Advanced micromechanical testing techniques of irradiated materials for Nuclear applications

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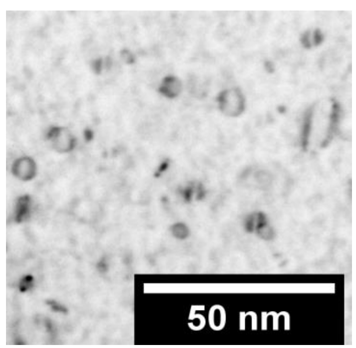
RaDIATE Collaboration Meeting, Geneva, 17-21 December 2018

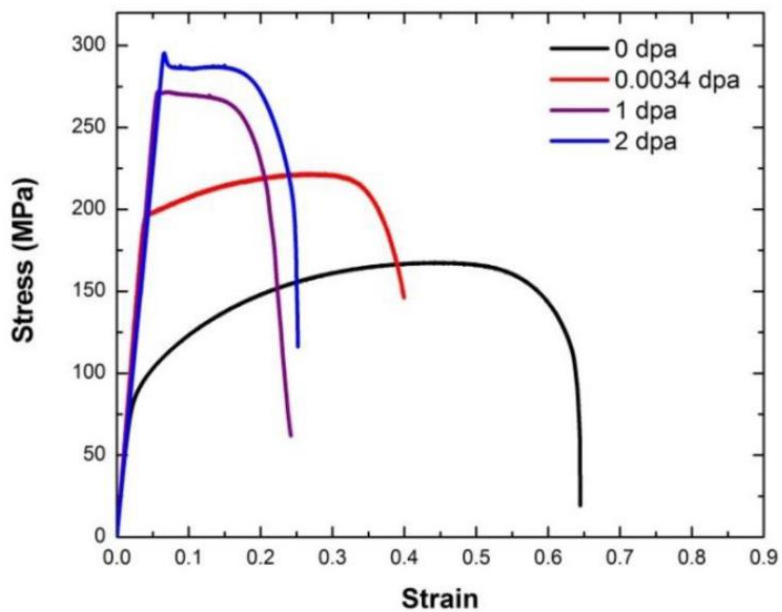
Radiation damage threats to structural materials

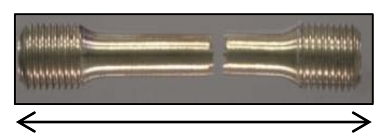
1) Reduction in ductility and work hardening capabilities
2) Segregation/precipitation leading to embrittlement
3) Plastic instability and prompt necking after yield

Microstrucuture of neutron irradiated Fe-9Cr

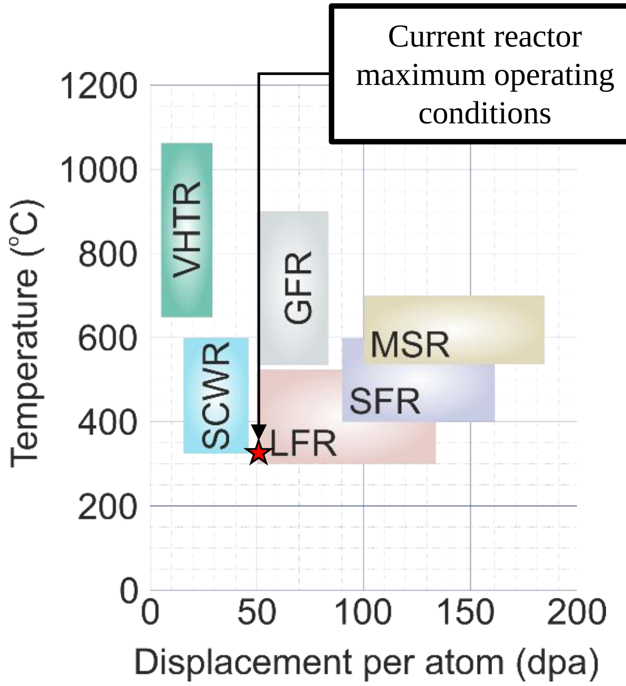


Typical uniaxial tensile behaviour of structural steels after irradiation

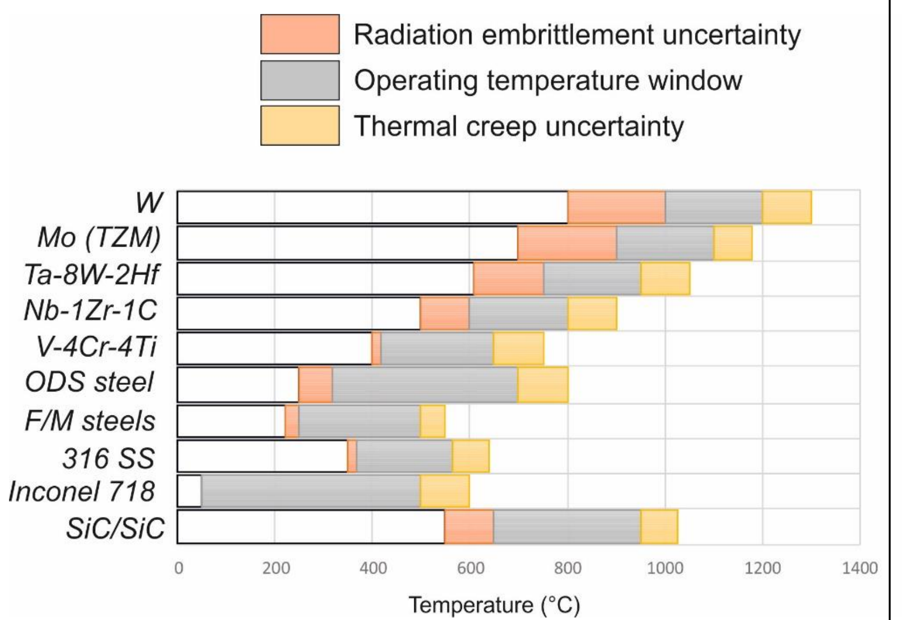




Materials issues in Future reactors



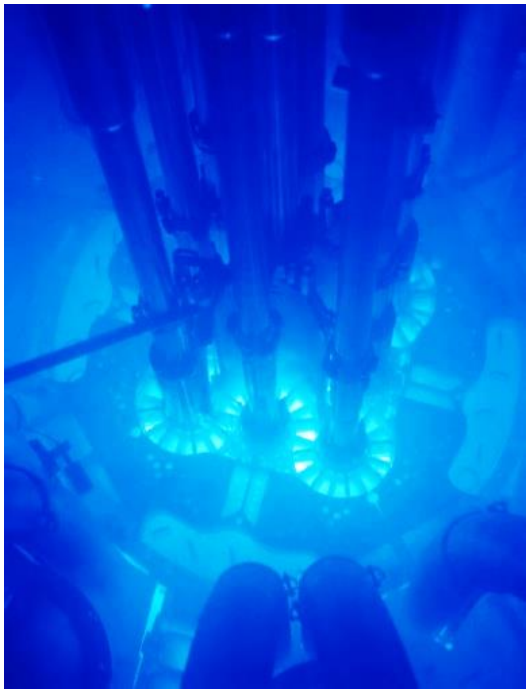
Overview of operating temperatures and expected damage levels for Generation IV reactor designs.



Estimated operating temperature windows for various structural materials in nuclear systems with damage levels of 10-50 dpa.

Zinkle, S. J. & Busby, J. T. Structural materials for fission & fusion energy Structural materials represent the key for containment of nuclear fuel. Mater. Today ***12,*** *12–19 (2009).*

Mechanical testing of reactor irradiated materials



Advanced test reactor (ATR)
Idaho national laboratory

Culham Materials Research Facility (MRF) – Hot cell facility

- Low dose rates **(200 dpa of damage in a test reactor would take decades to achieve)**
- Radioactive material handling / specialist facilities required for active material
- Not efficient process for **rapid characterisation** of mechanical response (expensive and time consuming)

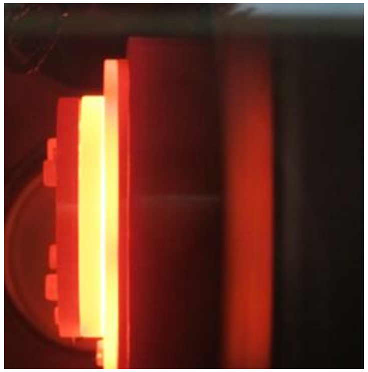
Mechanical testing of heavy ion irradiated material

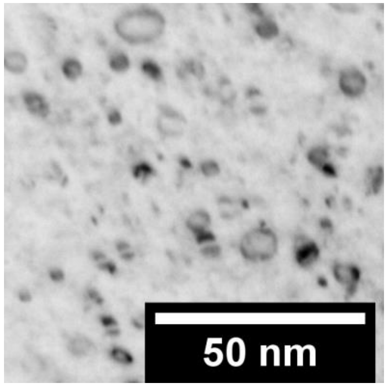
- Use ion irradiation to simulate neutron damage

H^+ and Fe^+

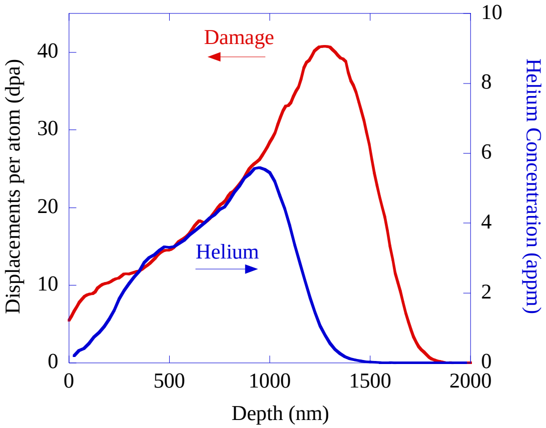
5MeV

420ºC





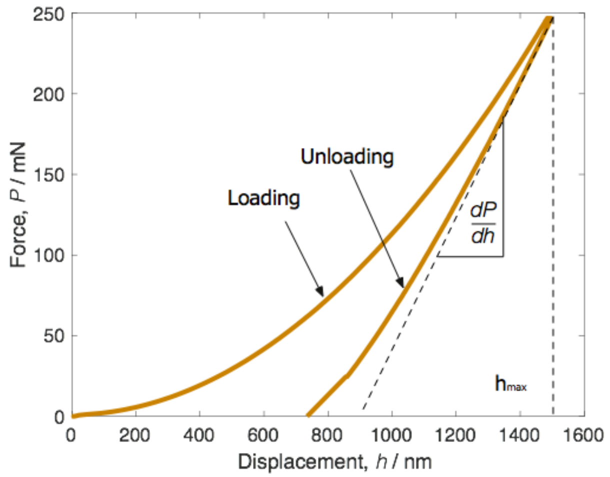
Ion irradiated damaged microstructure

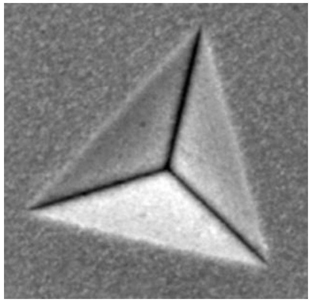


- Much higher dose rates (10^4 that of reactor irradiation)
- Negligible levels of activity
- Relatively cheap
- **However only small volumes of damaged material obtained…**

Need for small scale mechanical testing to predict bulk properties

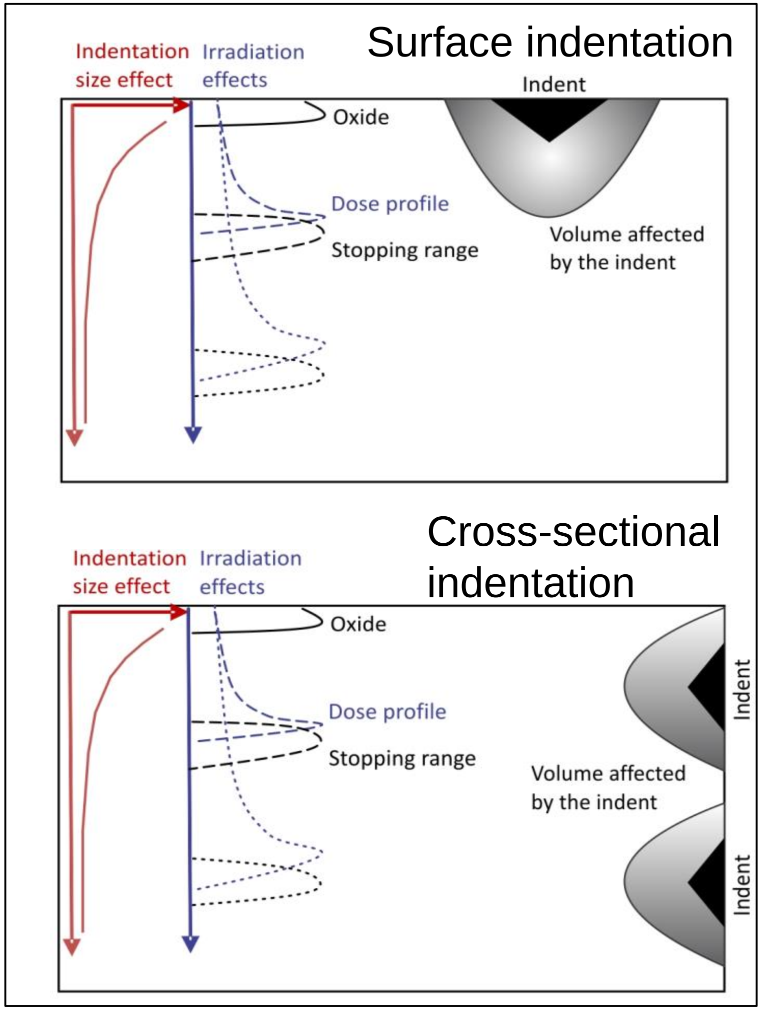
Nanoindentation: surface indentation vs. cross-sectional indentation





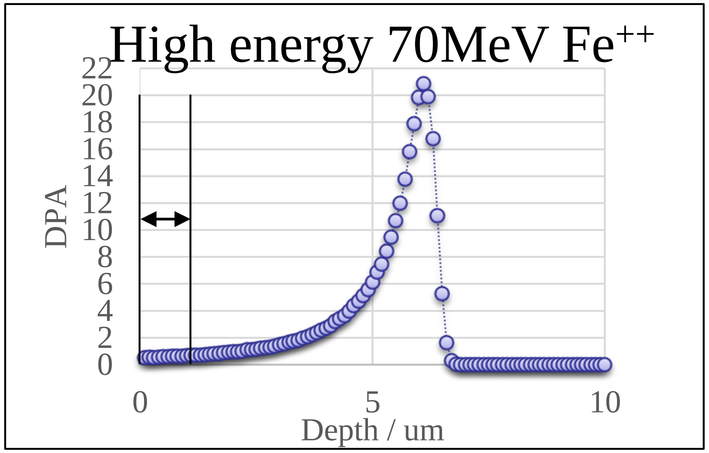
$$H = 3\sigma_y$$

- Measure hardness/yield strength and modulus
- Measure the relative increase in hardness due to irradiation damage
- Plastic zone extends into regions of unirradiated martial
- Indentation size effects for small indentation depth

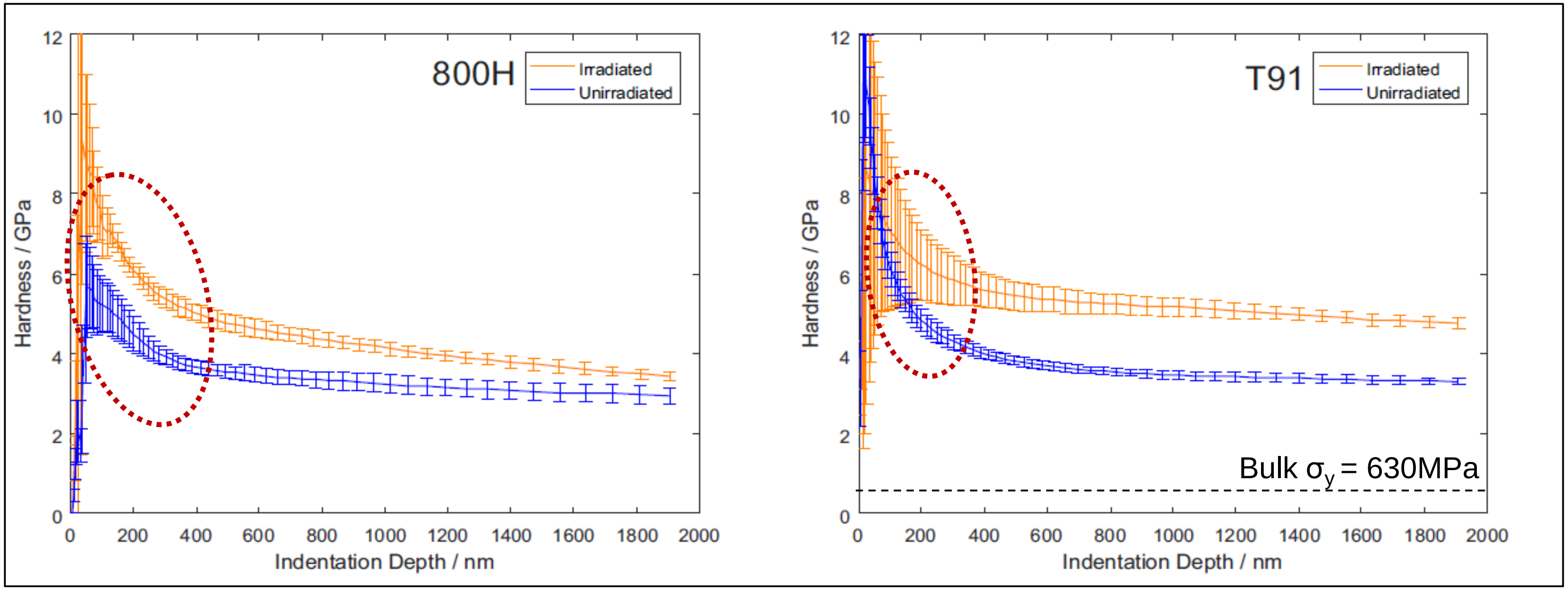


Kiener, Daniel, et al. "Application of small-scale testing for investigation of ion-beam-irradiated materials." Journal of Materials Research 27.21 (2012): 2724-2736.

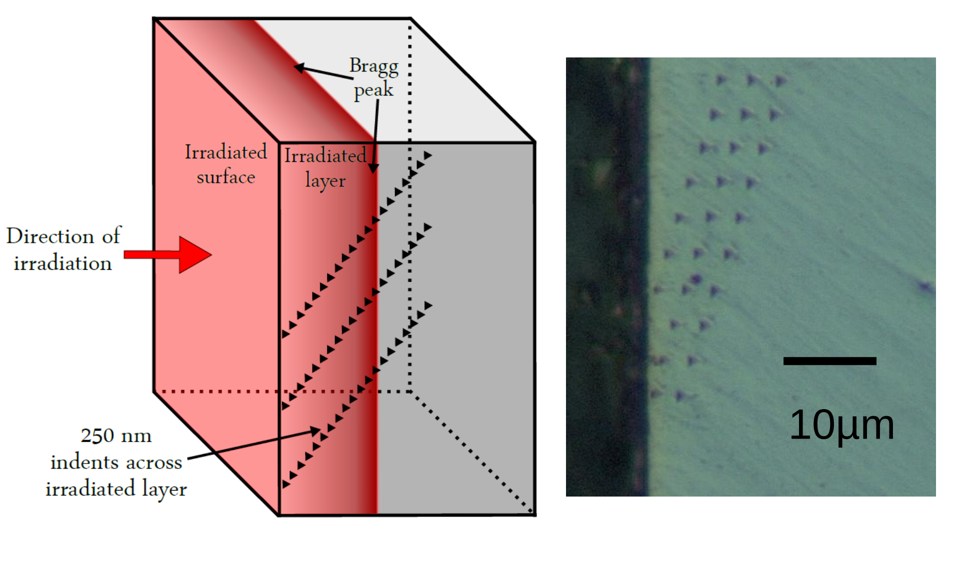
Nanoindentation of Fe^{++} irradiated candidate steels: **Surface indentation**

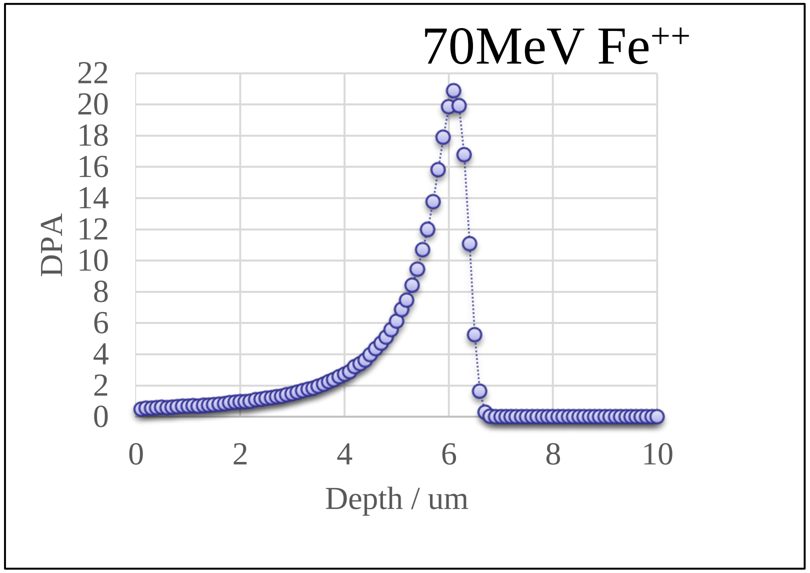


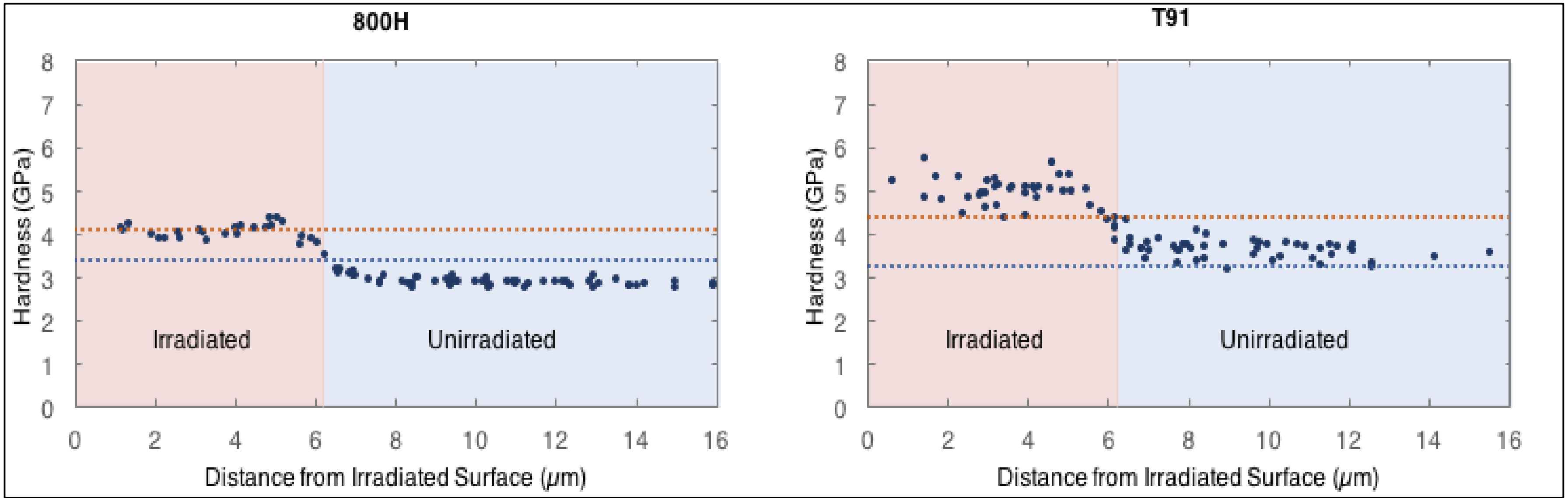
- Measure hardness/yield strength and modulus as a function of depth – dynamic indentation mode
- Measurement affected by the indentation depth → Indentation size effect **unless large damaged layer**



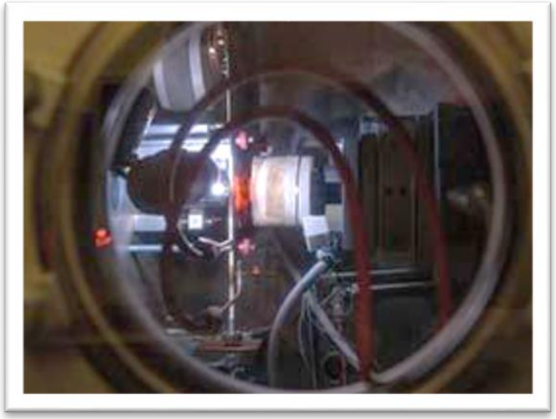
Nanoindentation of Fe^{++} irradiated candidate steels: **Cross-section indentation**





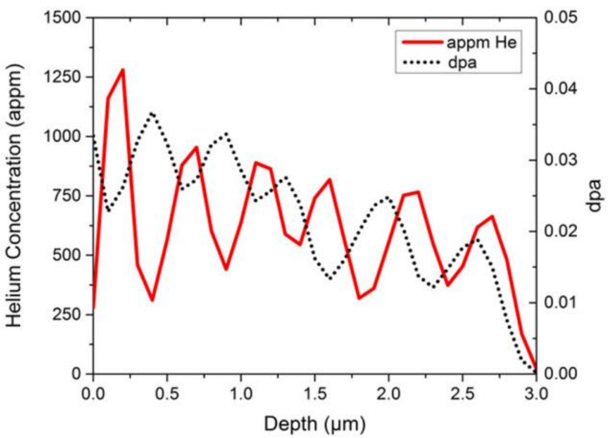


High temperature Nanoindentation

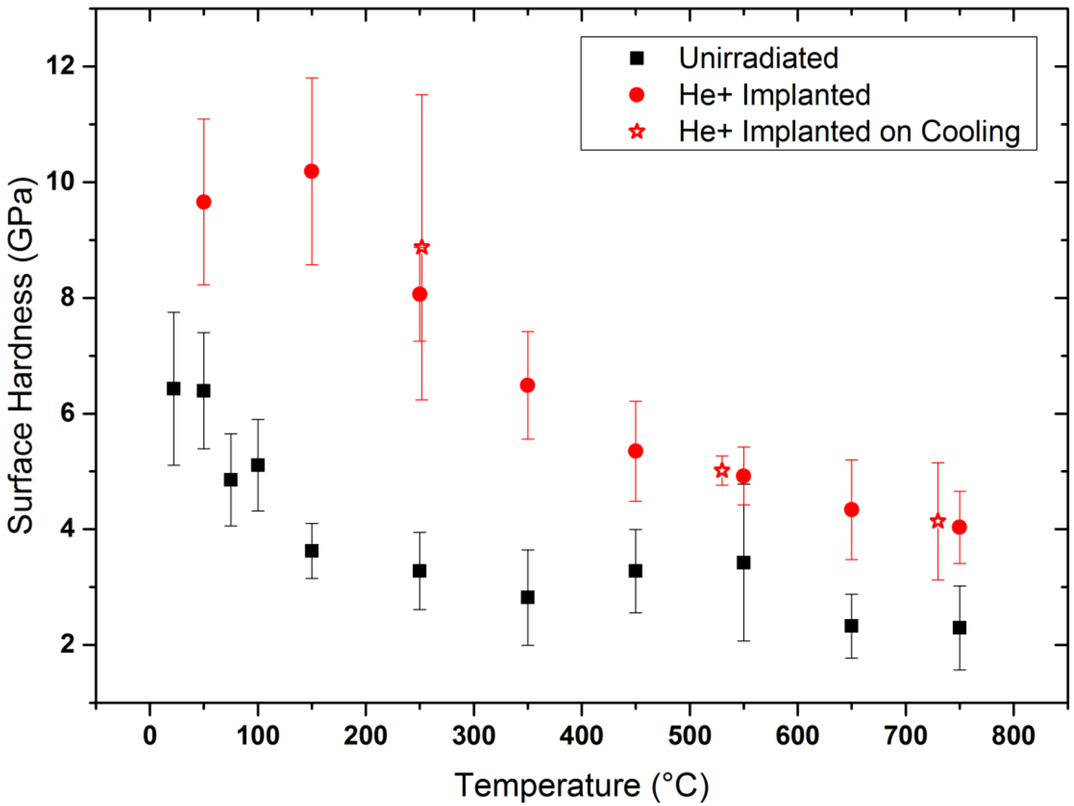


- Variable temperature indenter (-100~950 °C)
- Heats indenter tip and sample separately allow isothermal contact
- High vacuum - prevents oxidation (10^{-7} mbar)
- Extremely low thermal drift even at high temperature

High temperature Nanoindentation: He^{+} implanted Tungsten – Fusion applications

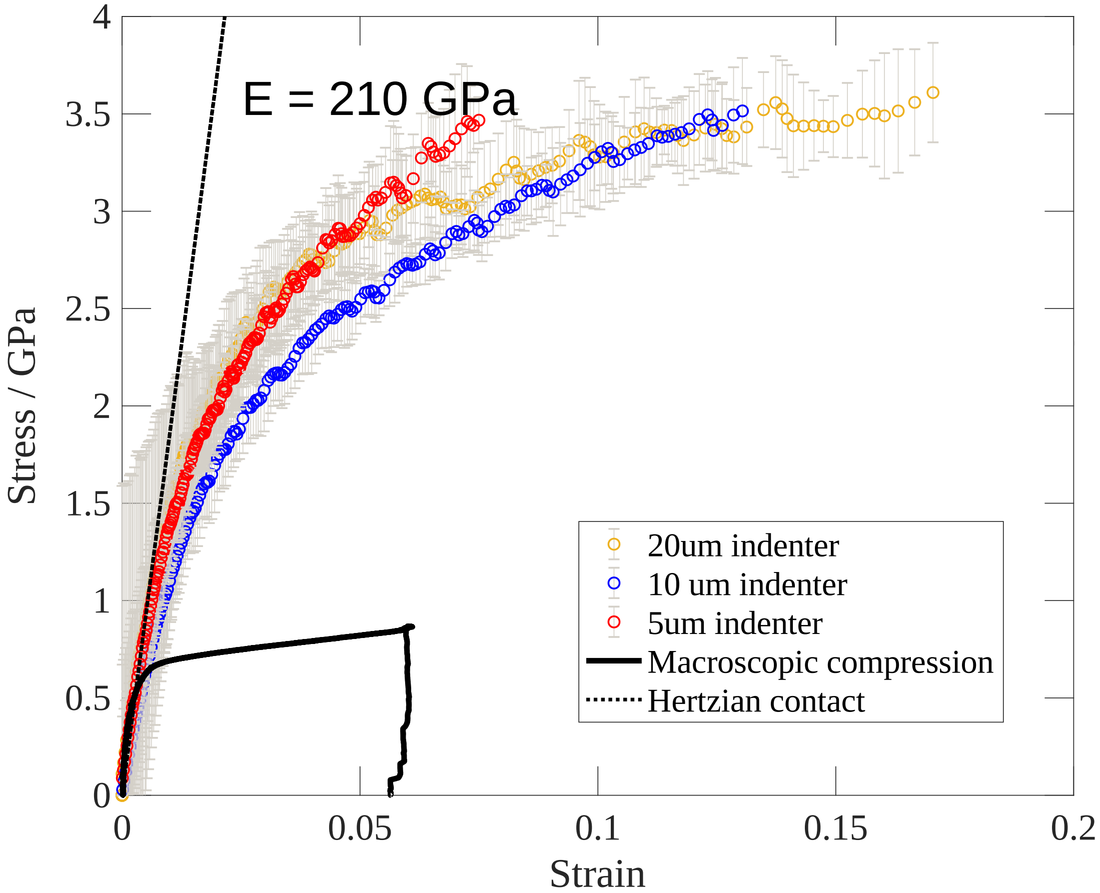


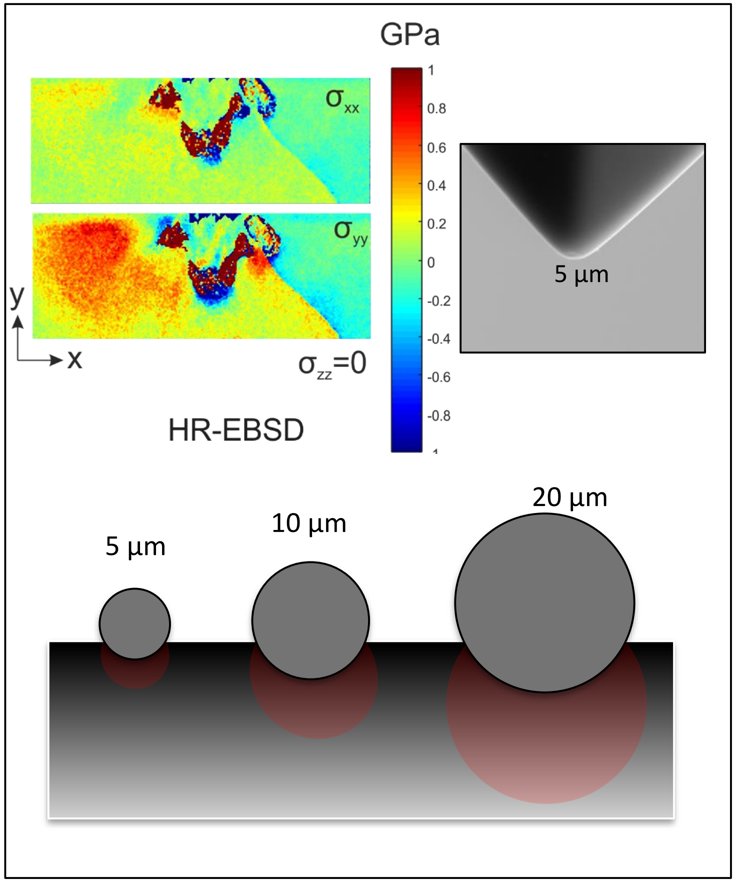
To simulate damage in fusion divertor helium ions implanted into pure tungsten at 850°C



JSKL Gibson, SG Roberts, DEJ Armstrong MSEA 625, 380-384

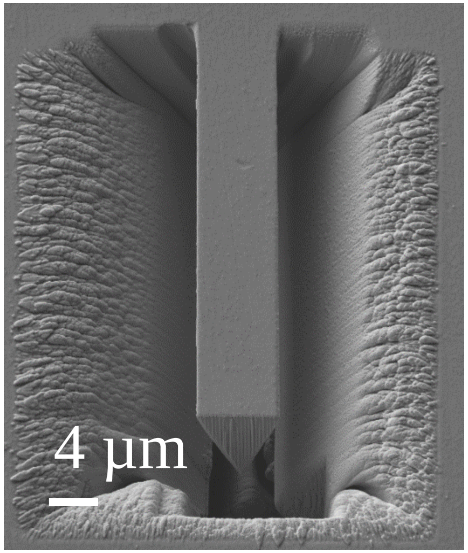
Spherical indentation/macroscopic compression in T91

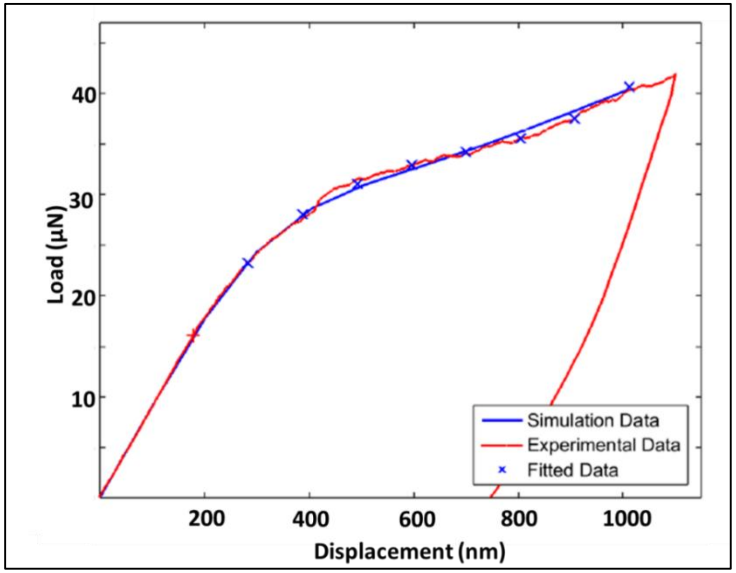


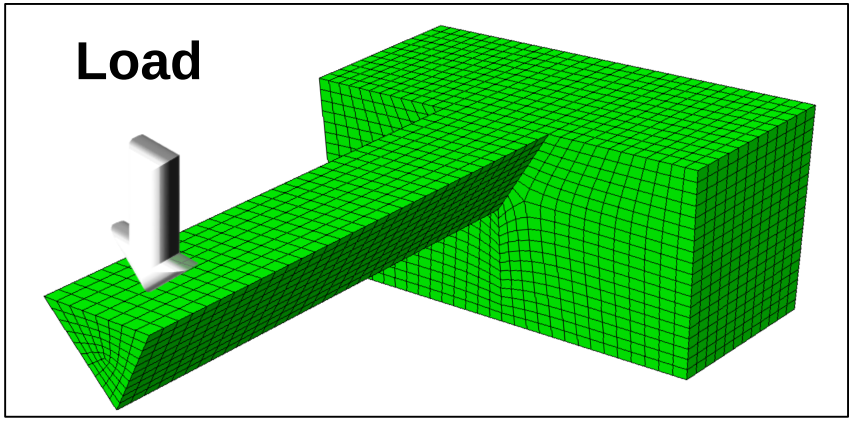


Microcantilver testing of ODS steel: Stress strain behaviour

- FIB machined micro-cantilevers in ODS and non ODS
- 20 x 4 x 5 micron with triangular cross section
- 0 dpa or 0.026 dpa Surrey at beam centre
- Stress-strain behaviour calculated using FEA model to account for non-fixed end







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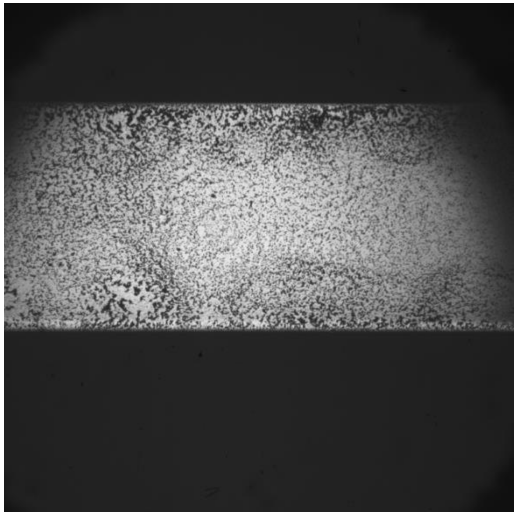
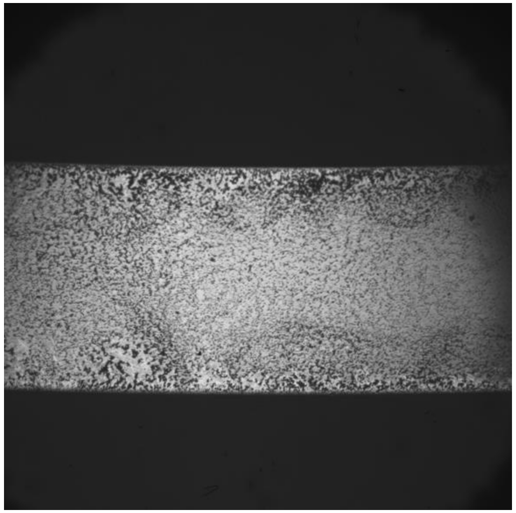
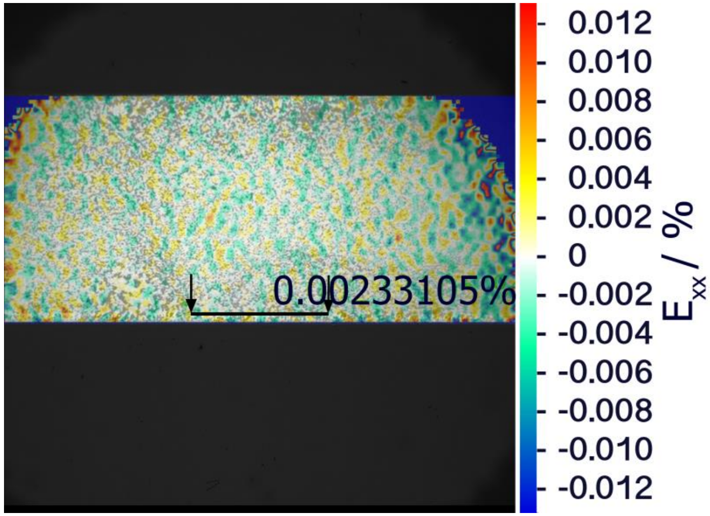
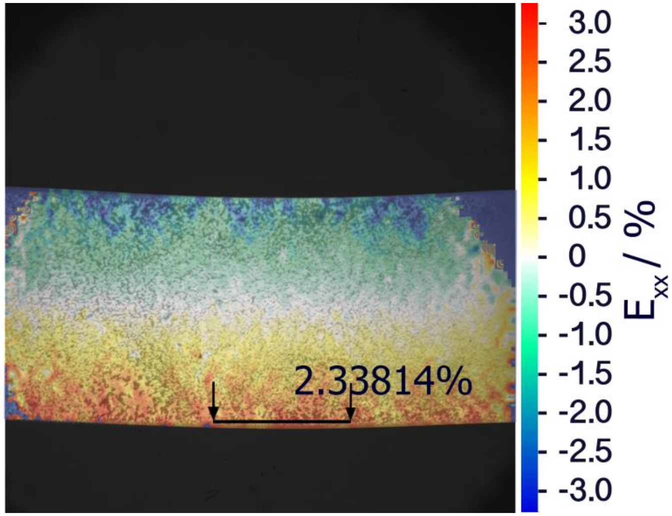
Macroscopic mechanical behaviour: ODS steels





- Samples 1x1x12mm
- Samples tested in 4-point bending
- Strain calculated using digital image correlation (DIC).





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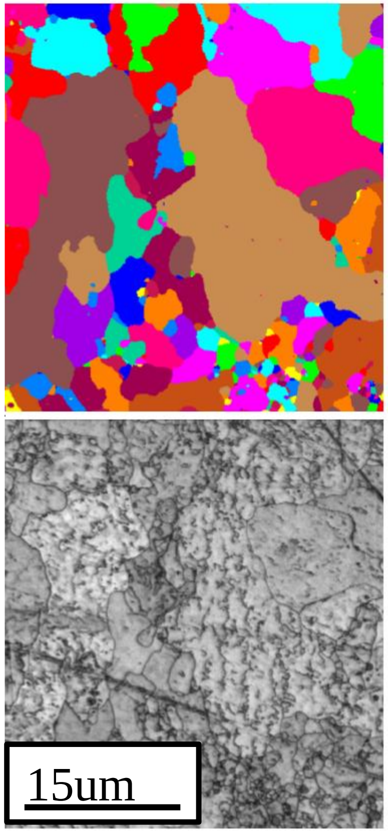
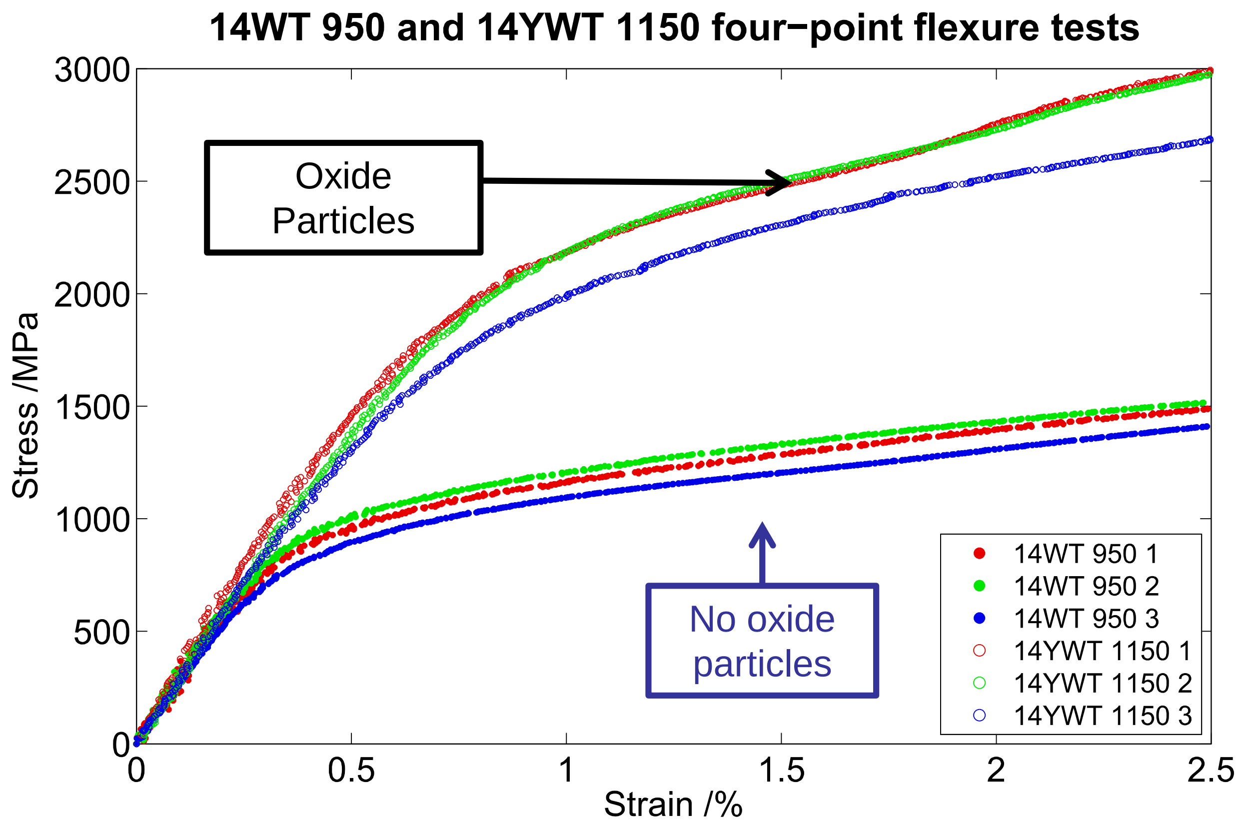
Macroscopic mechanical behaviour: ODS steels





Material	Average elastic modulus /GPa	Average yield stress /MPa
Non ODS	281 ± 6	$421\ ^{+35}_{-32}$
ODS	299 ± 6	$842\ ^{+63}_{-47}$

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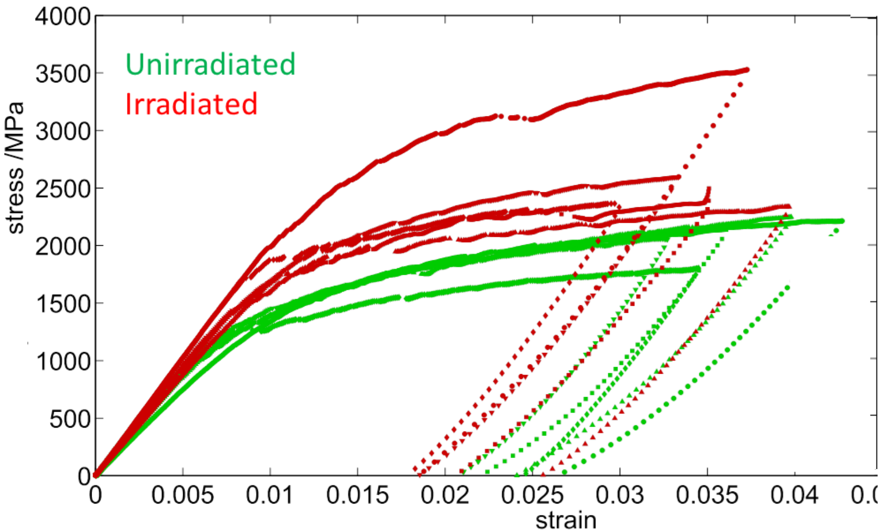
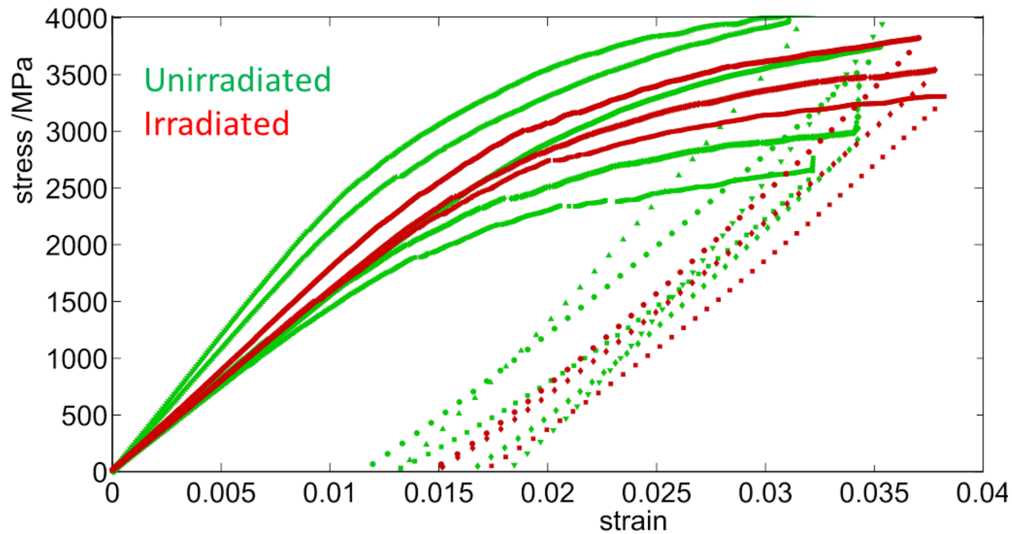
Microcantilever bend testing of proton irradiated ODS steels

Non-ODS



ODS



	Non - ODS		ODS	
	Modulus / GPa	Yield Stress/ GPa	Modulus / GPa	Yield stress/ GPa
Unirradiated	208±5	1.05±0.07	216±20	1.30±0.4
Irradiated	224±6	1.57±0.15	196±12	1.44±0.3

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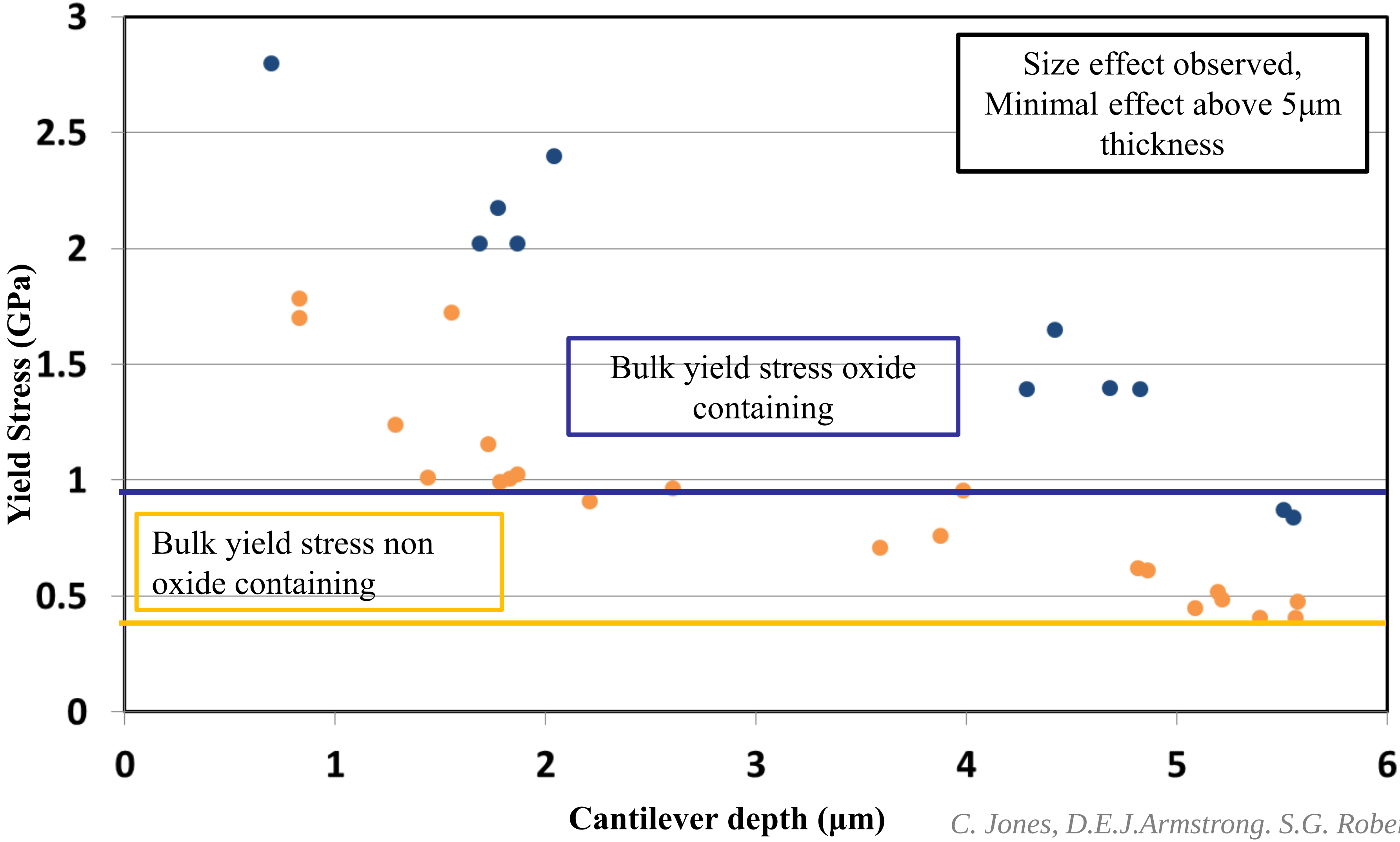
Microcantilever bend testing of proton irradiated ODS steels: Size effects



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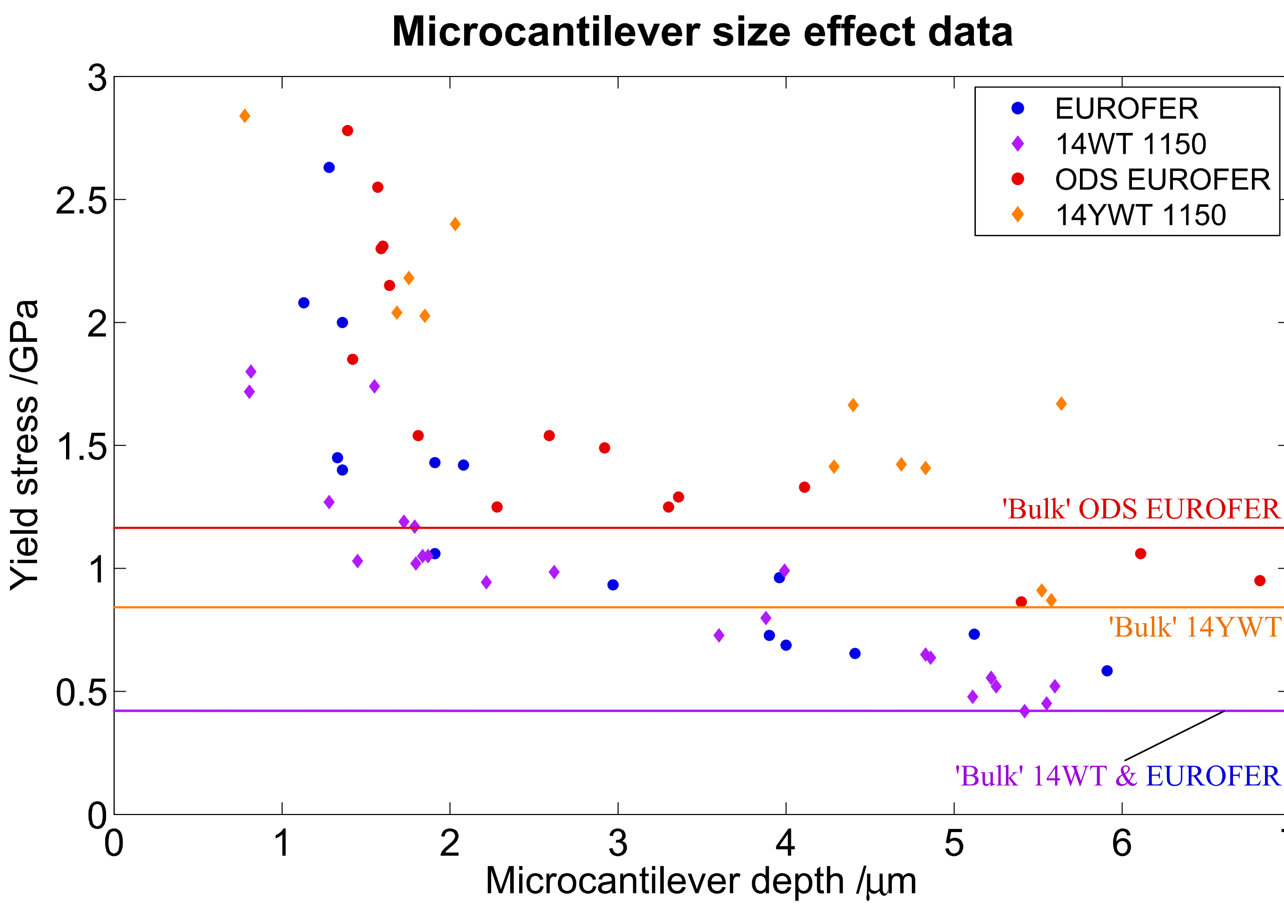
Microcantilever bend testing of proton irradiated structural steels: Size effects

The apparent strength of the microcantilevers appeared to drastically deviate from macroscale values (i.e. source limited) for beam depths ≤ 4.5 µm



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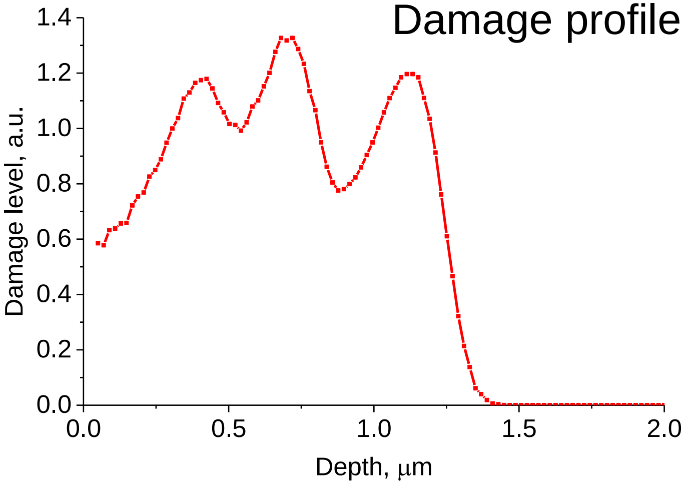
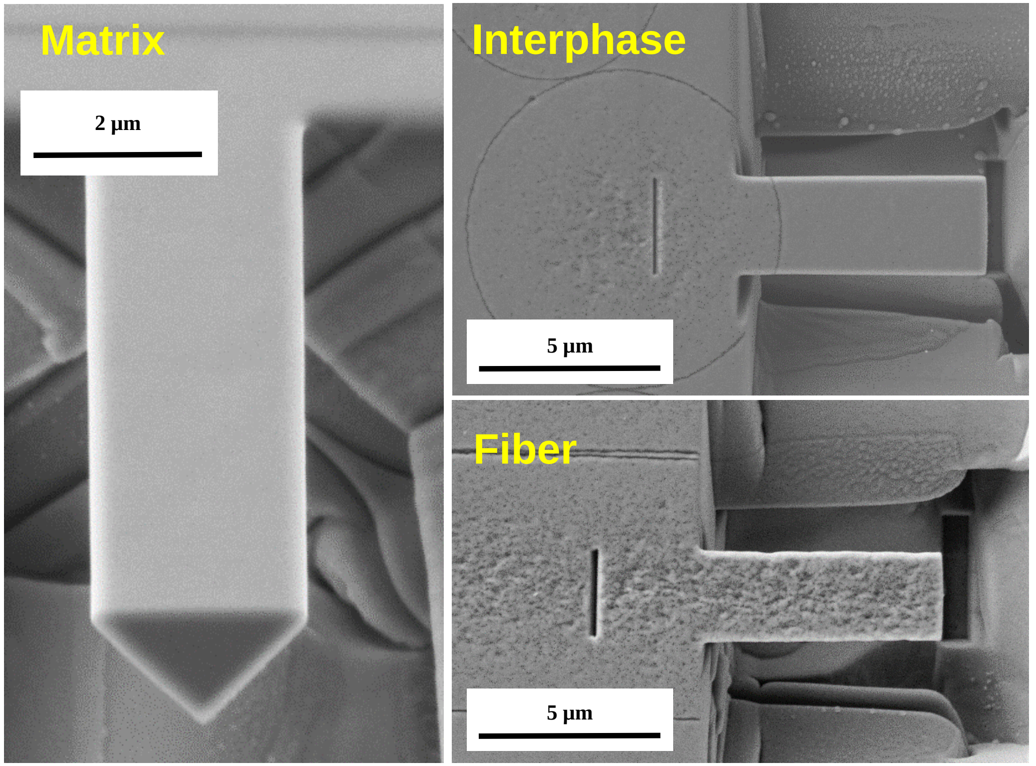
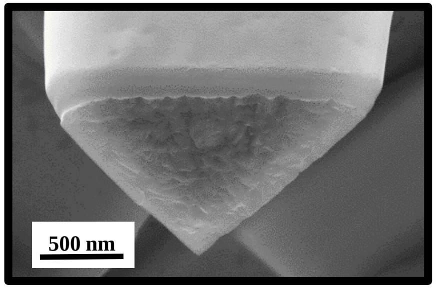
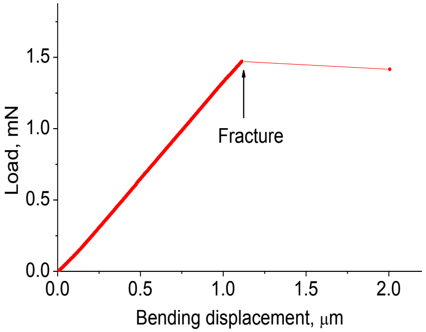
Ion irradiation of SiC-SiC composites for nuclear applications – fracture experiments

- Si ions.
- Three energies → 500 keV, 1 MeV, 2 MeV.
- Damaged zone ~1.4 µm deep.
- Dose ratio 3 : 2 : 1 → relatively uniform damage profile.
- 0.26 dpa – 300° C and 750° C
- Microcantilevers manufactured in the interphase, fibre and matrix of both irradiated and unirradiated material

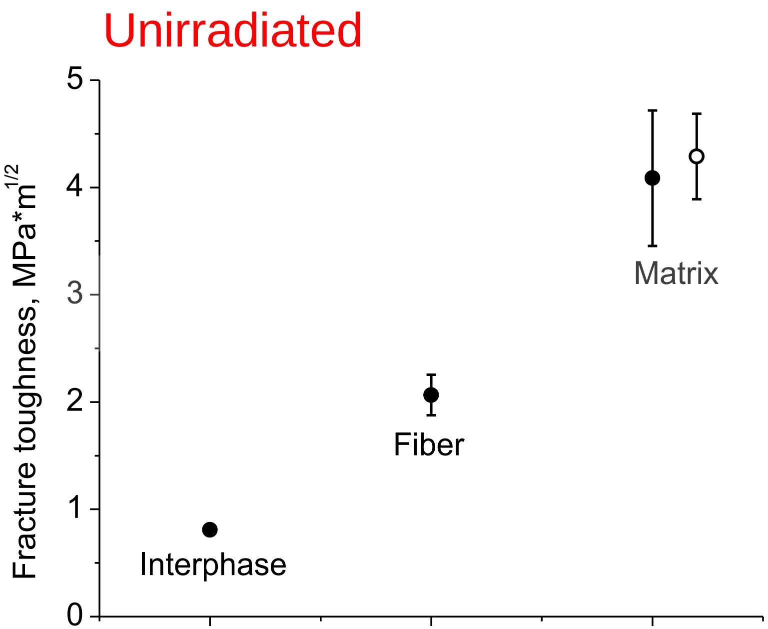


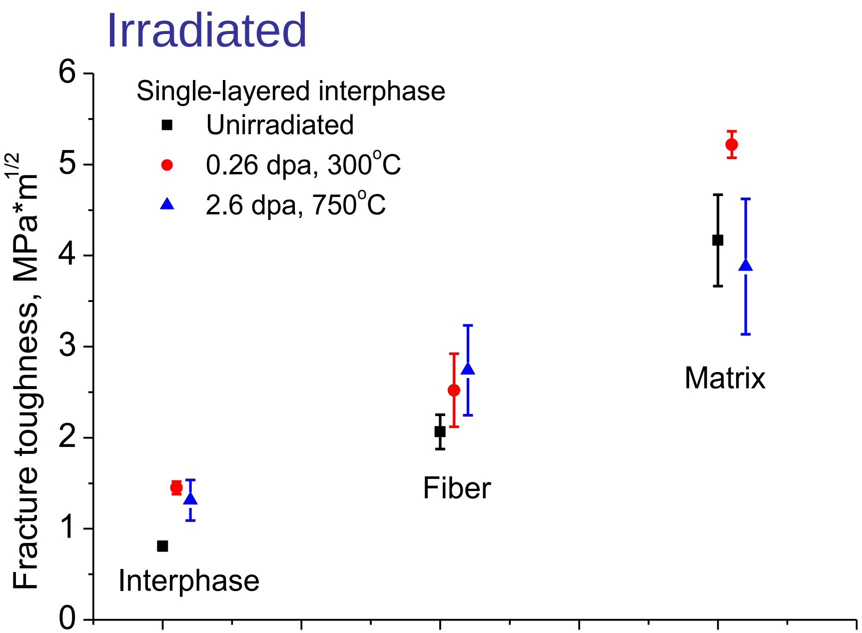






Ion irradiation of SiC-SiC composites for nuclear applications – fracture experiments





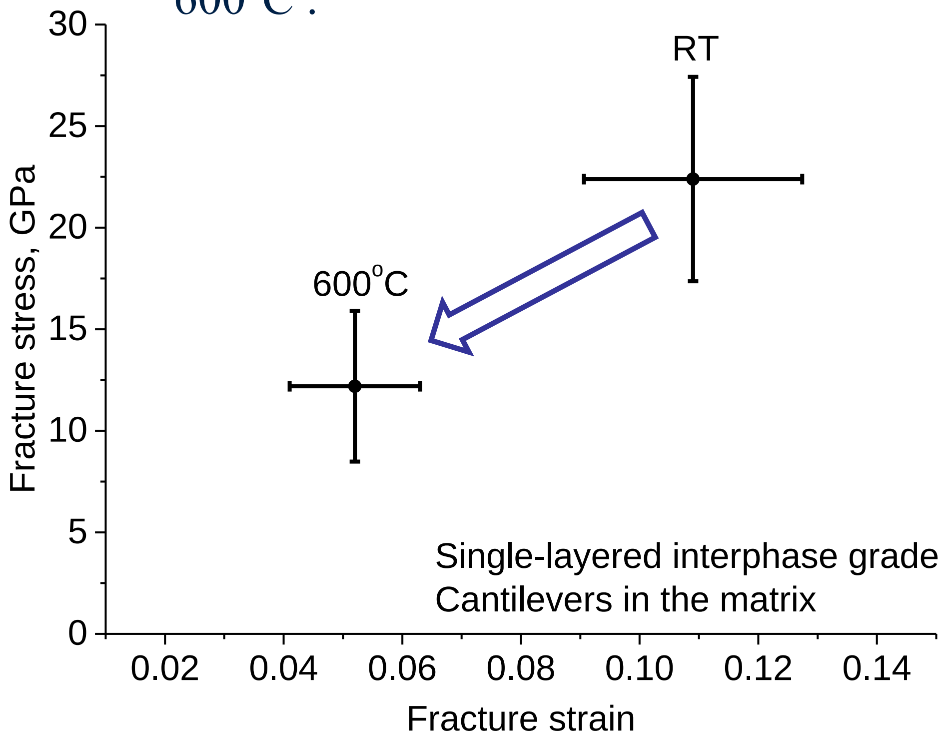
- Fracture toughness measurements – cantilevers with straight notch.
  - Matrix – 4.1 $MPa*m^{1/2}$
  - Fiber – 2.1 $MPa*m^{1/2}$
  - Interphase – 0.8 $MPa*m^{1/2}$.

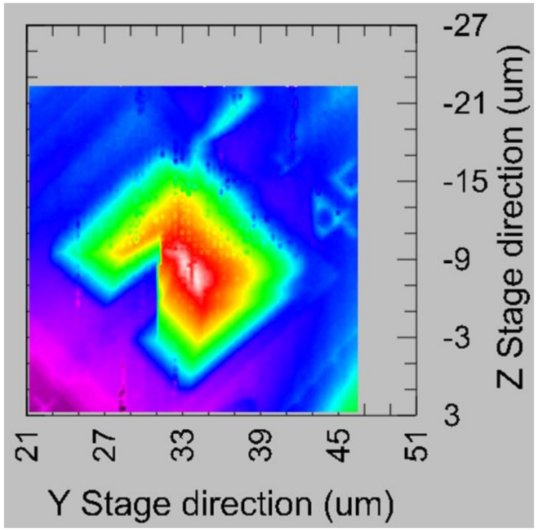
- Following the irradiations:
  - Interphase – toughness increases, doesn't noticeably change with the increase of dose.
  - Fiber – toughness progressively increases with the increase of dose.
  - Matrix – no clear trend.

Dr Y. Zayachuk

Ion irradiation of SiC-SiC composites for nuclear applications – fracture behaviour at temperature

Hot nanoindenter (Micro Materials NanoTest Xtreme) – vacuum tests at 600°C .





- Room temperature:
  Fracture stress – 21 GPa;
  Fracture strain – 13%.

- 600° C:
  Fracture stress – 12 GPa;
  Fracture strain – 5%.

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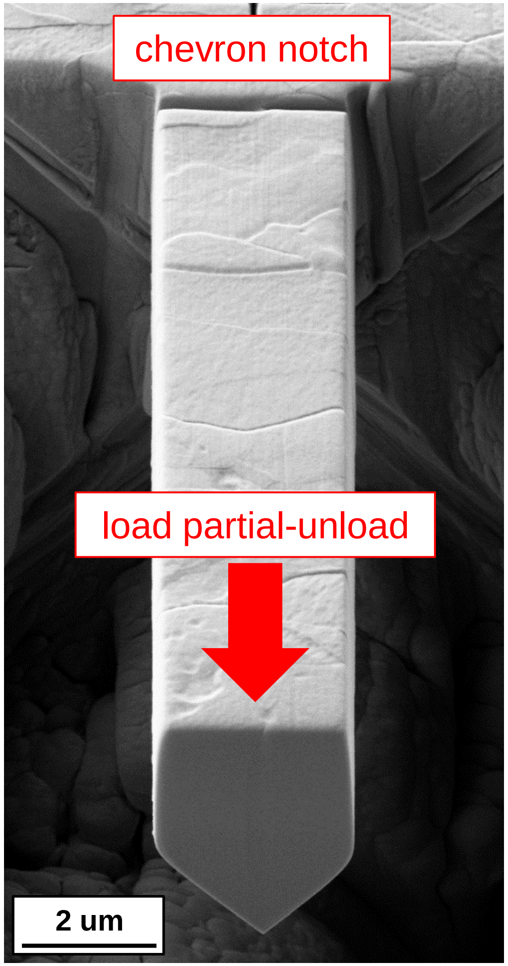
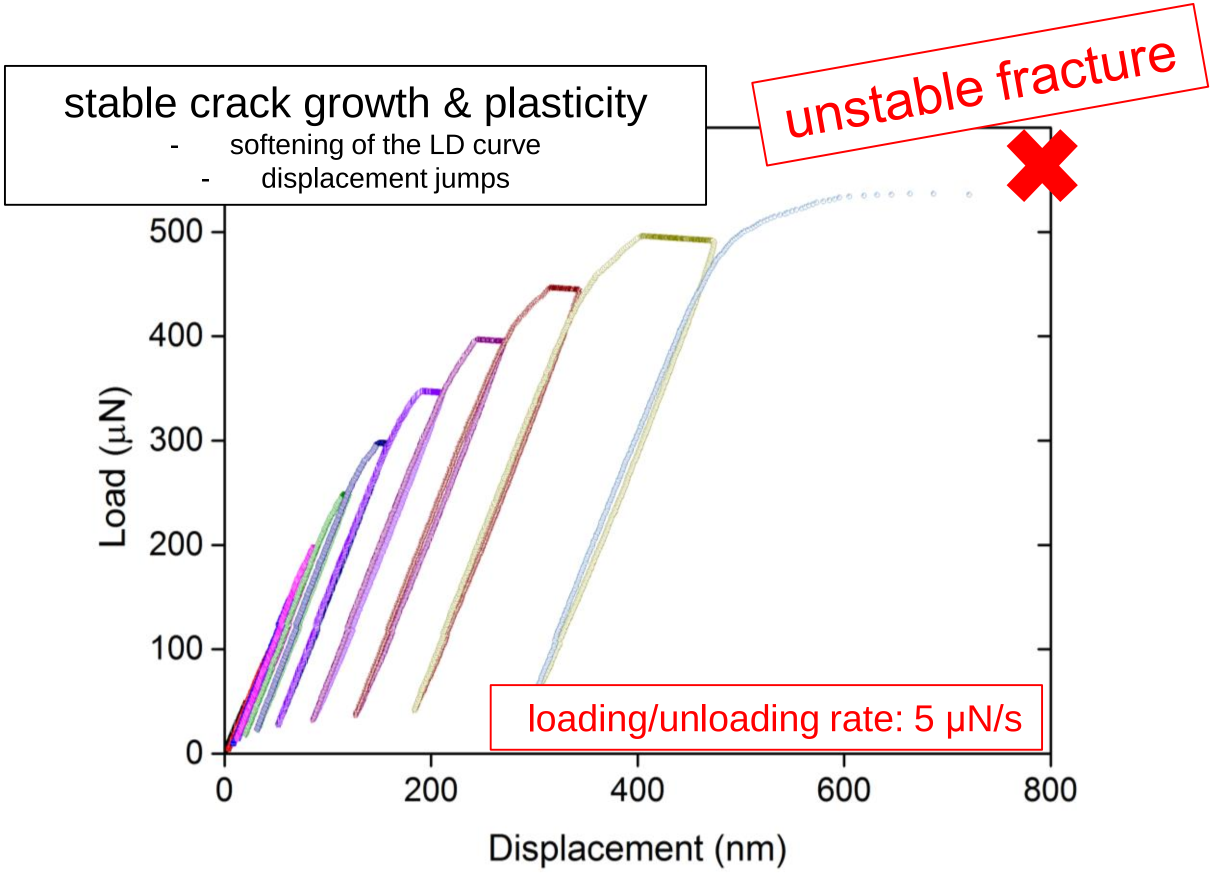
Microcantilever fracture experiments: load – unload in Tungsten





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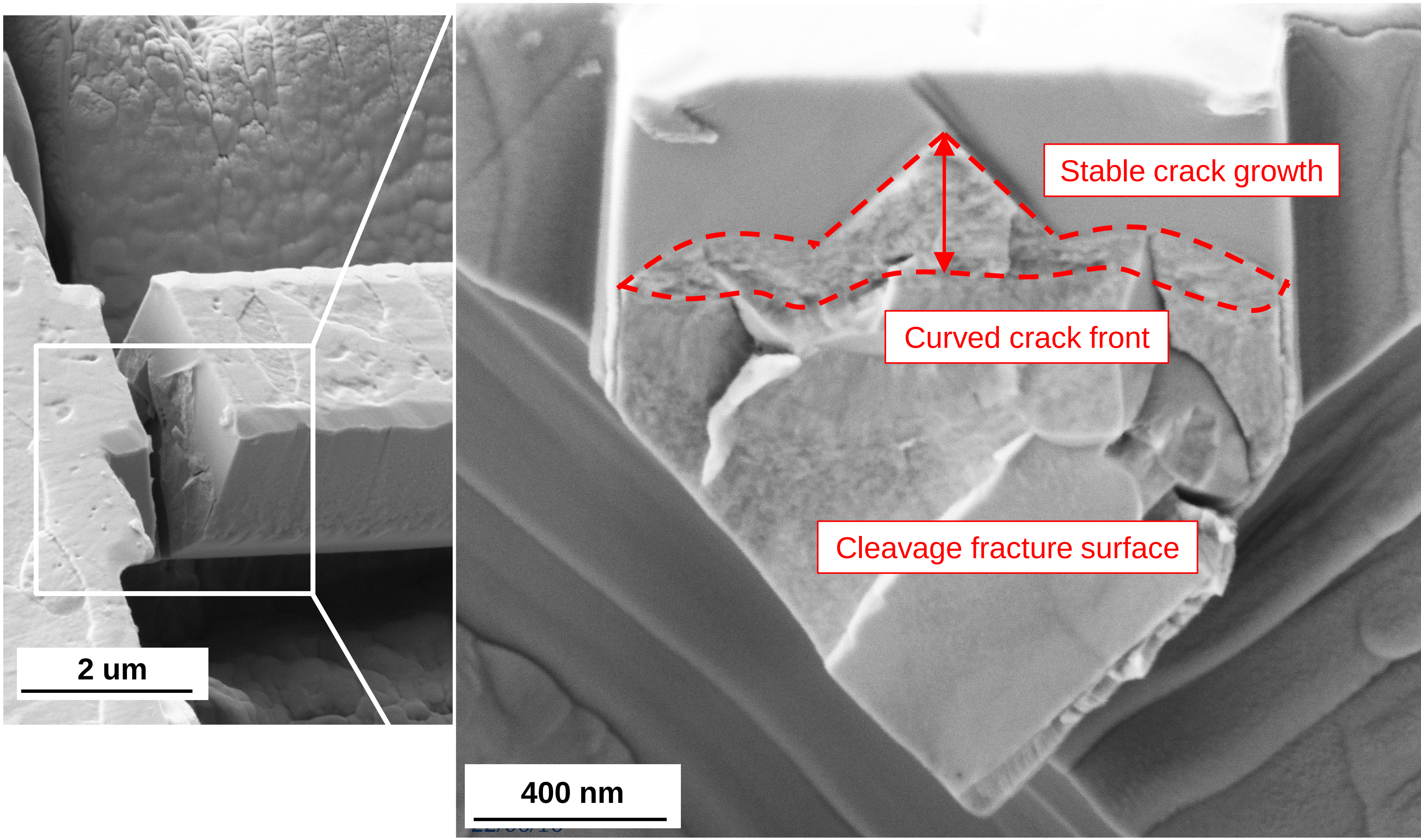
Microcantilever fracture experiments: Tungsten: Post-mortem fractography



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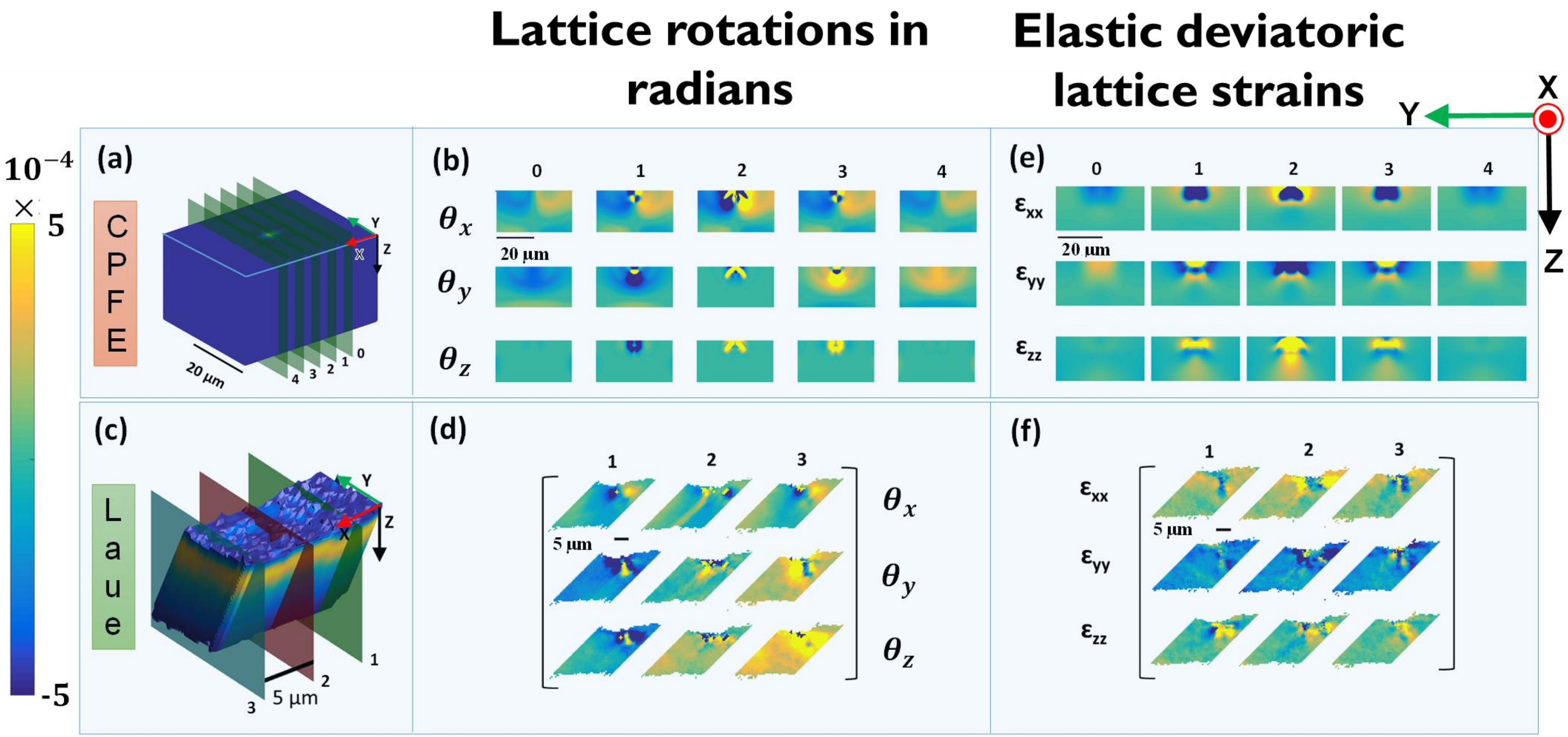
Dislocation based CPFEM to simulate Nanoindentation in implanted Tungsten



S. Das, F. Hoffmann, E.K.Tarleton

Summary

- By simulating reactor irradiation with charged particle accelerators – provide rabid screening of radiation damage in candidate materials for nuclear applications
- Micromechanical testing enables a **large** amount of data to be obtained from **small** volumes of material (either active material or where only small volumes are available or shallow irradiated layers)
- Nanoindentation is a rapid screening tool to measure the hardness/modulus of irradiated material, microcantilevers can be used to obtain mechanical properties beyond the yield point (full flow curve) and fracture behaviour
- Combining micromechanical tests with dislocation based modelling enables a full characterisation of the mechanical properties of irradiated materials

Thank you!