UK Atomic Energy Authority

MATERIALS RESEARCH FACILITY

UKAEA - Materials Research Facility SHRI Ion beam irradiation and characterisation workshop

Dr Ed Eardley
MRF Scientific Manager

UKRI Engineering and Physical Sciences Research Council

NNUF National Nuclear User Facility

HENRY ROYCE INSTITUTE

Fusion Grant 2022/27 EP/W006839/1

MRF is part of the National Nuclear Users Facility (NNUF) and the Sir Henry Royce Institute for Advanced Materials

Introduction

- Introduction to UKAEA.
- The MRF.
- MRF active sample procedures.
- MRF Use.
- Access funding for the MRF

Dr Ed Eardley

MRF Scientific Manager

UK Atomic Energy Authority

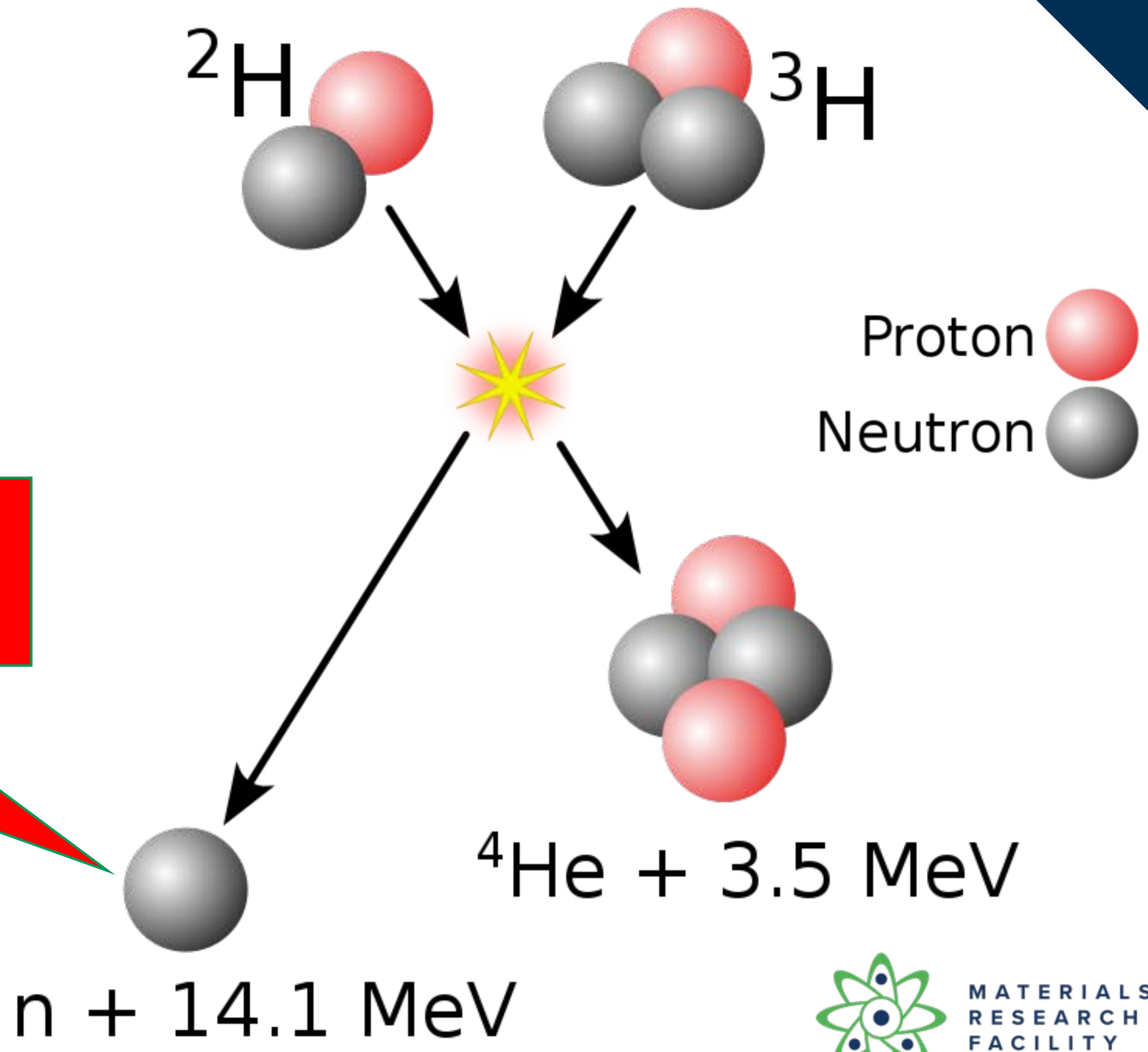
"To lead the delivery of sustainable fusion energy and maximise the scientific and economic benefit."

MATERIALS RESEARCH FACILITY

Fusion

$$^{3}_{1}H + ^{2}_{1}H \rightarrow ^{4}_{2}He + ^{1}_{0}n + 17.6MeV$$

- α particle 3.5MeV
  - Charged so confined by the magnetic field.
  - On cooling is exhausted as He
- Neutron 14.1 MeV
  - Not confined by the tokomak.
  - Carries a significant proportion of the released energy.
- Neutron damages materials through displacement and transmutation.
  - To study the former we need to deal with the later
- And then we're going to increase the temperature.
  - Improved thermal efficiency
  - Higher T waste heat



UKAEA Materials

- 3 Broad themes:
  - Study the damage.
    - MRF PIE facility.
  - Understand the damage.
    - Materials modelling group.
  - Prevent the damage.
    - Materials science group.

Facilitated by the MRF ability to deal with the transmutation.

- Materials development, assurance and manufacture for Fusion.
  - Superconductors, first wall, high temperature structural materials, coolant containment.
  - Multi length scales, atomic to engineering.
  - Theoretical as well as experimental.
  - MRF is the active experimental facility supporting

UK Atomic Energy Authority

UKAEA
Materials Research Facility
(MRF)

MATERIALS RESEARCH FACILITY

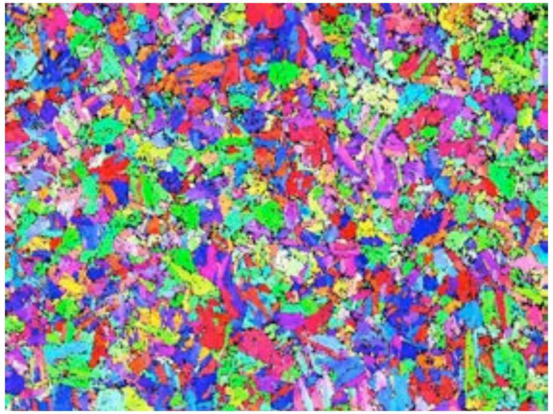
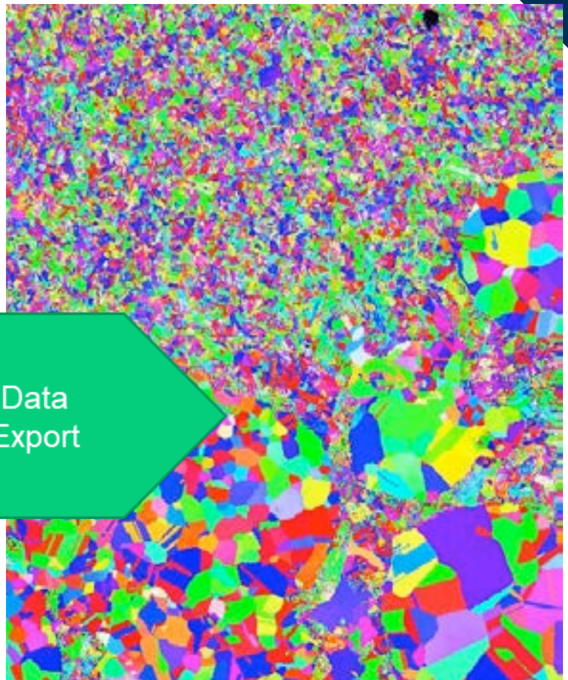
UKAEA - Materials Research Facility

- An integrated facility for the characterisation and testing of β/γ emitting materials as well as Beryllium.
  - Comparably small α limit.
- MRF bridges the gap between academic facilities and licenced nuclear sites.
- Focus is on post irradiation examination of structural and functional materials.
- High active operations with full remote sample handling
- Low active operations with manual handling
  - Governed by the MRF safety case and radiological RAs.
- MRF is a user facility offering external access to non UKAEA users.

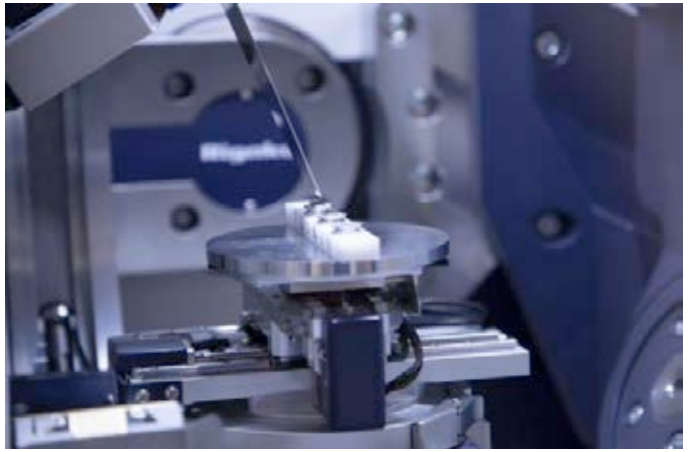
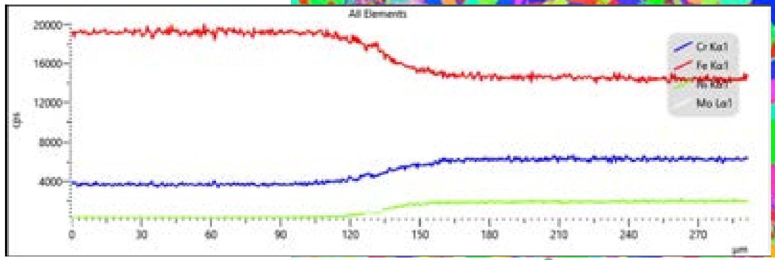
MRF PROCESS OVERVIEW

UK Atomic Energy Authority



Sample Receipt → Sample Prep → Sample Transfer → Testing → Data Analysis → Data Export

MATERIALS RESEARCH FACILITY

The Risks

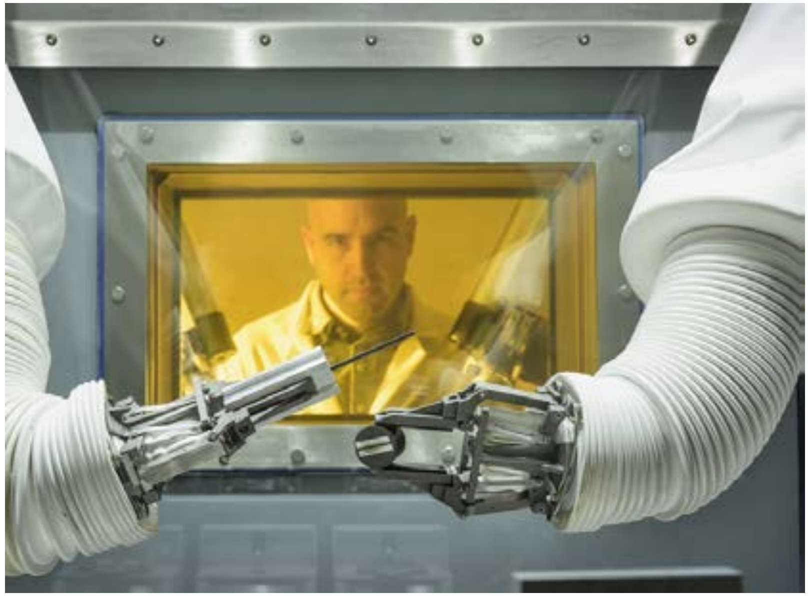
1. Radiation:
    - Energy emitted directly by the samples and absorbed by matter.
    - Absorption by matter results in damage.
    - In biological materials this has health consequences that can be potentially fatal.
    - Managed by the MRF shielded infrastructure.

- 2. Contamination:
    - Active material where it shouldn’t be.
    - Can be aerated and respirated.
        - Be is a specific non active contamination problem.
    - Managed by the MRF ventilation system.

MRF Main Hall

- MRF Facility:
  - HGV capable receipt facility including airlock.
    - 10 ton crane limit.
  - 3 hotcells (extension to 5 cells underway).
  - Shielded internal transport integrated into both hotcells and research rooms.
  - 23 shielded research rooms.
  - Managed ventilation system.

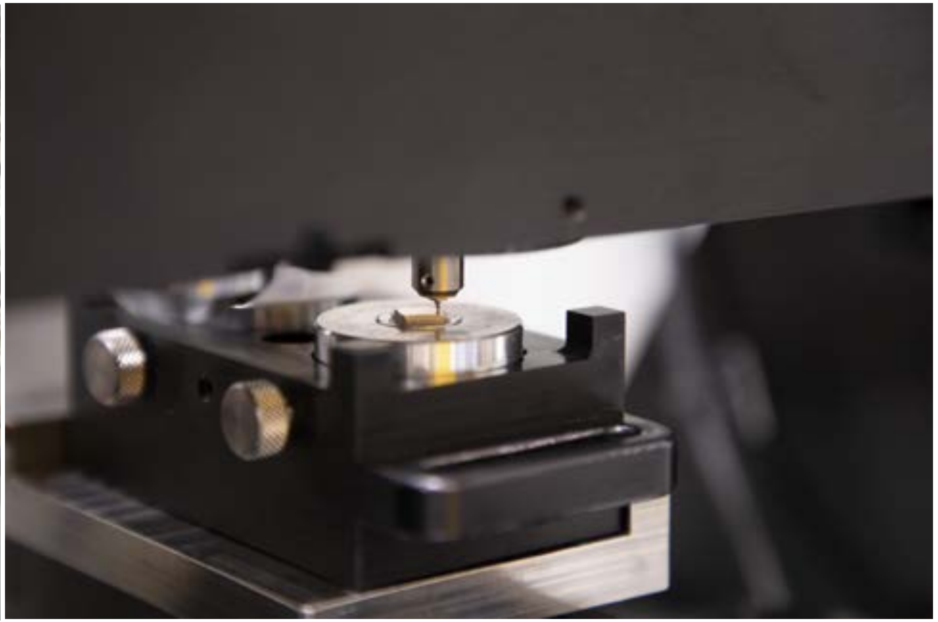
Shielded Remote Handling



- In cell gamma spec, dosimetry, sample prep and storage
- 3.75TBq ^{60}Co equivalent hotcell inventory limit.
  - β/γ facility
  - Small α inventory.
- 3.75GBq ^{60}Co equivalent transport trolley and research room sample limit.
  - Intention for remote handling capability within each research room.

Low activity samples

- Manual handling of low active samples in a contamination controlled area.
  - Additional monitoring in place.
  - BAU 10 μSv/Hr at 30cm dose rate limit.
- Low activity/Beryllium glovebox sample prep.
- Dose rate limit can be exceeded under a specific radiological risk assessment

UK Atomic Energy Authority
MATERIALS RESEARCH FACILITY

MRF Active Ventilation system

UK Atomic Energy Authority

- Key MRF infrastructure.
- Maintains air and thus contamination flow within the active areas.

Outside

↓

Hall

↓

Hotcells, research rooms and gloveboxes.

- Ventilation failure will lead to a closure of the active areas.
- Exhaust monitored and filtered prior to exhaust.

MATERIALS RESEARCH FACILITY

Control Room

- Remote operation of the instrumentation via a configurable network.
  - Separate data export network
- Removal of external users from the main hall
- MRF technical and scientific support

Available MRF Instrumentation

UK Atomic Energy Authority

Microstructure:
- Ga-FIB with EBSD
  - Cryo stage and transfer
- SEM with EDS, WDS and EBSD
- AFM
- CLM Raman
- XRD
- Plasma-FIB with EBSD and EDS in commissioning
- (S)TEM installation

Thermo Physical
- Physical properties measurement system
  - 14T and 1.8-1000K
- Thermal desorption spectrometry
- Laser flash analysis
  - -150 – 2000°C
- Dilatometer
  - -150- 2000°C
- Simultaneous thermal analysis
  - -150-2000°C
- Gas Pycnometer
- Ion exposure and impregnation system
- High-vacuum differential scanning calorimeter in procurement.

Mechanical testing
- Nano indentation with micromechanics nano positioning stage
- Instrumented indentation
- Micromechanical testing
  - 5kN frame with DIC and -100°C<T<600°C
- 10kN static load frame with high temperature capability
- DIC strain measurement
- 15kN dynamic load frame
- Ultrasonic fatigue rig (20kHz)
- Impulse excitation test (RT-900°C)
- In situ micromechanics stage in procurement
- 20kN in cell furnace equipped servo hydraulic loadframe in procurement.

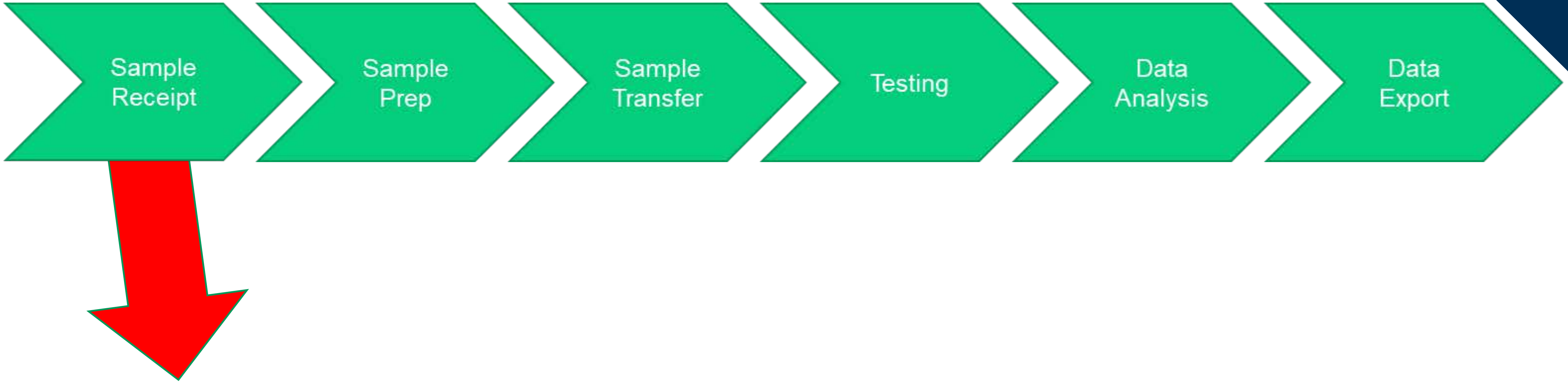
Sample Prep
- Non active.
- Low activity glovebox.
- High activity Hot cell.
- Gamma spectrometry.
- Dose rate.
- Sample cutting, mounting and polishing facilities.
- Active sputter coater.
- Active broad beam ion mill.
- Active TEM foil ion mill.

MATERIALS RESEARCH FACILITY

Materials Research Facility Active Sample Requirements

MATERIALS RESEARCH FACILITY

MRF Procedure



- Prior to sample receipt information required on:
  - Who you are.
  - What you want to do.
  - What do you want to do it to.
    - Subject to UKAEA Waste and health physics approval.

MRF - Customer Requirement Form

- First contact with the MRF experimental team.

UK Atomic Energy Authority

MATERIALS RESEARCH FACILITY

MRF Form	MRF-FOR-0002
MRF Job Requirements	Version: 5.0 Date: 26/05/2022 18:02:00 Approver: London, Andy

Materials Research Facility Enquiry reference	

Section 1	**Customer Information**
Company Name	
Address 1	
Address 2	
Address 3	
Town/City	
County	
Postcode	
Company Registration No	
Company VAT number	
Contact name for enquiry	
Contact email	
Email for invoices	
Accounts contact	

MRF - Customer Requirement Form

UK Atomic Energy Authority

- Section 3.
  - What you want to do.
- Section 2.
  - What you want to do it too.

Section 3	Scientific Evaluation Information
Sample prep requirements	
Required analysis	
Any specific sample storage requirements?	
Test Objectives	
Proposed timescales	
MRF or Customer to carry out analysis?	
Approval received from material owner to carry out analysis?	
IP/Data requirements	

Section 2	Material Information	
Material Composition		
Number of samples		
Sample size(s) and weights		
Sample geometry? (attach photographs if possible)		
Identified risks under COSHH		
Is the material radioactive?	Yes ☐ No ☐	If yes, please complete Section 5

MATERIALS RESEARCH FACILITY

MRF – Radioactive Sample information requirements

UK Atomic Energy Authority

- Section 4.
  - Transport courier details.
- Section 5 of the MRF Customer Requirements form.
  - Derived from UKAEA processes
- Required for all potentially active samples.
  - Includes proton and ion beam irradiations
- Dose rate.
- Assessment of contamination.
- Radionuclide fingerprint.

Section 4	Transport Information
Current sample location	
Transport contact name	
Transport contact email address	
Sample to be returned following analysis?	

Section 5	Radioactive Sample Information (See Appendix A for further requirements)
Radionuclide Fingerprint Per sample and consignment	
Reference date for Radionuclide Fingerprint	
How was the radionuclide fingerprint obtained?	
Total Activity (Bq)	
Maximum beta dose rate (µSv/h) at 30cm	
Maximum gamma dose rate (µSv/h) at 30cm	
Presence of nuclear material declaration	
Estimate and nature of any loose contamination	
How was the contamination assessed?	

MATERIALS RESEARCH FACILITY

MRF – Radioactive Sample information requirements

- Radionuclide fingerprint required along with source and date obtained.
  - Required for all samples that may be activated.
    - Includes all irradiations.
  - Required for work planning and waste management purposes.
  - May be empirical e.g gamma spec
  - Or calculated e.g FISPACT
  - Composition of the radioactivity declared down to a constituent specific activity of 0.001Bq/g.
  - **Willingness to accept will not be given without this information.**

Radionuclides	
H-3	Ra-226/Rn-222/Po-218/At-218/Pb-214/Bi-214/Po-214/Tl-210/Pb-210/Bi-210/Po-210
C-14	Ac-227
Cl-36	Th-229/Ra-225/Ac-225/Fr-221/Ra-221/Rn-217/At-217/Bi-213/Po-213/Tl-209/Pb-209
Fe-55	Th-230
Co-60	Th-232/Ra-228/Th-228/Ra-224/Rn-220/Po-216/Pb-212/Po-212/Tl-208
Ni-63	Pa-231
Sr-90/Y-90	U-232
Nb-94	U-233
Tc-99	U-234
Ru-106/Rh-106	U-235/Th-231
Ag-108m	U-236
Sb-125/Sb-126	U-238/Th-234/Pa-234m/Pa-234
I-129	Np-237/Pa-233
Ba-133	Pu-238
Cs-134	Pu-239/U-235m
Cs-137/Ba-137m	Pu-240
Pm-147	Pu-241
Eu-152	Pu-242
Eu-154	Am-241
Eu-155	Cm-243
Pb-210/Bi-210/Po-210	Cm-244
Ra-228/ Ac-228	

UK Atomic Energy Authority

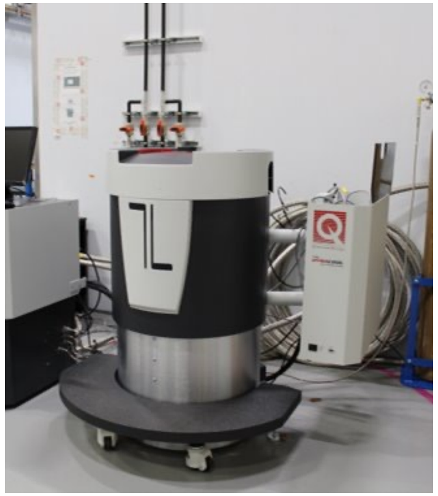
MATERIALS RESEARCH FACILITY

Past MRF Use

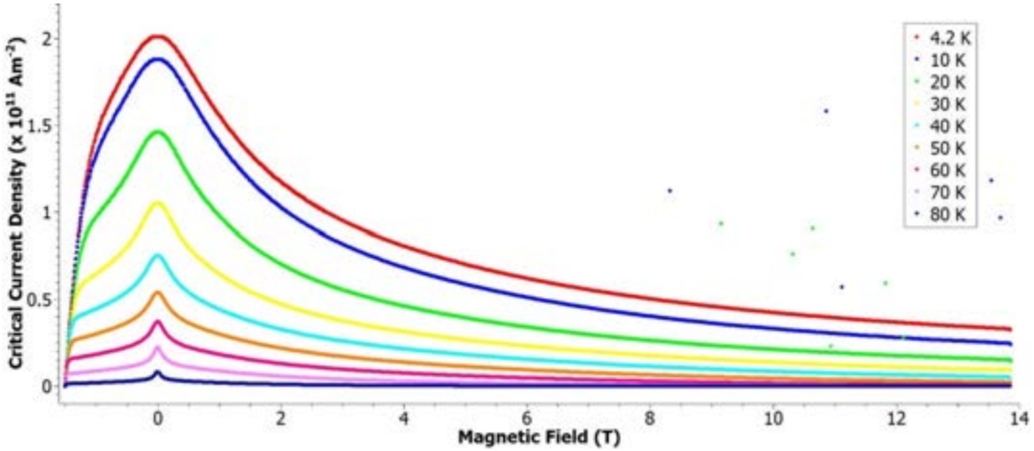
UK Atomic Energy Authority

MATERIALS RESEARCH FACILITY

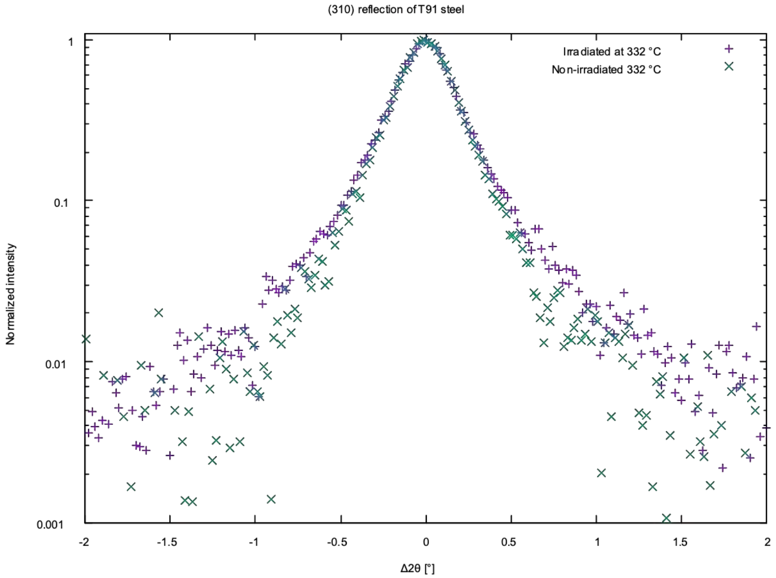
PPMS and XRD



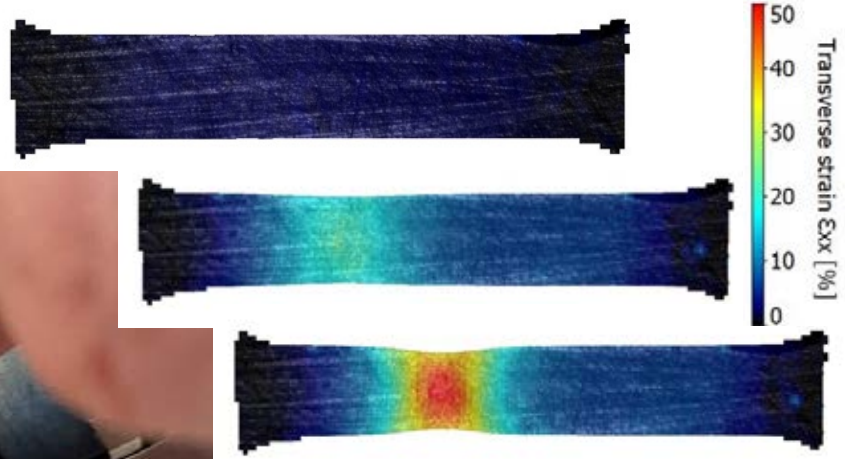
Physical Properties Measurement System Instrumentation specifically intended for the characterisation of superconductors, plots for critical current versus applied field for 4.2K<T<80K shown although can be used on other materials.



Xray Diffractometer
High brightness
Rotating anode source diffractometer optimised for the analysis of diffraction line broadening and defect characterisation of irradiation damage. Image shows broadening of the (310) reflection in a T91 steel.

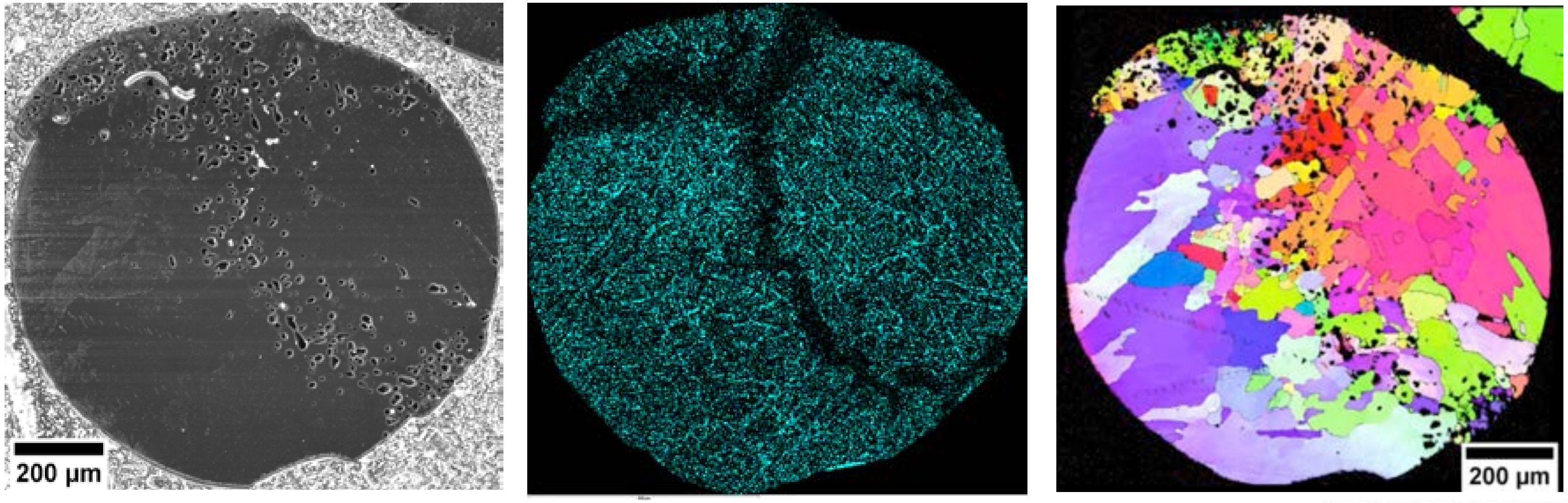


Small Sample testing
SSJ3 sample geometry

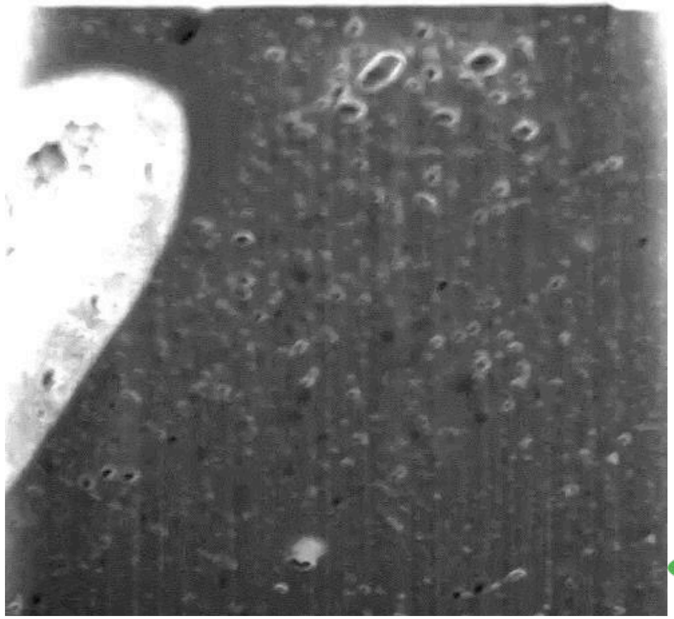


- 5kN load frame.
- -150°C to 550°C capability.
- Hi Vac or inert atmosphere.
- DIC equipped.

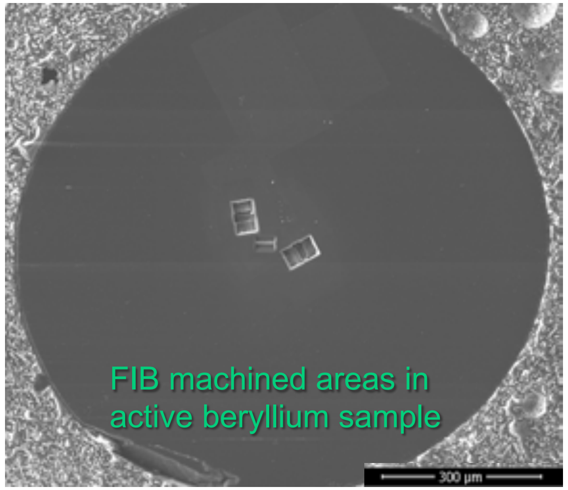
SEM characterisation
Neutron irradiated Be Pebbles microstructure

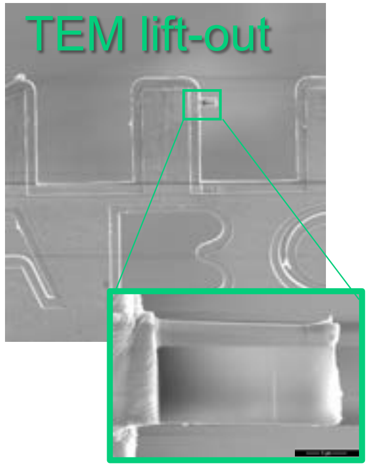


- Active Be samples.
- Correlative SEM, EDS (Al) and EBSD.
- Generated large pore structure associated with low Al high angle grain boundaries
- FIB tomography shows fine He bubble formation in grain bulk



Sample Volume Reduction

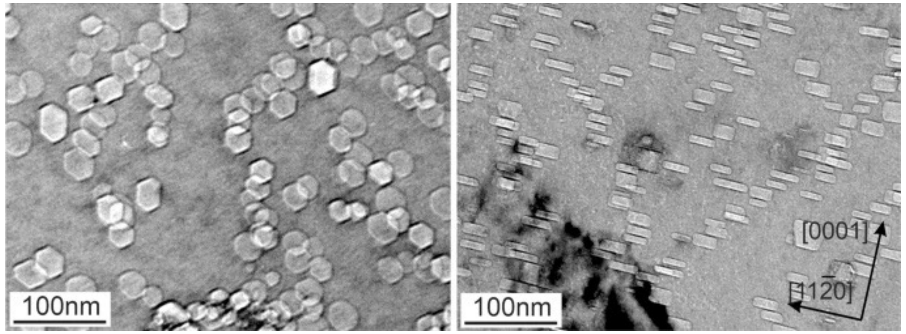




15 um

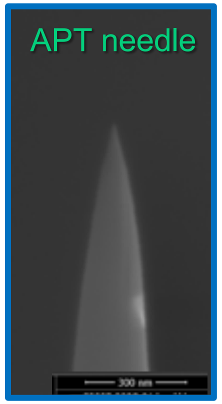
Collaboration with KIT (Germany)

n-irradiation, 480°C, 28 dpa

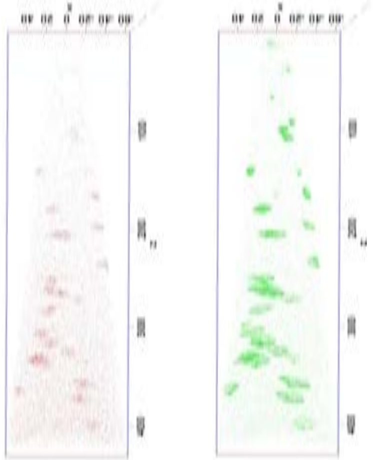


Klimenkov et al. Scientific Reports, 2020

Collaboration with Uni. Oxford



<1 um



3 Da (T?)

12 Da (Be-T complex?)

Kuksenko et al. to be submitted

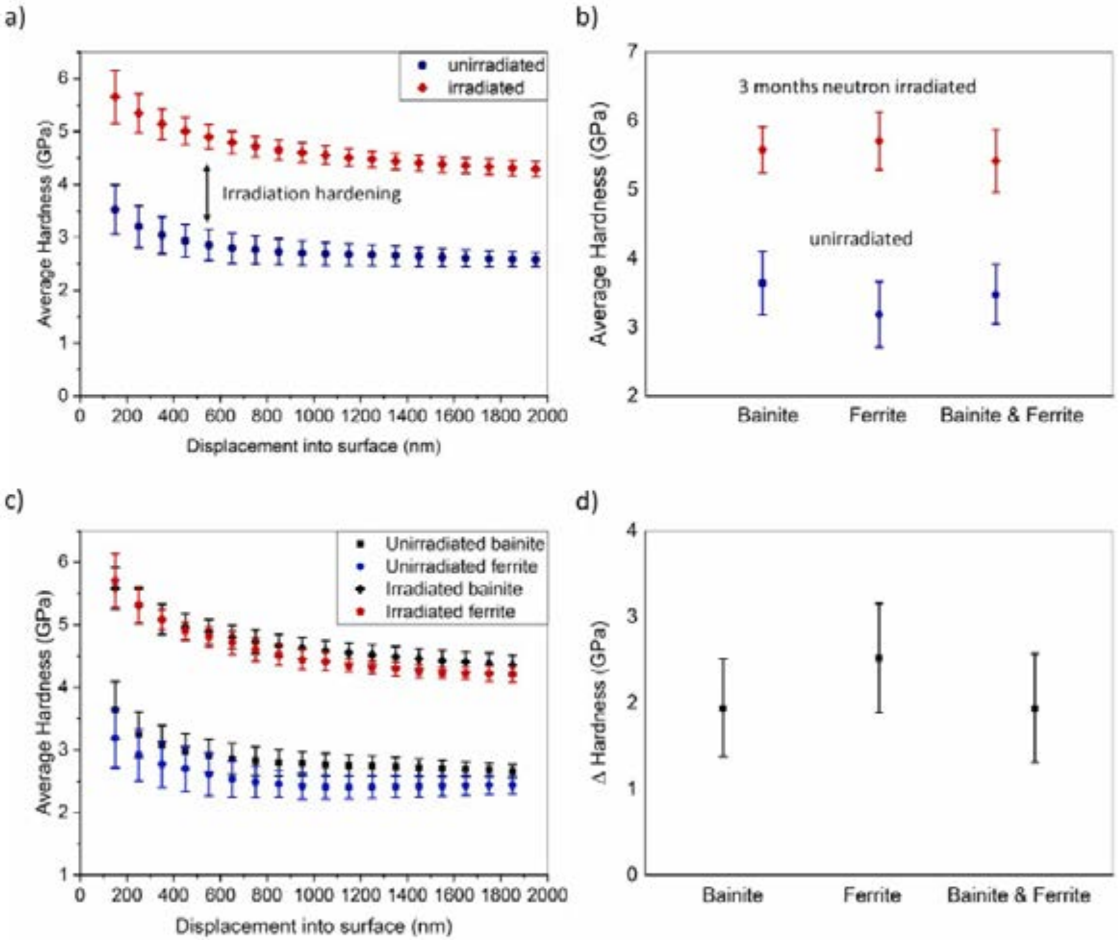
- FIB lift-out technique decreases **activity to < 1Bq per sample** and can be easily transported and investigated in non-nuclear laboratories
- Combination of TEM (KIT, Germany) and Atom Probe (Oxford) gives insights on transmutant helium and tritium distributions in highly irradiated beryllium

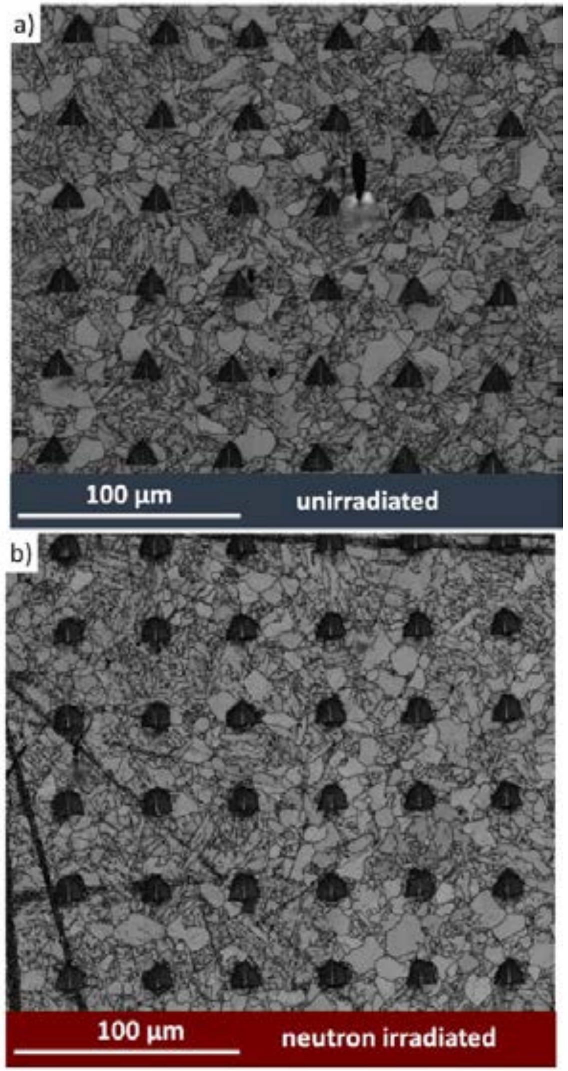
UK Atomic Energy Authority

MATERIALS RESEARCH FACILITY

Microscopy & Nanoindentation

On the influence of microstructure on the irradiation response of HIPed SA508 steel for Nuclear applications.
Carter et al.
Journal of Nuclear Materials 559 (2022)153435



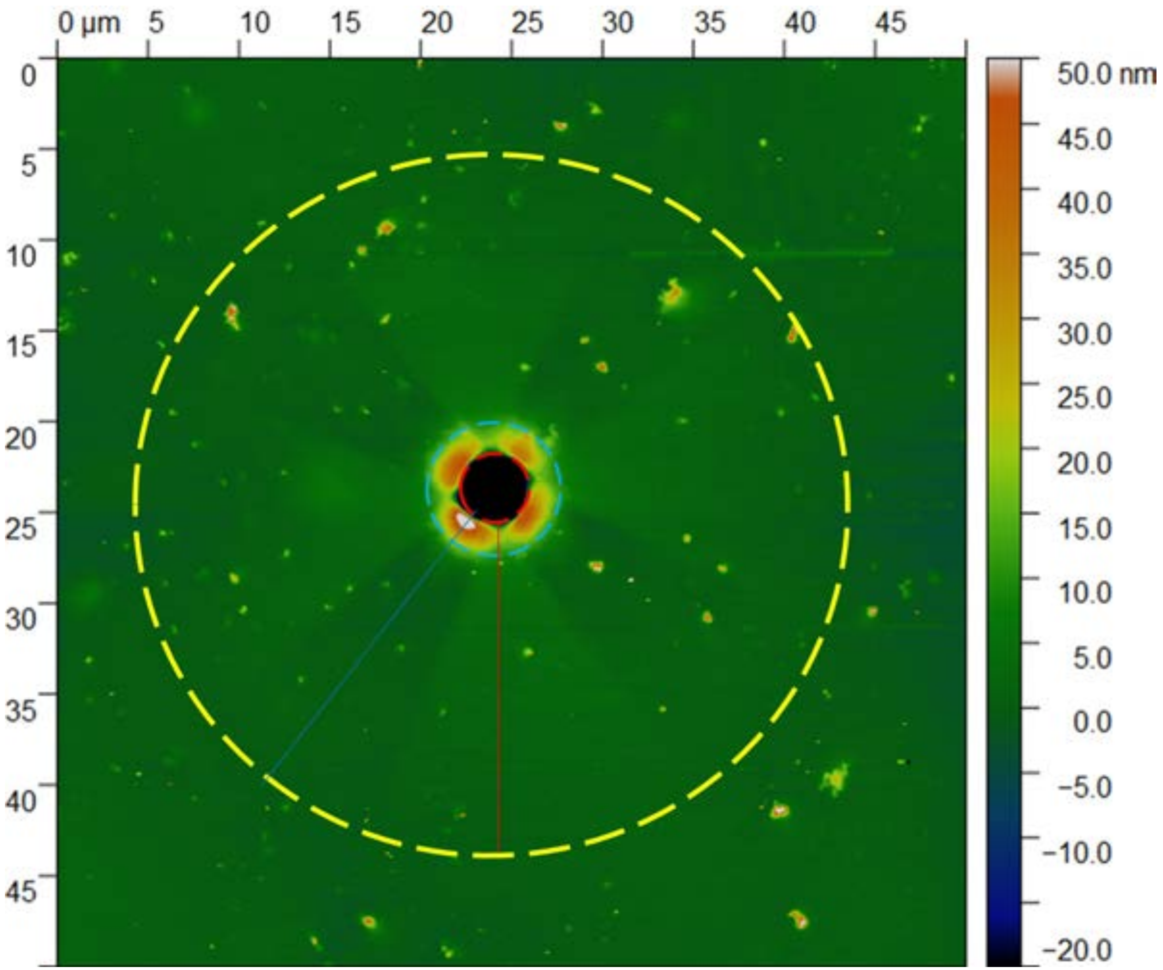


UK Atomic Energy Authority

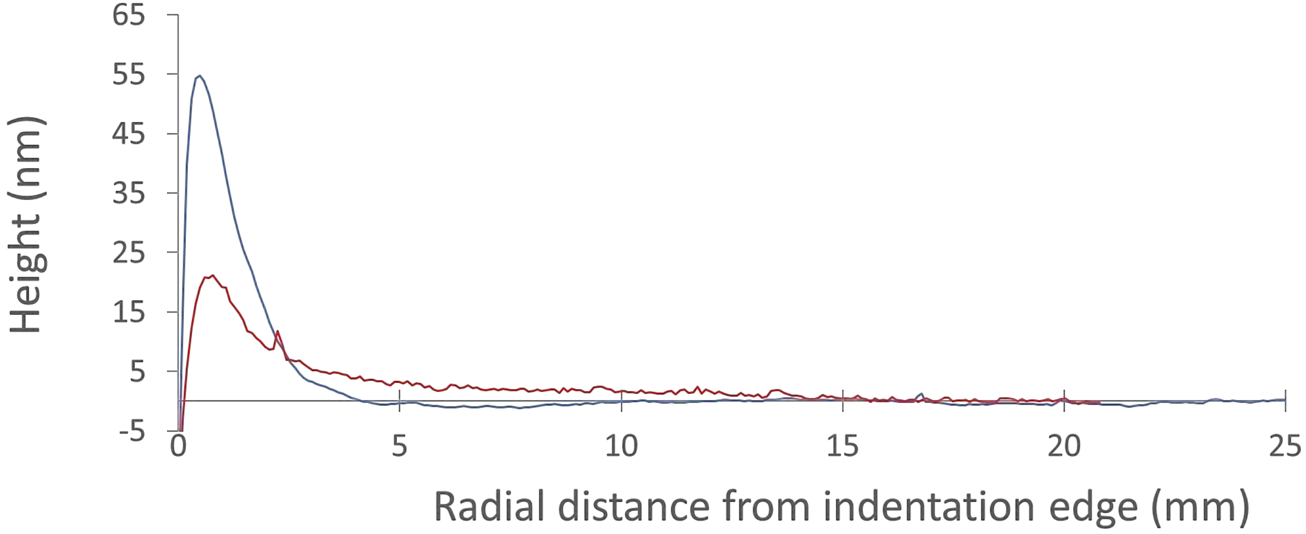
MATERIALS RESEARCH FACILITY

Official - Sensitive - Commercial

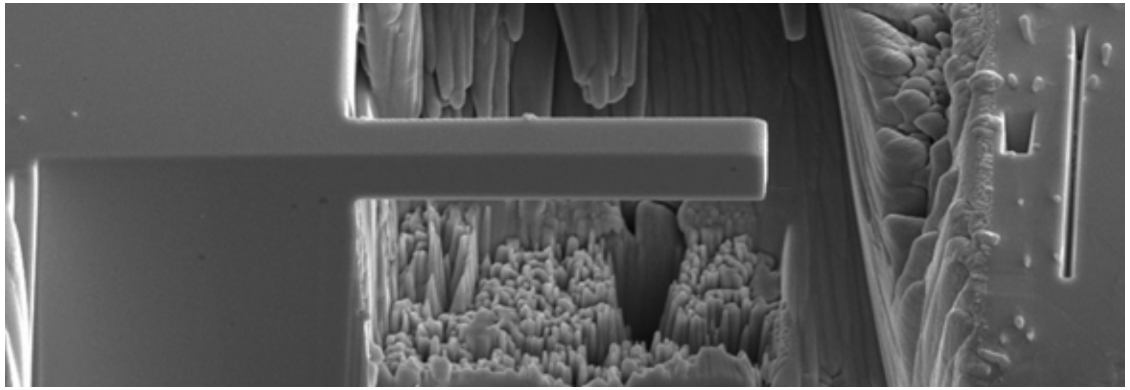
AFM - Nano Indentation Pile Up Characterisation



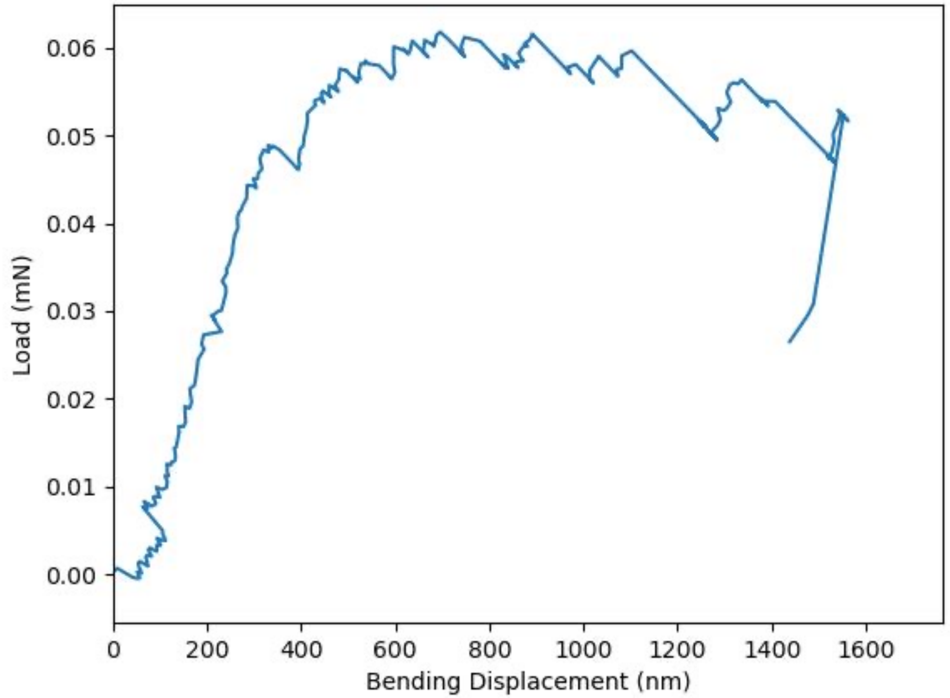
- AFM image of pile up around a spherical indentation in single crystal Tungsten.
- Crystallographic anisotropy evident.

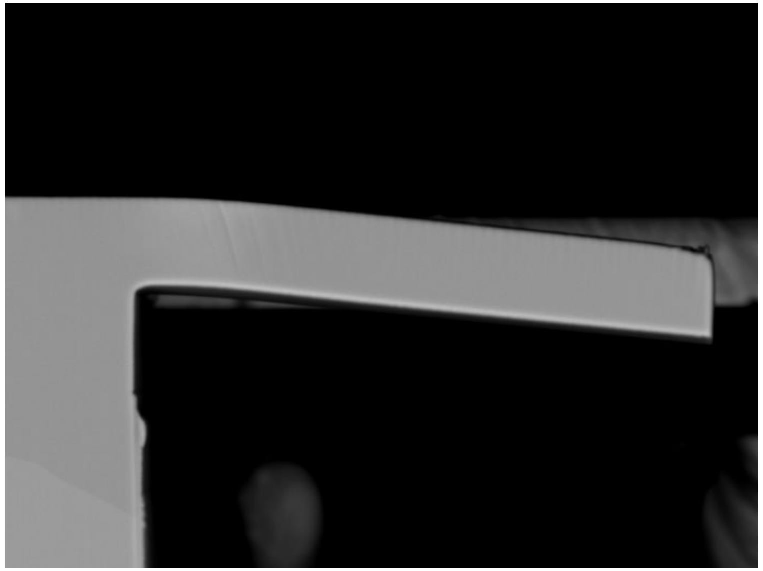


Micromechanics



- FIB micromechanics fabrication.
  - EBSD grain selection
- In-situ testing capability (June 2023).
- G200 nano indenter with nano positioning stage ex-situ.





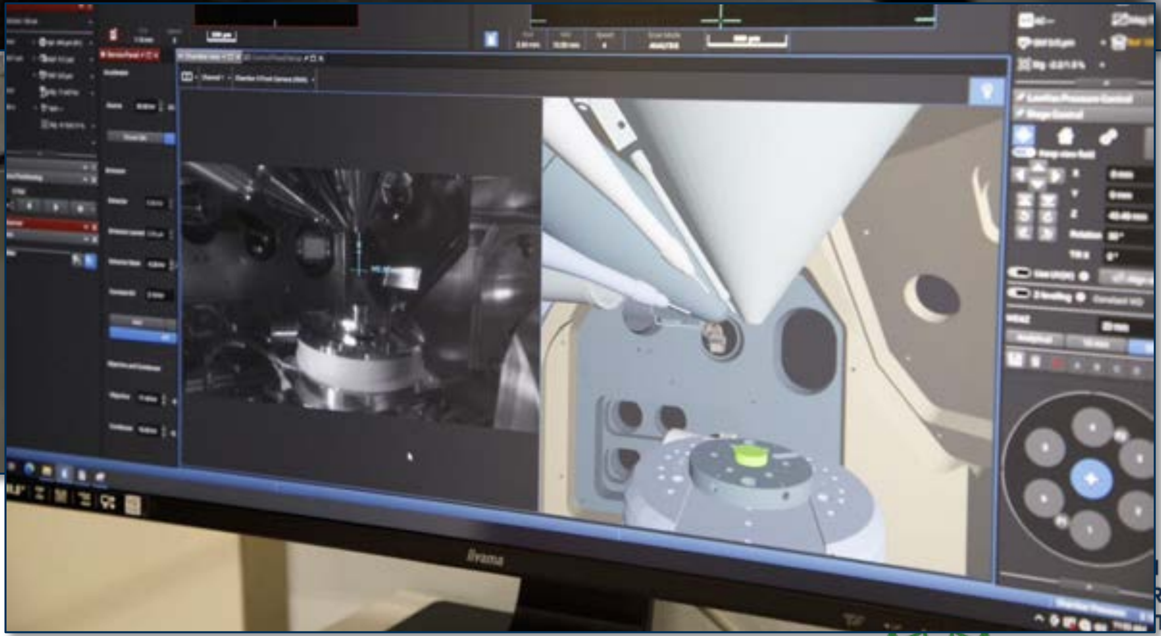
- FoV 18μm

The Future

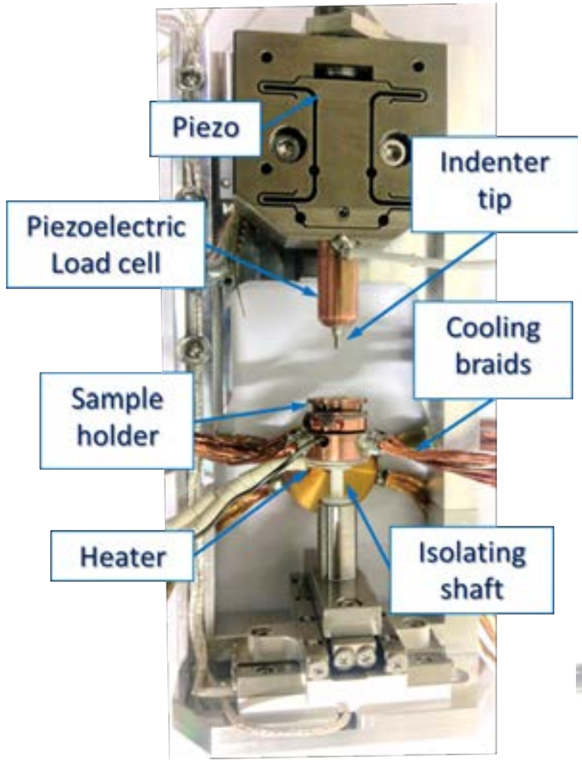
UK Atomic
Energy
Authority

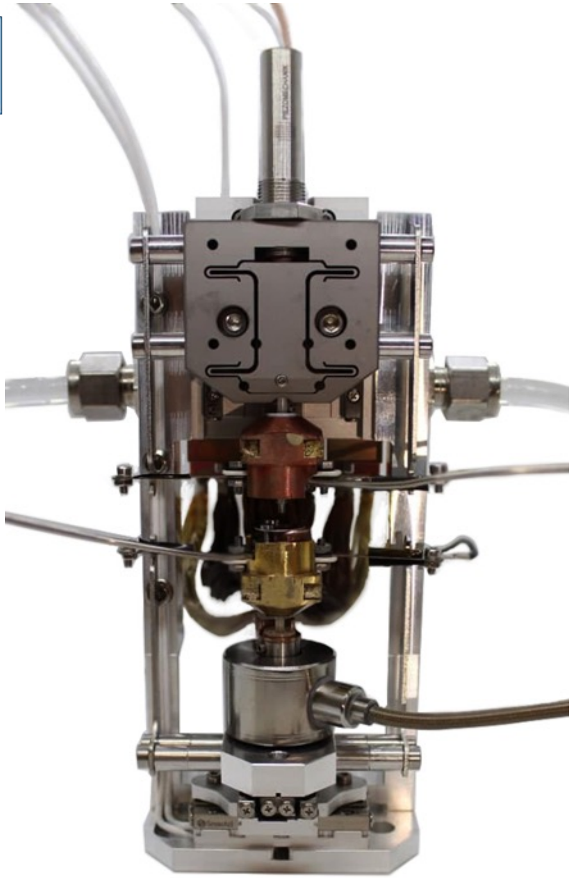
MATERIALS
RESEARCH
FACILITY

NEW TESCAN AMBER X – Xe Plasma FIB



High and low temperature In Situ tester





Fitted to MRF PFIB and SEM

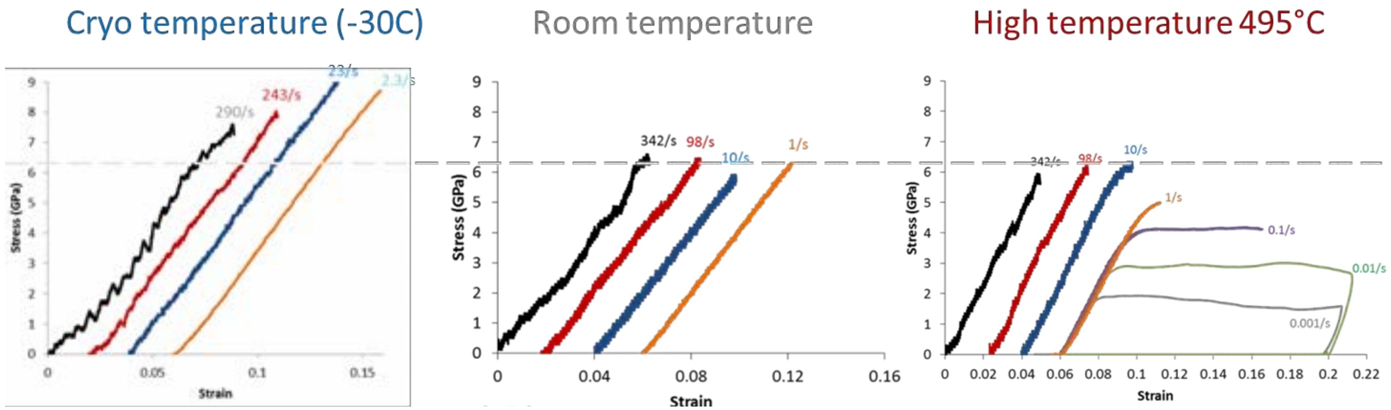
Potential additional hi vacuum chamber

In situ high and low T nano indenter.

-150°C to 1000°C

Offers multiple test modes:

Indentation, compression, tensile, scratch etc.



Ductile to brittle transition in Si

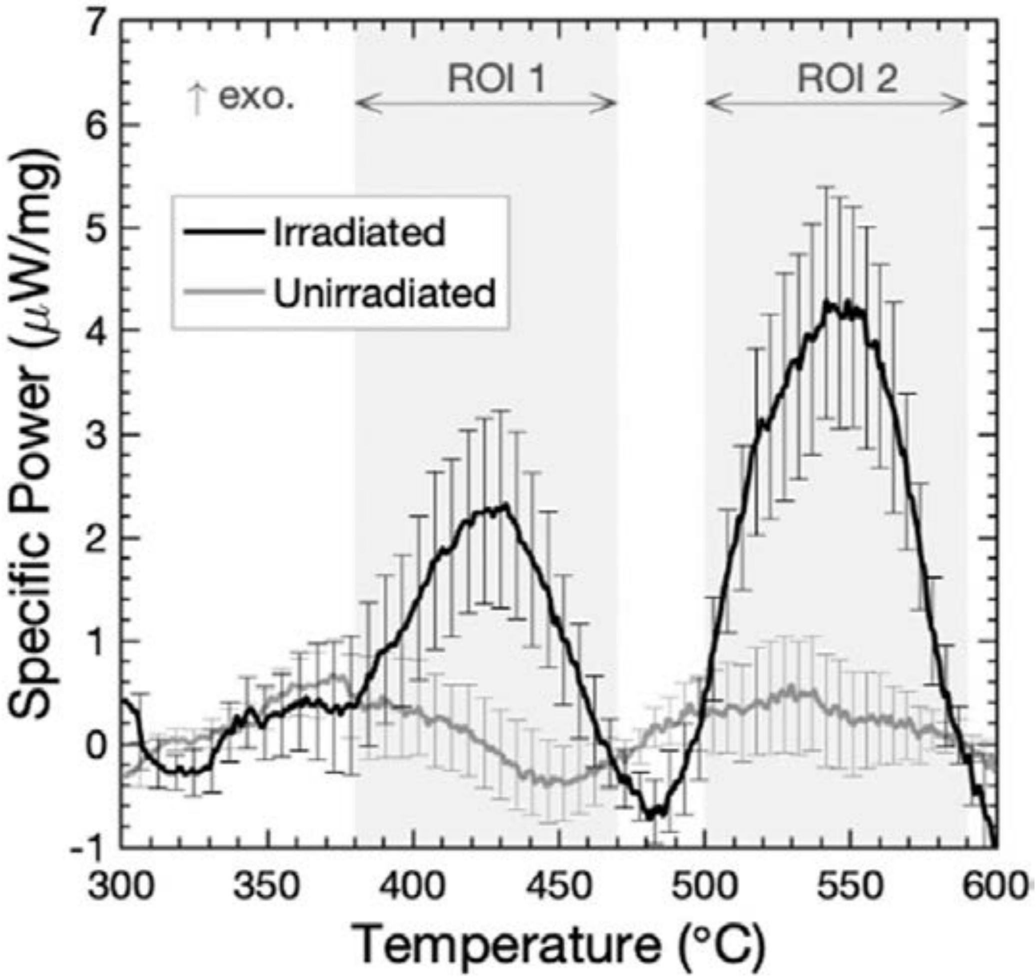
Si (6.82μm dia ~29μm height) micropillar testing

Courtesy of Raj Ramachandramoorthy et al., in press

UK Atomic Energy Authority

MATERIALS RESEARCH FACILITY

Future Active DSC



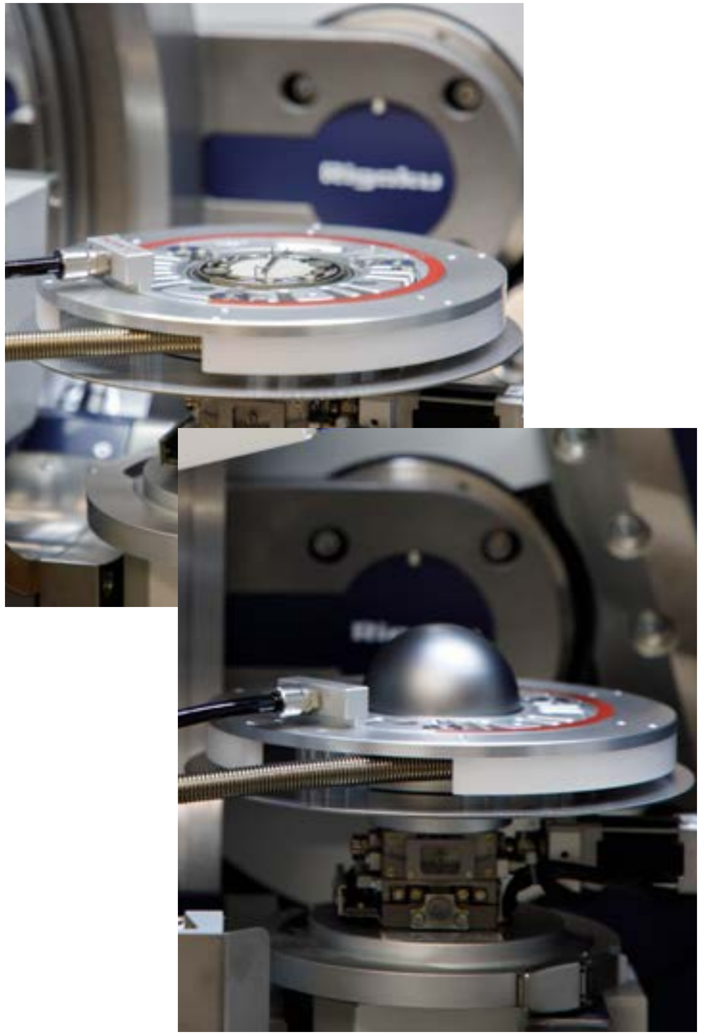
Graph showing the specific power difference between the firs
neutron-irradiated Ti sample and the mean subsequent of 2 to
5 annealing cycles.
From "Quantifying radiation damage through stored energy released during defect annealing in metals" Hirst 2015.

- Vacuum tight at 10^{-4}mbar
- High precision Pt Furnace
    - 25-1500°C
- High T SiC furnace with Mass spec 1-300amu

UK Atomic Energy Authority

MATERIALS RESEARCH FACILITY

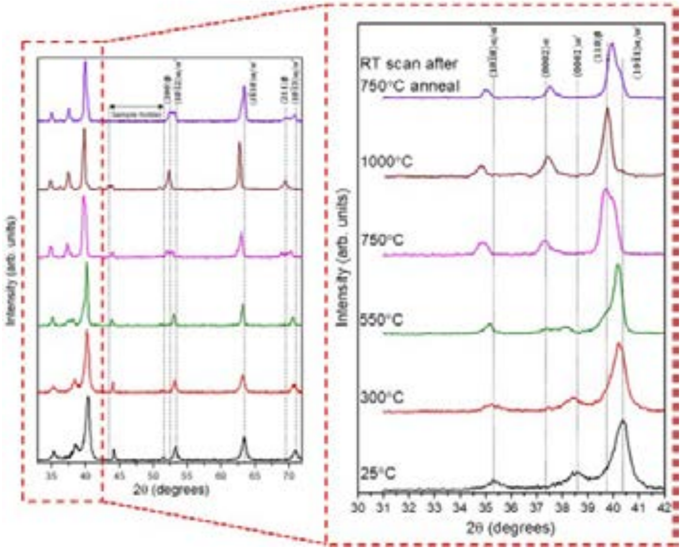
DHS 1100 Hot stage

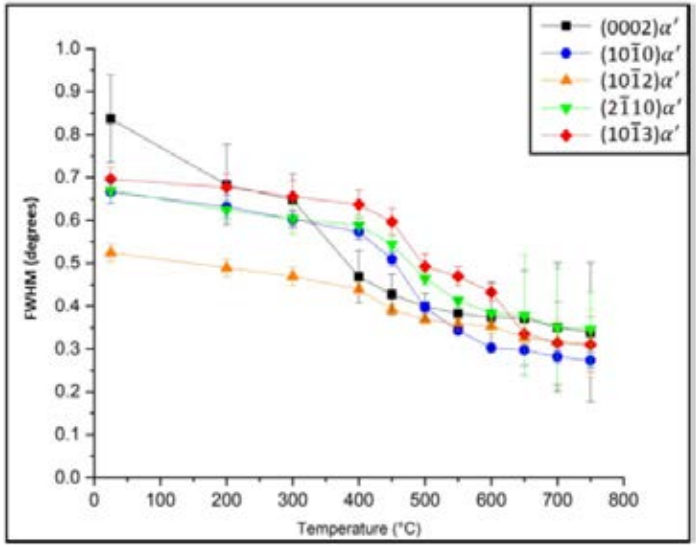


- Ambient to 1050°C
- Air, vacuum (0.1 mbar), nitrogen, or helium atmospheres
- Allows a full (±180°) rotation of the φ axis and use of in-plane arm

→ Suitable for stress, texture

- 10 mm to 25 mm sample diameter
- Max. 2 mm sample thickness





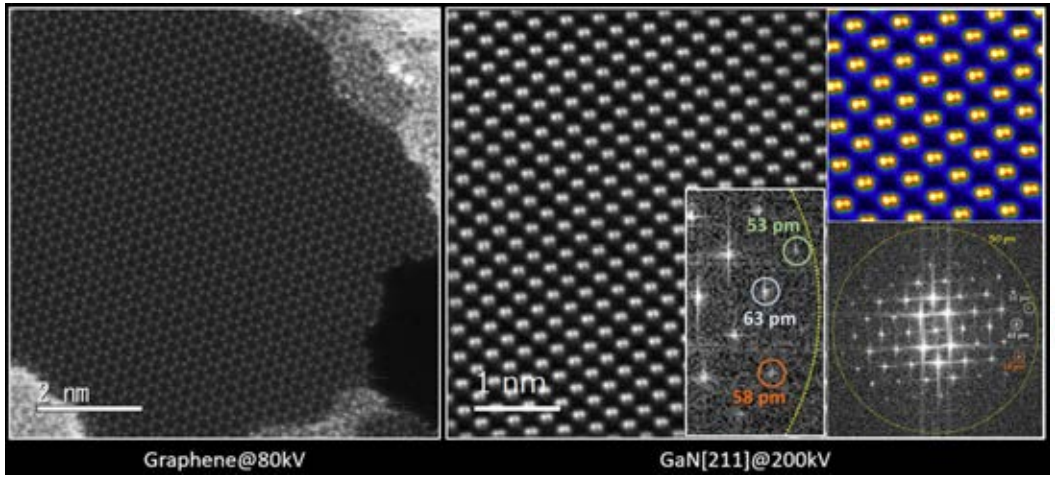
Kaschel et al: *Mechanism of stress relaxation and phase transformation in additively manufactured Ti-6Al-4V via in situ high temperature XRD and TEM analyses*, Acta Mat 2020+

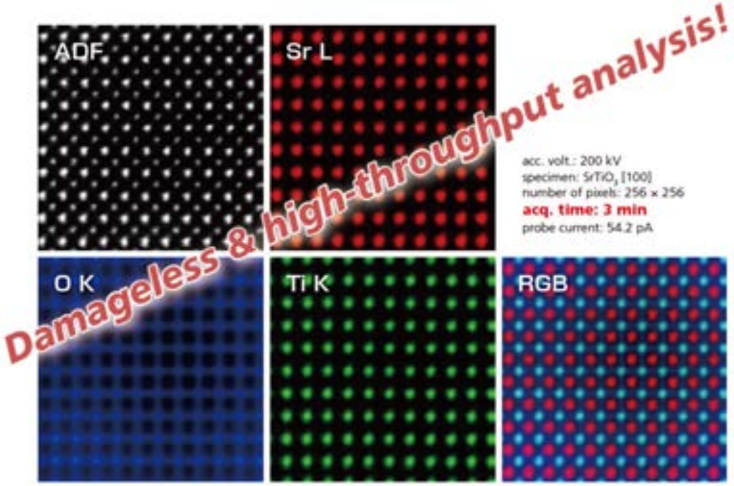
UK Atomic Energy Authority

MATERIALS RESEARCH FACILITY

Future Active TEM

- JEOL JEM ARM200F NEOARM from mid 2023.
  - Aberration corrected atomic resolution TEM.



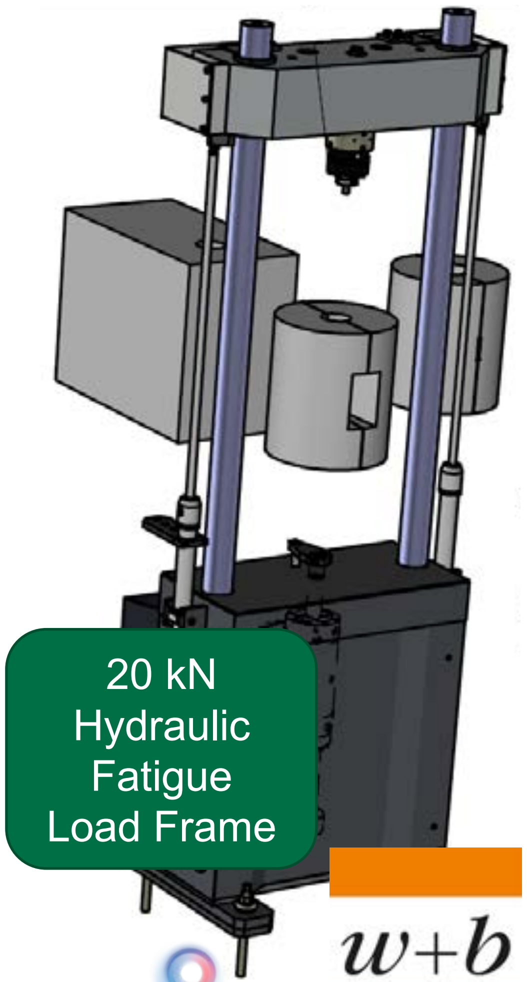


- Insitu environmental reaction cell.
- 1mbar to 1 bar pressure range.
- T control ~1000°C/s
- Mass spec to measure outgassing

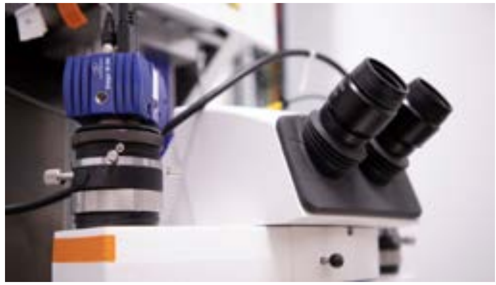
Hot Cell Extension – 2 new cells (2025)

UK Atomic Energy Authority

DWE Scientific Ltd

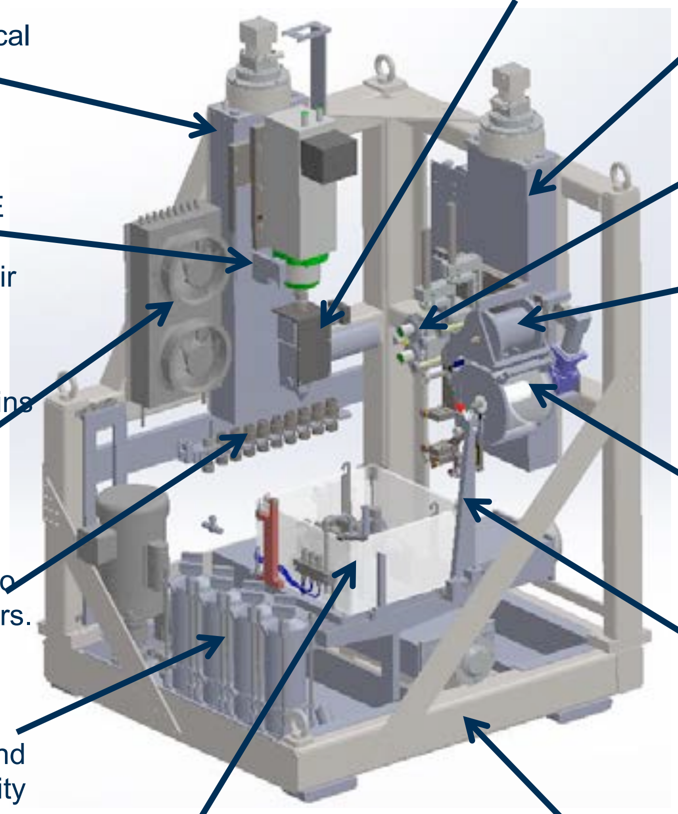


DIC System



w+b

severn thermal solutions



Microcutting Laser Head. Loaded into milling spindle for vertical movement.

Z-Axis. 400 mm vertical travel. Holds milling spindle.

Milling Spindle 5kW, 24,000 rpm, HSK-32E tool holder interface, pneumatic drawbar, air cooled.

Water Cooler. Maintains temperature of deionized water.

Tool Rack. Holds up to 10 HSK32E tool holders.

Filtration system. Removes particles and ions to maintain quality of deionized water.

Work Tank 303 (w) x 306 (d) x 165 (h) mm. Mounted on XY-stage with 400 x 400 mm travel.

W-Axis. 400 mm travel. Holds WEDM head.

WEDM Head with vertical and horizontal wire section.

Supply Spool. Standard spool loaded outside the cell into a frame that can be handled with the manipulator.

Takeup Spool. Loaded outside the cell into a frame that can be handled with the manipulator. Used to wind up the spent wire.

Wire Threader (shown in the active position). Used to automatically thread the wire from the supply spool around every element of the wire guiding system back to the takeup spool.

Welded Steel Frame filled with anti-vibration insert. 940 (w) x 960 (d) x 1250 (h) mm.

Viteris Technologies

MATERIALS RESEARCH FACILITY

Access Schemes

UK Atomic Energy Authority

MATERIALS RESEARCH FACILITY

Access arrangements

- MRF operates as a single integrated user facility for characterisation of active materials.
- Funding Routes:
  - NNUF (till early 2024 at present)
  https://www.nnuf.ac.uk/calls-access
  - Sir Henry Royce Institute researcher and SME facility access.
  https://www.royce.ac.uk/access-schemes/
  - Fusion Industry Partnership (FIP) Voucher.
    - £50k voucher to access UKAEA facilities for UK industry
- Support for grant applications via EPSRC and other funding bodies.
- Commercial user access.

email
info@mrf.ukaea.uk
Royce@mrf.ukaea.uk

UK Atomic Energy Authority

MATERIALS RESEARCH FACILITY