UK Atomic Energy Authority

UKAEA MRF

RaDIATE - UK Update

Phil Earp, Equipment Scientist, UKAEA

26th – 30th June 2023, Brookhaven National Laboratory, United States

HENRY ROYCE INSTITUTE

NNUF National Nuclear User Facility

EPSRC Engineering and Physical Sciences Research Council

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

MATERIALS RESEARCH FACILITY

Overview

UK Atomic Energy Authority

STFC

- STFC Status Update
- Titanium Irradiation Sample Environment

University of Oxford

- Meso-scale Mechanical Testing

University of Bristol

- Thermal Conductivity Measurement

UKAEA Materials Research Facility

- Ultrasonic Fatigue Update
- NNUF2a Investment

Dr Phil Earp
Equipment Scientist (Mechanical Testing)
Materials Research Facility
UK Atomic Energy Authority
philip.earp@ukaea.uk

MATERIALS RESEARCH FACILITY

RaDIATE
STFC status

Chris Densham

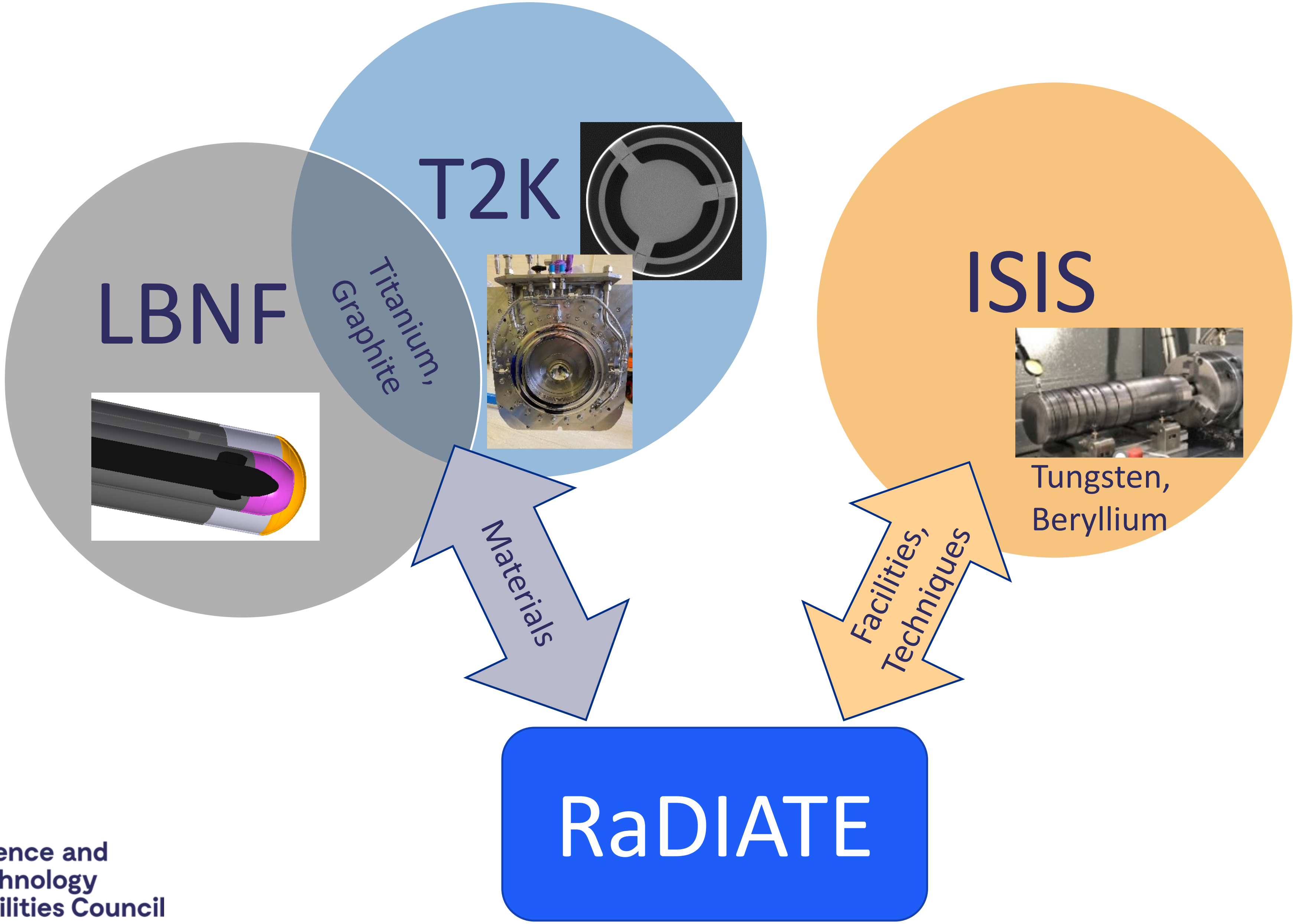
for T2K-UK and LBNF-UK project teams and ISIS at STFC Rutherford Appleton Laboratory

Science and Technology Facilities Council

WARWICK
THE UNIVERSITY OF WARWICK

DUNE
DEEP UNDERGROUND NEUTRINO EXPERIMENT

Overlapping Interests in RaDIATE for STFC Projects and Facilities



Science and
Technology
Facilities Council

STFC support and coordination of target & window materials research

Contributions to RaDIATE materials science program

- Initial funding via HyperK-UK project
  - Kick-started academic materials science projects
- Project now supported by LBNF-UK
  - Supports all outstanding HK-UK activities (ie BLIP irradiated samples)
- Coordinate activities with US-JP collaboration

- RAL (UK coordination)
- ISIS (Bristol study of NuMI graphite on ISIS/ENGIN-X instrument approved)

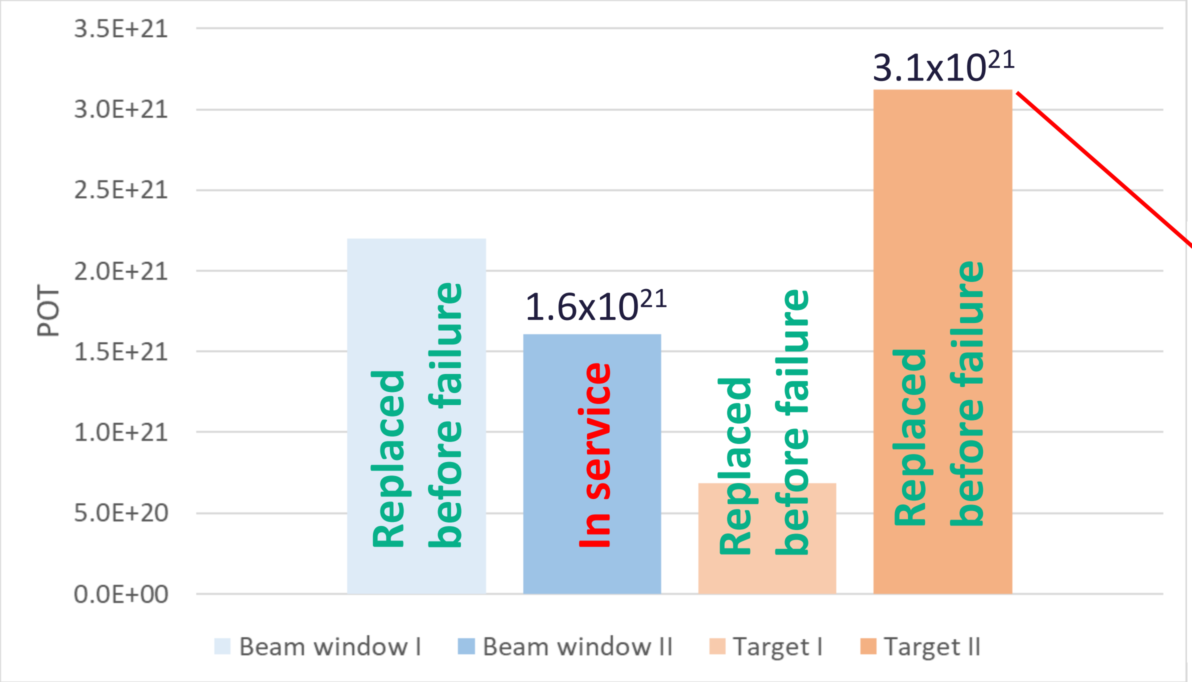
Bristol University (graphite research)

Birmingham University, PDRA + PhD (MC40 cyclotron for sample irradiation, c.30 MeV protons)

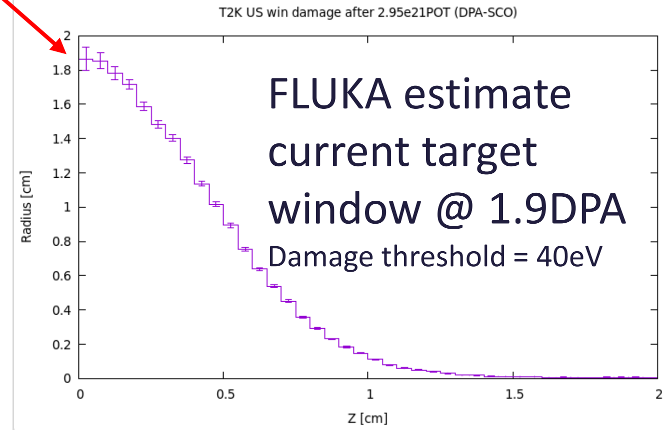
Oxford University PDRA (micromechanics) PhD (titanium - our irradiated samples)

Culham Laboratory (Materials Research Facility for all Post-Irradiation Examination)

Good experience so far on T2K at 500 kW



- How long will a Ti-6Al-4V window/target last?
- Target currently has highest POT ~3 x 10^{21} POT
- C.2 DPA – c.10x BLIP sample data so far(c.0.24 DPA)

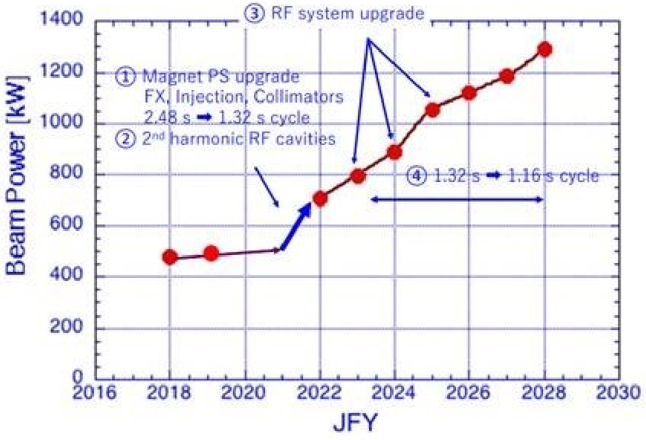


Future plans	PPP	rep rate	current (A)	Beam power (kW)	Run time (mths)	POT/yr
2021	2.64E+14	2.48	1.71E-05	512	4	7.28E+20
2022	2.20E+14	1.32	2.67E-05	801	3	8.55E+20
2023	2.48E+14	1.32	3.01E-05	903	4	1.29E+21
2024	2.24E+14	1.16	3.09E-05	928	4	1.32E+21
2025	2.80E+14	1.16	3.87E-05	1160	2	8.26E+20
2026	2.96E+14	1.16	4.09E-05	1227	4	1.75E+21
HK	3.20E+14	1.16	4.42E-05	1326	6	2.83E+21

Similar DPA/year for LBNF at 1.2 MW

J-PARC

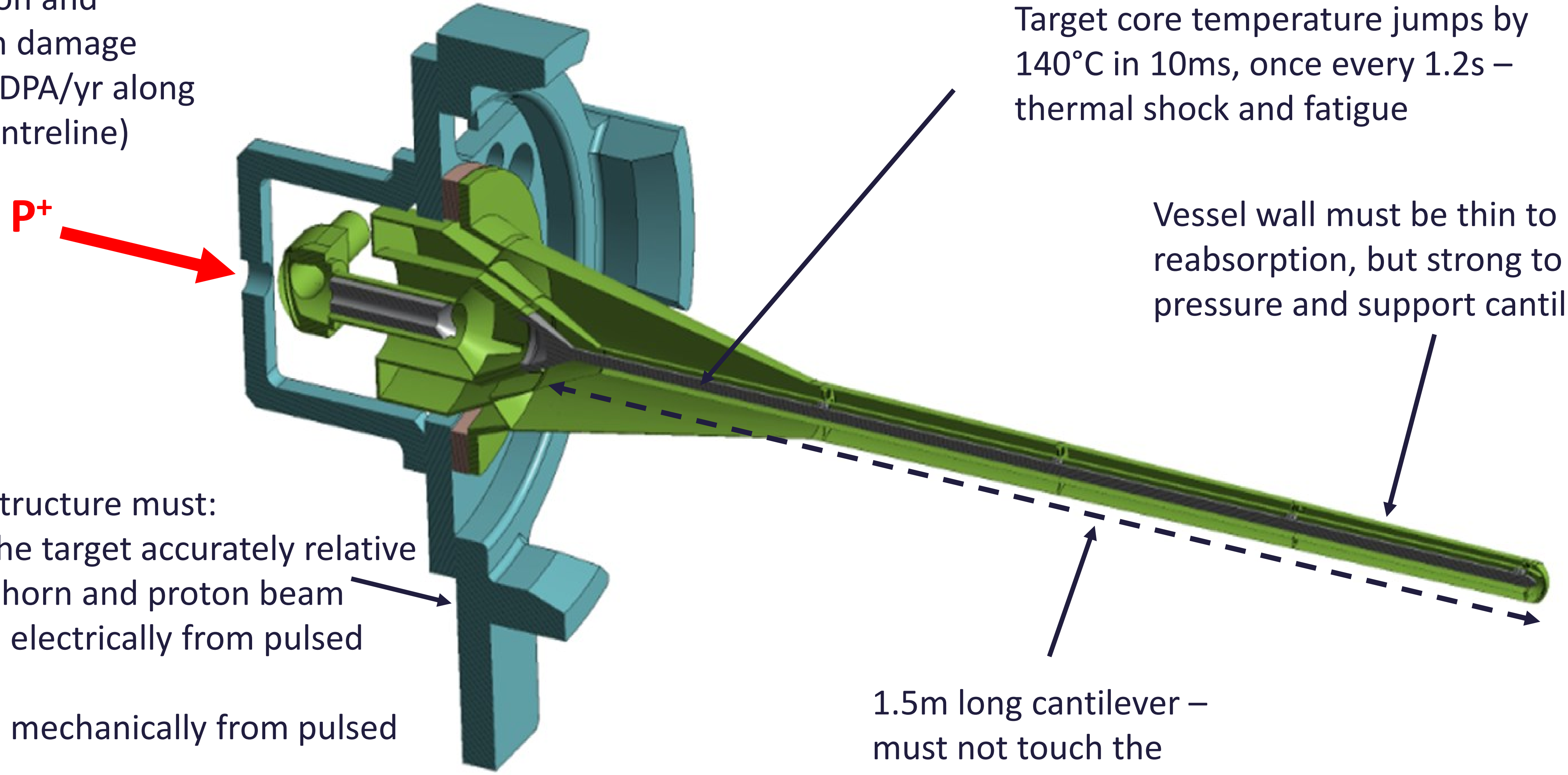
MR FX Beam Power Projection



c/o Mike Fitton

LBNF Target Challenges

Proton beam causes very high heat deposition and radiation damage (several DPA/yr along beam centreline)



Target core temperature jumps by 140°C in 10ms, once every 1.2s – thermal shock and fatigue

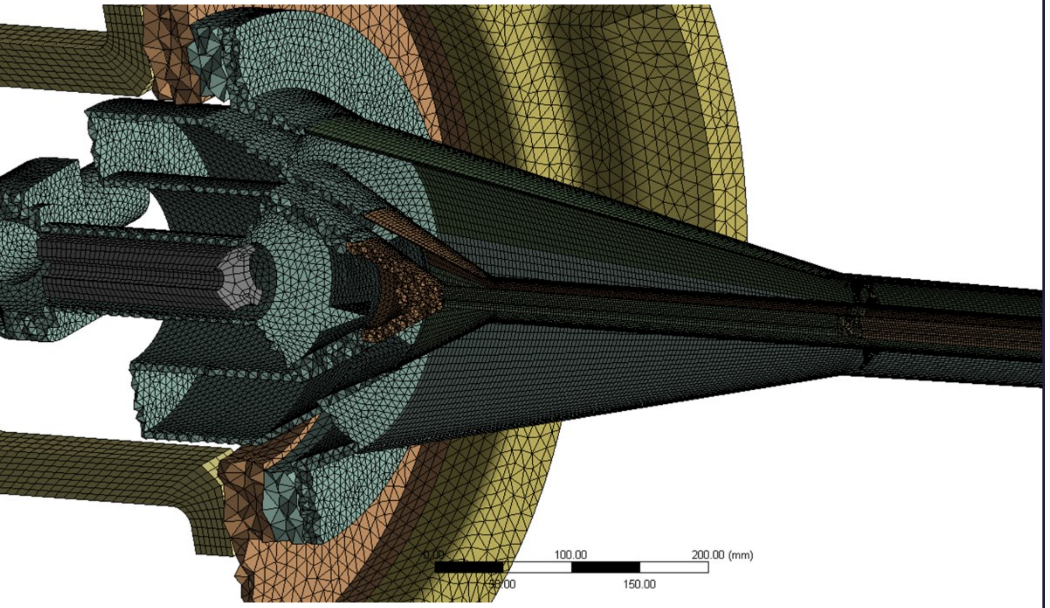
Vessel wall must be thin to prevent pion reabsorption, but strong to contain pressure and support cantilever

Support structure must:
- align the target accurately relative to the horn and proton beam
- Isolate electrically from pulsed horn
- Isolate mechanically from pulsed horn

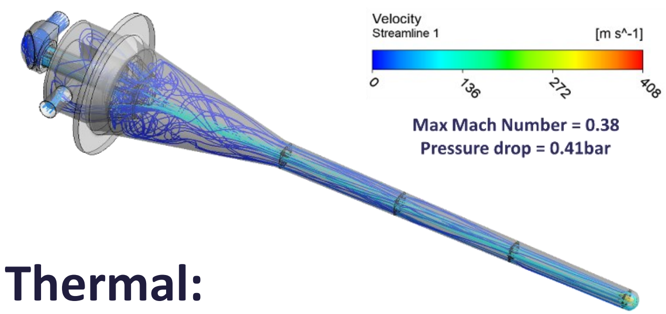
1.5m long cantilever – must not touch the inside of the horn

Detailed Target Analysis for 1.2 MW operation

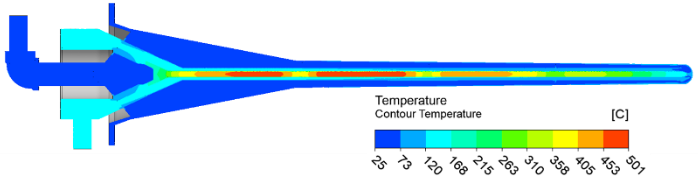
Mesh:



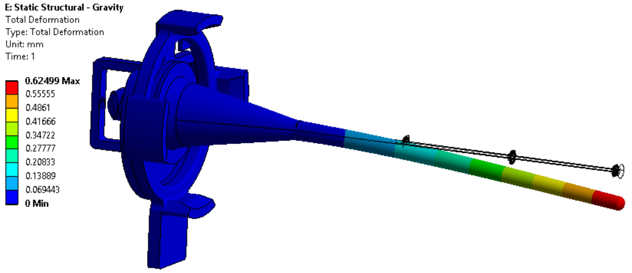
Fluid:



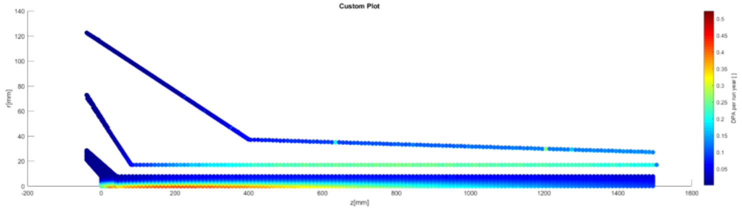
Thermal:



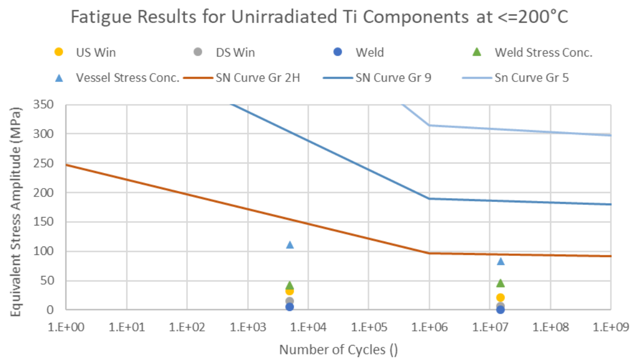
Structural:



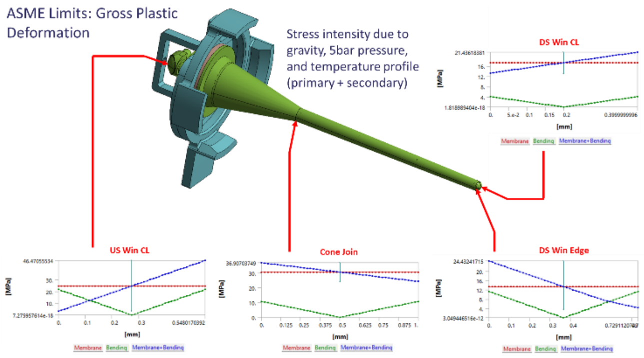
Irradiation:



Fatigue:



Stress Linearisation:



Path to Pressure Vessel Code Approval

(FESHM and ASME BPVC)

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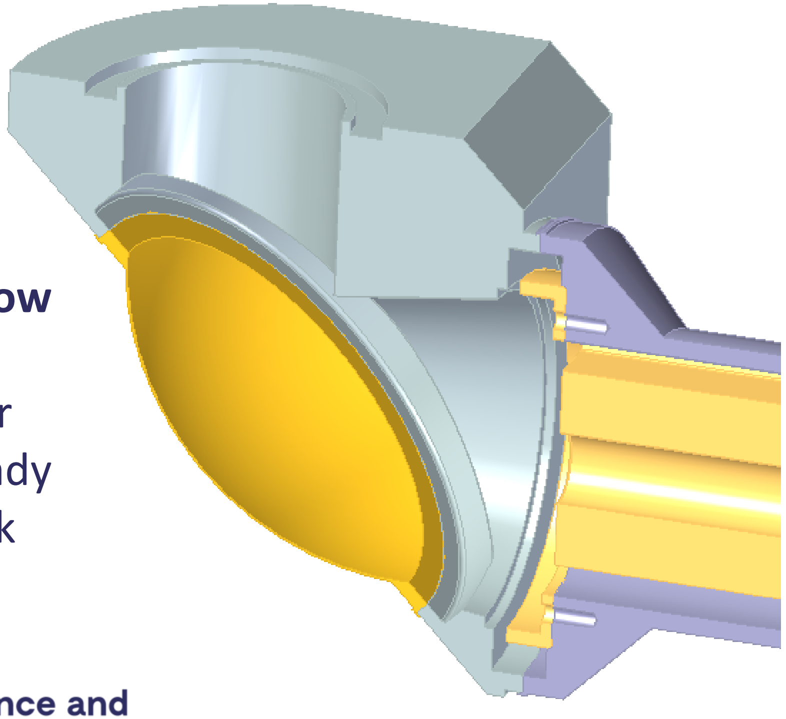
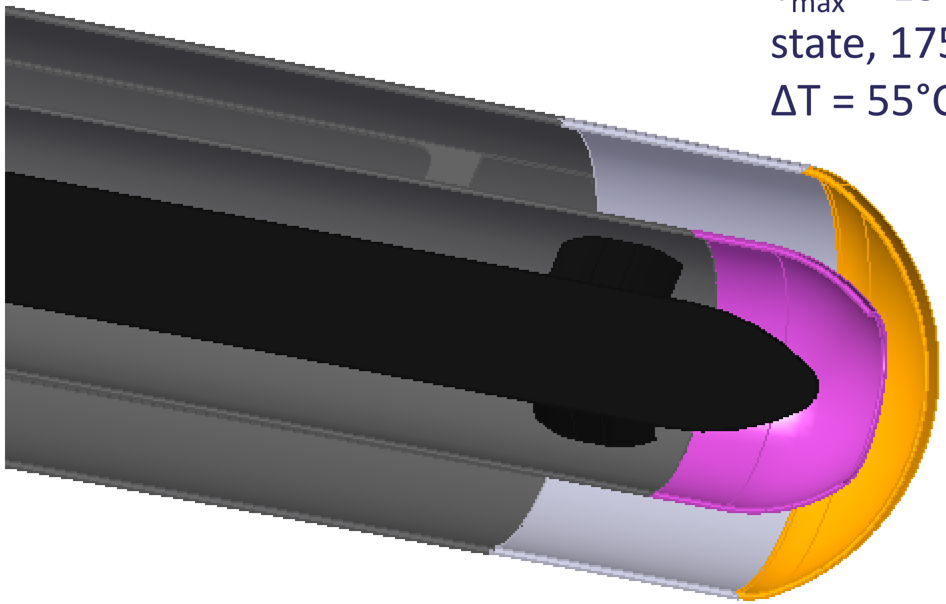
LBNF: Beam Window Ti alloy/grade Selection

- Windows have higher temperature, radiation damage and fatigue than the container
- Available radiation damage data: up to **0.24 dpa**, mostly for grades 5 and 23
- Peak dose: **0.14dpa/yr in container**, **2.5 dpa/yr in upstream window**



Upstream Window
t_{min} = 0.3mm
Dose = 2.5dpa/yr
T_{max} = 115°C steady state, 150°C peak
ΔT = 70°C/pulse



Downstream Window
t_{min} = 0.4mm
Dose = 0.7dpa/yr
T_{max} = 150°C steady state, 175°C peak
ΔT = 55°C/pulse

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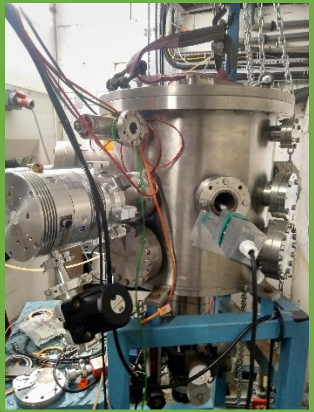
Irradiated titanium fatigue collaboration

Sample & environment



Sample design **(Oxford)**
Sample environment **(RAL & Birmingham)**

Irradiation at MC40 cyclotron (Birmingham)

Proton and He irradiation at high current, low energy

Post-Irradiation Examination (PIE)
(Oxford, Birmingham @Culham)

- Micro/nano indentation
- TEM/XRD
- High cycle fatigue testing

Challenges:
- Maintain sample temperature at 160 °C under high energy proton e.g. 30 MeV/30 µA

Solutions:
- High-flow rate nitrogen cooling of foil samples

Challenges:
- Produce irradiated Ti samples with a high dpa, e.g. 2 dpa;
- He/H production.

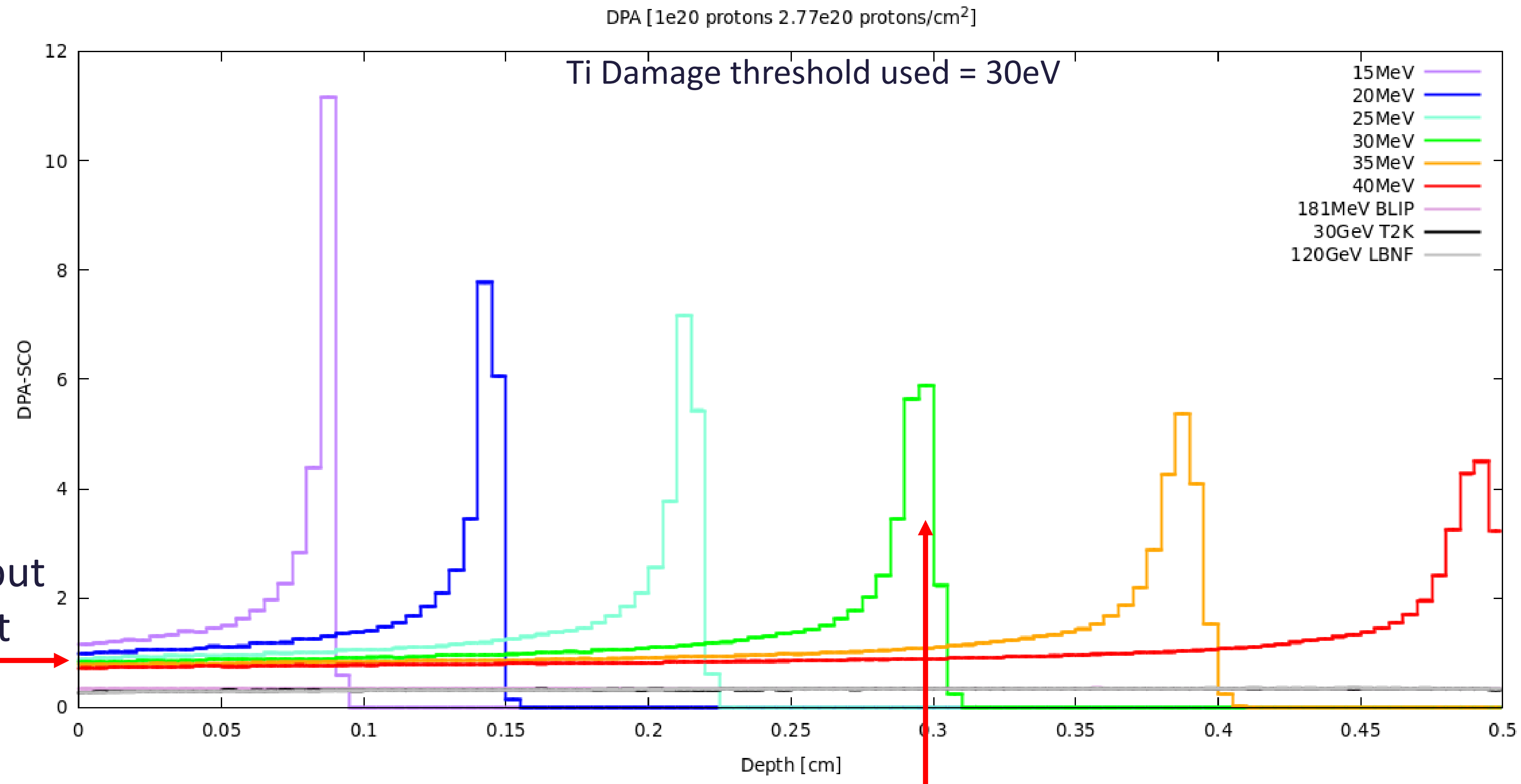
Solutions:
- Long term exposure
- Proton/He implantation
- Vary beam energy

Challenges:
- Handling, mechanical and microstructural testing of active samples.

Solutions:
- MRF has expertise and equipment to handle and test active samples

Irradiation of titanium test samples at Birmingham MC40 cyclotron

Optimum performance of MC40 cyclotron c. 28-30MeV, maximum beam current 30uA



30 days irradiation: ~1DPA in titanium.

Damage is higher at Bragg peak but gas production is unrealistic (next slide)

At 30MeV the range in titanium is approximately 3mm

c/o Mike Fitton

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He & H production in titanium test samples at Birmingham MC40 cyclotron

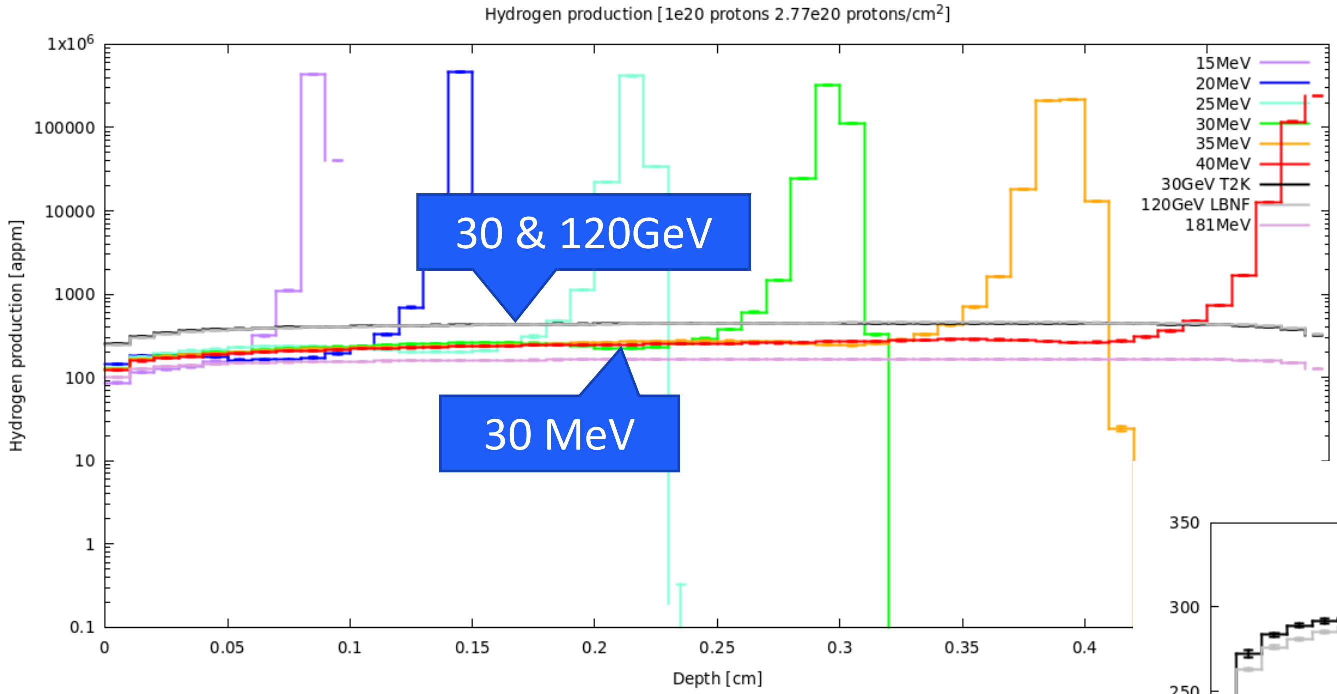
H / He production

Hydrogen production ~1/5th at 30MeV compared to T2K & LBNF (GeV range) except at the Bragg peak where it is much higher (x45+!)

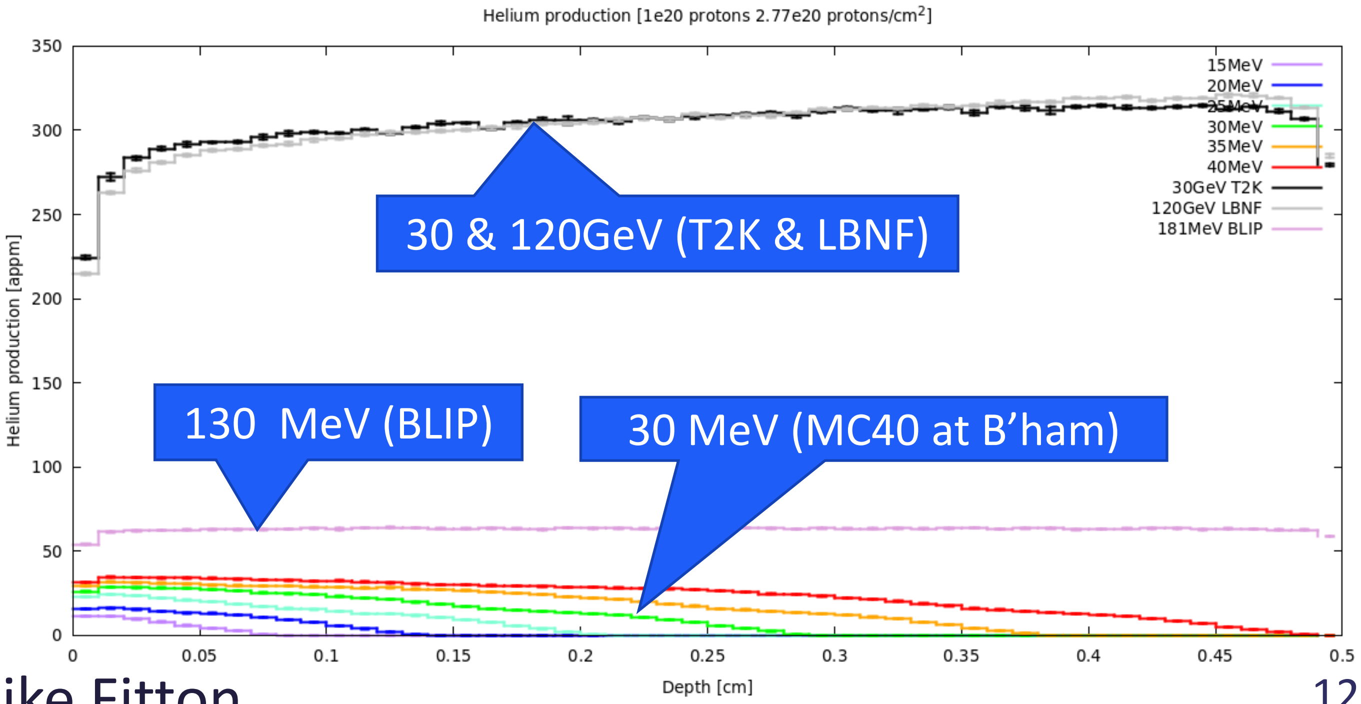
- Avoid the Bragg peak as H production is unrealistic. This could cause swelling, cracking, etc

Helium production at 30MeV is much lower (~1/30th) than irradiation in the GeV range

- Could implant He but range is very limited

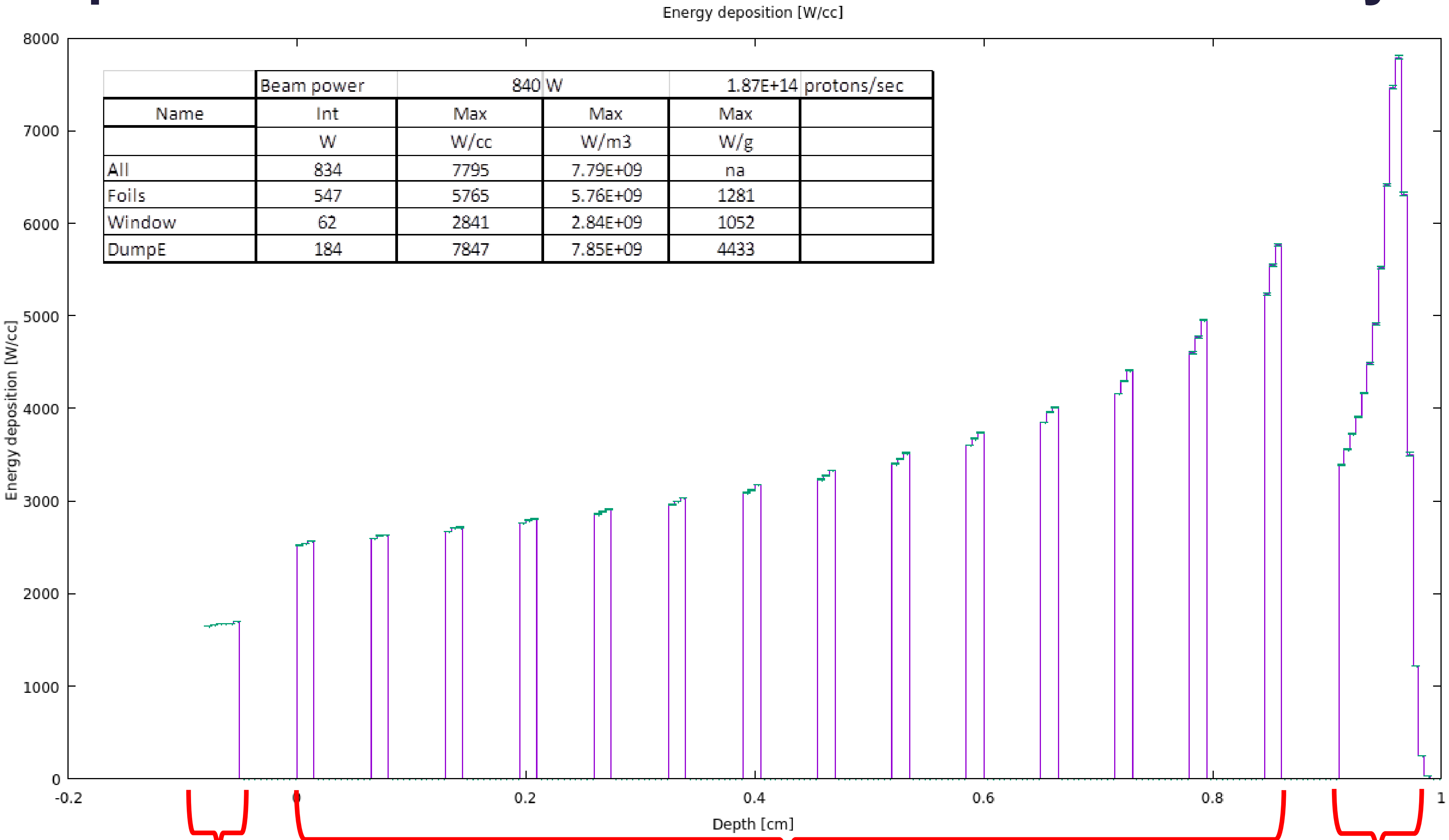


	At 1mm depth		At Max (Bragg)	
	He/DPA [appm/DPA]	H/DPA [appm/DPA]	He/DPA [appm/DPA]	H/DPA [appm/DPA]
15MeV	na	na	1.0	38750.0
20MeV	3.9	140.0	2.1	59102.6
25MeV	14.1	227.0	3.4	55972.2
30MeV	25.1	259.8	4.9	56271.2
35MeV	34.1	285.5	5.9	39629.6
40MeV	41.8	293.5	7.7	52888.9
30GeV	964.5	1335.5		



c/o Mike Fitton

Sample environment FLUKA simulation for MC40 cyclotron



	Beam power	840 W		1.87E+14 protons/sec	
Name	Int	Max	Max	Max	
	W	W/cc	W/m3	W/g	
All	834	7795	7.79E+09	na	
Foils	547	5765	5.76E+09	1281	
Window	62	2841	2.84E+09	1052	
DumpE	184	7847	7.85E+09	4433	

Aluminium window

Titanium foil samples

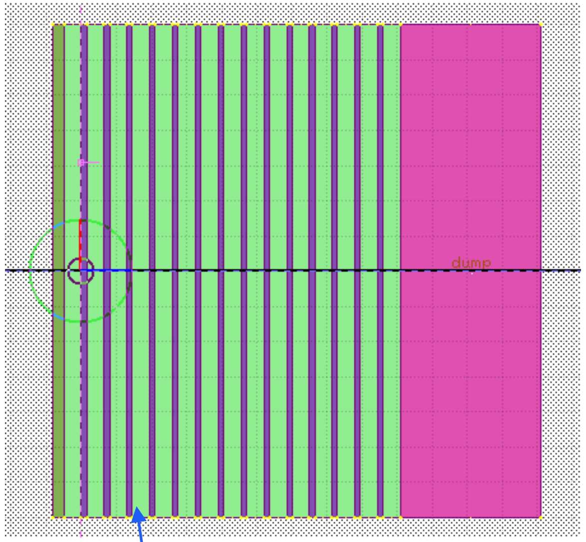
Graphite beam dump

28MeV @ 30 microamps

Beam area = 0.75cm^2

1.87e14 protons per second

840W beam power



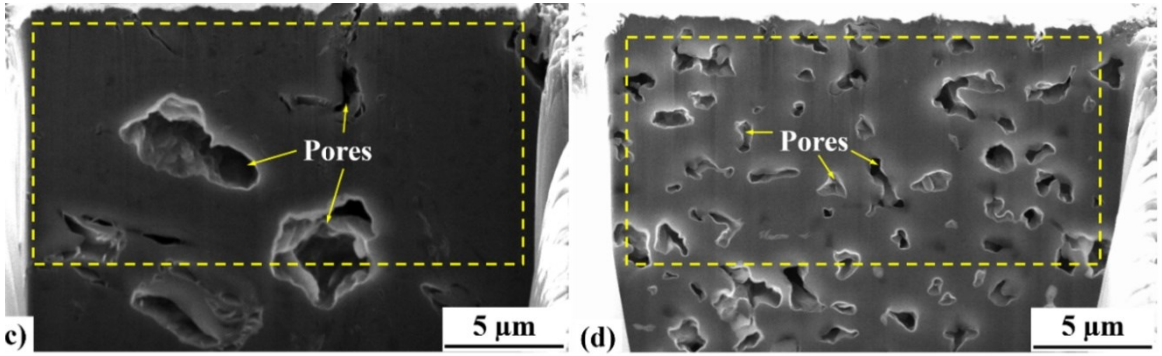
Nitrogen cooling gas between foils

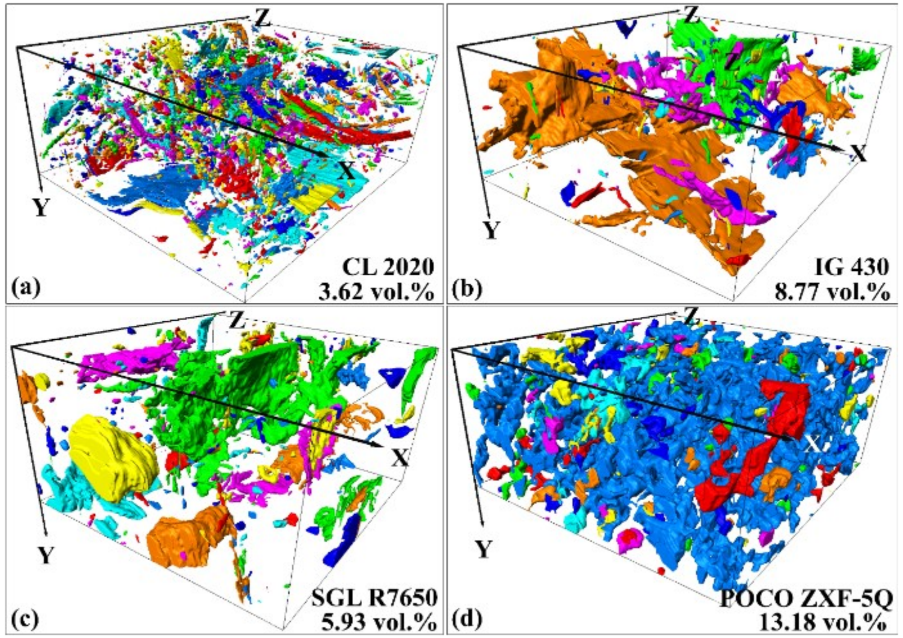
c/o Mike Fitton

Prototype target graphite selection

- Long lead times and procurement challenges for some nuclear grade graphite
- Recommendation requested based on current knowledge?
- Can use a different grade for '1st operating' target
- But want prototype to be viable if manufacture is successful

Sub-micrometre (FIB/SEM tomography)





Bristol University (Dong Liu)

Science and Technology Facilities Council

Titanium Irradiation Sample Environment

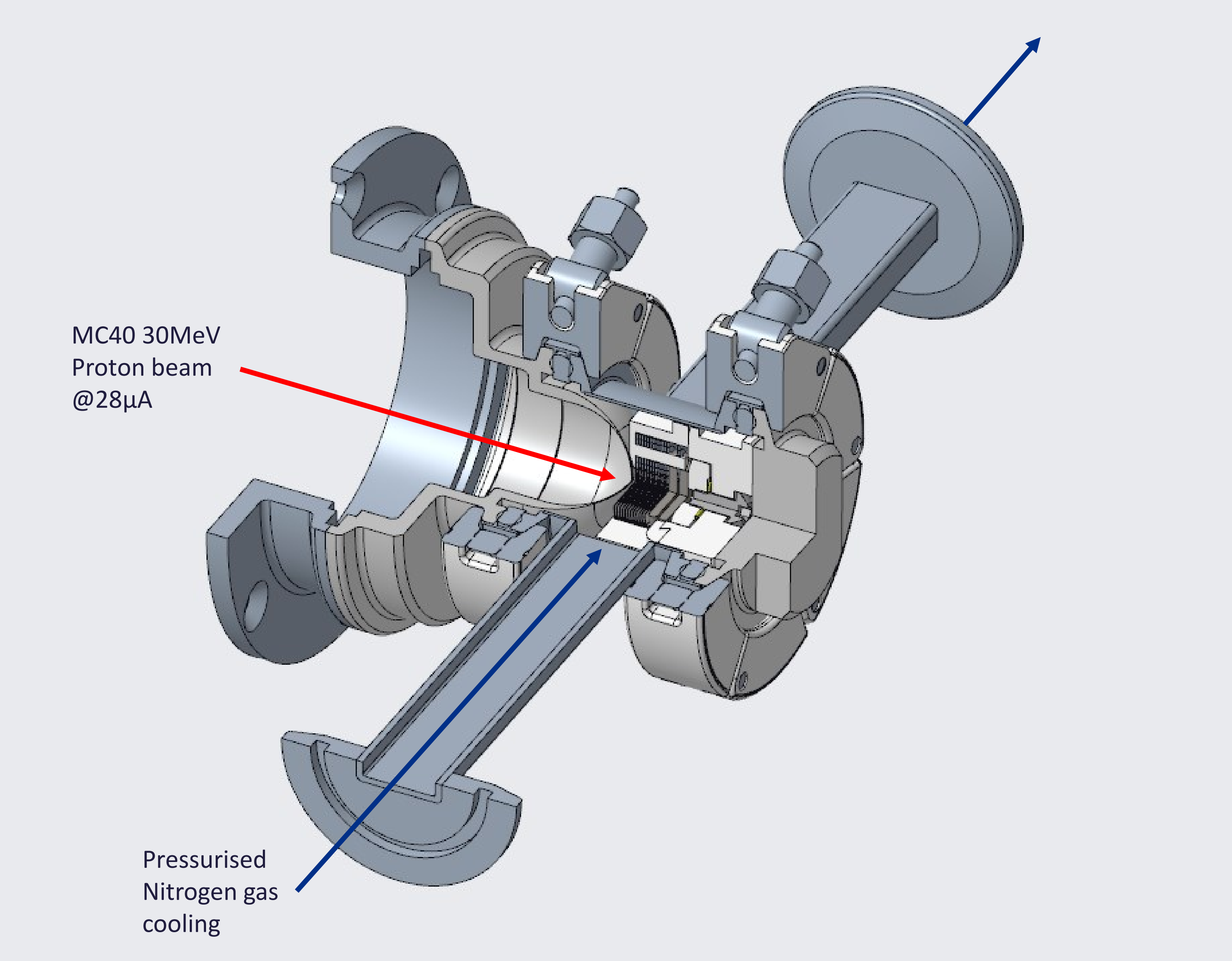
Eric Harvey-Fishenden

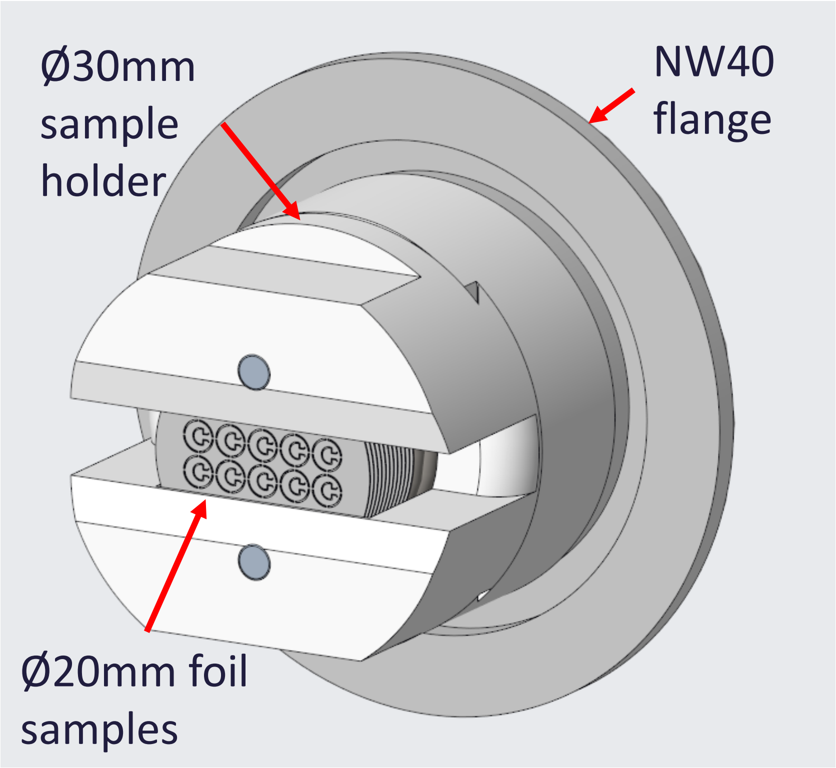
June 2023

RAL High Power Targets Group

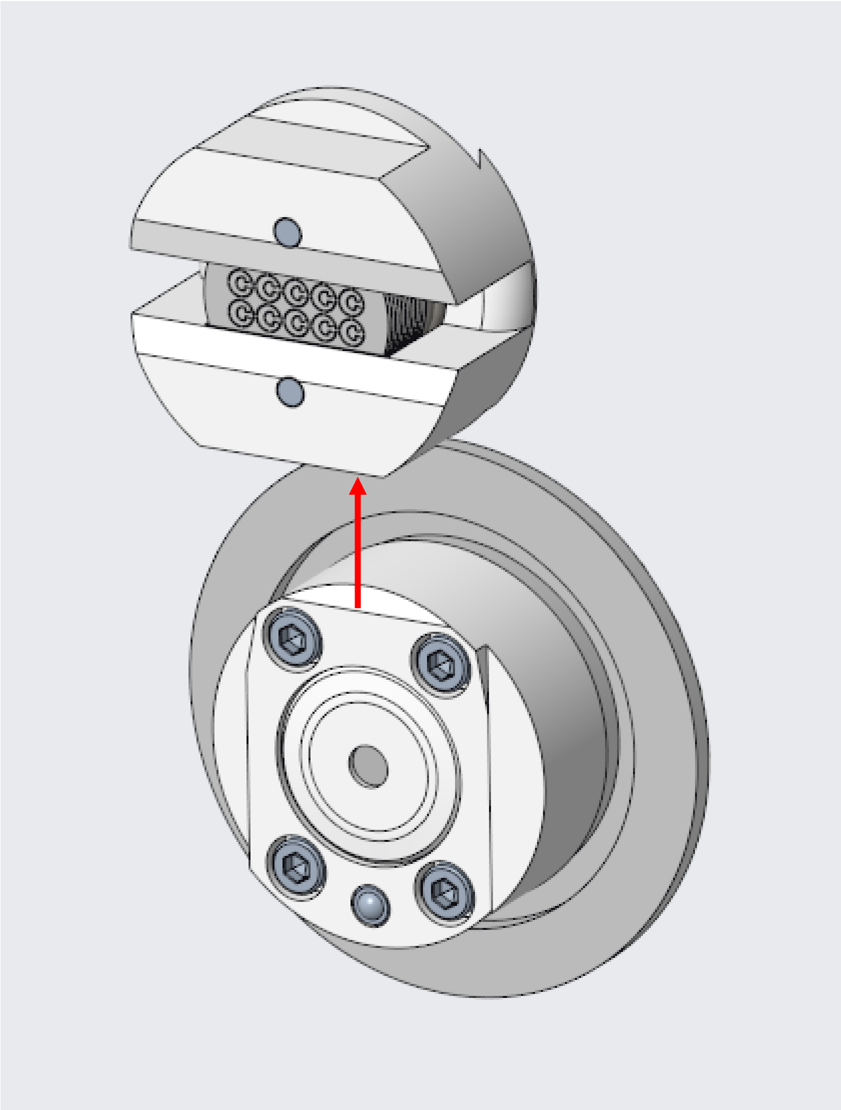
Concept Overview:

- Nitrogen gas cooled samples installed onto MC40 beamlines in a quick-disconnect module
- 10-14 Titanium foil (0.15mm) samples held in a container with several mm of graphite located behind to act as a beam stop
- Aluminium domed window to separate MC40 beamline vacuum from pressurised nitrogen cooling environment

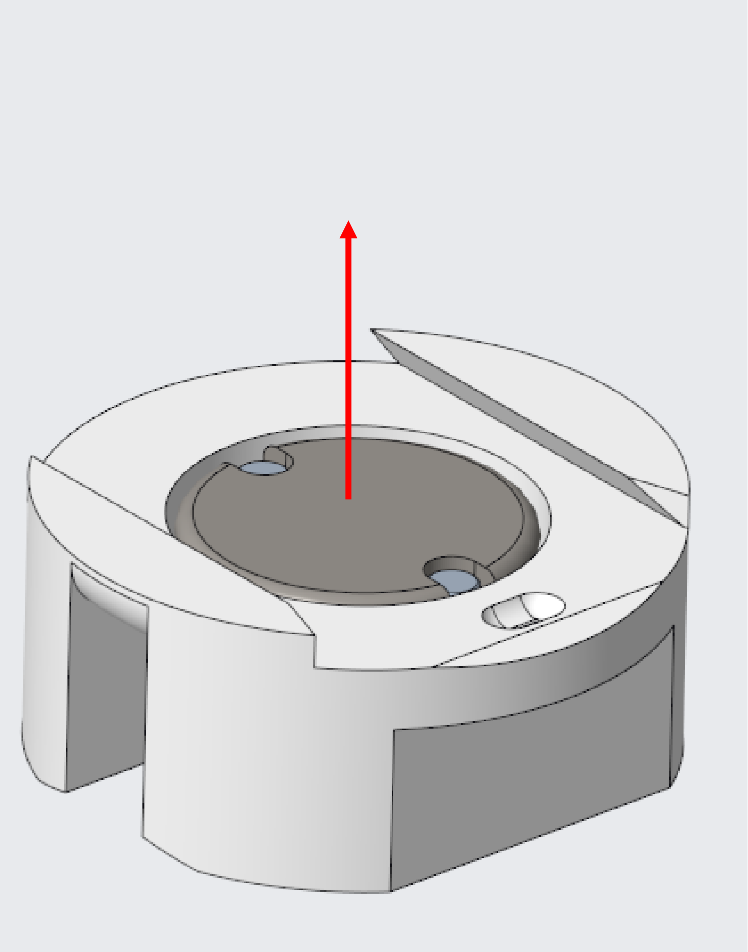




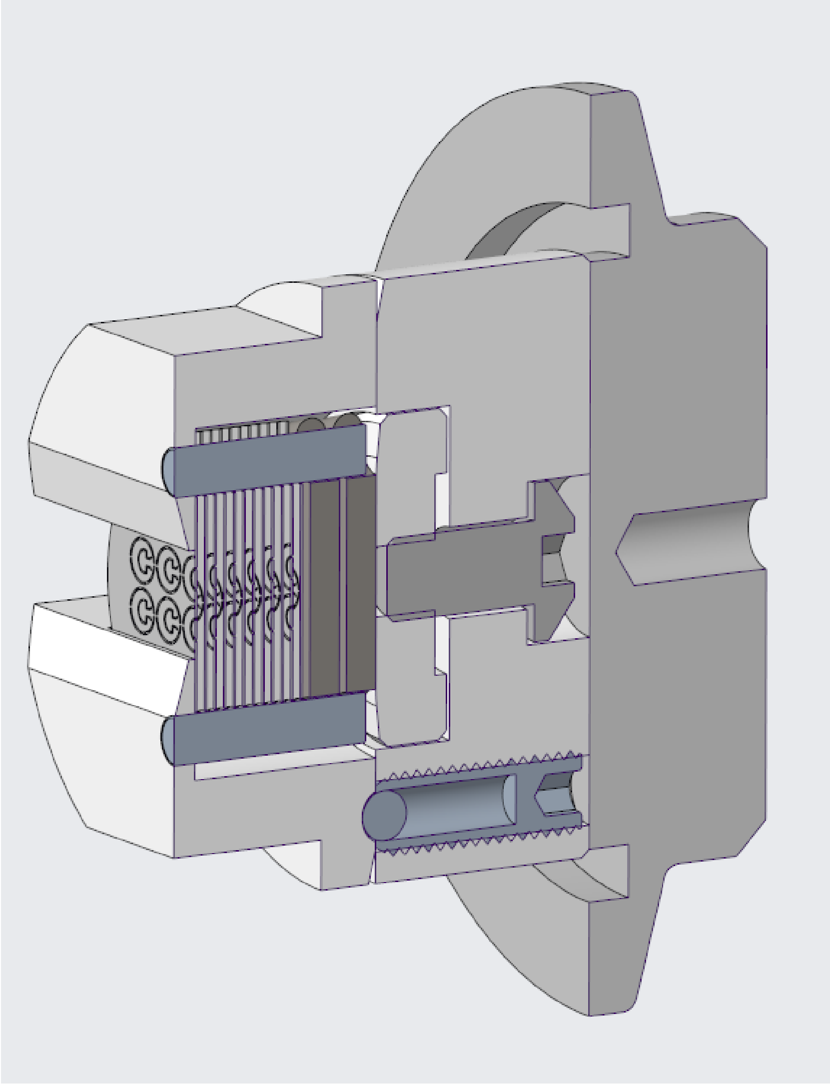
This is the assembly shipped to MRF in transport cask

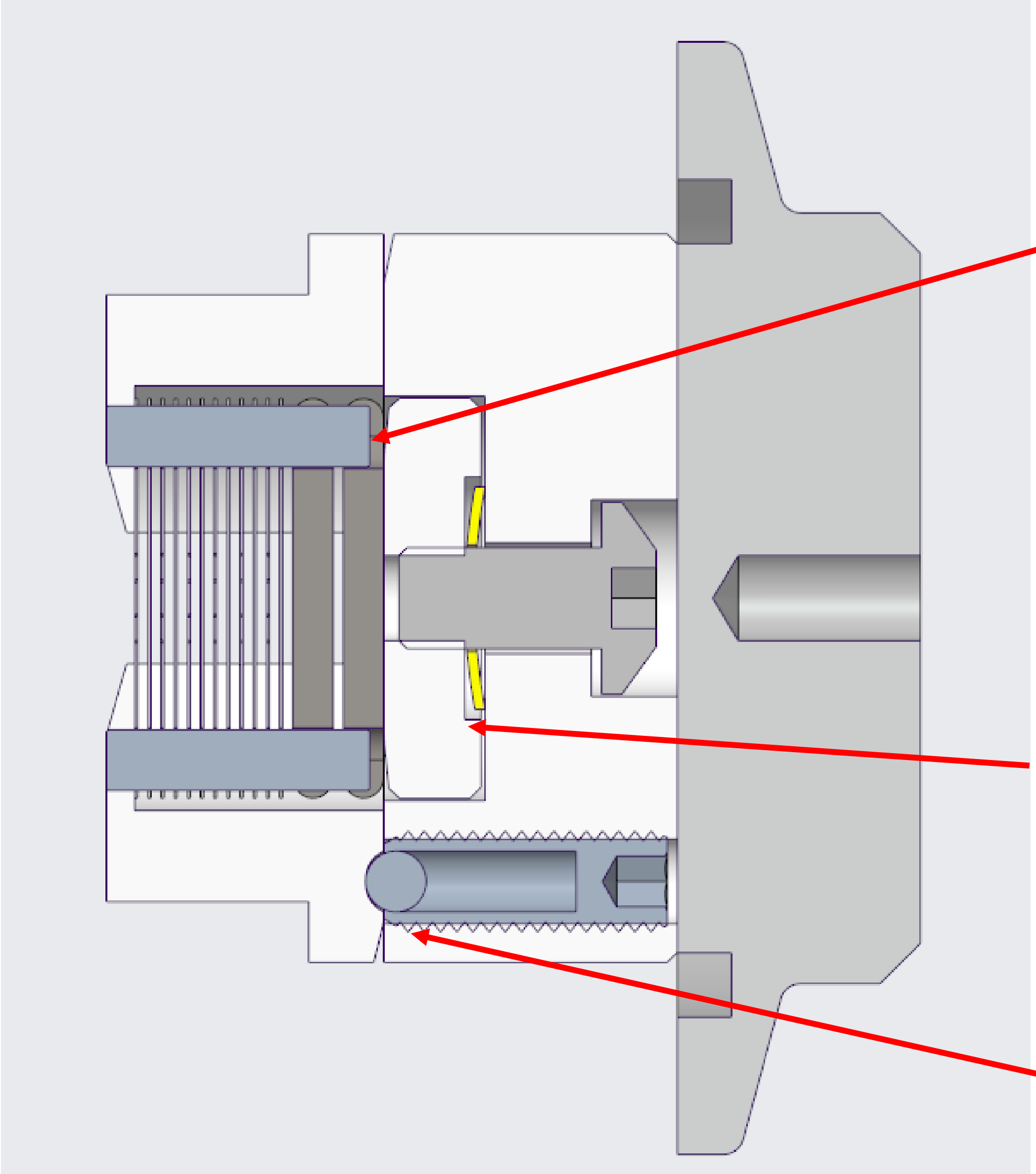


Sample holder is removed from flange in hot cell – sliding dovetail joint (retained with a spring plunger)



Samples are removed (graphite and titanium foils) from holder using vacuum tweezers





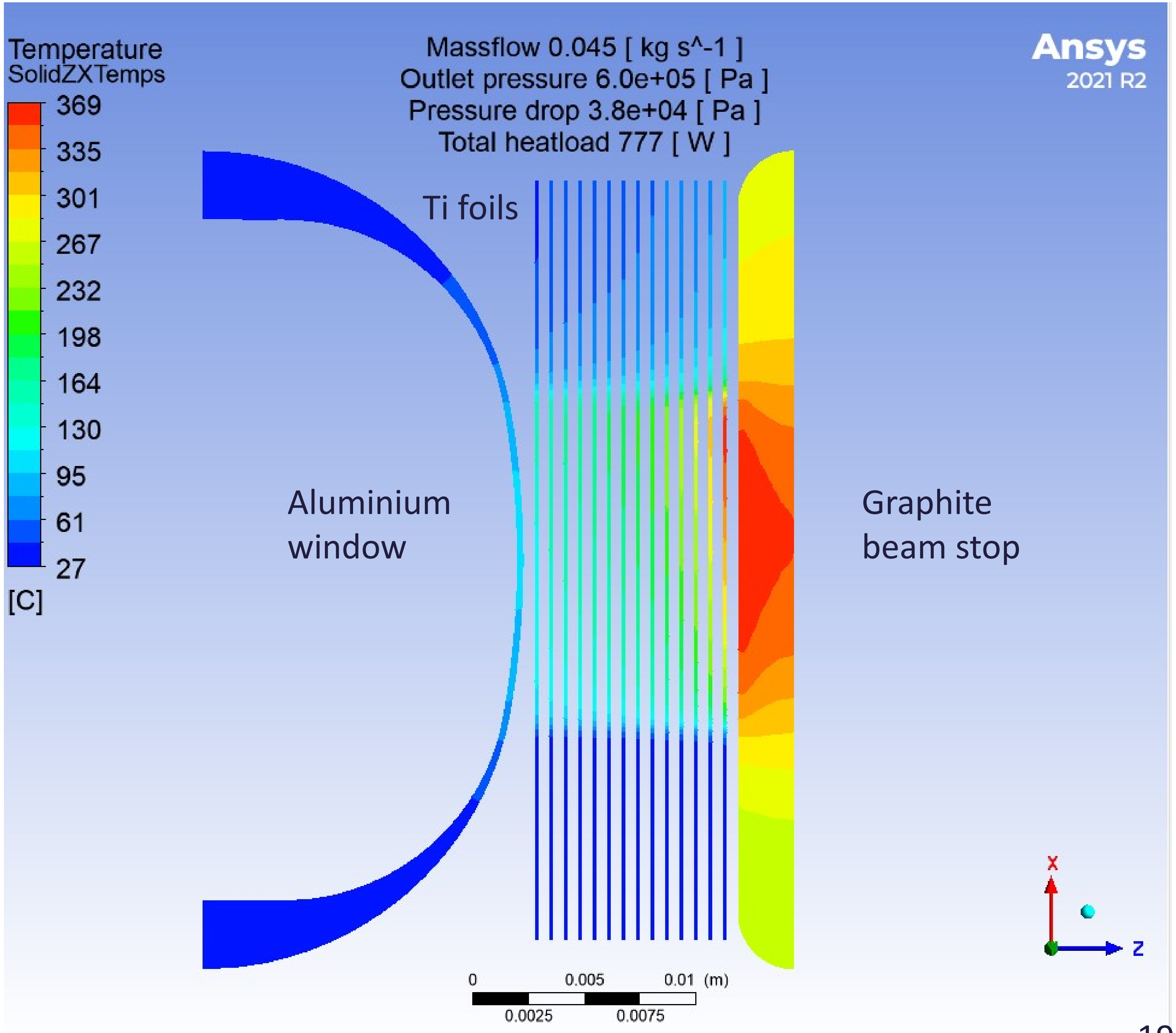
Samples aligned on two pins and separated with 0.5mm spacers (not shown)

Wave washer & preload block constrain samples within holder & allows for thermal expansion

Spring plunger for retaining sample holder

Thermal Analysis:

- Updated heat load – material properties for Aluminium beam window have been corrected since last analysis
- Since window was previously the limiting factor, this has reduced the cooling requirement significantly (x2)
- Have also agreed to reduce the number of foils (14 to 12) to reduce the temperature/damage gradient within the samples



Birmingham Irradiation Project

Sample Holder Cooling – Flow Test Results

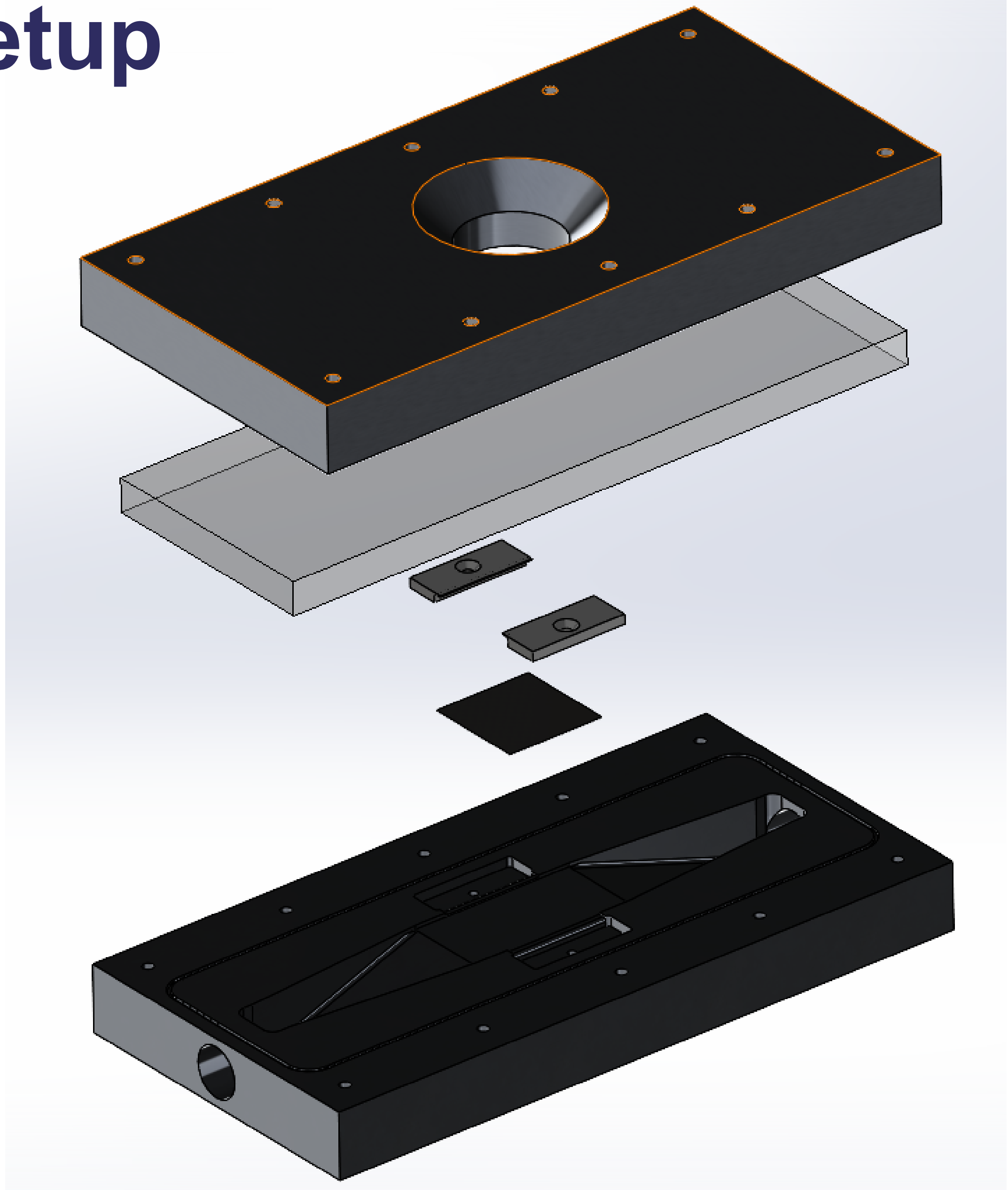
Richard Cowan, STFC

Science and Technology Facilities Council

UNIVERSITY OF BIRMINGHAM

Test Setup

- Sample held clamped with 0.5mm gap on each side to allow flow
- High-strength glass on one side for viewing
- Aluminium pressure manifold



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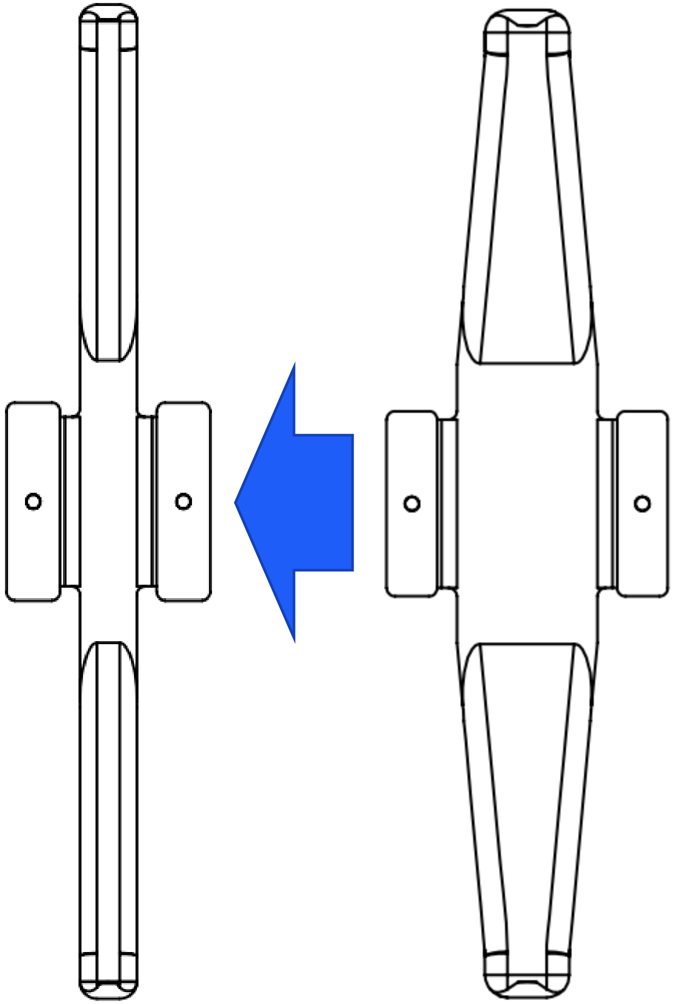
Previous Results

- Recap: first runs in February
- Tested between 2 bar to 6 bar.
- Static buckling seen at all pressures.
- Flutter seen at 2 and 3 bar.
- Samples detached and lost in pipework in later test
- Showed permanent deformation around ~0.5mm and impact damage at downstream edge due to hitting glass viewing pane

Updated Clamping

- Improved clamp to hold sample closer, reduce 25mm gap to 10mm, increasing stiffness of foil in flow

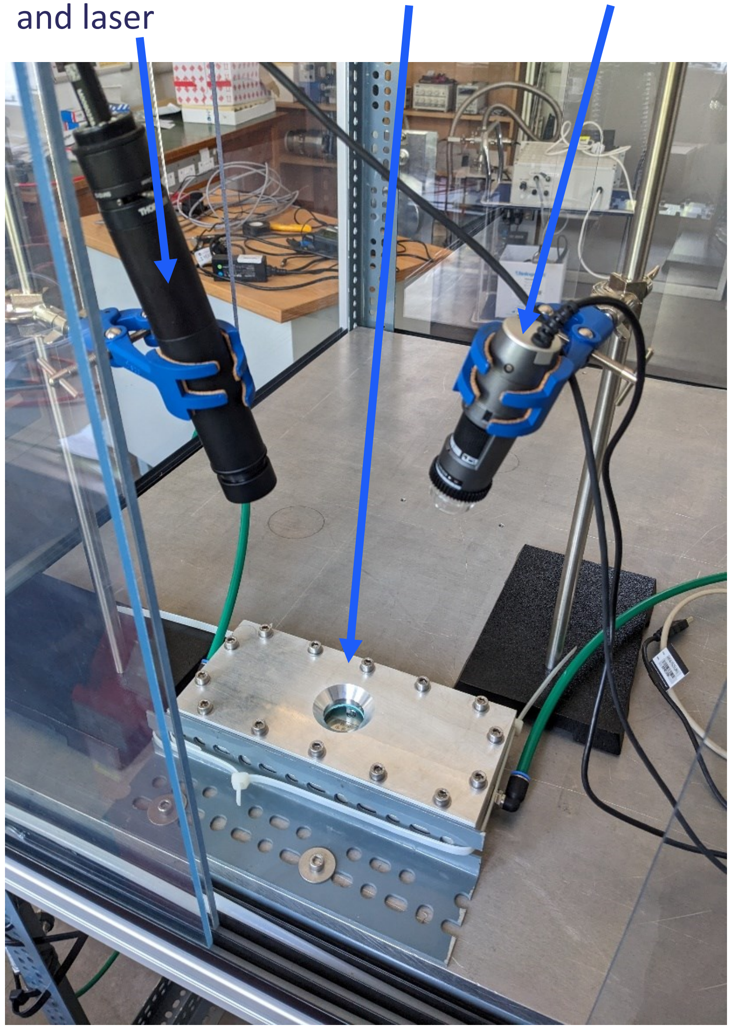
- Increased sample separation to 1.5mm
- Increased sample holding tabs – 3 -> 4 and 2x thickness



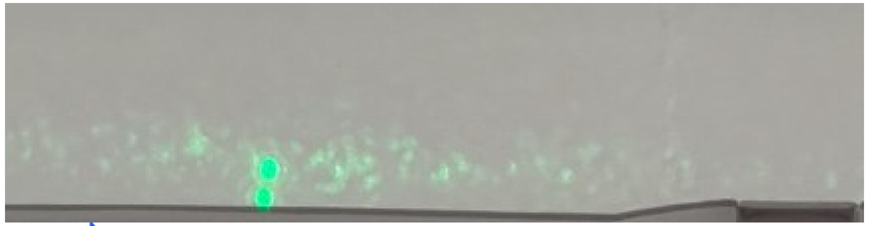
Tests with new clamping

- Tested both flat plate foil and sample foil between 2 and 6 bar.
- No flutter seen at any pressure or flow rate!
- Exceeded required flow rate by a large margin at lower pressures.
- Pressure drop slightly higher than expected but likely a result of pressure sensor location
- Small amount of deflection seen using laser spot deflection (unnoticeable on direct viewing cameras)

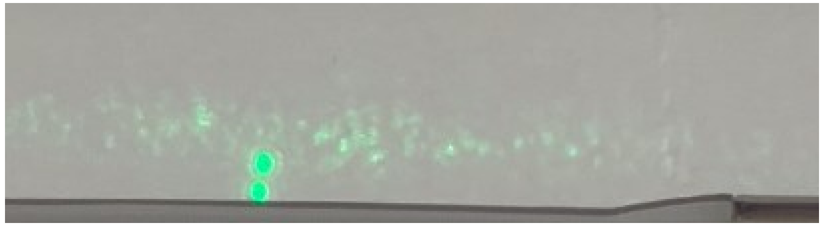
Testing setup with pressure vessel, microscope and laser



No Flow - speckled line is reflection from sample



With Flow – speckled line has moved slightly up

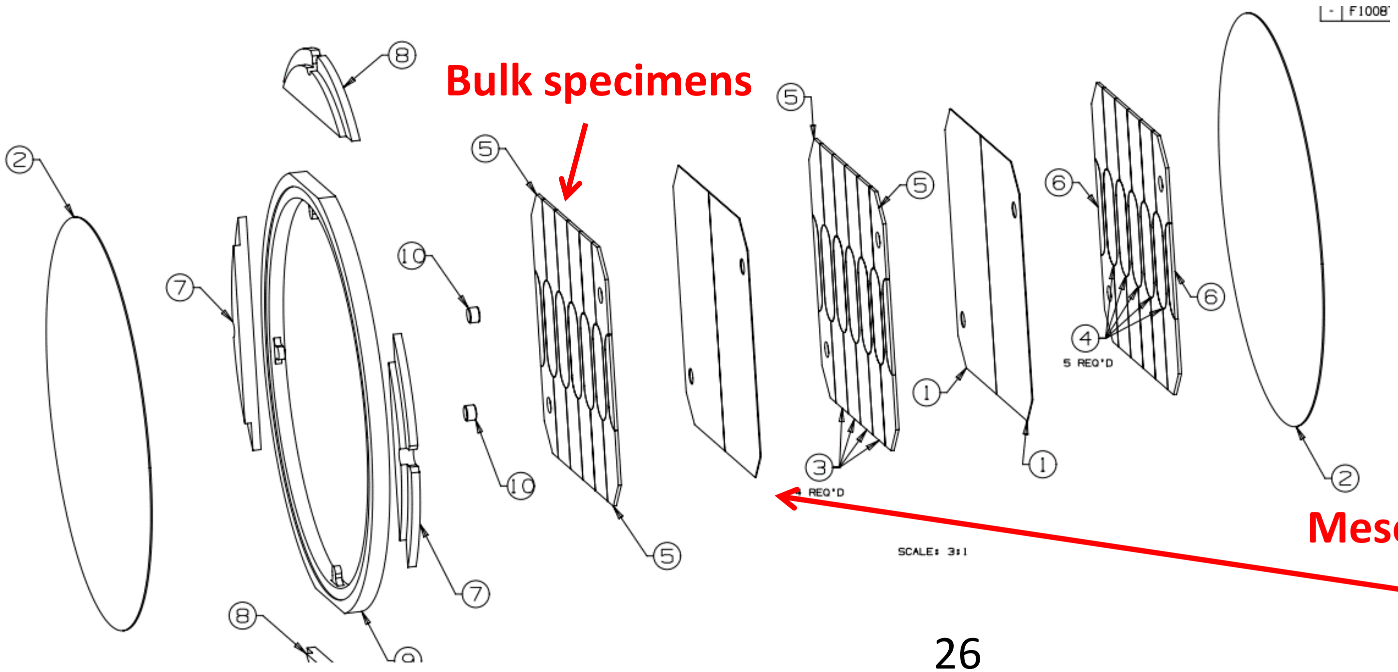


Meso-scale Mechanical Testing of Proton Beam Window Materials

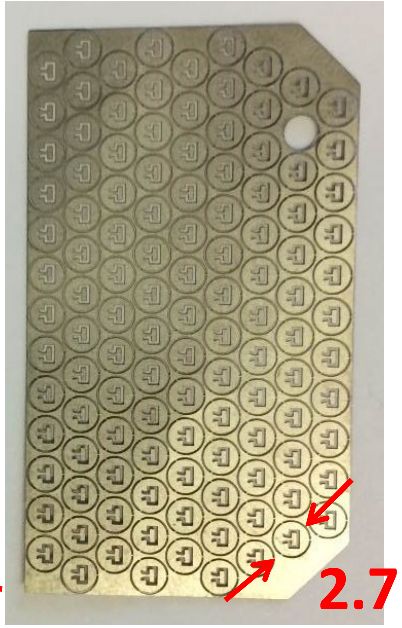
Lazuardi Pujilaksono, Angus J Wilkinson, Jicheng Gong
Micromechanics Group, Oxford University
26/06/2023

Why meso-scale mechanical tests?

- Significantly more specimens
- 1000 times smaller. Low activity and easy to handle and dispose
- Low cost
- More flexibility
- The size is comparable to the real component



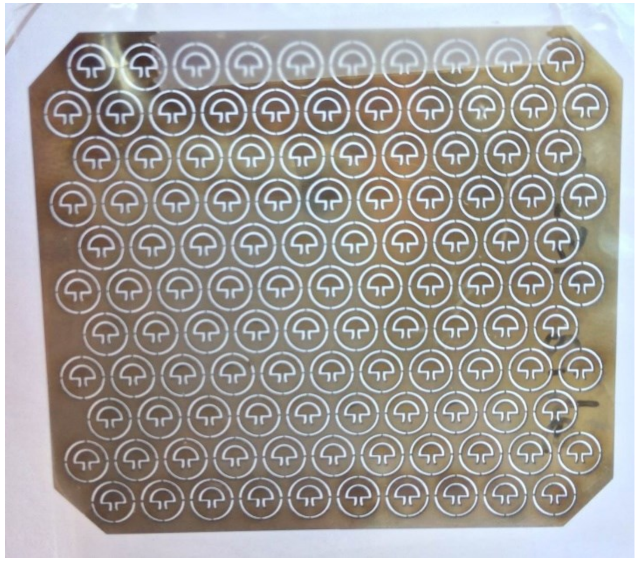
Meso specimens

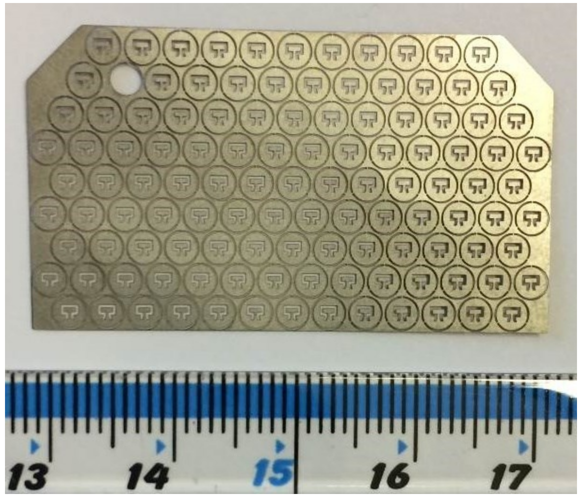


2.7 mm

Two batches of irradiated titanium meso-cantilevers

- Down stream Ti1: Ti-6Al-4V. This is the same material as the proton beam window at J-PARC. The irradiation level is 0.25 DPA
- Down stream Ti2: Expanded the materials from Ti-6Al-4V to CP-Ti and Beta titanium alloys Ti15 and Ti-333. The irradiation level is 1 DPA.





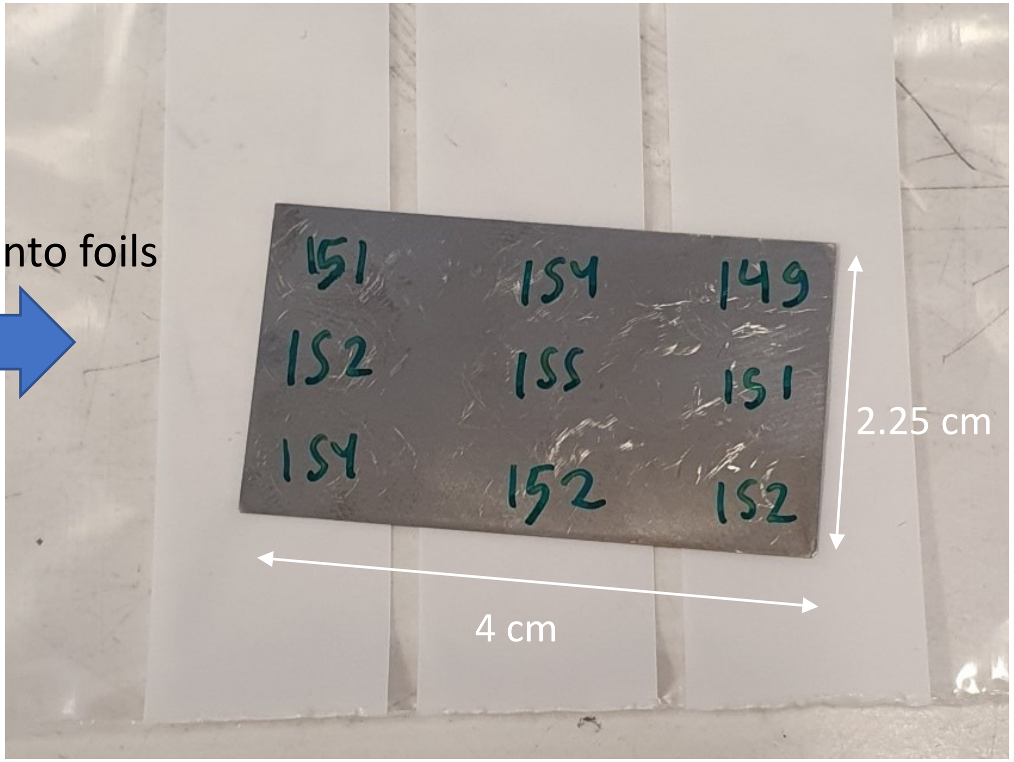
O.M.G.

UNIVERSITY OF OXFORD

Current work on non-irradiated Ti-6Al-4V materials from RAL

Dummy proton beam windows

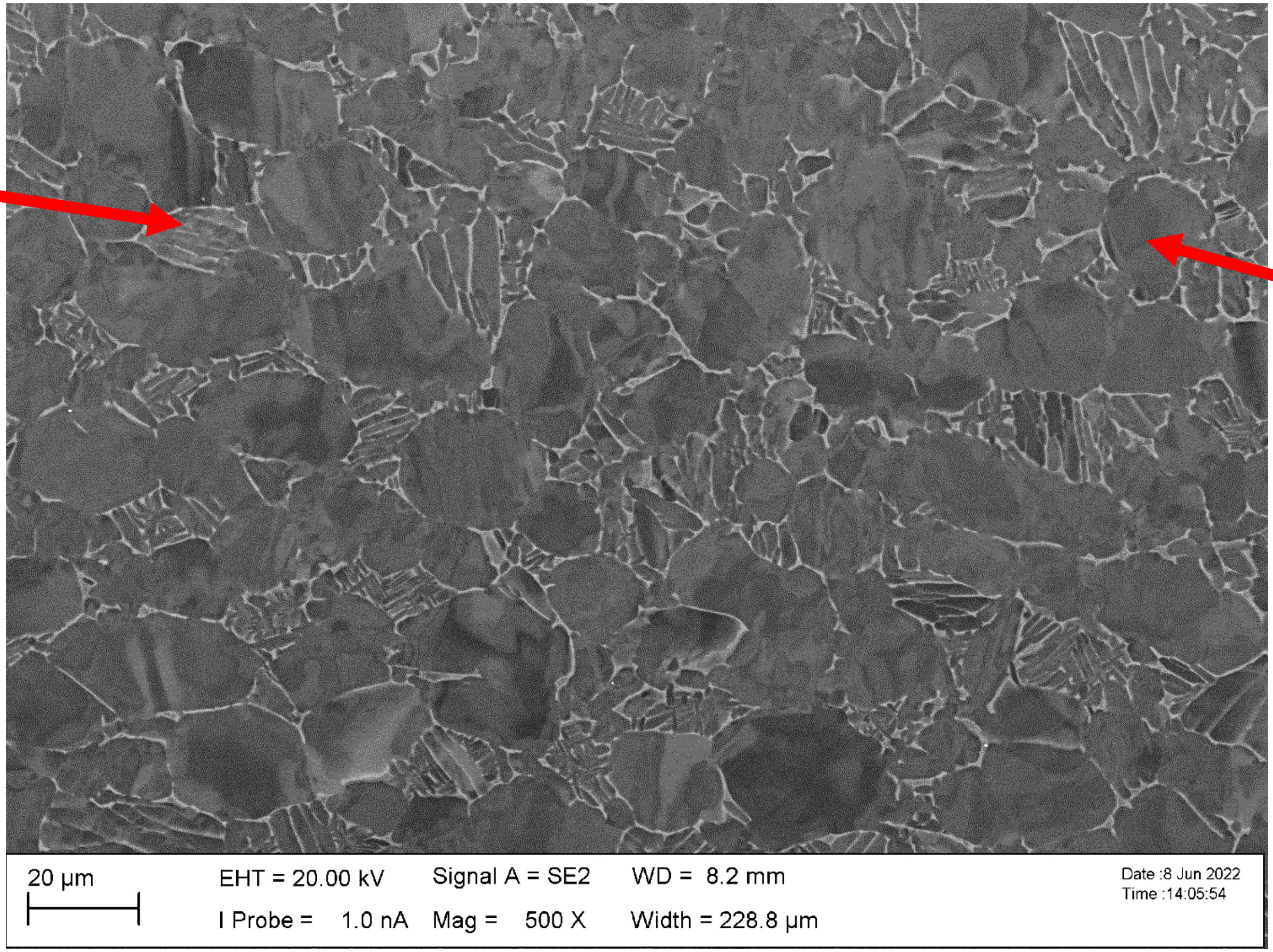
Machined into foils



The samples used in this study came from new Ti-6Al-4V beam windows; therefore, the studies we conduct have identical microstructures to real components

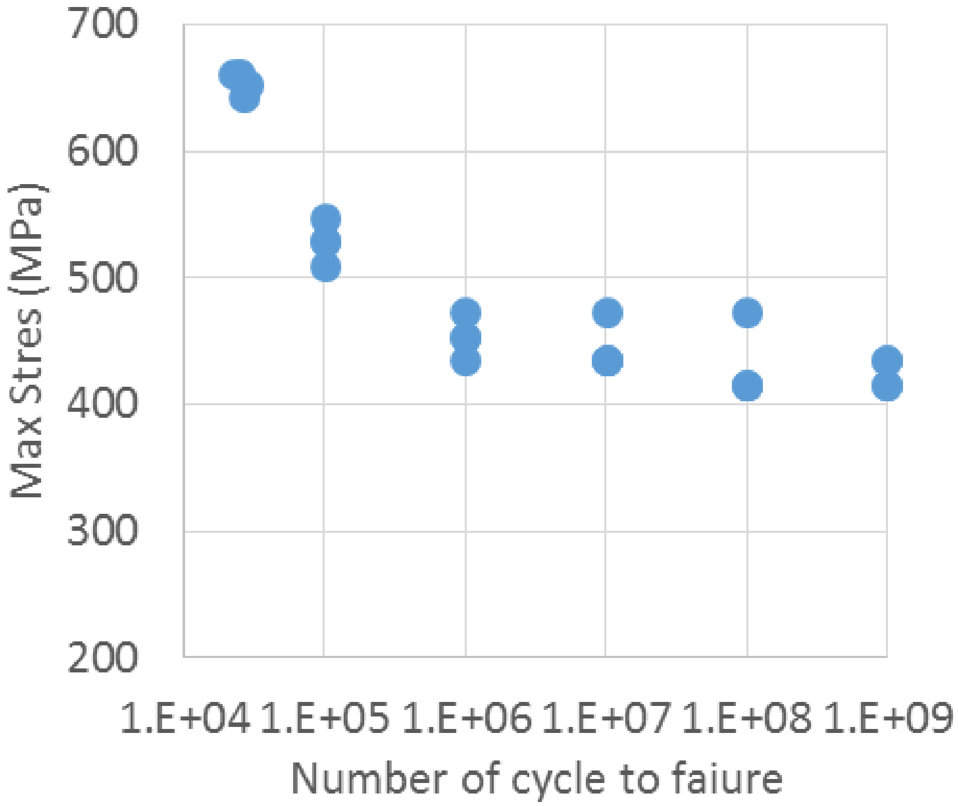
Bimodal microstructure of Ti-6Al-4V

Transformed β



Primary α

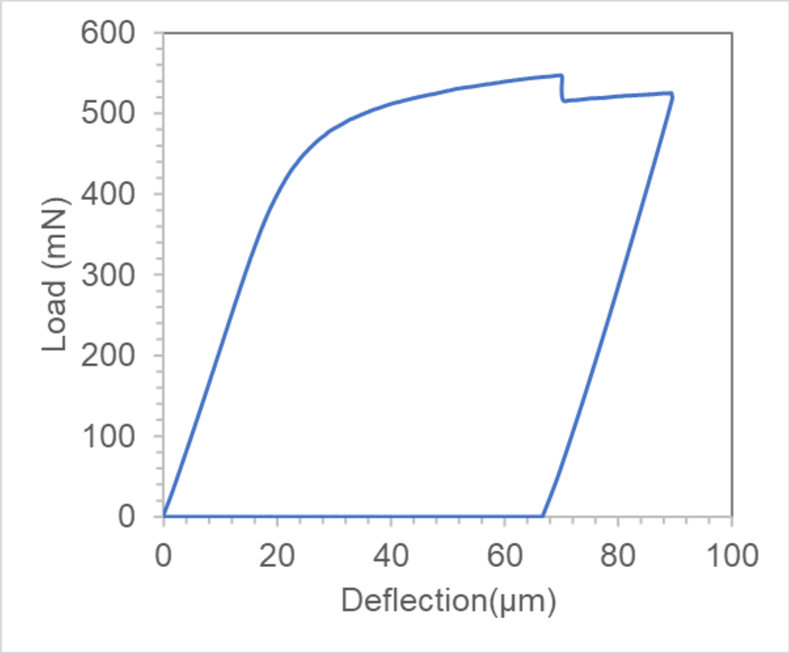
Ti-6Al-4V Meso-cantilevers from dummy beam windows



Future plan 1: Static meso-cantilever tests, irradiated vs. non-irradiated

Premier nanoindenter

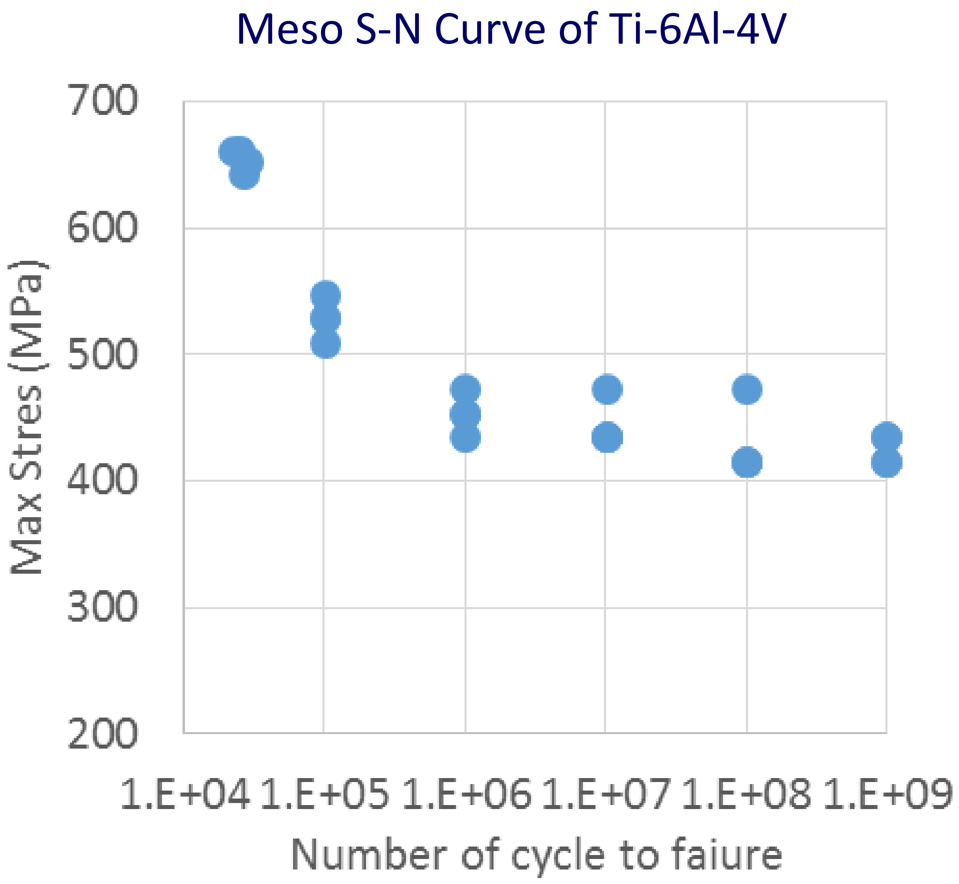
Load displacement response of a meso-cantilever



	Material
Foil one	Grade 2 CP-Ti Cold rolling and annealed
Foil two	Ti15 Solution treated
Foil three	Ti-64 Annealed at 595 ^{0}C for 3 hours
Foil four	Ti-64 Annealed at 595 ^{0}C for 3 hours
Foil five	Ti-64

Static deflections tests of five proton irradiated materials and make comparison with non-irradiated titanium

Future plan 2: Ultrasonic fatigue testing of irradiated meso-cantilevers



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Foil four	Ti-64 Annealed at 595 ^{o}C for 3 hours
Foil five	Ti-64

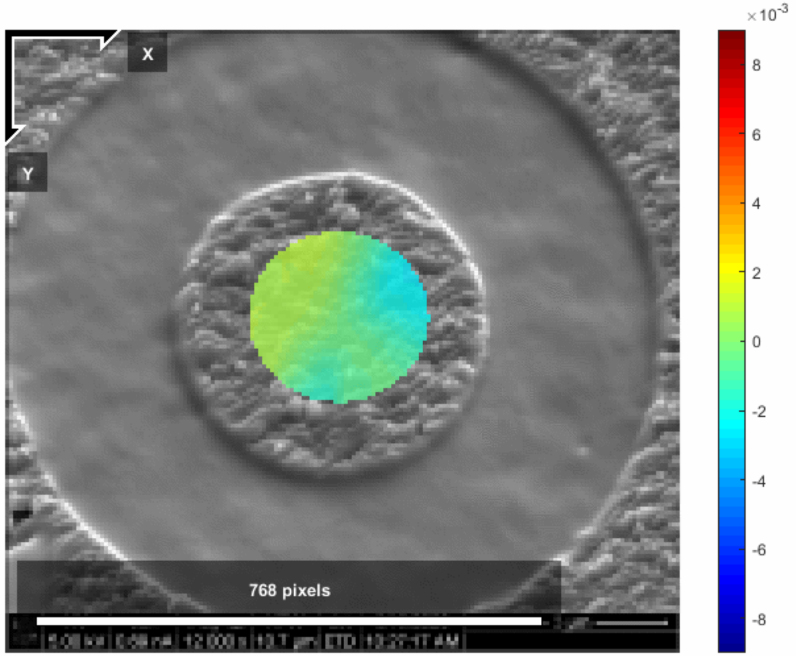
- ❑ High cycle fatigue tests of two candidate materials (Ti6Al4V and one other Ti material)

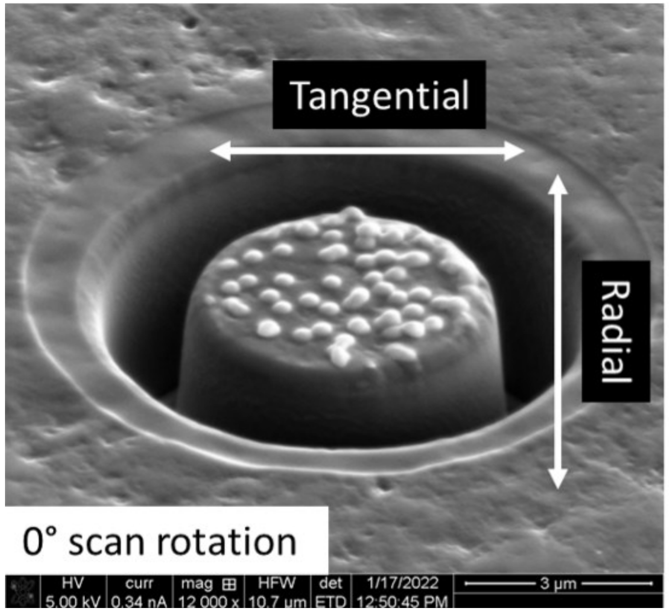
Future plan 3: Design foils that can be irradiated at the cyclotron irradiation facility (MC40) at the University of Birmingham

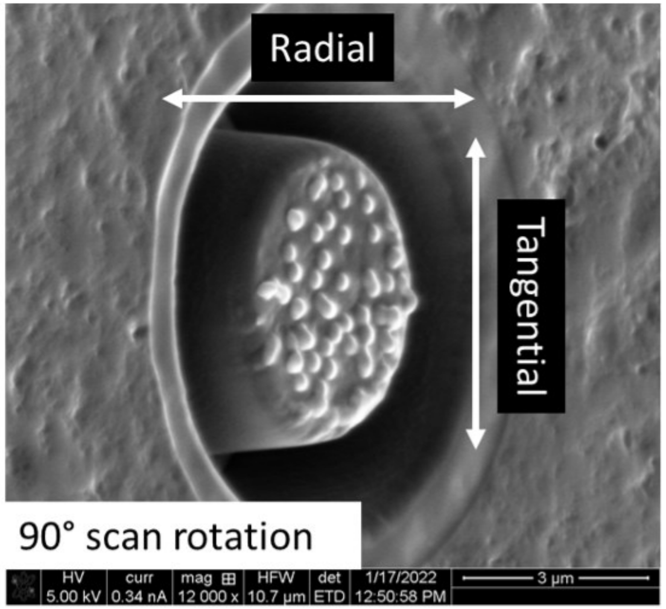
- Help design the foils, enabling irradiation of meso-cantilevers at the cyclotron irradiation facility at the University of Birmingham

Local residual stresses in thin ceramic coating layers

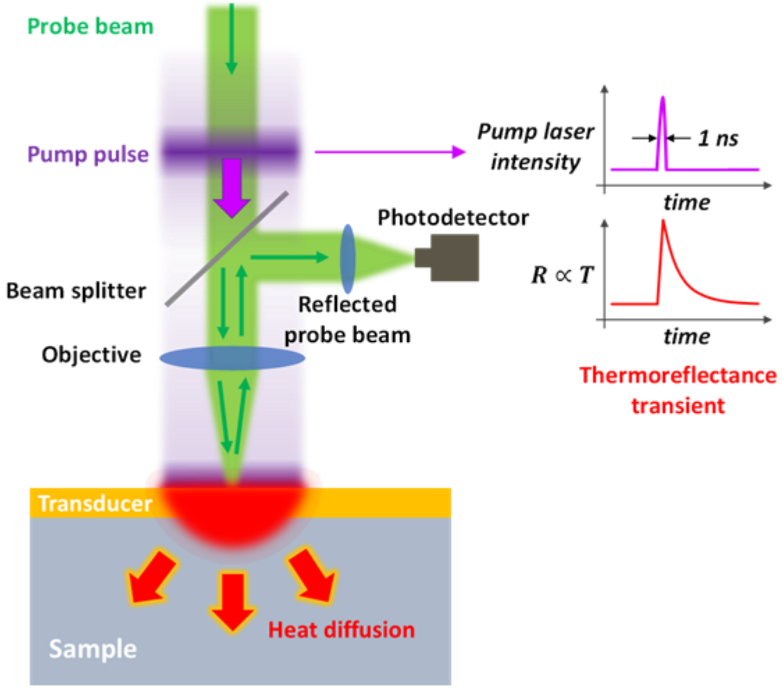
FIB/SEM-DIC (Ring-core method) for local residual stress measurements
In the PyC layer of PYCASSO TRISO particles







Local thermal conductivity measurements in thin ceramic coatings

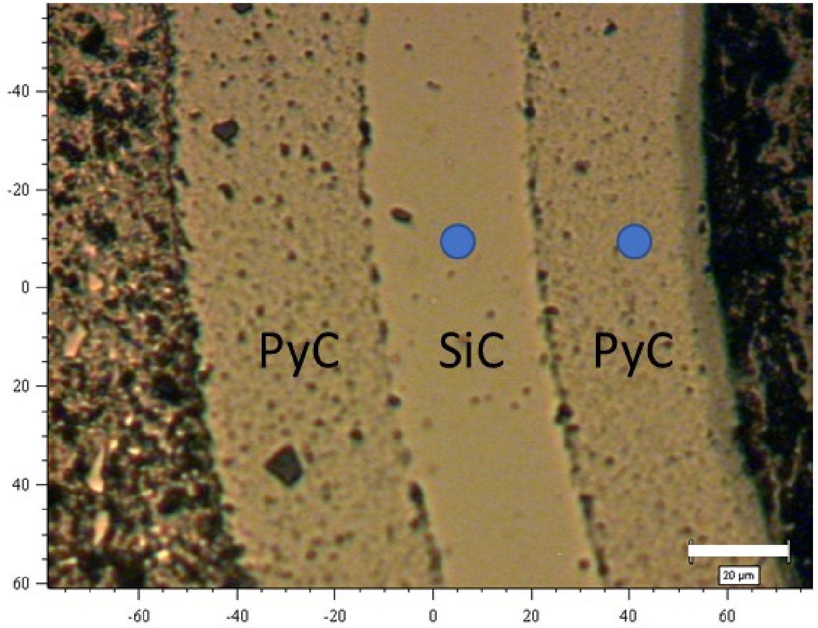


TherMap Solutions

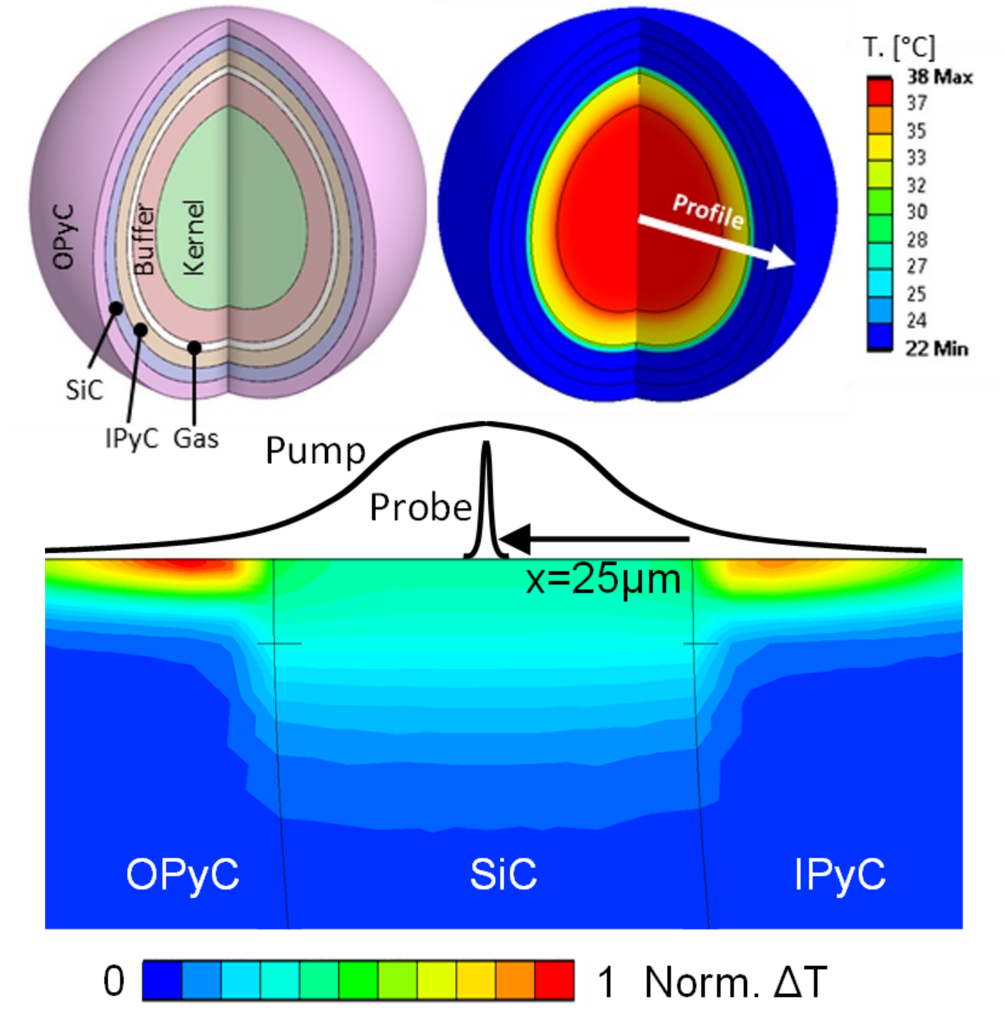
Centre for Device Thermography and Reliability (CDTR), Bristol

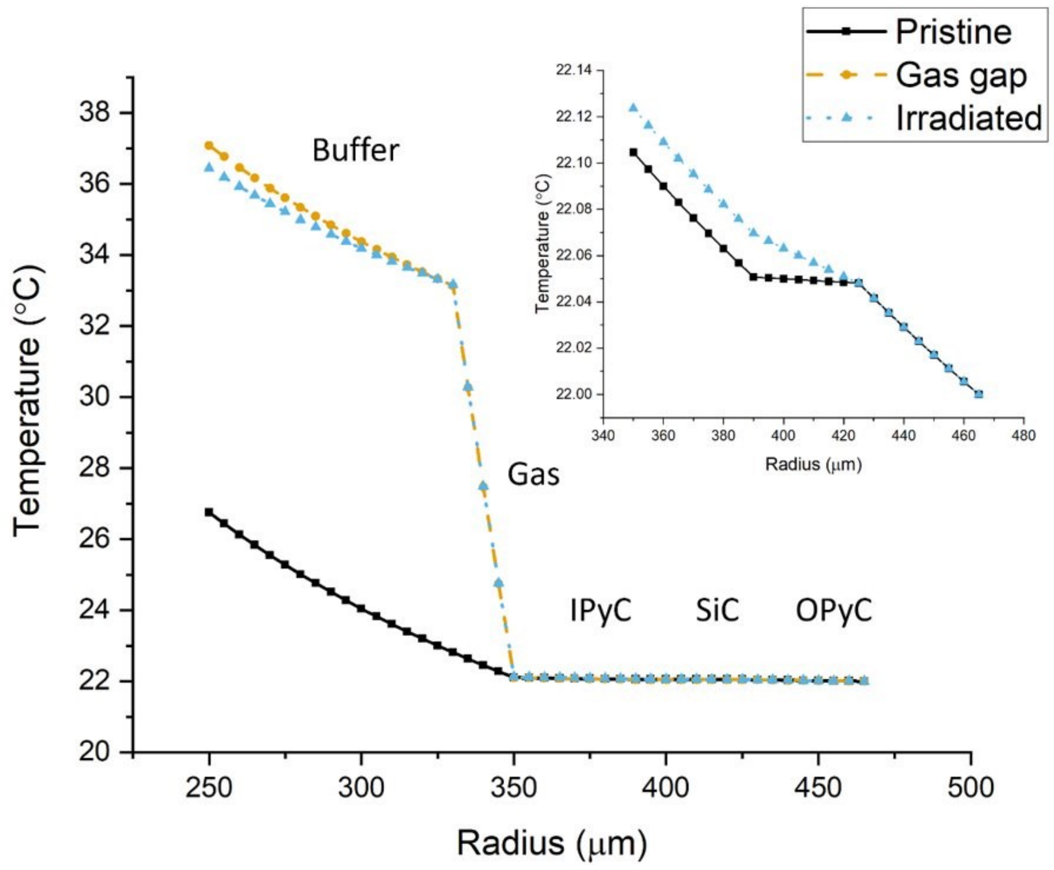
Nanosecond transient thermoreflectance (**TTR**) method

Thermal conductivity measurements on cross-section of a TRISO particle



Local thermal conductivity modelling in thin ceramic coatings





Paper submitted currently under review

UK Atomic Energy Authority

UKAEA MRF

Facility Update

Phil Earp, Equipment Scientist, MRF

Ultrasonic Fatigue Rig

UK Atomic Energy Authority

Ultrasonic Fatigue Rig

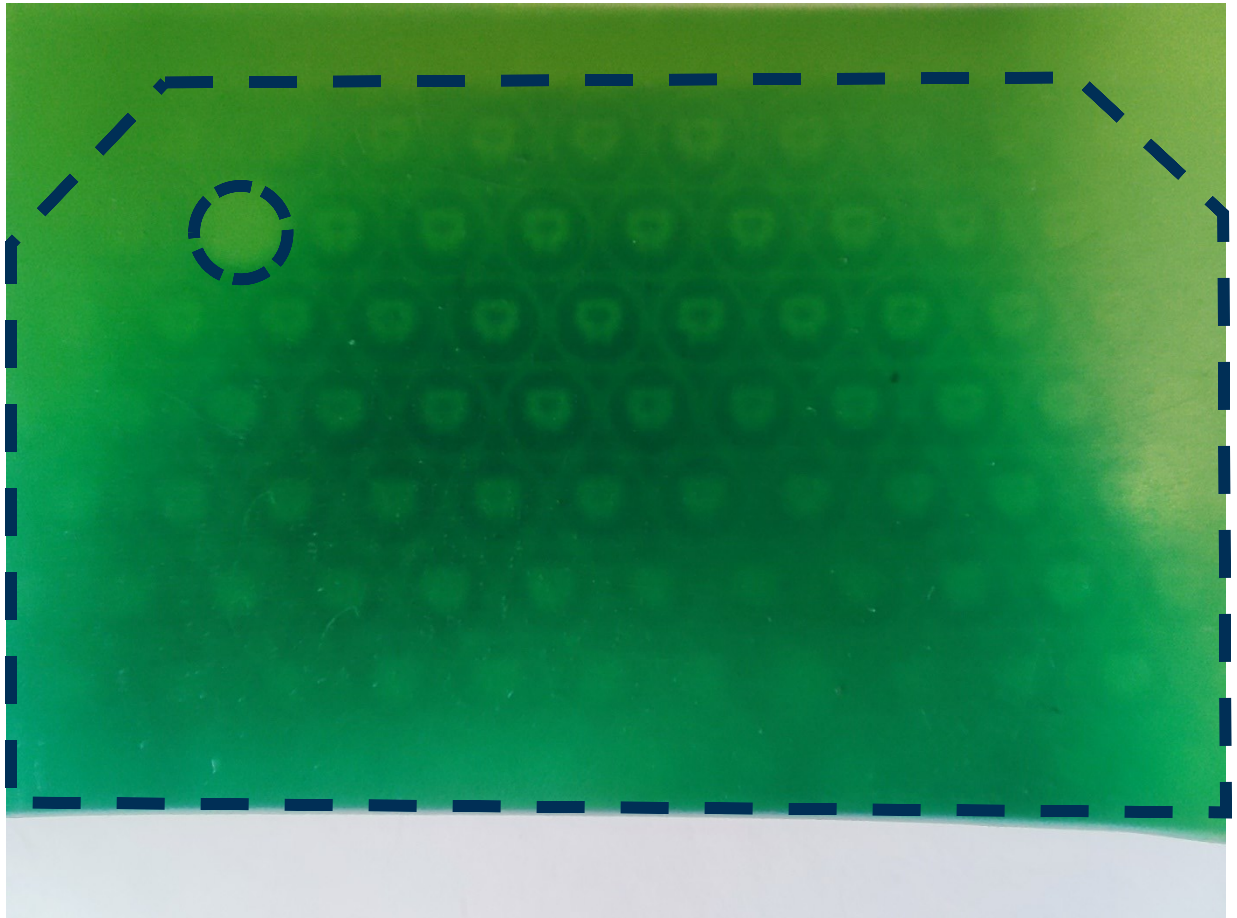
- Simplified Optics System
- Thanks to Richard Cowan from STFC
- Ongoing Compressed Air System Issues

FIB Holder for Foils

- Direct mounting of foils in FIB

Film Dosimetry

Aim to identify the BLIP irradiation beam centre



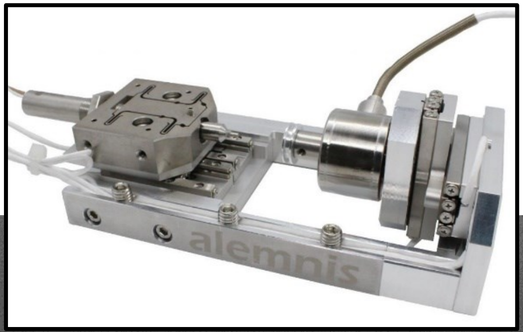
Foil 2: Ti-15-3-3-3 irradiated half-foil film dosimetry

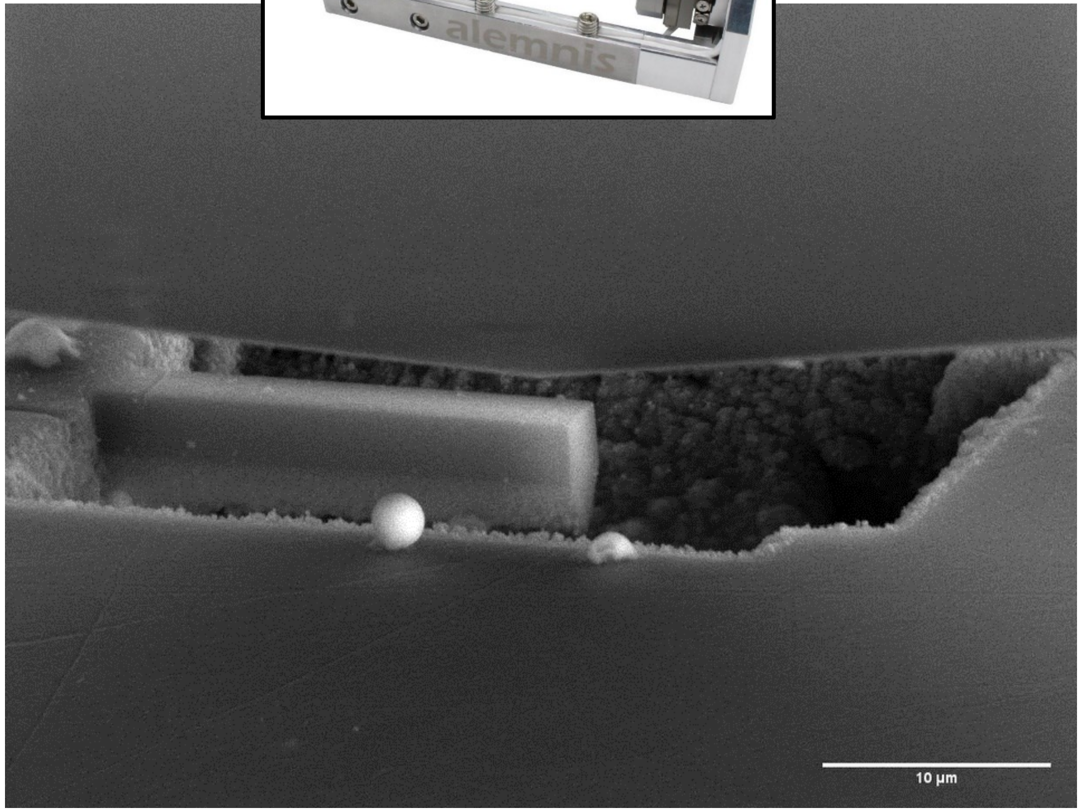
MATERIALS RESEARCH FACILITY

NNUF2a - FaSCiNATe

Objective: Deliver three instruments to expand capability in microstructural, mechanical and thermo-physical testing

- **In-situ Piezo Stage (IPS)** with high-temperature capability for simultaneous mechanical testing and microstructural imaging of materials samples.
- **High temperature stage** and focusing optics for **XRD** for thermo-physical properties measurements.
- **High Vacuum Differential Scanning Calorimeter** for characterisation of materials phase transitions and measurements of specific heat capacity.



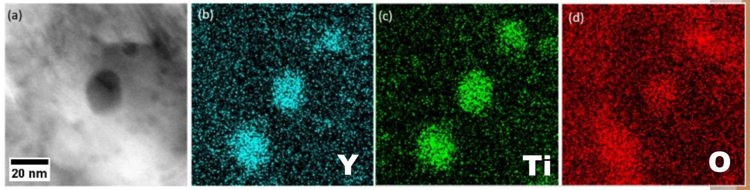


Cantilever Fracture test of Be
Courtesy of Dr Slava Kuksenko & Chris Bearcroft

NNUF2a - TEM

Dual EDS

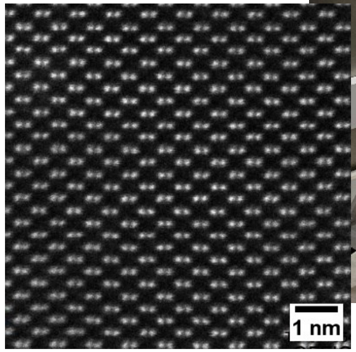
- Can be used for chemical characterization of materials.
- Useful for observing segregation of alloying elements in structural materials such as oxide dispersion strengthened ferritic steel.



Y, Ti and O in ODS steel. From [1].

Atomic Resolution

- Powerful tool for visualizing atomic structures.
- Can be used to further understand structural defects including dislocation loops in plasma facing materials such as beryllium.

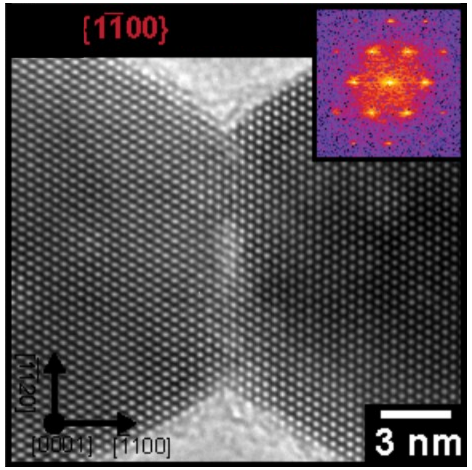


Si <110>

JEOL NEOARM STEM

4D STEM

- Can be used to record bright-field, dark-field, and diffraction patterns simultaneously.
- Useful for assessing many crystallographic orientations at once.
- 4D STEM can be used to observe strain in irradiated materials.



Atomic columns in CdSe with simultaneously acquired diffraction pattern. From [2].

[1] J. Ribis et al., “Nano-Structured Materials under Irradiation: Oxide Dispersion-Strengthened Steels,” Nanomater. 2021, Vol. 11, Page 2590, vol. 11, no. 10, p. 2590, Oct. 2021, doi: 10.3390/NANO11102590.
[2] J. C. Ondry and A. P. Alivisatos, “Application of Dislocation Theory to Minimize Defects in Artificial Solids Built with Nanocrystal Building Blocks,” Acc. Chem. Res, vol. 54, p. 18, 2021, doi: 10.1021/acs.accounts.0c00719.

Bringing it all together:

Jan 2023

UK Atomic Energy Authority

SEM
- High resolution imaging
- Analytical capability (BSE, EBSD, EDS)

PFIB
- High volume throughput
- Analytical capability (BSE, STEM, EBSD, EDS)

GaFIB
- Precision milling
- EBSD for site-specific liftouts and TKD
- Cryo stage and transfer

(S)TEM
JEOL NEOARM 200kV
- Aberration probe-corrected
- Analytical capability (Atomic STEM, EDS)
- In-situ annealing

In-situ tester
- In-situ indentation and micro-tensile testing (0.5 – 2.5 N)
- High temperature (up to 1000 °C)
- Cryo-capable to -150 °C
- Fast indentation 2D-mapping

XRD
- Strain measurement
- Micro-focusing
- In-situ annealing

DSC
- Stored energy
- Cp measurement

External Equipment
- (S)TEM microscopes
- Atom probe
- Synchrotron

NNUF
National Nuclear User Facility
NEW

April 2023

MATERIALS RESEARCH FACILITY

Summary

STFC
- STFC Status Update
- Titanium Irradiation Sample Environment

University of Oxford
- Meso-scale Mechanical Testing

University of Bristol
- Thermal Conductivity Measurement

UKAEA Materials Research Facility
- Ultrasonic Fatigue Update
- NNUF2a Investment

Dr Phil Earp
Equipment Scientist (Mechanical Testing)
Materials Research Facility
UK Atomic Energy Authority
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UK Atomic Energy Authority

MATERIALS RESEARCH FACILITY