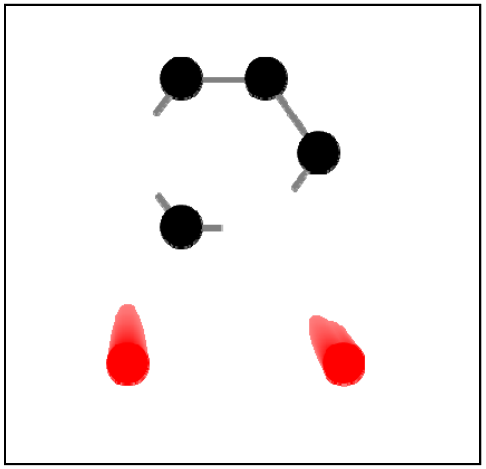

Status Report: Experimental investigation of beryllium

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UNIVERSITY OF OXFORD · Fermilab · Science & Technology Facilities Council Rutherford Appleton Laboratory

Content

- Context of the research
- Materials, points of interest
- Status of different parts of the experimental program
- Conclusions

http://www-radiate.fnal.gov

RaDIATE Collaboration

Radiation Damage In Accelerator Target Environments

Investigation of the radiation response of structural window and target materials in new highly intensity proton accelerator particle sources

Beryllium **is a promising candidate because of:**

- good "nuclear" properties;
- appropriate mechanical properties
- good "thermal" properties (conductivity, specific heat, melting point);
- high oxidation resistance;
- positive experience from existing beam windows

What about new working conditions?

Long-Baseline Neutrino Experiment (LBNE)

Application	Operating conditions					Proton beam parameters
	Avg. T (°C)	Peak T (°C)	Total DPA	Gas production (appm/DPA)		
				He	H	
Beam window (vacuum to air)	200	300	~ 0.23/yr	1030	2885	700 kW; 120 GeV; ~1 Hz; σ_{rms} = 1.3 mm
Target	375	450	~ 0.23/yr	1030	2885	700 kW; 120 GeV; ~1 Hz; σ_{rms} = 1.3 mm

Size:

Target: L = 950 mm, D = 15.3 mm (48 sections)

Window: 25.4 mm diameter, 0.25 mm thick

PF-60

	Max impurities, appm
Al	170
C	450
Fe	130
Mg	810
O	2900
Si	130
N	195
Be	balance

S-200-F

	Max impurities, appm
Al	335
C	1130
Fe	210
Mg	130
O	5445
Si	195
Be	balance

S-65

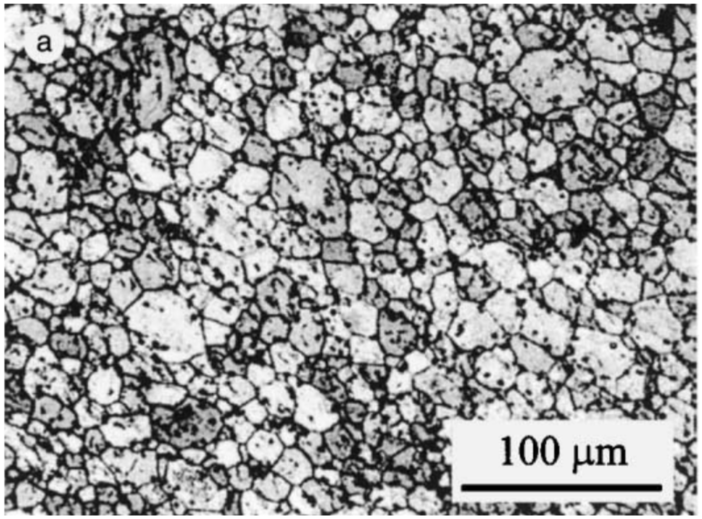
	Max impurities, appm
Al	170
C	680
Fe	130
Mg	15
O	3260
Si	145
Be	balance

Goodfellow order:

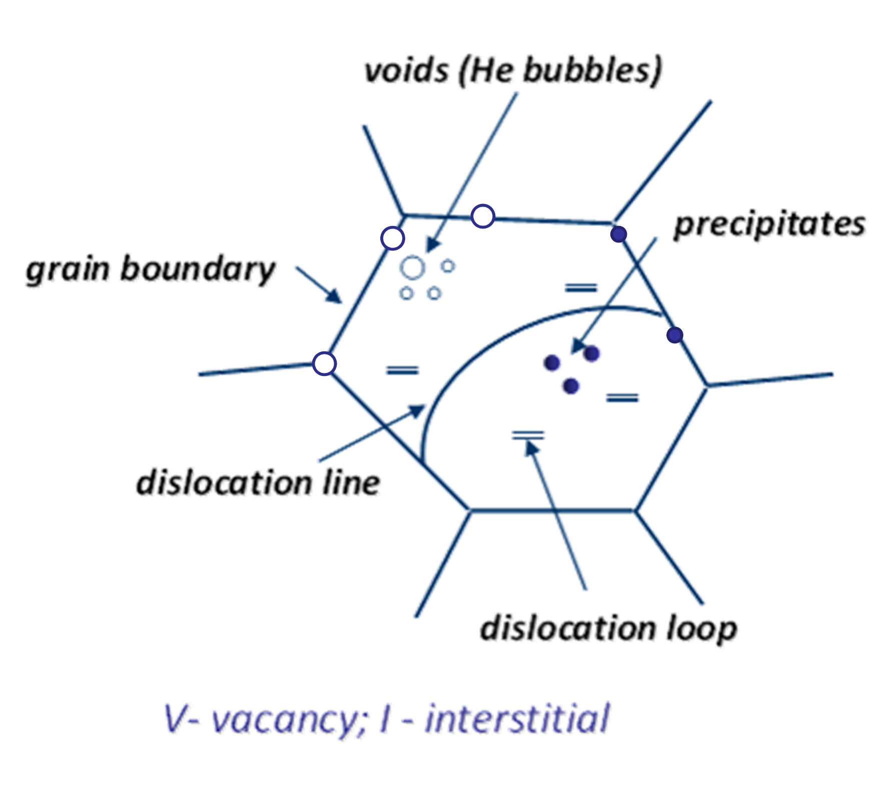
PF60 5x5x0.5mm	25 samples	⇦ Arrived, stored in RAL
S-200F 5x5x0.5mm	10 samples	
S-65 5x5x0.5mm	10 samples	

Method of manufacture: vacuum hot pressing

What can we expect during irradiation?



TE-56 beryllium, Chakin and Ostrovsky / JNM 2002



Microstructural response:

- *creation and agglomeration of point defects;*
- *creation of transmutation products;*
- *segregation (precipitation) or depletion on point defect sinks*

Possible irradiation effects:

- *reduction of fracture toughness*
- *irradiation induced hardening*
- *reduction of ductility*
- *reduction of thermal conductivity*

Experiments:

Investigation of the as-received Be

Investigation of the existing proton Be windows

- “real” GeV proton irradiation;
- irradiated volume is big enough for microstructural investigations and micromechanical tests

Simulation with ion irradiation experiments

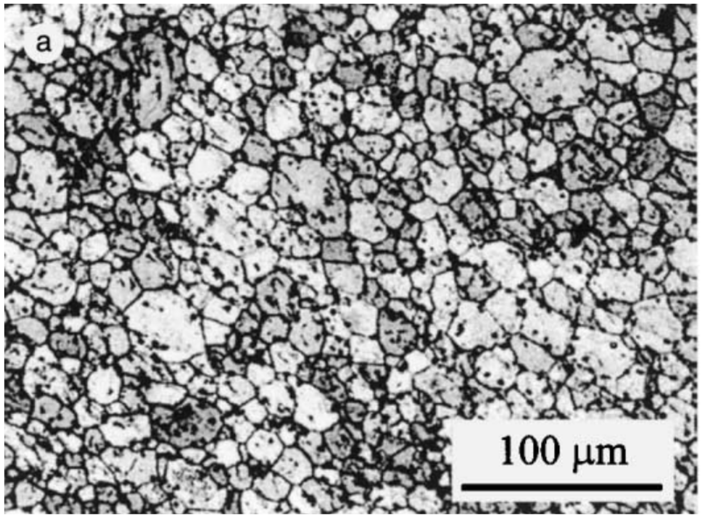
- flexibility of irradiation conditions
- observations of the evolution of the microstructure;
- reasonable correspondence of He/dpa ratio.

Low energy in-situ irradiation:

- easy variation of irradiation parameters;

High-energy irradiation + PIE

- microstructural and micromechanical tests data will be available



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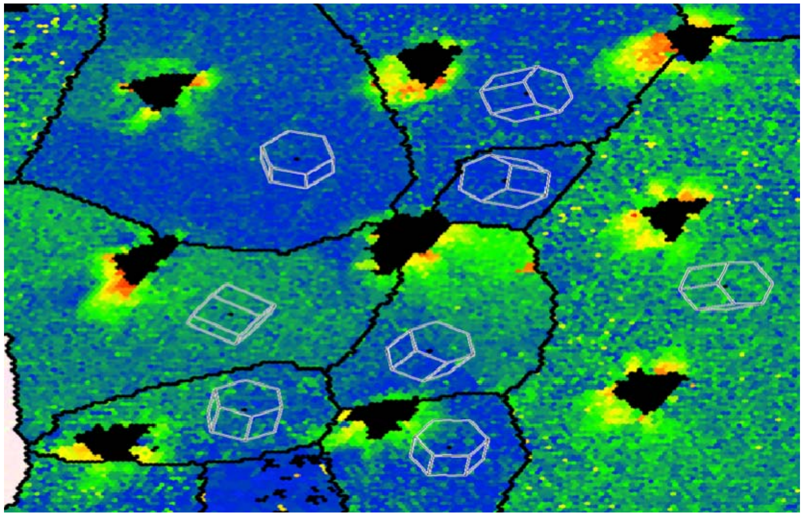


Local misorientation around indents made in pure Zr measured using EBSD
From http://energy.materials.ox.ac.uk/nuclear-projects/previous-projects/hydride-cracking-in-zirconium.html

Grain size

Texture

Precipitates

Dislocation structure

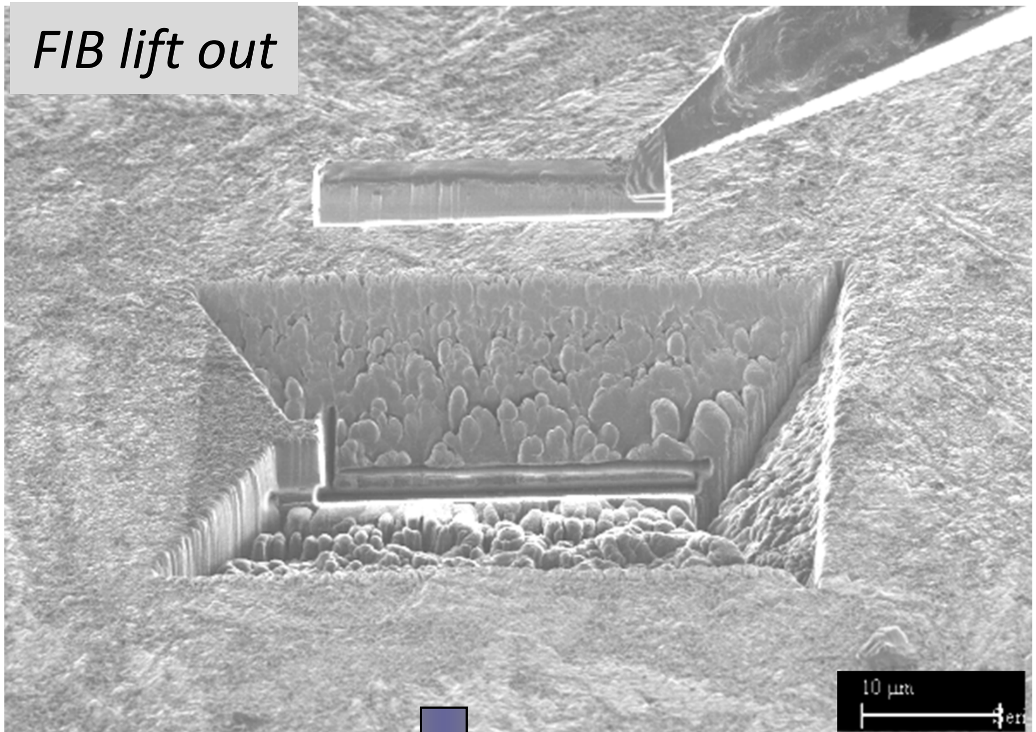
SEM + EDX + EBSD (Grain size, Texture, Precipitates)

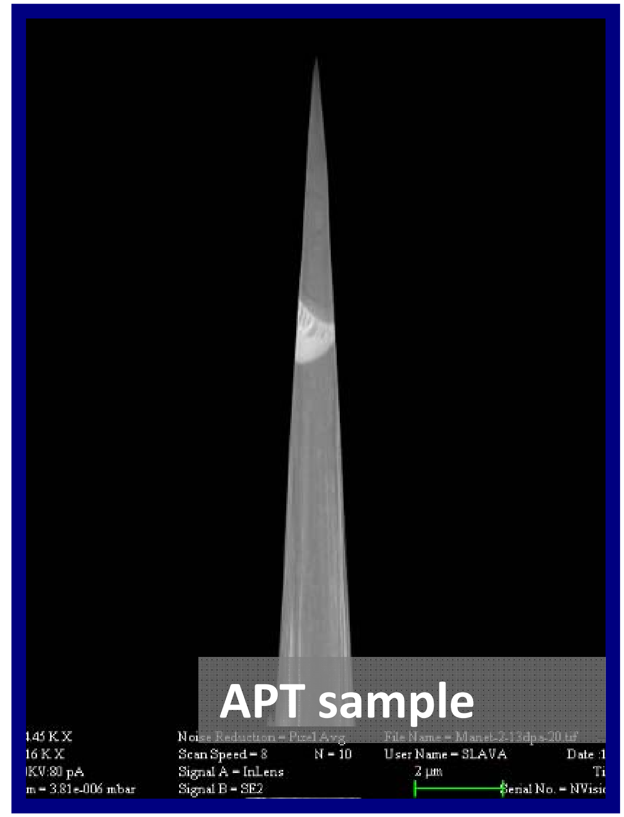
TEM (+ APT) (Precipitates, Dislocation structure)

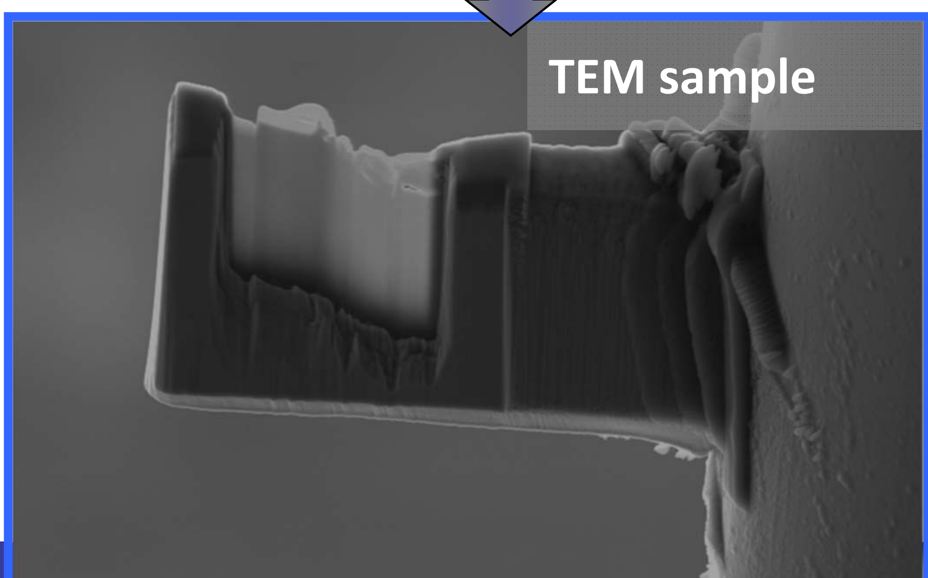
Samples preparation

Difficulties: Be dust is very toxic

TEM and APT – focused ion beam technique. The place is under consideration.
Most probably in CCFE lab







FIB lift-out

- minimize the toxicity of samples

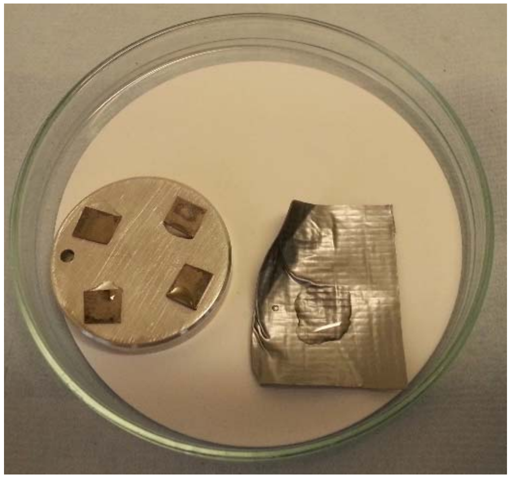
Samples preparation

Difficulties: Be dust is very toxic

TEM and APT – focused ion beam technique. The place is under consideration.
Most probably in CCFE lab

SEM+EDX+EBSD: should be mechanically mirror-polished up to colloidal silica (for EBSD)
- under development

Polishing machine for Be - repaired and cleaned.
Polishing procedure was developed



Be polishing will be made in the clean lab (used for Ta activity) at Science park in Begbroke

The procedure of the mechanical polishing will be made in several steps:

- primary polishing with SiC paper (Grade P2500),
- polishing with diamond paste (6 μm and 1 μm)
- final polishing with colloidal silica (0.06 μm).

Test polishing of non-hazardous samples was approved by the Safety office.

Some modifications will appear at the new site, final check with the safety office will be done

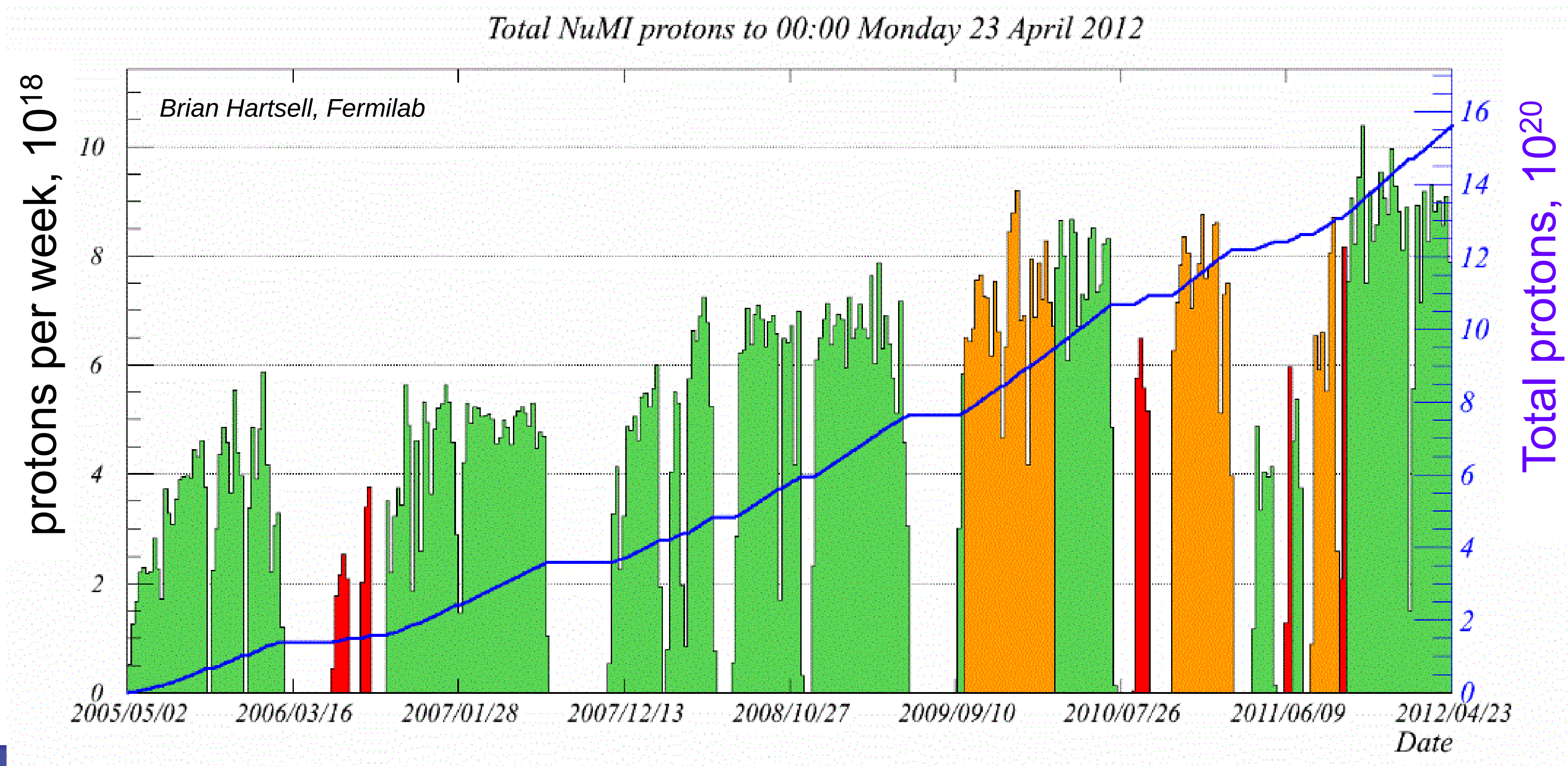
Occupational Health assessment - waiting for the information
Measurements of contamination level (air and surface) should be organised

Microstructure characterisation: EBSD trainings are in progress. The quality of Zr samples was relatively low, probably because of lack of water during the last polishing stage

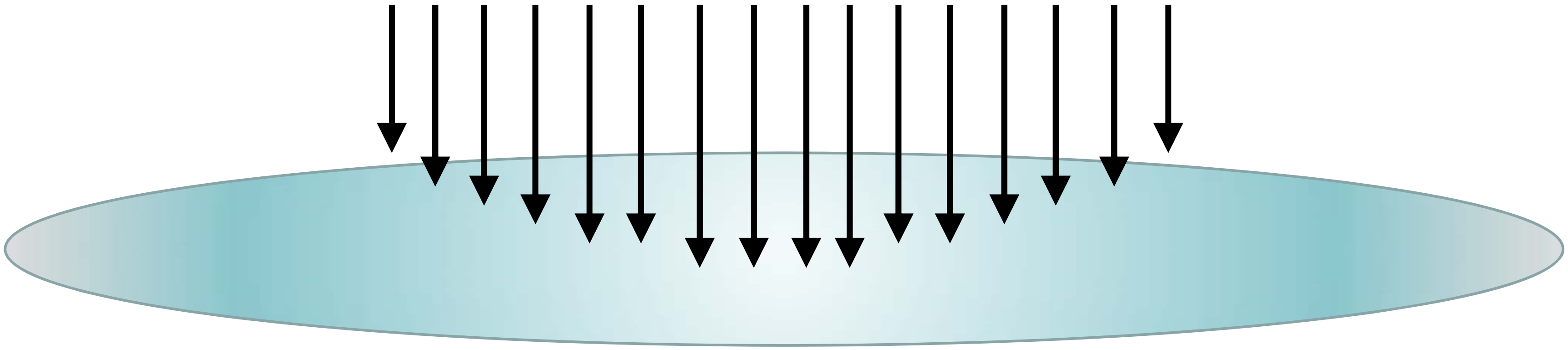
300 kW NuMI beam window

(MARS calculations of Brian Hartsell, Fermilab)

- 120GeV proton beam
- about 3×10^{13} protons per pulse, 0.5 Hz
- 1.57×10^{21} protons during its lifetime
- 1.1mm beam sigmas, X and Y
- T ≈ 200ºC

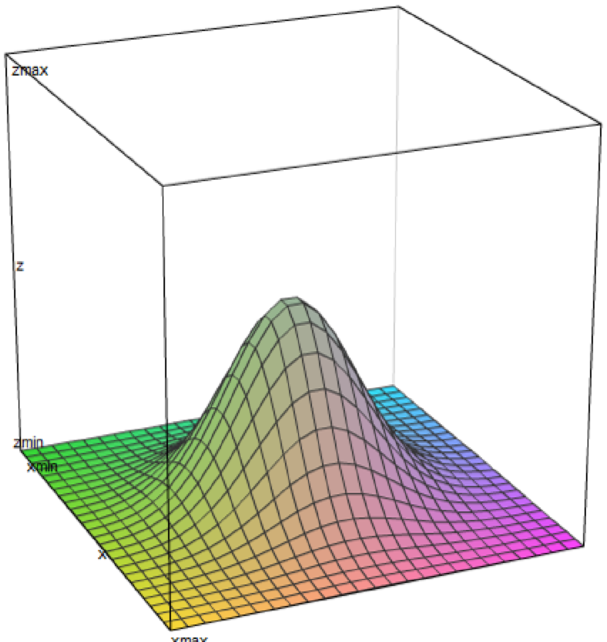


Gaussian distribution of the beam

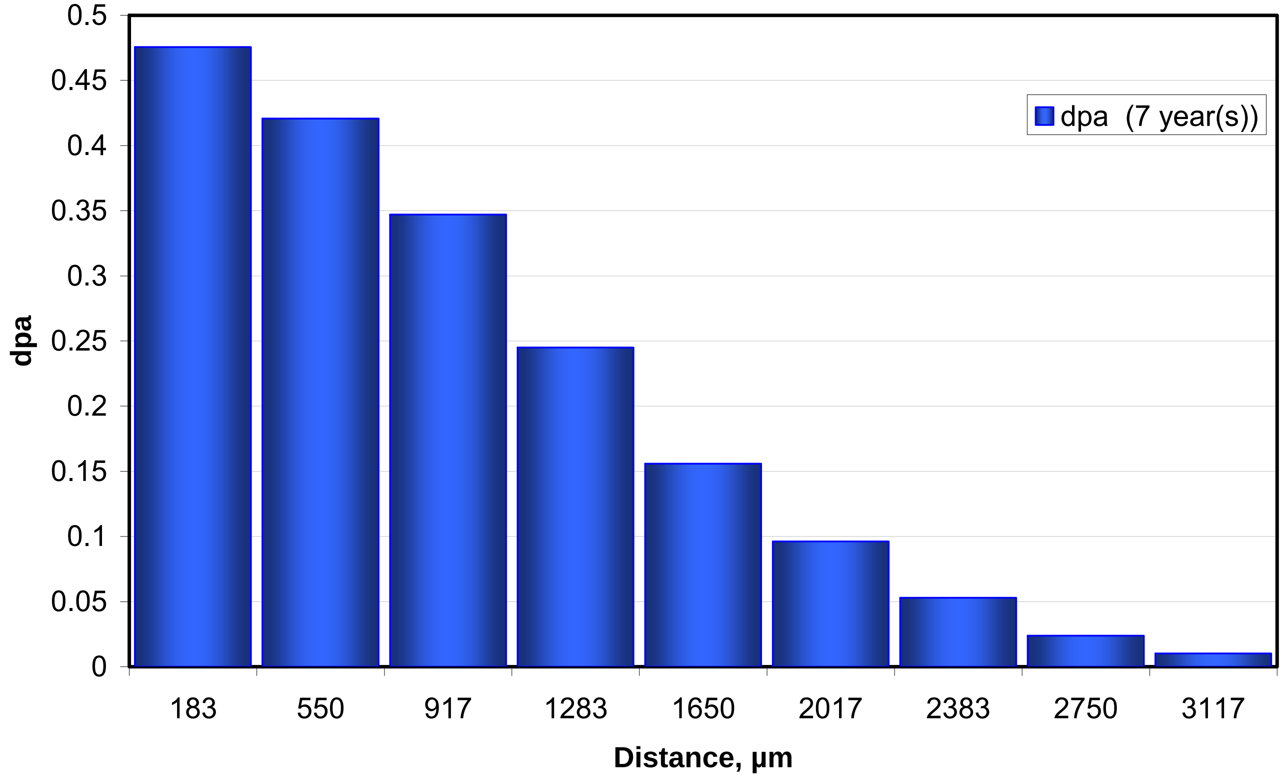


•**Radiation damage distribution is not monotonic**

We need to quantify the exposure



http://www.livephysics.com



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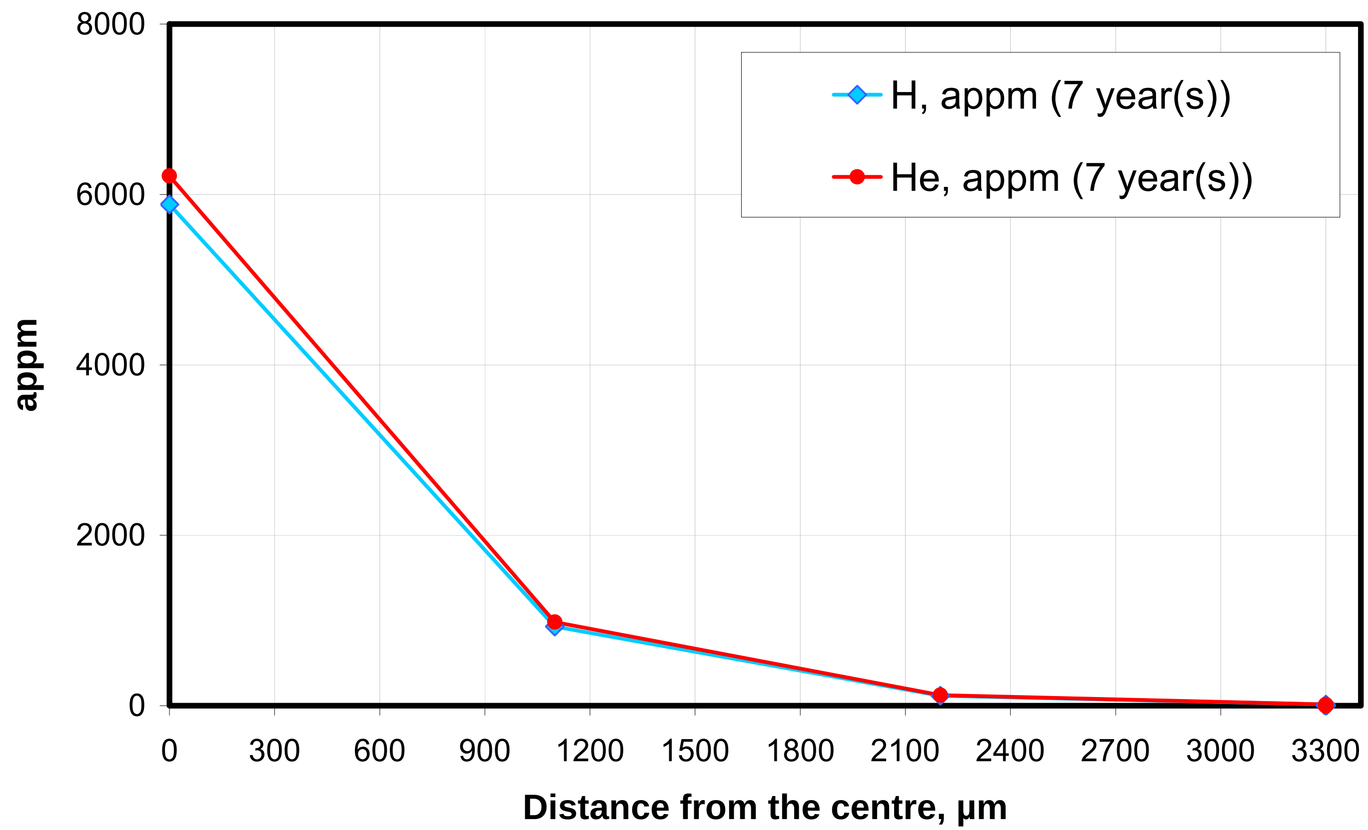
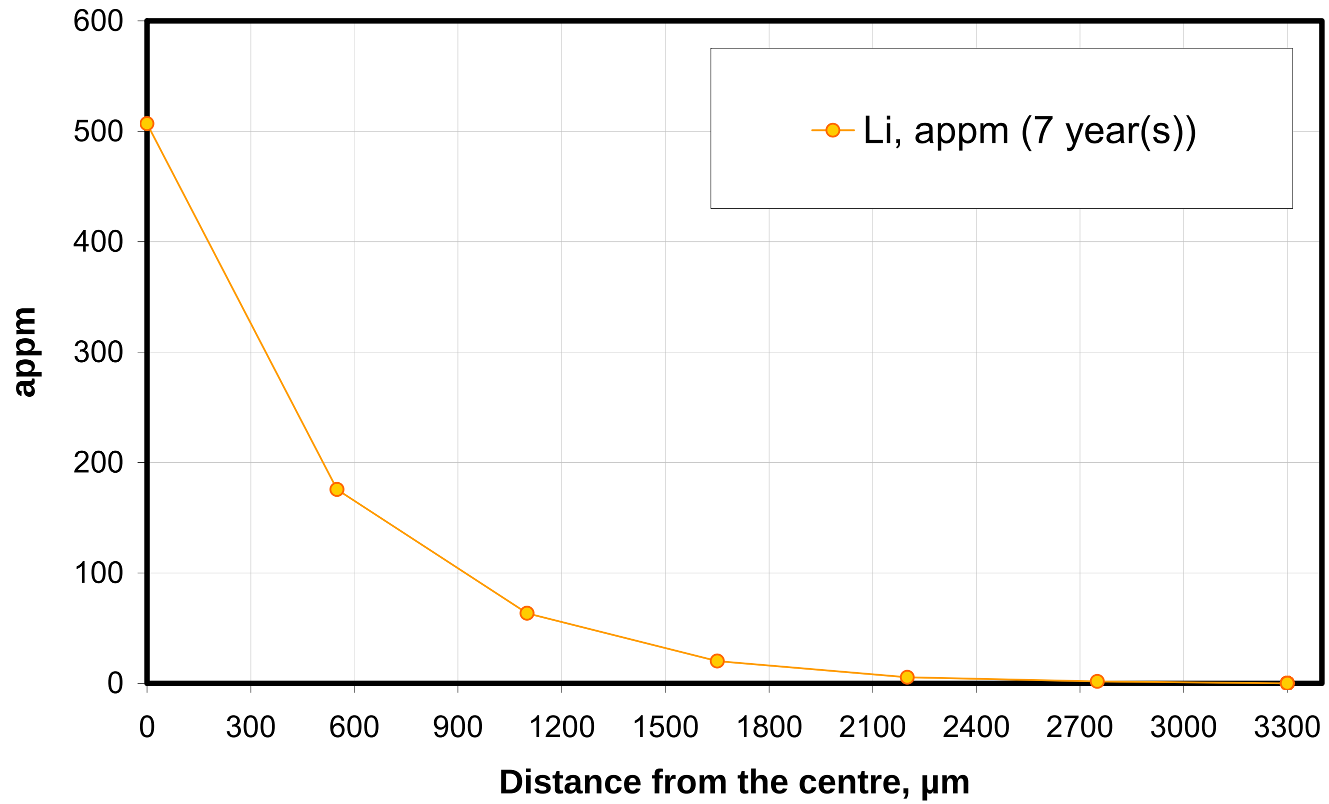


The main transmutation products are He and H

300 kW NuMI beam window

(MARS calculations of Brian Hartsell, Fermilab)

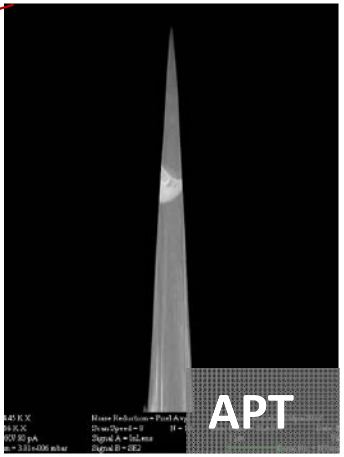
- 120GeV proton beam
- 1.57×10^{21} protons during its lifetime

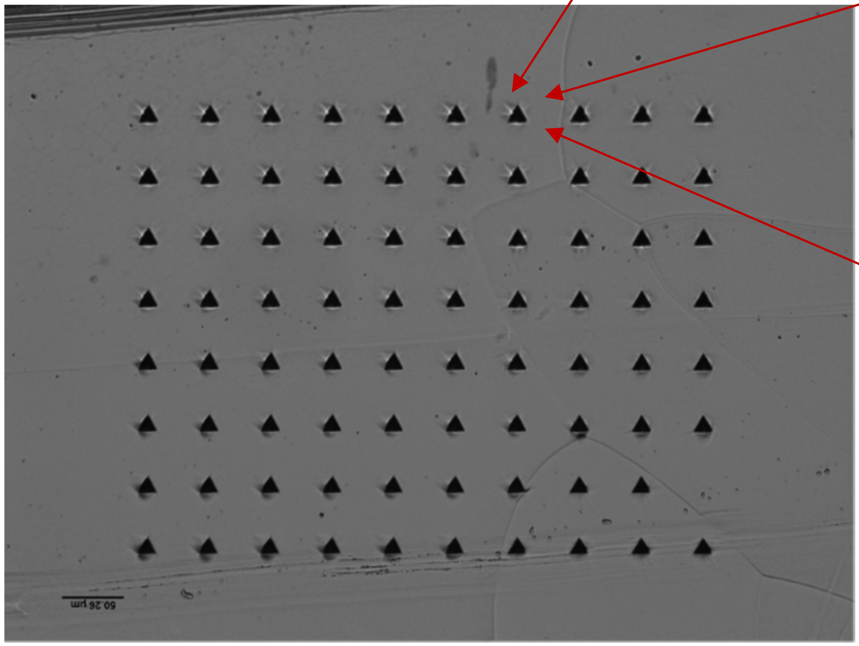


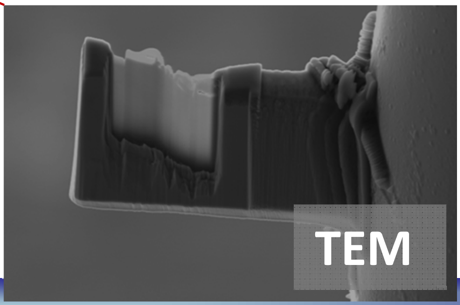
- The quantity of Li is not negligible (up to 500 appm in the center)
- APT for experimental validation of MARS code

Nano-hardness measurements:
- to find the Gaussian peak
- to estimate the irradiation effect

Local microstructural investigations







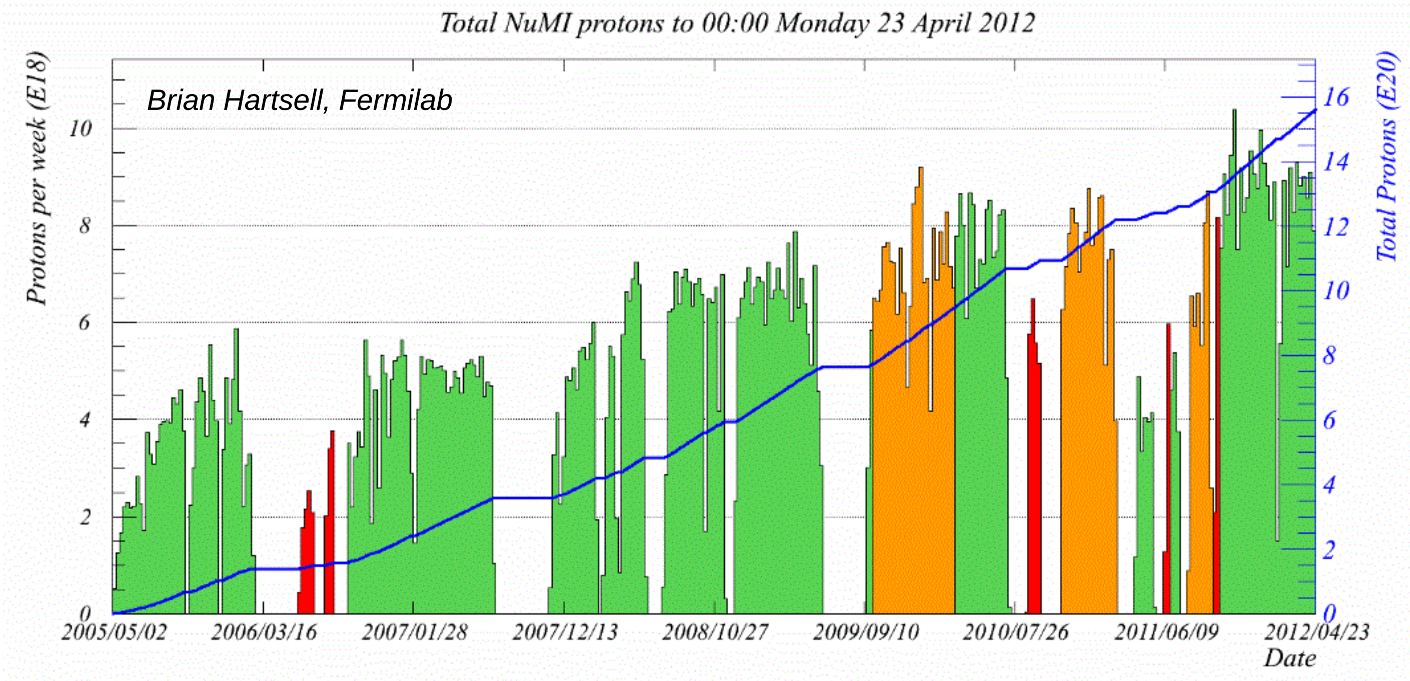
300 kW NuMI beam window

Spreadsheet for dose/appm is done

The activity calculations need to be crosschecked: Fermilab, my calculations, M.Gilbert (CCFE)

Can the activity be measured?

Activity of the window will affect the further steps, first of all – the place of samples preparations and experiments



We need to know the evolution of radiation effects over the time

Collaboration with HiRadMat project

Ion irradiation experiments

Experiments:

Simulation with ion irradiation experiments

- flexibility of irradiation conditions
- observations of the evolution of the microstructure structure;
- reasonable correspondence of He/dpa ratio.

Low energy in-situ irradiation:

- easy variation of irradiation parameters;

High-energy irradiation + PIE

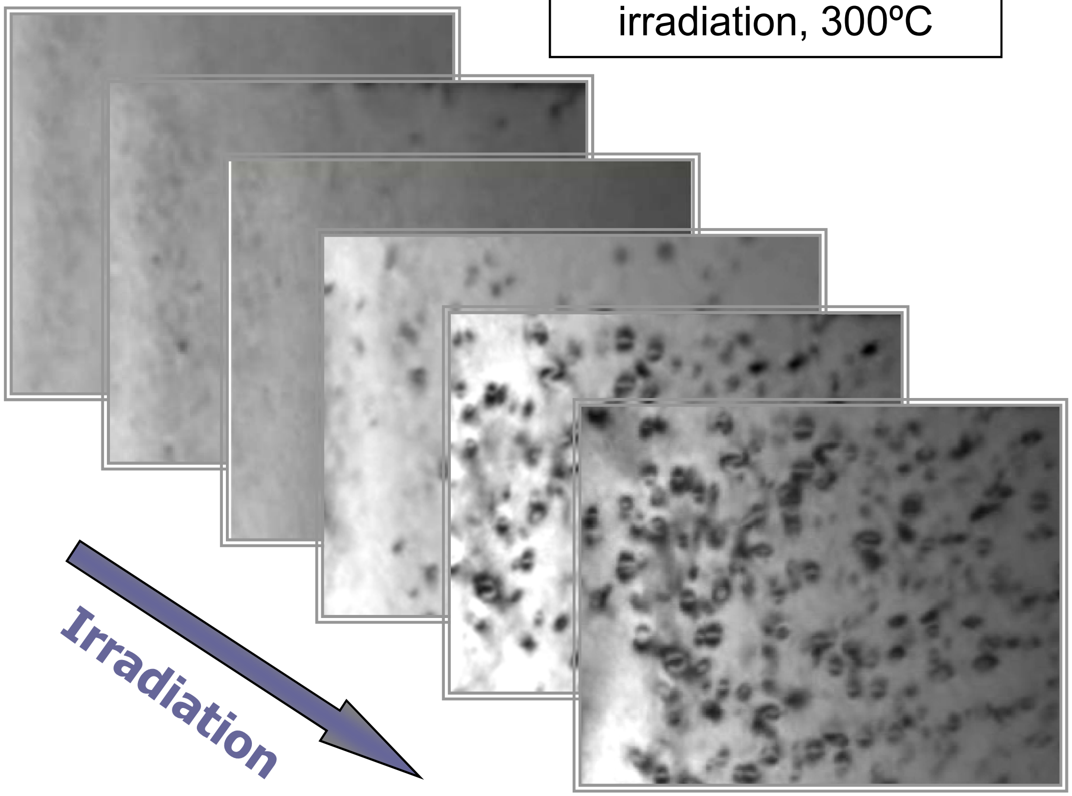
- microstructural and micromechanical tests data will be available

University of HUDDERSFIELD

Microscope and Ion Accelerator for Materials Investigations facility (MIAMI) University of Huddersfield , UK
(collaboration with Prof. S E Donnelly)

Ions: He+
Beam energy: ~ 10keV => peak of damage in the middle of TEM foil (SRIM)
Dose: up to 1 dpa
Temperature: 200ºC (300ºC, 600ºC)

Fe-9Cr alloy,
150keV Fe^{+} ions
irradiation, 300ºC



In-situ observations of the evolution of the microstructure

- evolution of number density and size of dislocation loops and/or He;

- Burgers vector and loops nature determination*

But: effect of the surface

Irradiation of APT tips?

Low energy ion irradiation:

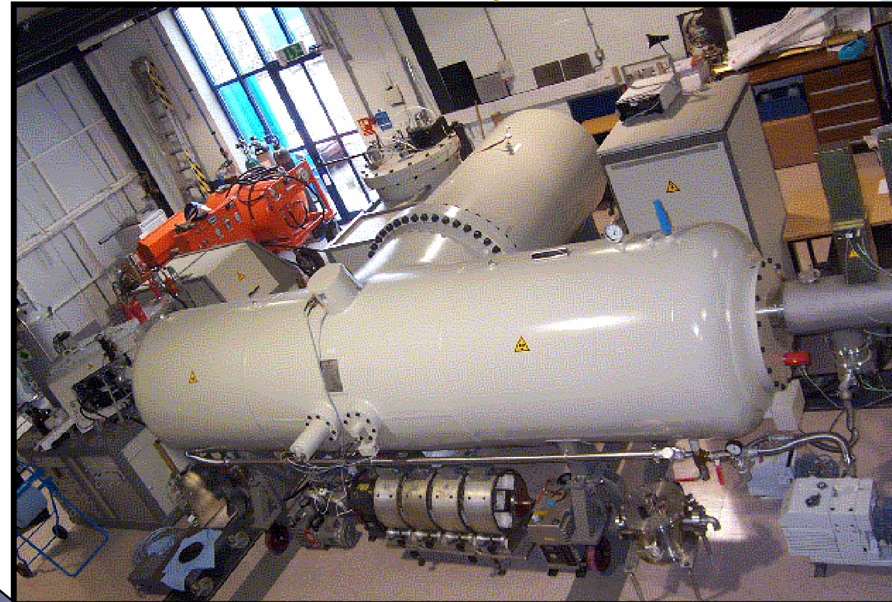
Approval of CCFE for Be FIB activity should be received

Next step: preparation of samples.

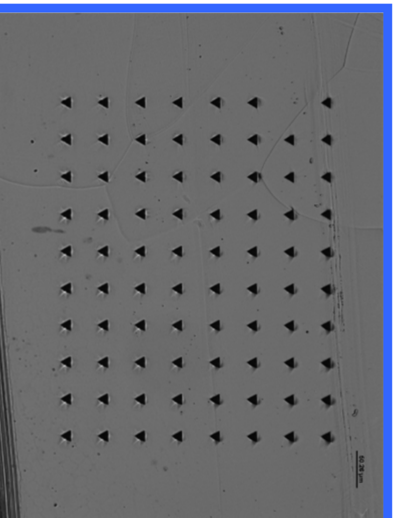
First samples – without flash polishing.

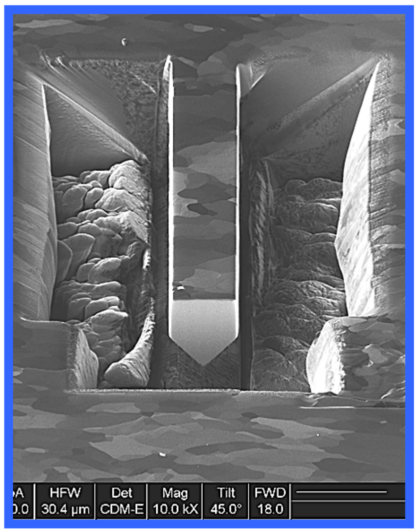
Surrey Ion Beam Centre, UK
(collaboration with Prof. R.Gwilliam)

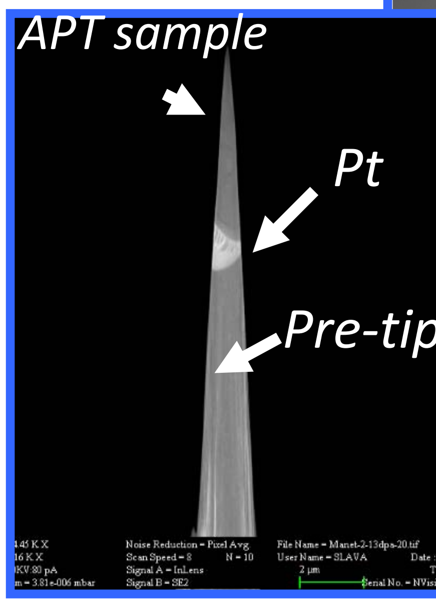
Ions: He+
Maximum beam energy: 2 MeV => 7.5µm implantation depth (SRIM)
Dose: up to 1 dpa
Temperature: 200ºC (100ºC, 600ºC)

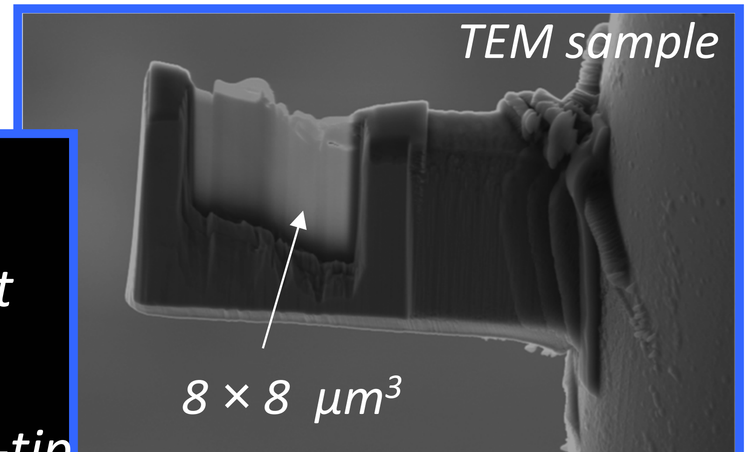


Micromechanical tests



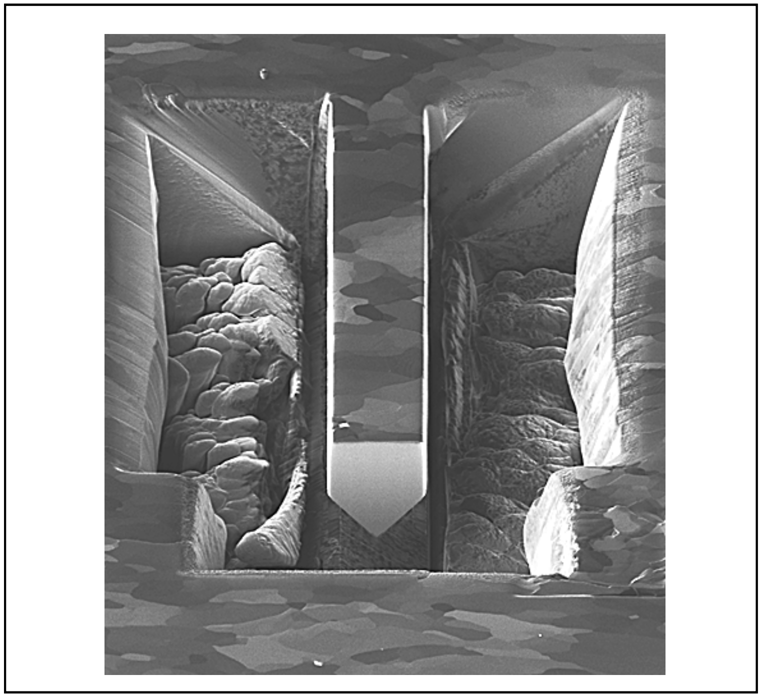




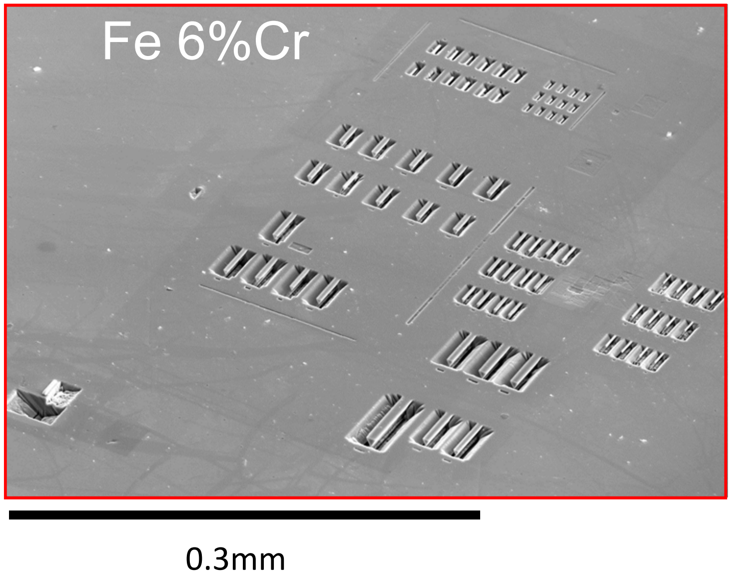


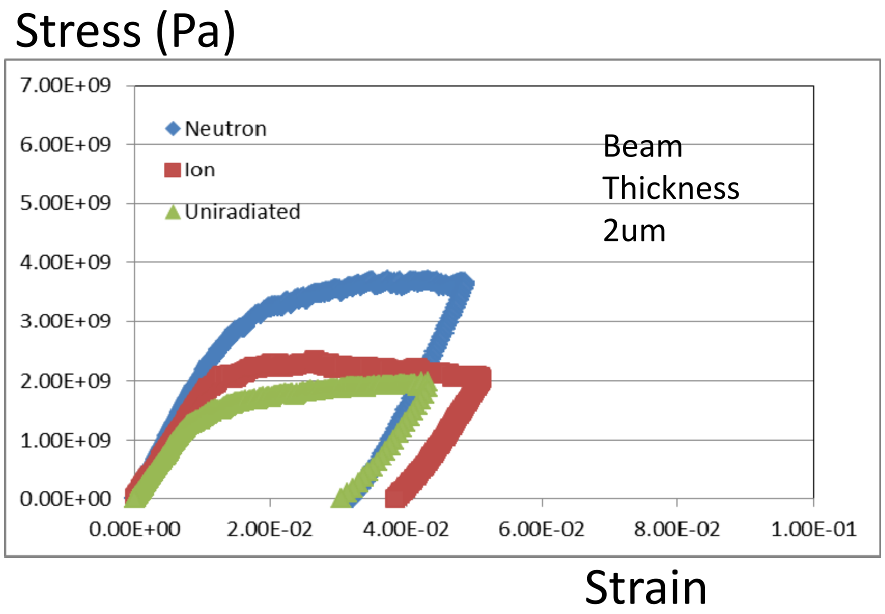
Why use micro-cantilever testing?

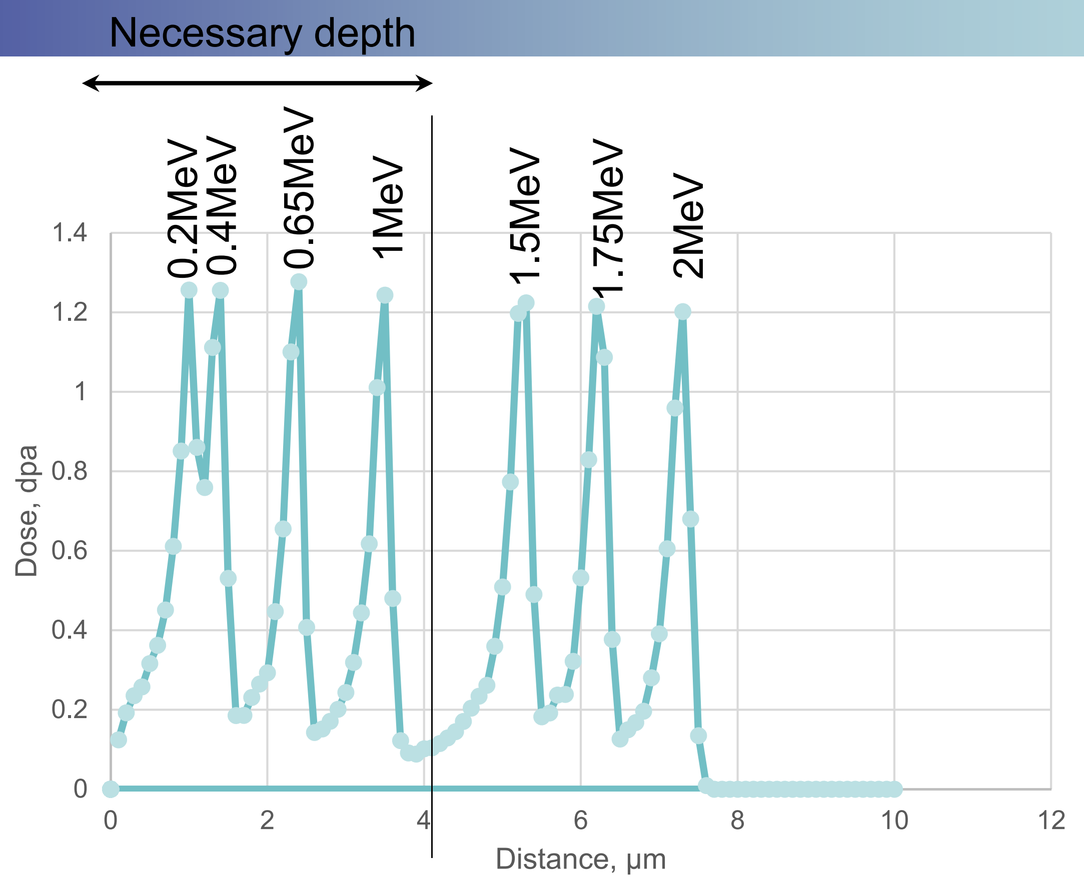
- Useful where only small samples are available (implanted layer)
- Need for a sample design that can be machined in surface of bulk samples
- Geometry that can be manufactured quickly and reproducibly



Chris Hardie
University of Oxford







High energy ion irradiation:

Damage peck from He implantation is very narrow:

- chain implantation (15 energies);
- tilt of the sample;
- use of degrader (Al foil)

High energy ion irradiation:

- mechanical polishing procedure should be finished;
- irradiation conditions should be determined.

Conclusions

Experimental investigation of beryllium within Radiate project should cover 3 main goals:

- characterization of existing GeV proton irradiated Be samples;
- simulation of proton irradiation effect by ion implantation experiments;
- prediction of the microstructural evolution for new irradiation conditions.

Top-priority steps:

- final approval of the mechanical polishing procedure (will clear the way for microstructural investigations and samples preparation for high-energy ion irradiation experiments);

- clarity with FIB (for TEM, APT, micromechanical tests and low-energy ion irradiation experiments).

The main difficulty: time consuming safety aspects

Thank you for your attention!

Beryllium in as-received state: grain size, existing precipitates, dislocations, homogeneity of impurities.

Beryllium in irradiated state:

Investigation of the existing Be window (NuMi from Fermilab)

Ex-situ and in-situ irradiation experiment

A) What do we expect to do?
- Investigation of the existing Be window
- One of the main difficulties – preparation of the samples - FIB.

a) Microstructure, TEM, APT, SEM+EBSD.

A-a Voids and loops.

A-b-Transmutation products exp vs simulation

A-c Amorphisation (clusters, BeO)?

b) Mechenical properties.

B-a Microhardness, microcantelivers.

What do we know?

enhancement of phase transformation

$$D^* = \alpha_V D_V C_V^* + \alpha_x D_x C_x^*$$

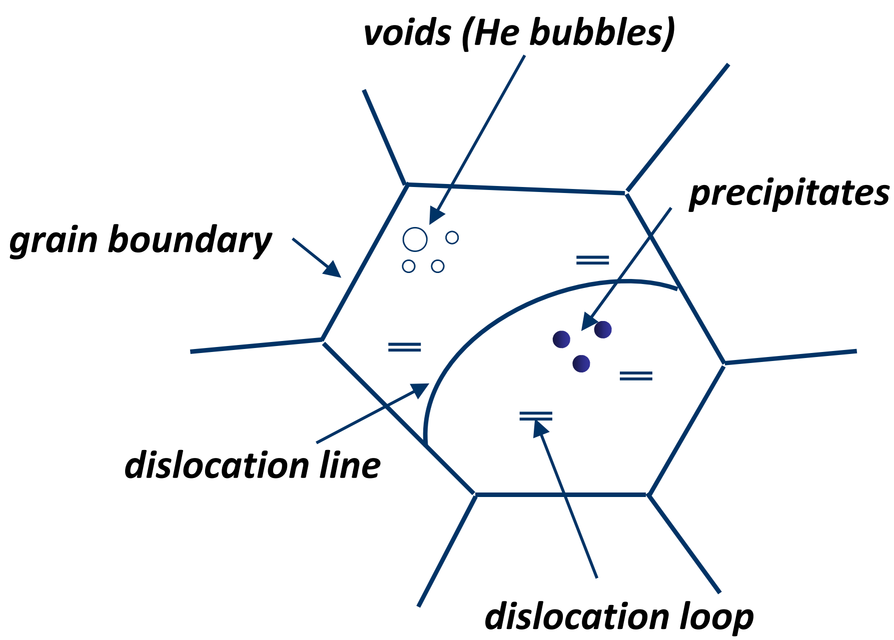
$$C_V^* > C_V^T$$

X - self interstitial atom; clusters of point defects

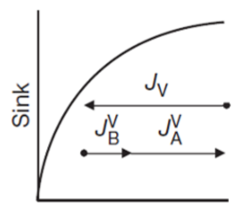
agglomeration of point defects

- **• self-interstitials**
  - clusters;
  - dislocation loops.
- **o vacancies**
  - voids;
  - dislocation loops.

segregation (precipitation) or depletion on point defect sinks

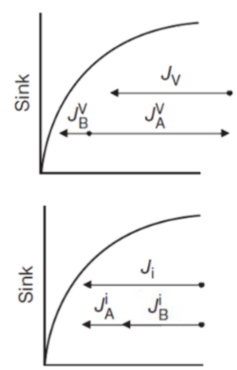


V- vacancy; I - interstitial



inverse Kirkendall effect

If $D_B^V < D_A^V$ ⇒ depletion of A atoms

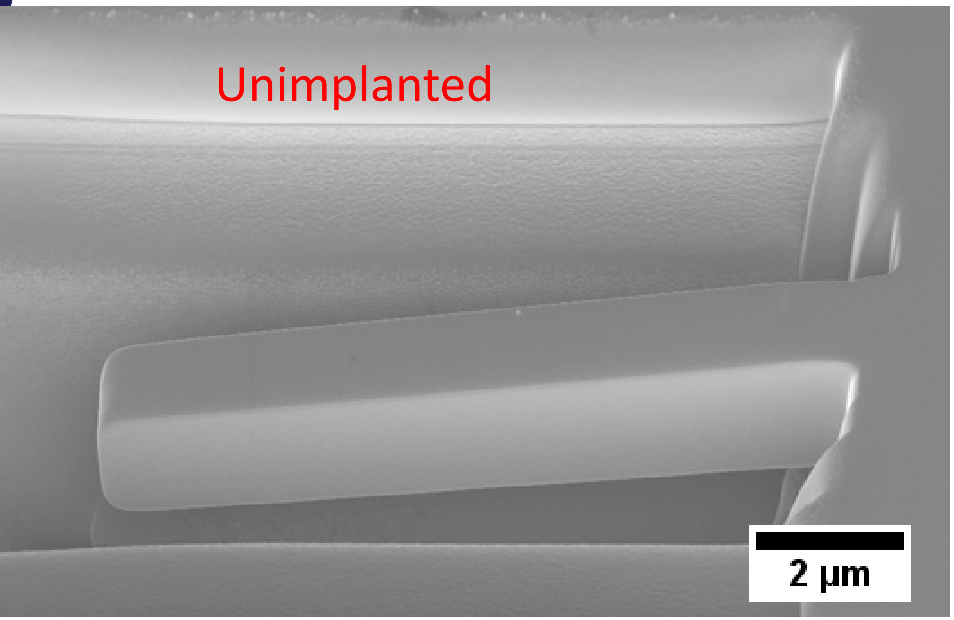


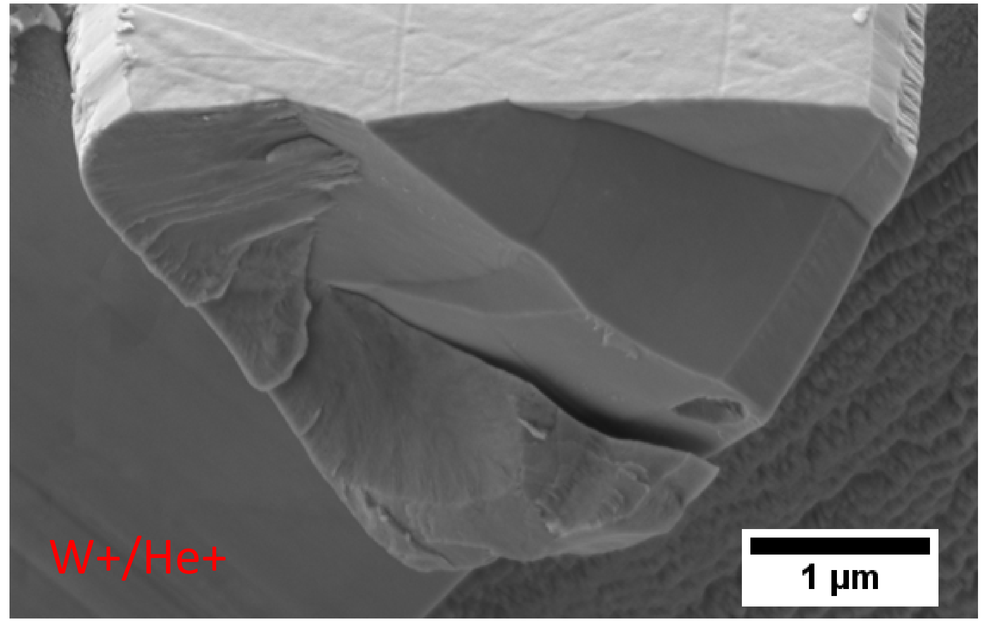
drag effects

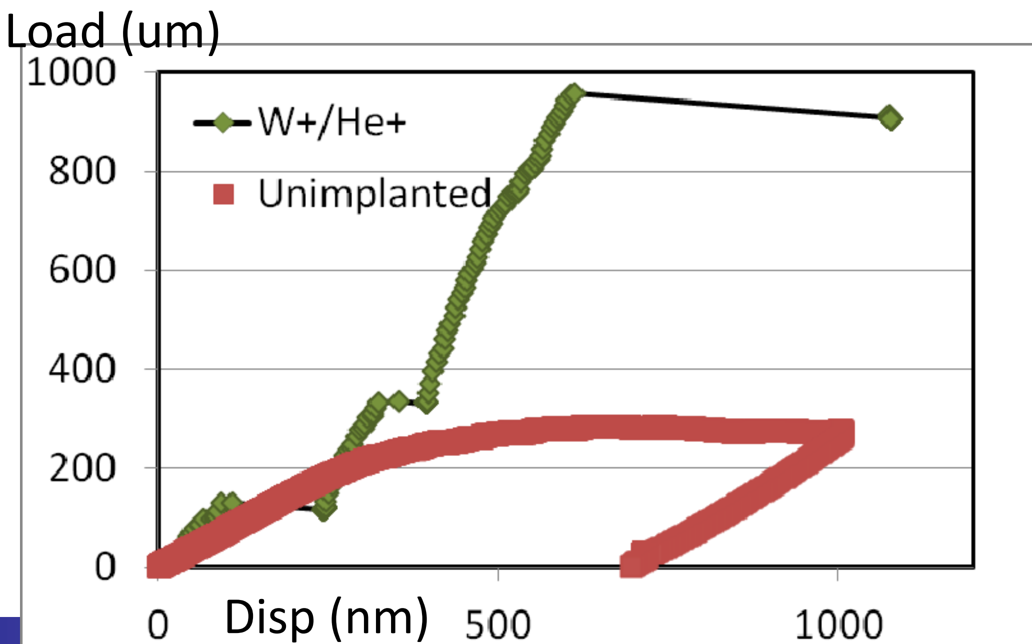
B-V complexes
or
B-I complexes

⇒ Segregation of B atoms

Tested Cantilevers







Helium has complex effects on both yield and fracture properties of tungsten

Differences between results for micro-cantilevers and nanoindentation show the difficulty of relying on one type of test

Content

Beryllium

Irradiation conditions for beryllium

Experimental program

A) 3 Be grades. We need to classify: grain size, precipitates, dislocations, texture, homogenity of elements.

B) What do we expect to do?

- Investigation of the existing Be window
- One of the main difficulties – preparation of the samples - FIB.

a) Microstructure, TEM, APT, SEM+EBSD.

A-a Voids and loops.

A-b-Transmutation products exp vs simulation

A-c Amorphisation (clusters, BeO)?

b) Mechenical properties.

B-a Microhardness, microcantelivers.

c) in-situ ion irradiation of (Simulation of proton irradiaiton by He implantation experiments); Evolution of the microstructure as a function of flux, fluence and temperature

Current and future Be spallation target operating conditions*

Application	Operating Temperature °C	Dpa dose	Helium ppm/year ppm/dpa	Hydrogen ppm/year ppm/dpa	Beam Conditions
LBNE Window	200/300	0.23/year total 7 10^{-4} to 7 10^{-9} dpa.s^{-1}	He 659 appm/year He/dpa = 2865	H 235 appm/year	700kW, 120GeV, 1Hz, σ_{rms} = 1.3mm
LBNE Target	350/550	0.5/year total 2.5 10^{-3} to 2.5 10^{-8} dpa.s^{-1}			2.3MW 120GeV1Hz, σrms = 1.3mm

Possible irradiation effects:

- reduction of fracture toughness
- irradiation induced hardening
- reduction of ductility
- reduction of thermal conductivity

** From: Literature Review On The Irradiation Response Of Be, W And Graphite for Proton Accelerator Applications/ R.B. Jones (BazNutec), G. Hall (University of Manchester), B. Marsden (University of Manchester) and C. A. English (NNL)// . NNL (13) 12703. Issue 1*

Radiation damage → dose, temperature and He concentration dependence

PIE of Be windows/targets

Ion irradiation and implantation experiments

TEM → *defect clusters and He bubbles*

APT → *behaviors of impurities (precepitations, segregations at point defect sinks*

Micromechanical tests → *changes of mechanical properties*

Precipitates **Fe and Al rich precipitates** may affect **ductility** and **creep strength** (A.W. Jones, R.T. Weiner, J. Common Met. 6 (1964) 266.)

Grain, twin and sub-grain boundaries and dislocations can be the preferential places for precipitation of **Fe-rich phases** during ageing of Be-0.25%Fe. Dislocation can locked by precipitates leading to the increase of **hardness** (S. Morozumi, N. Tsuno, S. Koda, Trans. Jpn. Inst. Met. 10 (1969) 64.)

Intermetallic **Fe/Al/Be inclusions** are the preferential sites for **corrosion** pit initiation, some corrosion pits had also initiated at elemental **Si** and **carbide** inclusions. (J.S. Punni, M.J. Cox, Corros. Sci. 52 (2010) 2535)

Al and Mg can form **low melting point eutectics** in Be, that might influence the mechanical behaviour of Be.

$e^{(-((x^2)/3+(y^2)/3))}$