Fe-14Cr-3W-0.2Ti-0.25Y_2O_3 (named 14YWT)

Grain structure bimodal and micro-mechanical properties bimodal

- What is influencing the bimodal nature of these alloys?
  - Yttrium-oxide particle distribution
  - Inhomogeneous temperature during sintering
  - Inhomogeneous dislocation density distribution after mechanically milling

- Understanding why these alloys are bimodal will provide essential input to optimising the manufacturing process
- Once properties are predicable, then it can become a true engineering alloy for the nuclear industry

14YWT EBSD bimodal grain structure



C. Jones. 2017

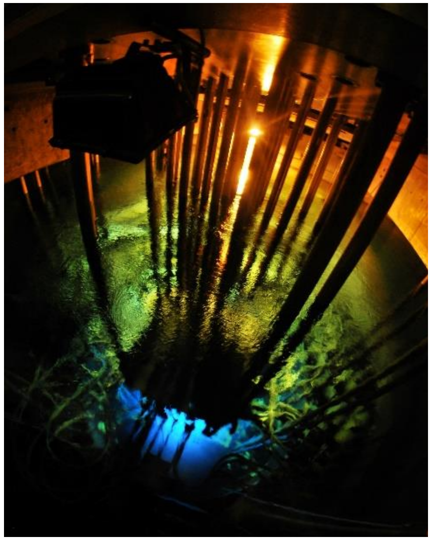
Neutron Irradiation: MARIA REACTOR

MARIA experimental nuclear reactor, Świerk, south-east of Warsaw, Poland

Pool type reactor 20-30MW

Water and Beryllium block moderated

Neutron flux: 1.0 × 10^{14} n/cm^{2}s (thermal)
1.0-1.5 × 10^{14} n/cm^{2}s (fast)

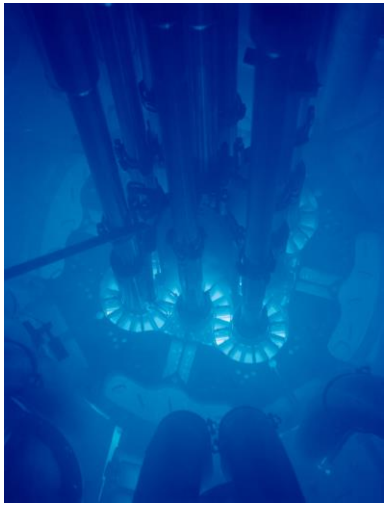


Due late 2019

	Radiation Dose (approximate) [dpa]			
Temperature	0.05	0.15	0.2	0.25
100C	Materials: 14WT 14YWT Fe-14Cr T91 316L			
200C				
300C				
400C				

Neutron Irradiation 2: Idaho National Laboratory

- Summer 2019 – 1 month at Idaho National Laboratory
- Neutron irradiated T91 steel
    - 4 – 10 dpa
    - 300 – 500C
    - Irradiated between 2000-2010 in the Advanced Test Reactor
- FIB, Atom probe and nanoindentation will be used
- Investigate the effect of temperature and flux on the segregation of elements in T91
- Collaboration with Professor Peter Hosemann at the University of California, Berkeley



NSUF Nuclear Science User Facilities

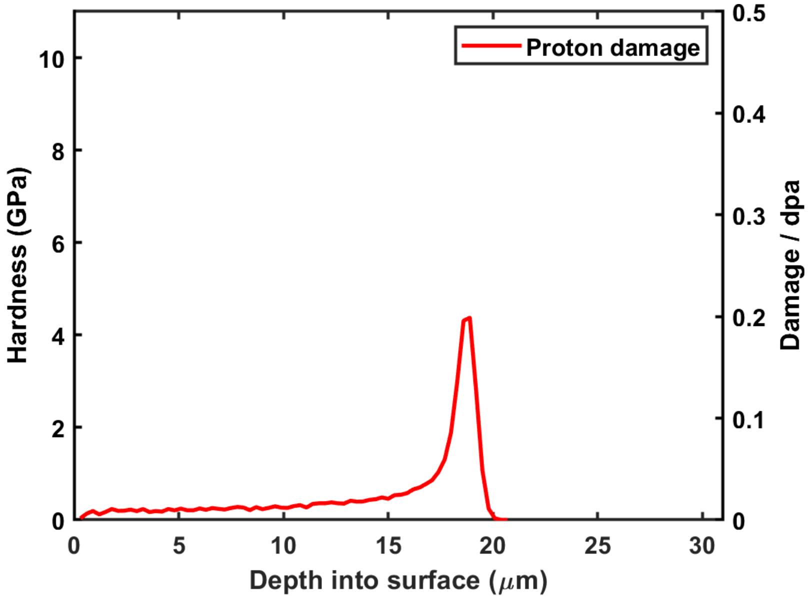
U.S. DEPARTMENT OF ENERGY | Nuclear Energy

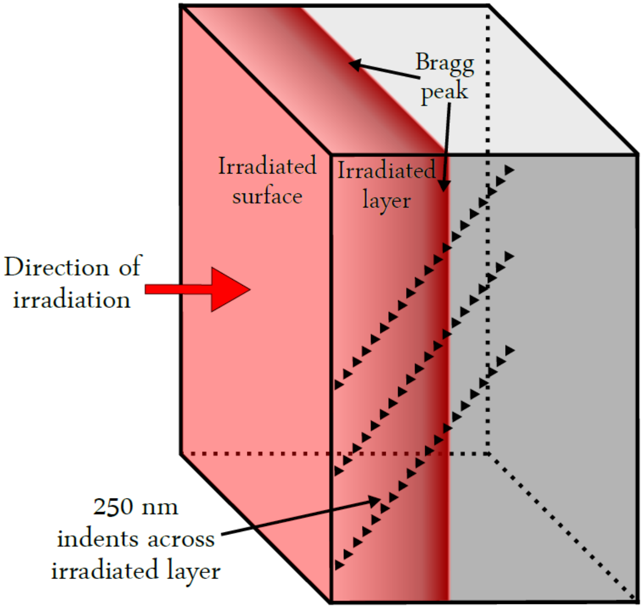
4. Analysis of proton irradiated ODS

ODS: how resistant is the material to radiation damage?

How resistant is ODS to irradiation damage?

- Proton irradiation to 0.2 dpa at 300ºC at the Ion Beam Centre, University of Surrey
- 14YWT ODS Steel (Fe-14Cr-3W-0.20Ti-0.25Y_2O_3)
- Cross-sectional nanoidentation technique used

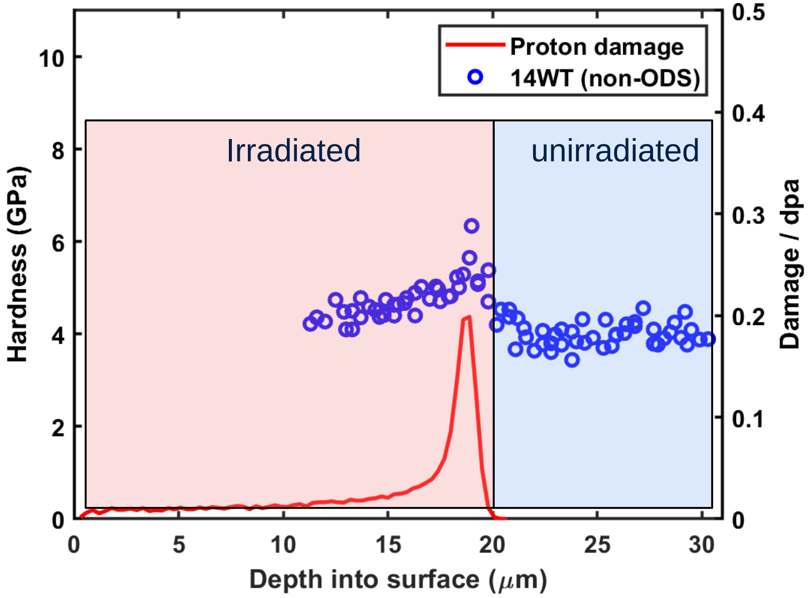




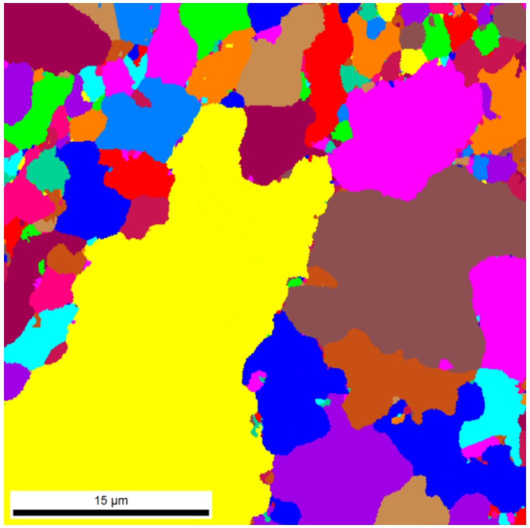
G200 nanoidentor's optical micrograph

First: Non-ODS model alloy

- ‘Non-ODS’ model alloy is manufactured the same of 14YWT but without the 0.25 Y_2O_3 additions
- Non-ODS = 14WT alloy (Fe-14Cr-3W-0.2Ti)
- Proton irradiated to 0.2 dpa at 300 at 300^oC at the Ion Beam Centre, University of Surrey



14WT EBSD grain structure



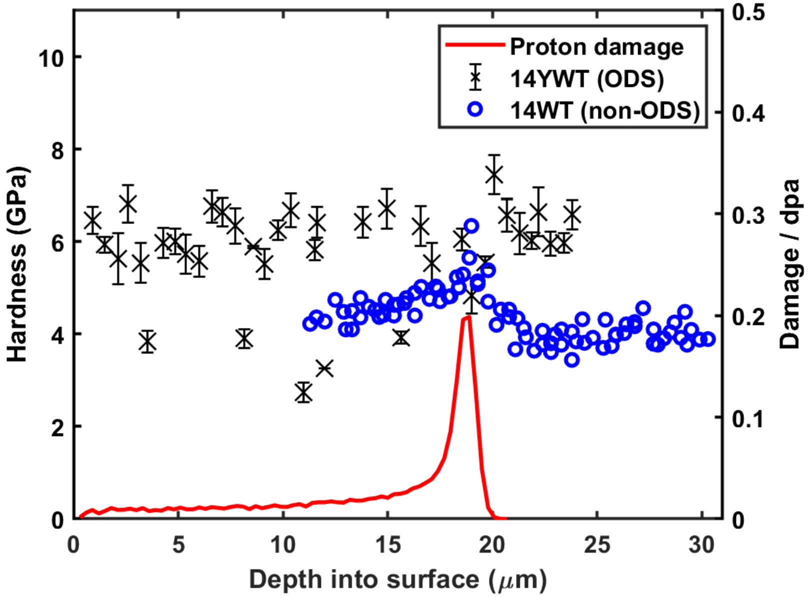
Observations:

- Clear proton damage profile observed
- 4.2 GPa in implanted region
- 6.2 GPa peak hardness

Nanoindentation of proton irradiated ODS

Questions:

- How ‘resistant’ is ODS steel to radiation damage?
- Can we observe this with nanoindentation?



Observations:

- No hardness increase in implanted layer
- No hardness increase at Bragg peak

Clear that Y-Ti-O nano-oxide particles create a resistant alloy to irradiation damage. However, what is the limit of this resistance?

Will investigate the heavy ion irradiated samples from DCF in the coming months