Summaries of Material-Studies Status Reports

Steve Roberts
U. Oxford
May 19, 2015

2nd RaDIATE Collaboration Meeting
RAL

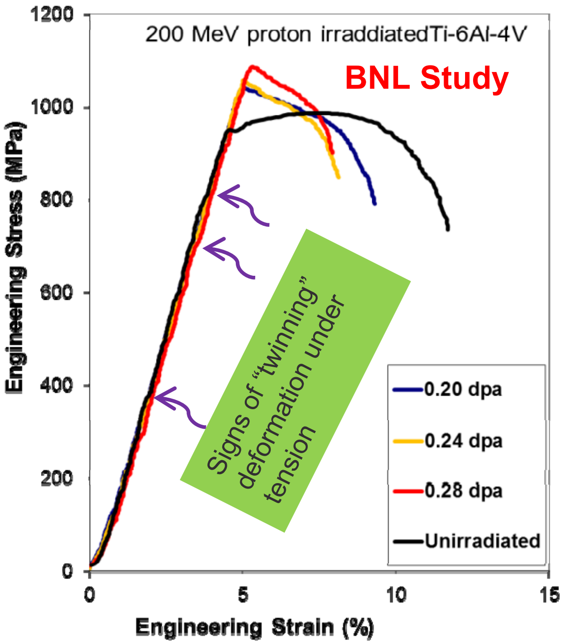
Ti Alloys

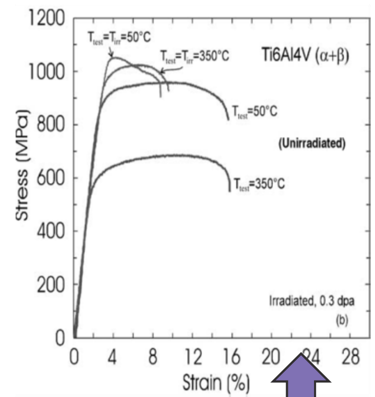
Nick Simos, Brookhaven NL

Titanium Alloy Irradiation/Characterization Studies at BNL

- An array of irradiation damage and post-irradiation characterization studies have been under way at BNL for Ti-alloys that include
  - The (α + β) Ti-6Al-4V alloy
  - The β-titanium alloy Gum metal (Ti-21Nb-0.7Ta-2.Zr-1.2O)
- Both alloys were investigated as candidates for HP targets in the Neutrino Factory initiative
- The (α + β) Ti-6Al-4V has also been studied as a substrate of ceramic nano-structured coatings for potentially nuclear applications (fast neutron and elevated temperatures)
- 200 MeV protons and spallation generated fast neutrons at the BNL complex were used for irradiation induced damage
- Macroscopic post-irradiation and EDXRD/XRD studies at the BNL synchrotrons were employed to study microstructural changes and damage

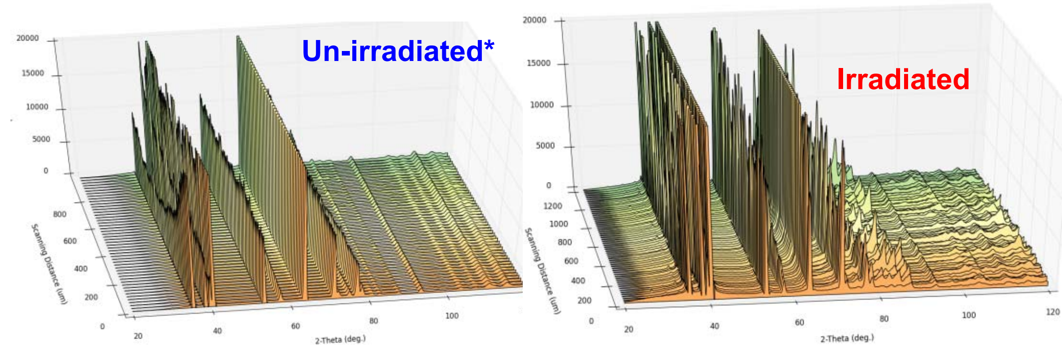
Titanium Alloy Irradiation/Characterization Studies at BNL – Ti-6Al-4V

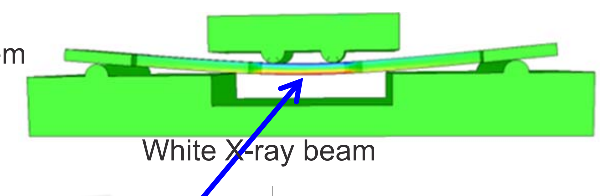


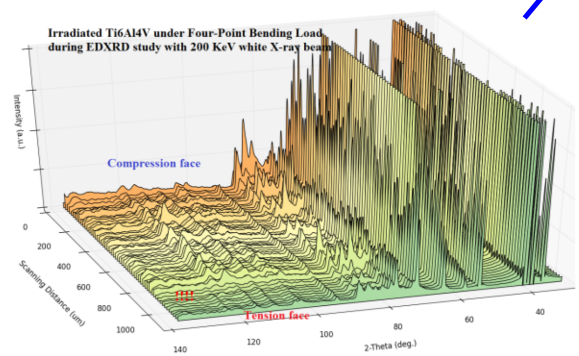


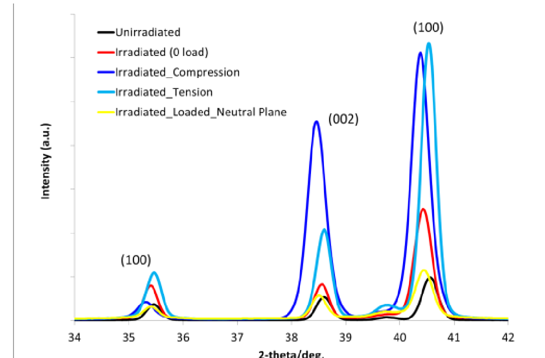
Tensile and fracture toughness properties of unirradiated and neutron irradiated titanium alloys, S. T€ahtinen et al *Journal of Nuclear Materials* 307–311 (2002) 416–420

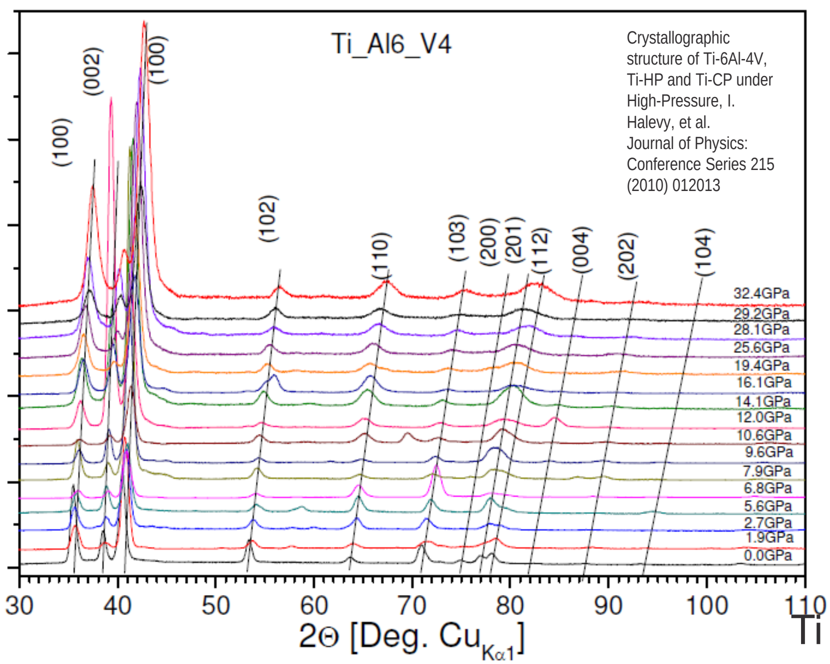
EDXRD Studies of proton-irradiated Ti6Al4V





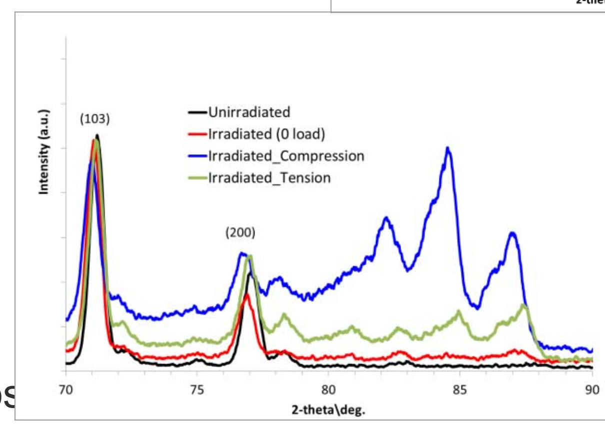






Crystallographic structure of Ti-6Al-4V, Ti-HP and Ti-CP under High-Pressure, I. Halevy, et al. Journal of Physics: Conference Series 215 (2010) 012013

Confirmation of phase evolution/appearance under compression



Titanium Alloy Irradiation/Characterization Studies at BNL - Gum Metal

Ti-Nb-based multi-functional alloys, known as **gum metals**, exhibit extraordinary properties of super-elasticity, super-plasticity, low elastic modulus and high strength.

HOWEVER, serious debate exist as to which mechanism is responsible for its deformation (martensitic transformations or rather unconventional localized lattice distortions.

Stress and thermally-induced martensite transformations and their role in super-elasticity and super-plasticity of the multifunctional β alloy Ti-21Nb-2Ta-3Zr-1.2O are being explored

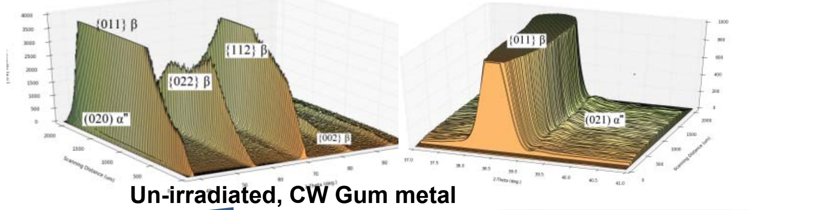
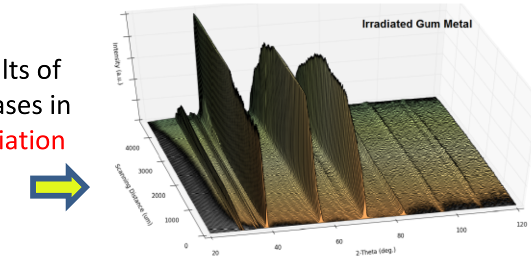


Fig 1b: EDXRD results of alpha and beta phases in the alloy plus irradiation induced phases



Summary of Macroscopic and Microscopic Observations

- XRD measurements on the multi-functional β alloy Ti-21Nb-2Ta-3Zr-1.2O post-plastic deformation established:
  - role of stress-induced α“ and/or γ martensite transformations in the dislocation-free deformation
  - origin of the pronounced tension-to-compression asymmetry
  - reversible nature of thermally-induced transformations responsible for macroscopically observed transition of the beta-alloy between 380-560 C
- Correlation of the EDXRD and XPD results revealed presence of the alpha phase as a result of cold-working (Fig. 1)
- Stress-induced beta phase evolution is accompanied with the appearance of α“ and γ phases that respectively control the compressive and tensile deformation (Fig.2)
- Radiation-induced phase evolution and phase appearance observed
- Thermally induced transformations currently under study and data analysis

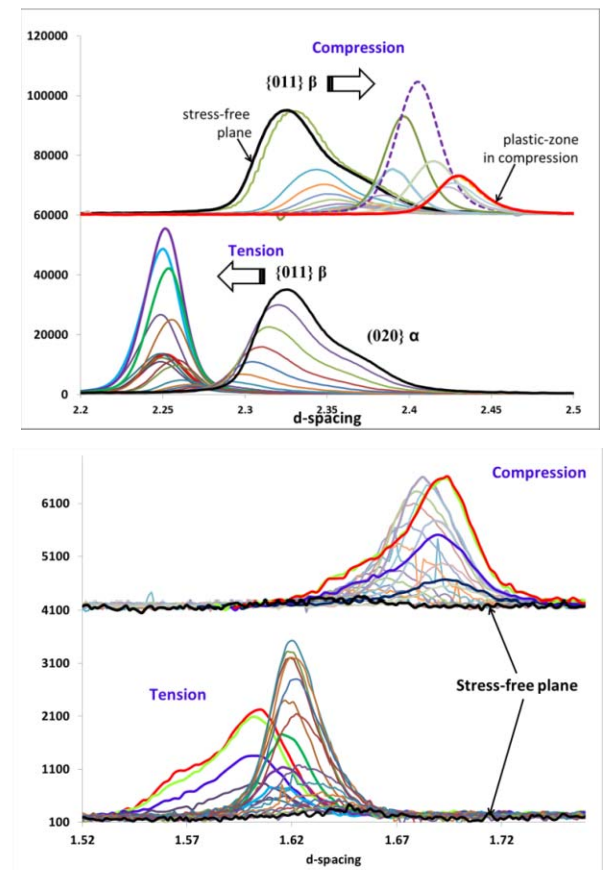


Fig 2: Evolution of {011} β (top) due to compression and tension accompanied by the martensite transformations (α“ and/or γ) at bottom

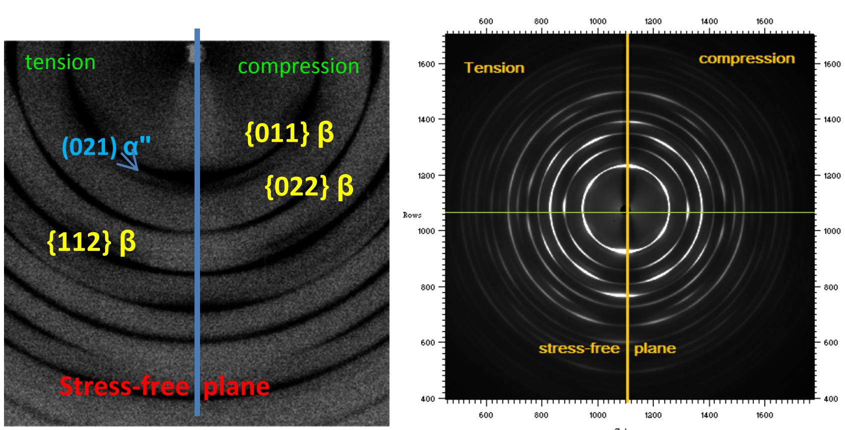


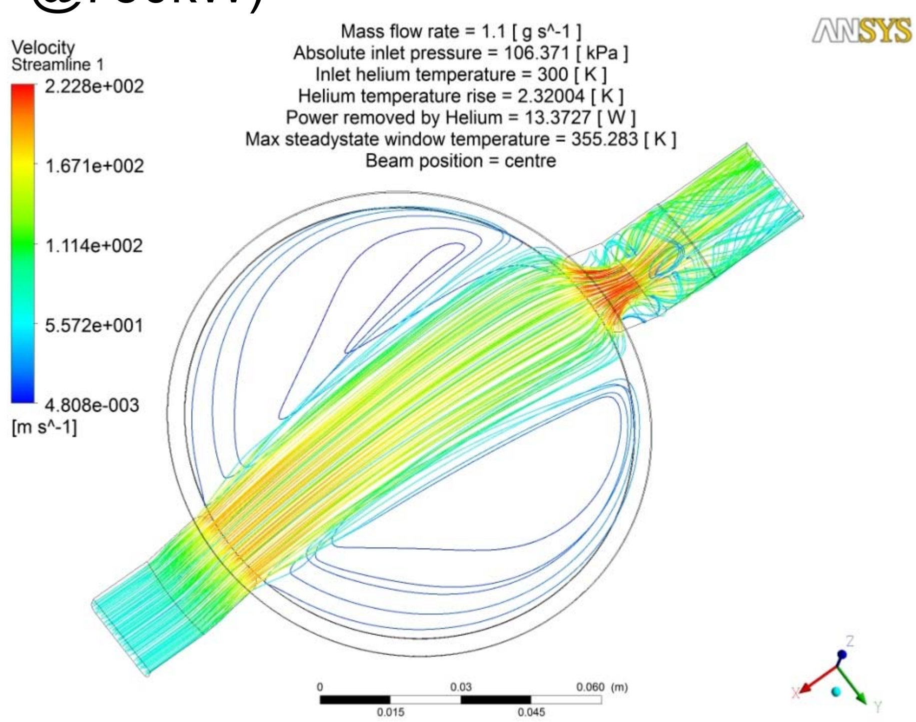
Fig 1a: XPD analysis of the gum metal following plastic deformation with detector at far (left) and near (right)

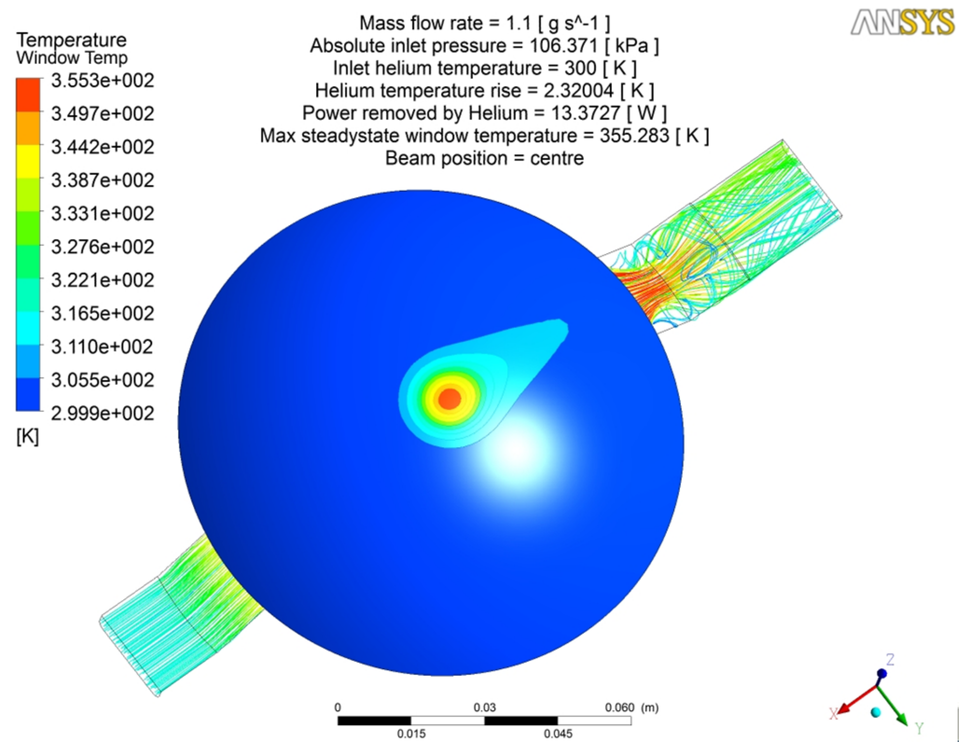
T2K Ti-6Al-4V Beam Window

Mike Fitton, Rutherford Appleton Lab.

T2K Beam window

Design: 2 x 0.3mm thick titanium domes cooled by helium flow
Material: Titanium alloy bar Ti6Al-4V (Grade 5) (Windows I & II)
Proton beam : 30GeV, 4.2mm sigma
Beam power: 345kW (750kW window design power)
Number of protons to date: 1.04×10^{21} (May 2015 and still in service)
Max temp (at beam centre): 52°C estimate at current beam power (82°C @750kW)

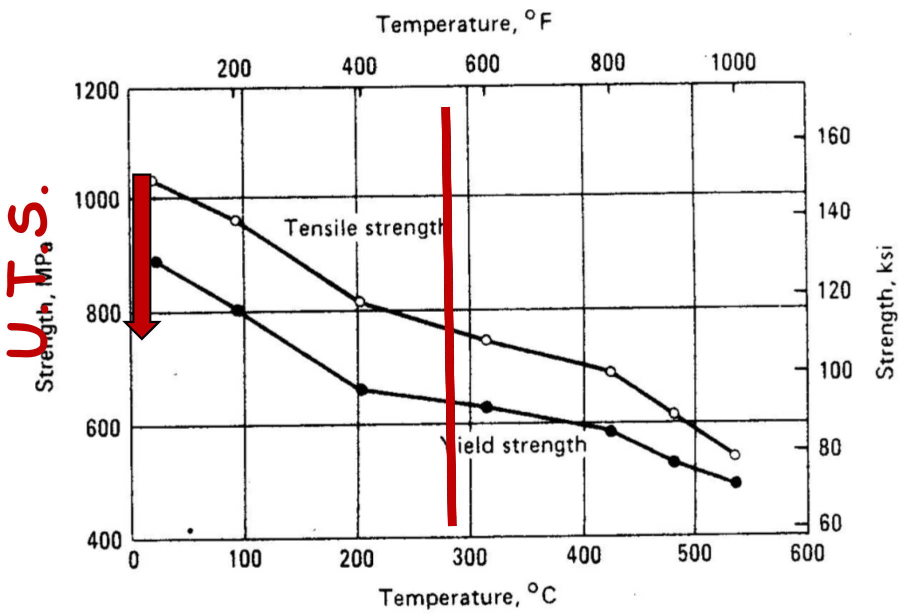


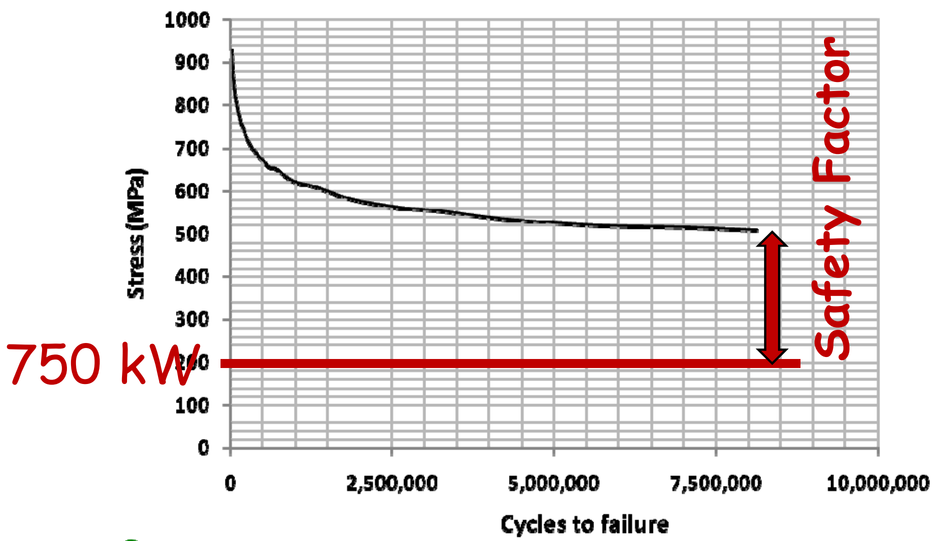


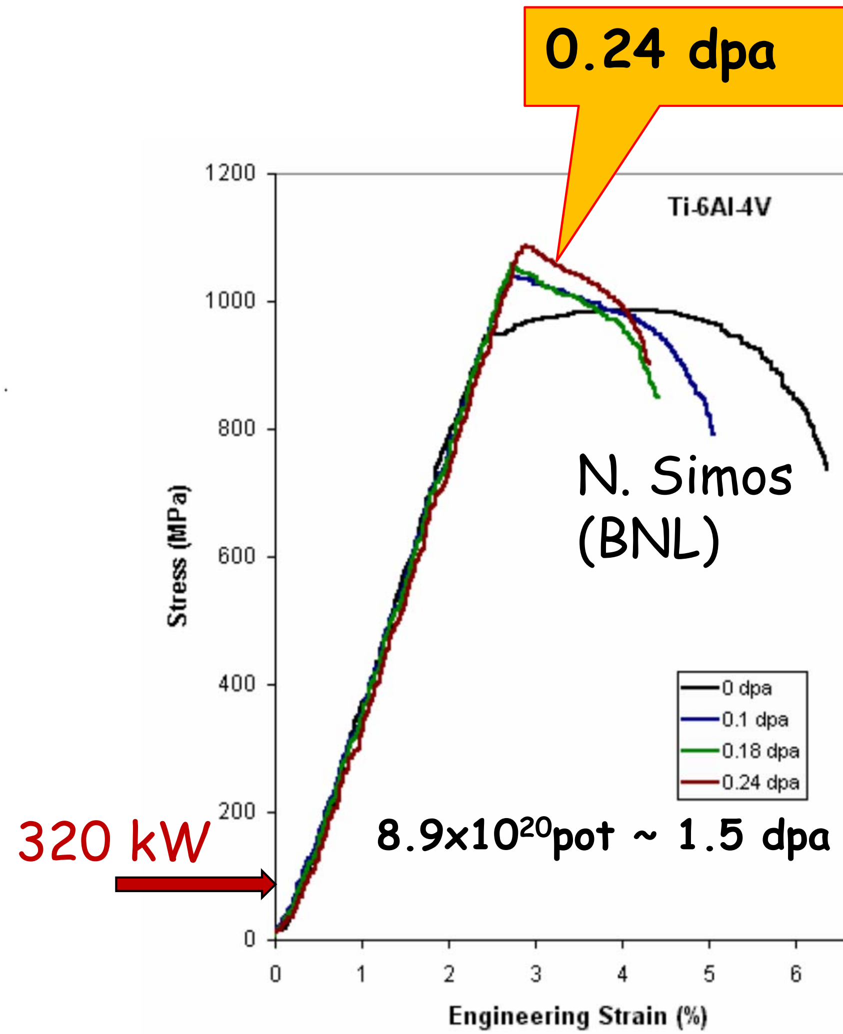
High Power Targets Group
Mike Fitton

Science & Technology Facilities Council

Effects of elevated temperature, fatigue and radiation damage on T2K beam window







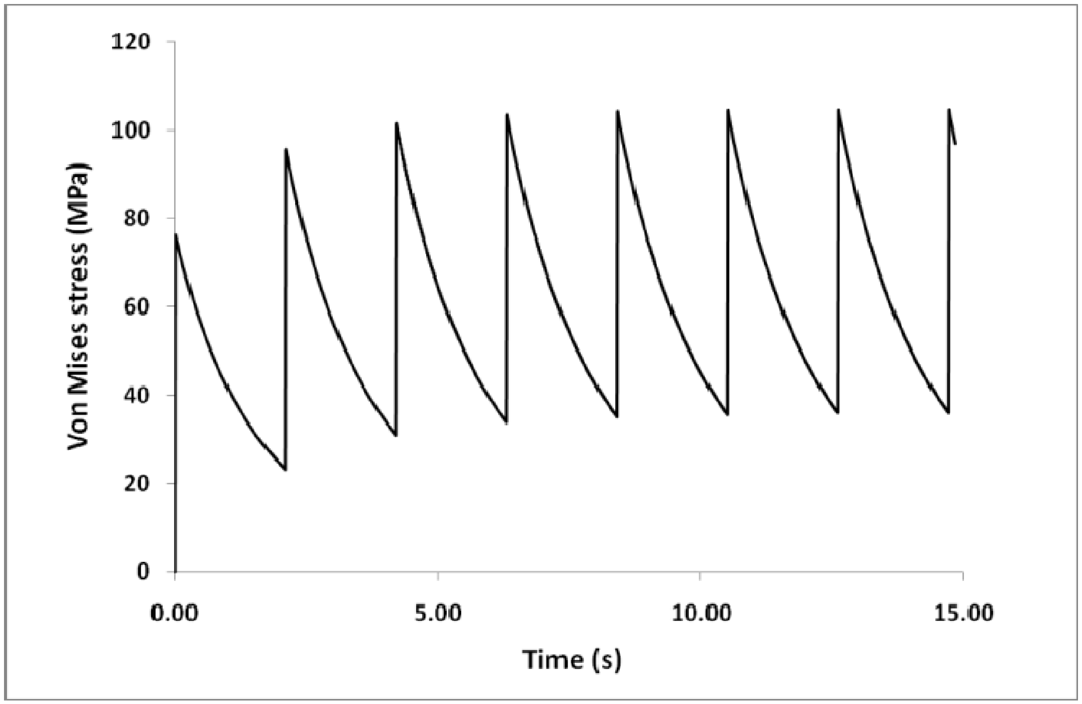
Significant loss of ductility at 0.24 dpa
Now likely to be entirely brittle at 1.5 dpa
Does it matter?
Low stress at moment

Science & Technology Facilities Council
Rutherford Appleton Laboratory

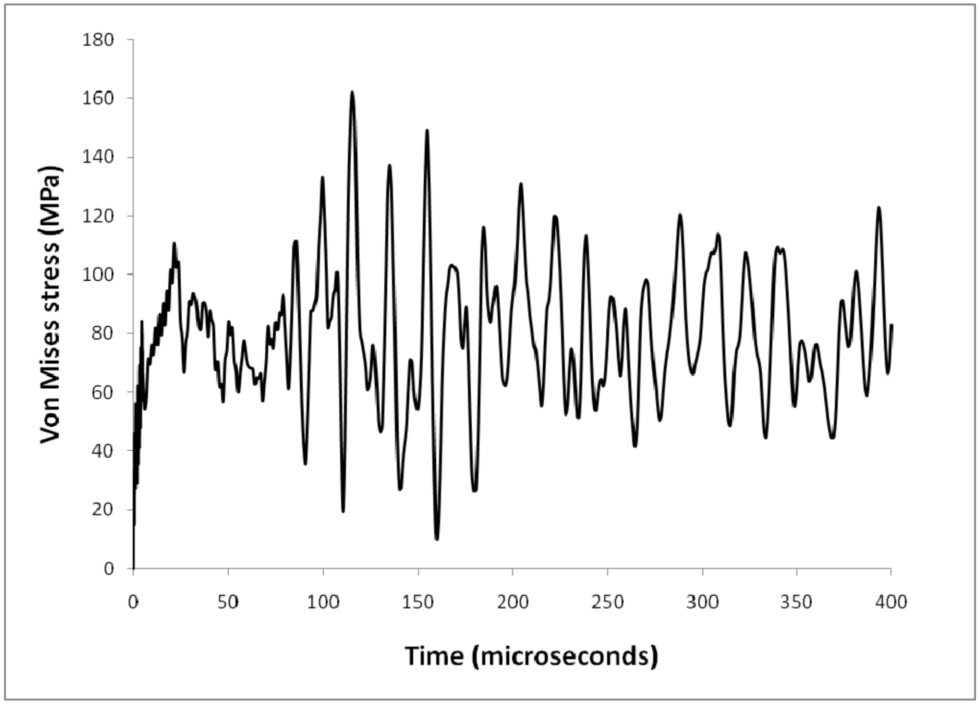
T2K Beam window

Results for 750kW simulations

Thermal stress cycling



Dynamic stress waves due to rapid beam heating



Estimate of current conditions at 345kW
Peak stress ~ 50MPa
Fatigue cycles ~ 0.5×10^6 @ 0.5Hz

High Power Targets Group
Mike Fitton

Science & Technology Facilities Council

T2K beam window III

Domes made from Ti-6Al-4V ELI (Grade 23)

Plate used instead of bar

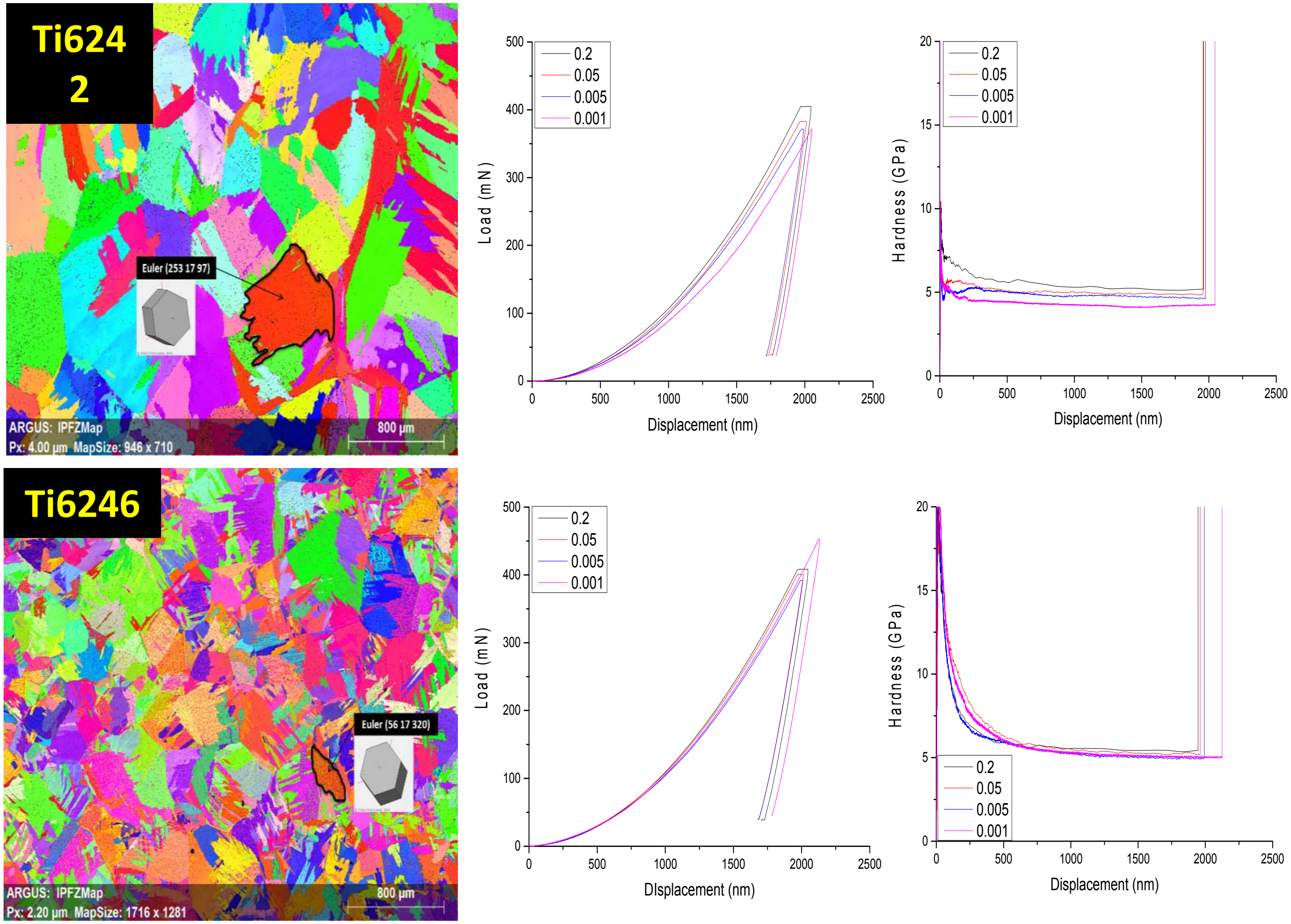
Material offcuts kept for
future radiation damage studies.

Irradiated Ti-6Al-4V

Ben Britton, Imperial College

Frederique Pellemoine, Facility for Rare Isotope Beams (FRIB, Michigan State University (MSU)

Nanoindentation strain rate tests



Indentation tests done with continuous stiffness measurement (CSM) technique

Need to perform strain rate jump (SRJ) tests

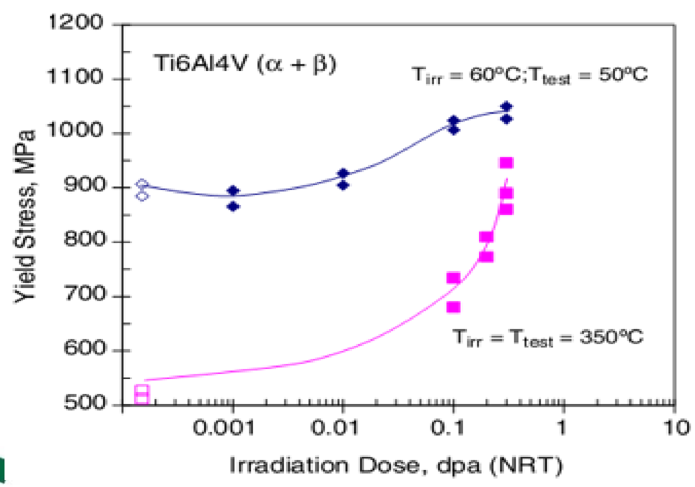
T. Jun, D. Armstrong, B. Britton

Imperial College London

Irradiation damage in Ti-6Al-4V

Effect of dose and temperature on the microstructure of neutron irradiated Ti-6A-4V (Tähtinen *et al.* , Sastry *et al.*, Peterson)

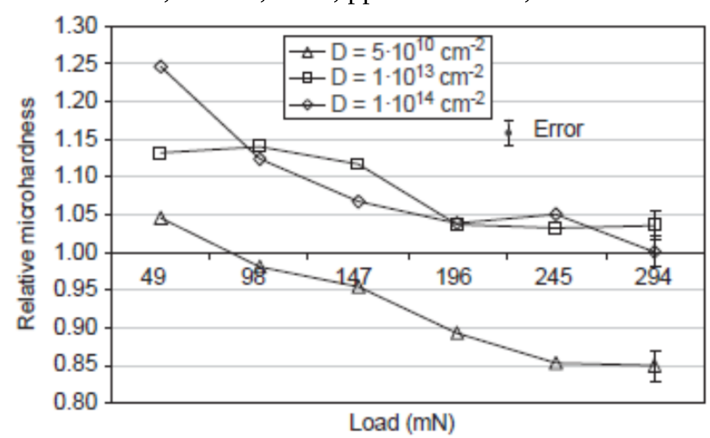
Temperature and dose level	Microstructure change observations
50ºC , 0.3 dpa	A high concentration of uniformly distributed defect clusters in the α-phase
350º C, 0.3 dpa	Dislocation loops Vanadium precipitates
450ºC, Dose 2.1 and 32 dpa	Dislocation loops β-phase precipitates in α phase
550ºC 32 dpa	Extensive void formation Coarse β-precipitates



Dose dependence of yield strength of Ti-6Al-4V irradiated with neutrons

Different hardening mechanisms operate at 50ºC than at 350ºC.

P. Budzynski, V. A. Skuratov, and T. Kochanski, "Mechanical properties of the alloy Ti–6Al–4V irradiated with swift Kr ion," *Tribol. Int.*, vol. 42, no. 7, pp. 1067–1073, Jul. 2009.



Relative micro-hardness in Ti-6Al-4V irradiated with swift 250Mev Kr^{+26} at different fluences

MICHIGAN STATE UNIVERSITY

Tähtinen et al. / *Journal of Nuclear Materials*, 367-370 (2007), 627–632
Sastry et al / Fourth International Conference on Titanium, Kyoto, Japan, 1980, vol. 1, p. p. 651.
D.T. Peterson, / *Effects of Radiation on Materials: 11th International Symposium*, Philadelphia, PA, 1982, p. p. 260.

FRIB

Hardness measurements

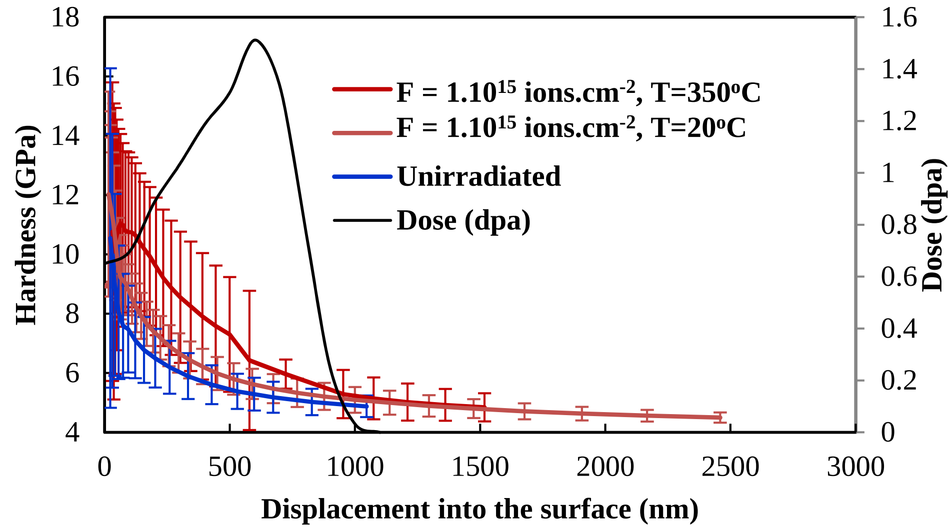
Nano-indentation

Obtain the properties of the materials in depth.

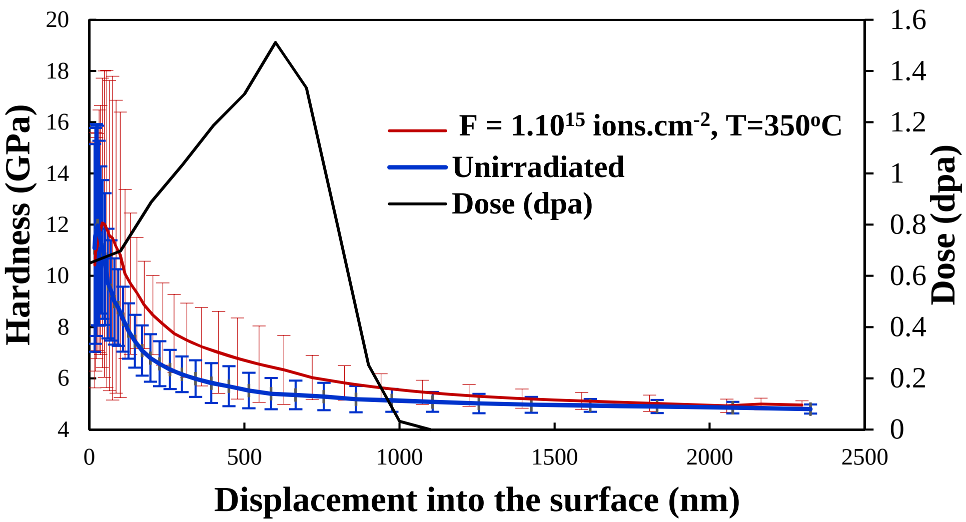
Parameters:

- Berkovich tip
- Strain rate : $0.05 s^{-1}$
- Poisson ratio=0.33
- Distance between indents: 50µm

Ti-6Al-4V



Ti-6Al-4V-1B



Nano-indentation results for Ti-6Al-4V and Ti-6Al-4V-1B irradiated with ^{36}Ar @36 MeV at fluence of 1.10^{15} ions.cm^{-2}with the CP –Ti foil on the surface.

Boron addition to Ti-6Al-4V did not change its irradiation resistance

➢ A slight increase in hardness observed for the sample irradiated with a higher fluence (1.10^{15} ions.cm^{-2}) and lower temperature (T = 350°C) for the higher doses

Graphite

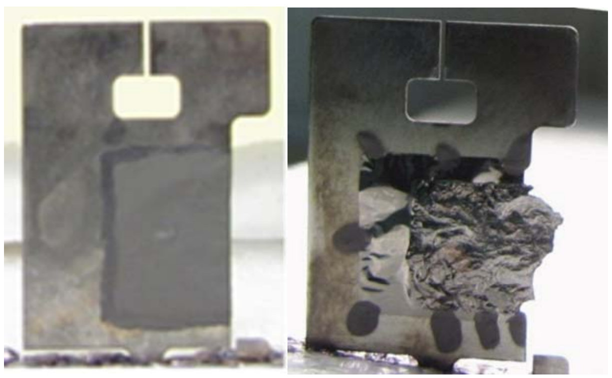
Nick Simos, Brookhaven NL

Frederique Pellemoine, Facility for Rare Isotope Beams (FRIB, Michigan State University (MSU)

FRIB

Radiation Damage and Annealing in Graphite Stripper Foils

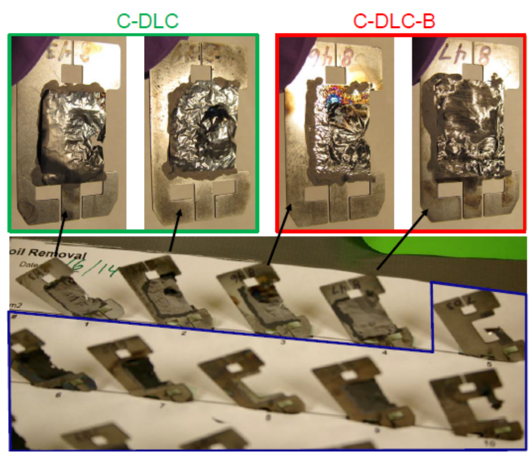
- Quick deterioration of graphite foil under heavy ion bombardment at NSCL



Carbon stripper foils on their frame before (left) and after irradiation (right) to a 8.1 MeV/u Pb beam at a fluence of 4.5 x 10^{16} ions/cm².

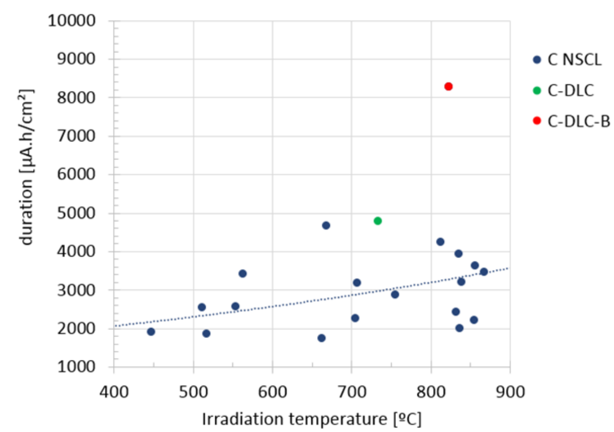
NSCL irradiation tests of UHV Technologies multi nano-layered foils

- C-DLC: alternate layers of nano-crystalline carbon and diamond like carbon
- C-DLC-B: ordered mixture of carbon, diamond, boron nano-layers



Stripper foils after irradiation (right) to a ^{78}**Kr^{14+} at 13 MeV/u** at NSCL. Standard carbon foils (blue box) present more damages compare to the C-DLC (green box) and the C-DLC-B (red box)

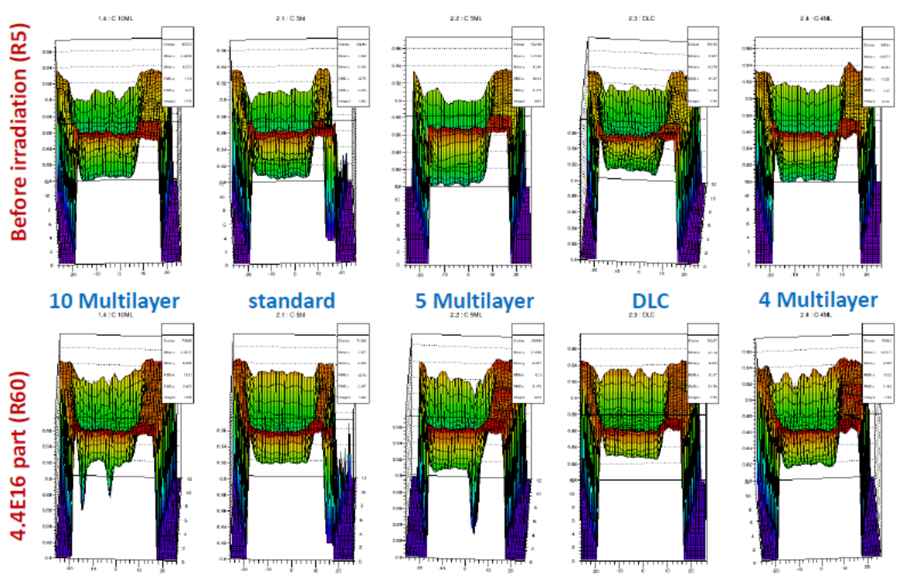
C-DLC-B lifetime superior to standard foils



Lifetime time (µA·h/cm²) as a function of the irradiation temperature and the microstructure of graphite stripper foils.

Irradiation at GANIL

- Foil irradiation on rotating target wheel (500 rpm) with ^{78}Kr at 10 MeV/u
- 5 and 10 multilayer foils developed small cracks
  - Indicated by spikes in the 5 and 10 lower row multilayer foils



Electron gun scan of the stripper foils before (upper row) and after irradiation with a GANIL ^{78}Kr beam at 10 MeV/u up to a fluence of 4.5 x 10^{16} ions in a rotating target holder.

Summary

- Thin ~500 μg/cm^2 carbon foils irradiated at different temperatures at NSCL and GANIL with swift heavy ions
- Increase of lifetime at higher temperatures was observed with standard stripper foils
- Further increase of lifetime for new multi-layer nano-crystalline carbon foils

Future studies

- Improved fabrication method of multi-layer foils to avoid cracks during GANIL irradiations
- New irradiations planned in the K1200 cyclotron at NSCL
- New irradiations with heavy ion beams under discussion

N. Simos

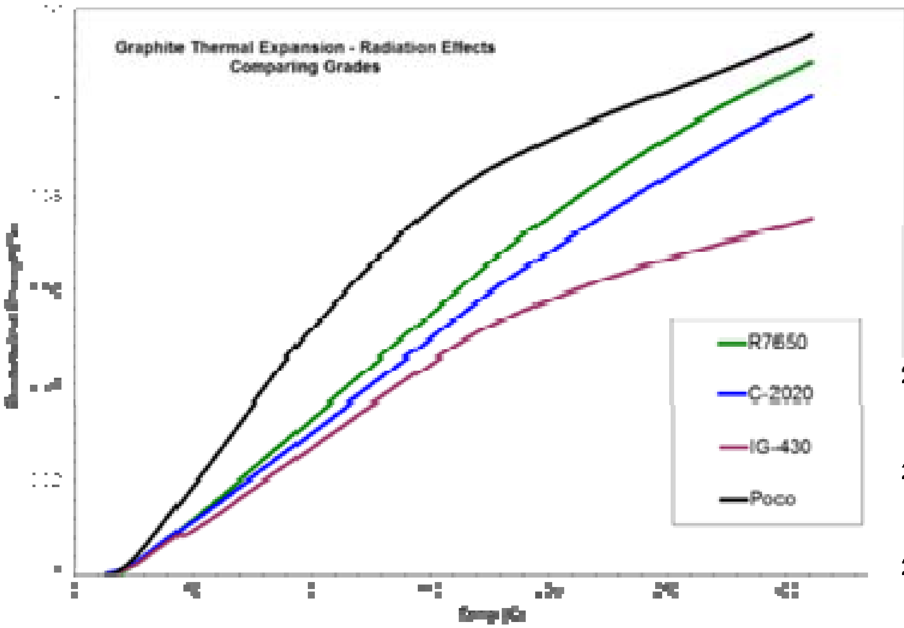
Graphite & Carbon-based Material Irradiation/Characterization Studies at BNL

- An array of irradiation damage and post-irradiation characterization studies have been under way at BNL for graphite and carbon-based structures
- Brookhaven has a long history in the study of nuclear graphite
- Studies were prompted by (a) Next Generation Fast Nuclear Reactor needs, (b) Neutrino Factory, (c) LHC and (d) LBNE
- BNL accelerator complex facilities (200 MeV Linac/BLIP and Tandem accelerator) provide proton, spallation fast neutron and ion irradiation beams)
- Macroscopic post-irradiation characterization utilizes the Isotope Extraction Facility (hot cells, remote handling and testing)
- Microscopic post-irradiation is performed at the BNL Synchrotron facilities (NSLS using white and monochromatic x-ray beams and now NSLS II) aided by multi-faceted characterization at the Center of Functional Nanomaterials

Graphite & Carbon-based Materials

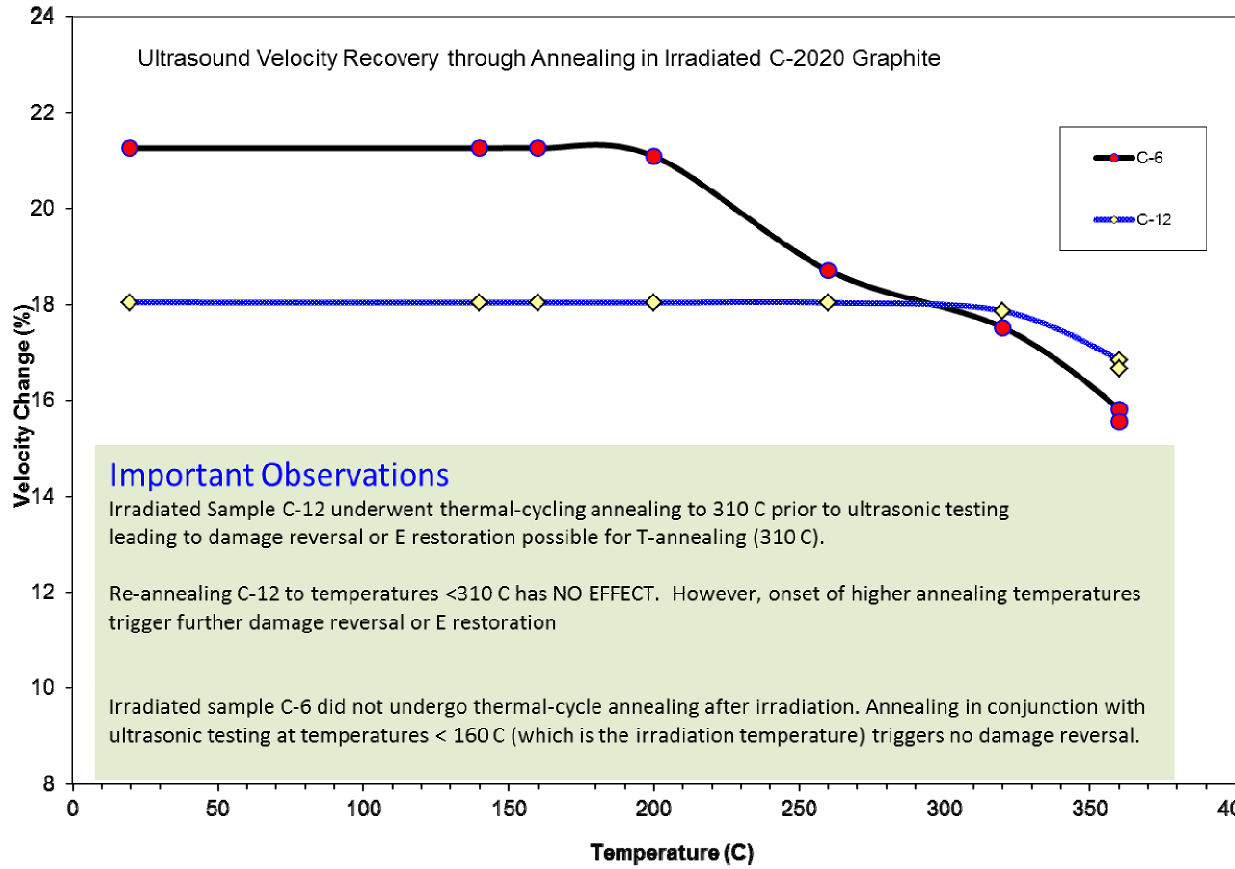
- Reactor-grade graphite (IG-43, IG-430) under fast neutrons and protons
- Carbon fiber composites (2D C/C and 3D C/C) + SiC/SiC
- HP Target bound graphite (LBNE) – 4 grades (POCO, IG-430, Carbone and R7650)
- Newly developed structures such as Mo-GR

Some PIE results



Dimensional change for various graphite grades during the 1st post-irradiation annealing cycle which reverses damage

Annealing in proton-irradiated graphite and restoration of E modulus using ultrasonic techniques.

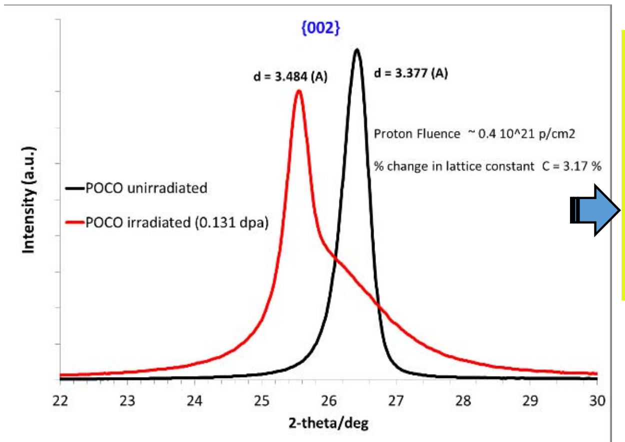


- Increase in **E** observed for all grades
  - Dislocation pinning and "tightening up" of the aggregate structure due to irradiation growth.

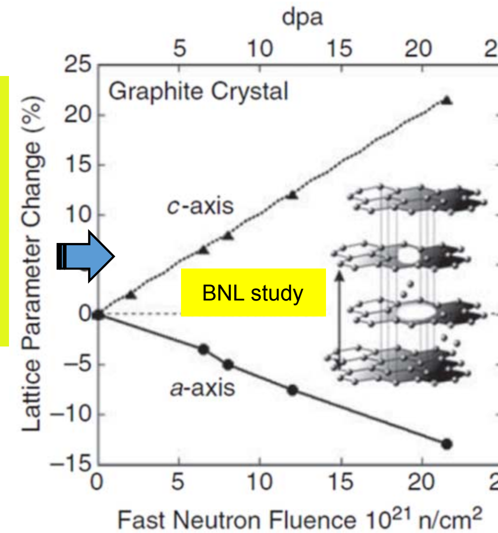
- The change in elastic modulus cannot be annealed out.
  - Observations in agreement with reactor neutron-induced deformation of graphite.

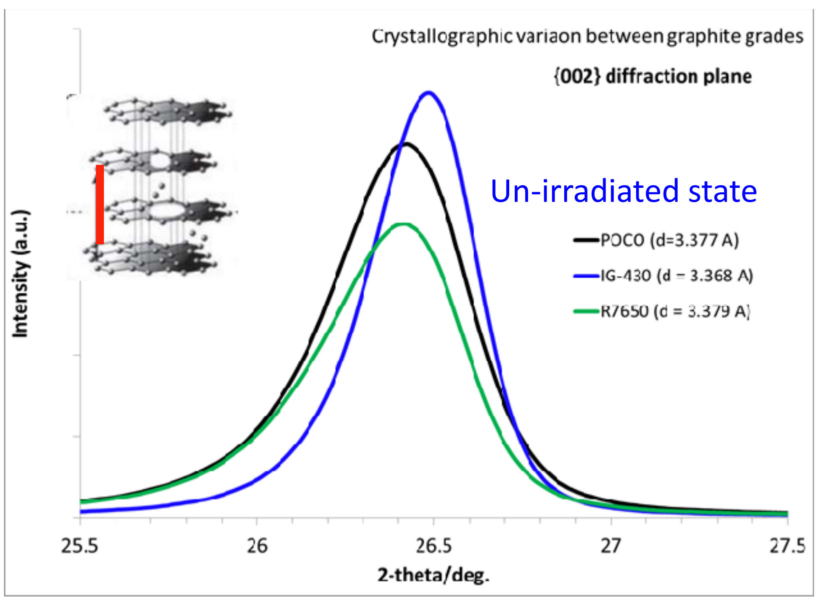
X-ray Diffraction Studies of Irradiated graphite

- EDXRD studies of irradiated graphite with/without load
- XRD (monochromatic x-ray beam studies
- Assess irradiation damage and annealing effects on graphite crystal aggregate

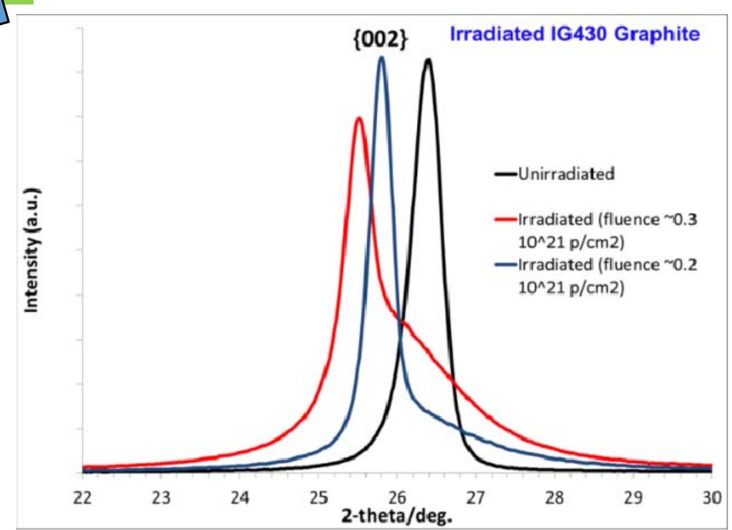


Damage expressed in terms of MEASURABLE quantities (i.e. crystal lattice changes) is achieved much faster and at much lower FLUENCE or DPA by energetic protons than fast neutrons.
BNL finding is set to a factor of ~10





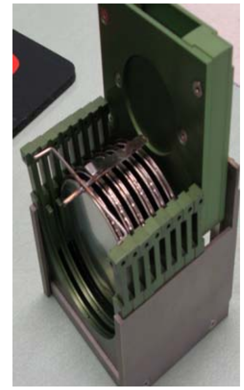
Proton irradiation damage on IG-430 With increasing fluence

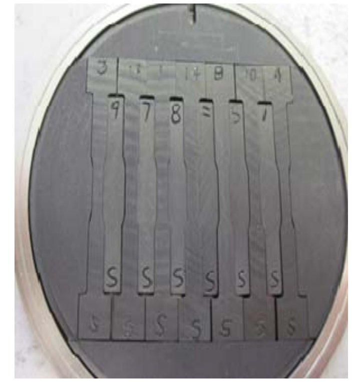


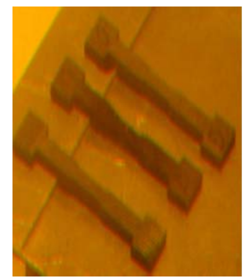
Variability in crystal structure of un-irradiated graphite

FNAL LBNE BLIP run – PIE status

- **Graphite grades:** POCO, IG-430, SGL R7650, C2020, 3D C/C composite
- 9 weeks irradiation at:
  - 180 MeV
  - $\sigma_x \sim 10$ mm, $\sigma_y \sim 7$ mm
  - **Peak DPA: 0.1**
  - **Peak temperature: 200 °C**







PIE partially completed

- Some tensile tests
- 3-point bending tests
- Ultrasonic tests for elastic modulus
- Dimensional changes
- EDXRD

Future tests

- Complete 3D C/C composite flexural tests
- Thermal conductivity measurements, with new resistivity fixture
- New round of EDXRD experiment

Radiation Damage Studies in Graphite [1]

Annealing of Damage at High Temperature (> 1300ºC)

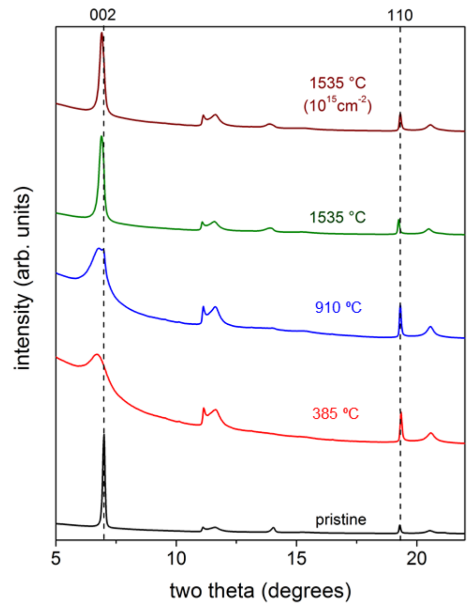
1 A - 350°C
10^{14} cm^{-2}

11 A - 750°C
10^{14} cm^{-2}

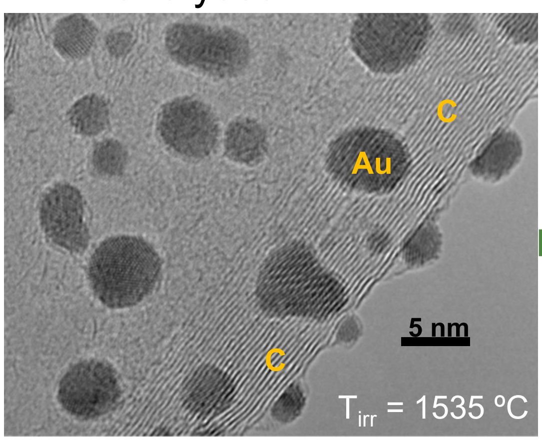
25 A - 1205°C
10^{14} cm^{-2}

35 A - 1635°C
10^{15} cm^{-2}

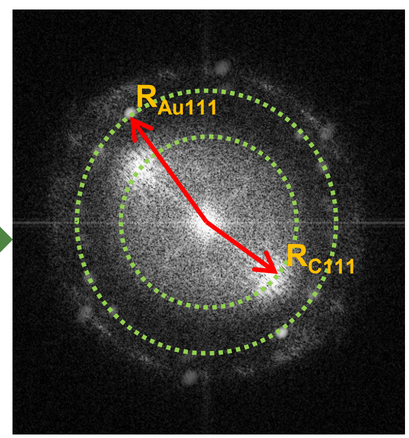
X-Ray Diffraction analyses

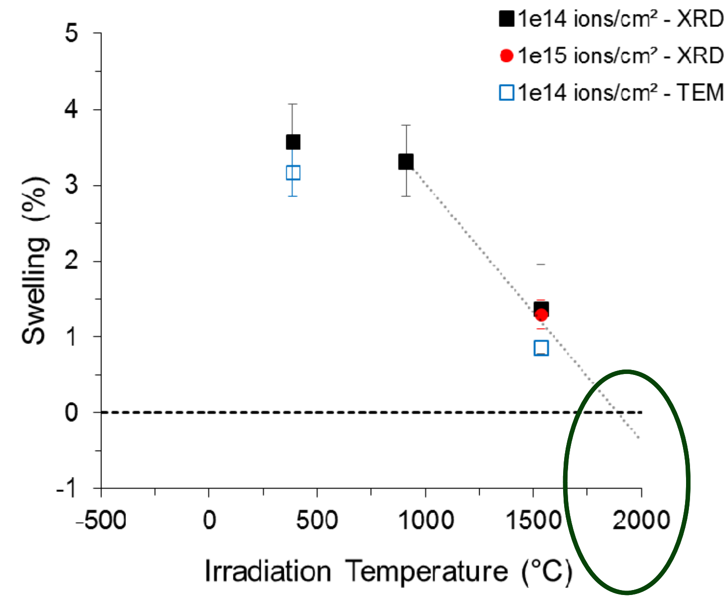


TEM analyses



FFT

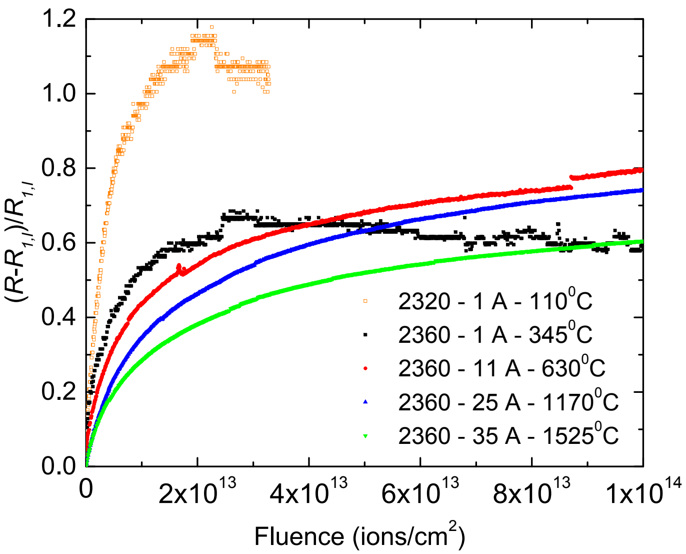


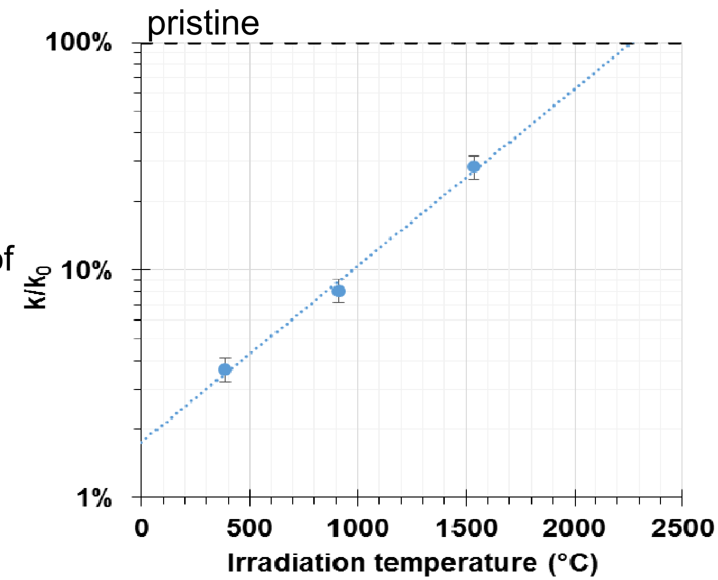


Swelling is completely recovered at 1900ºC

Radiation Damage Studies in Graphite [2]
Annealing of Damage at High Temperature (> 1300ºC)

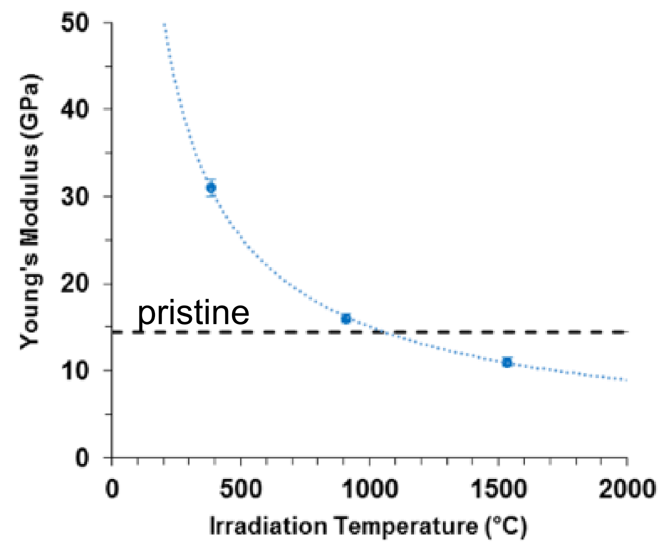
Electrical resistance change of irradiated graphite samples - ^{197}Au





Thermal conductivity change of irradiated graphite samples - ^{197}Au fluence 10^{14} ions/cm²

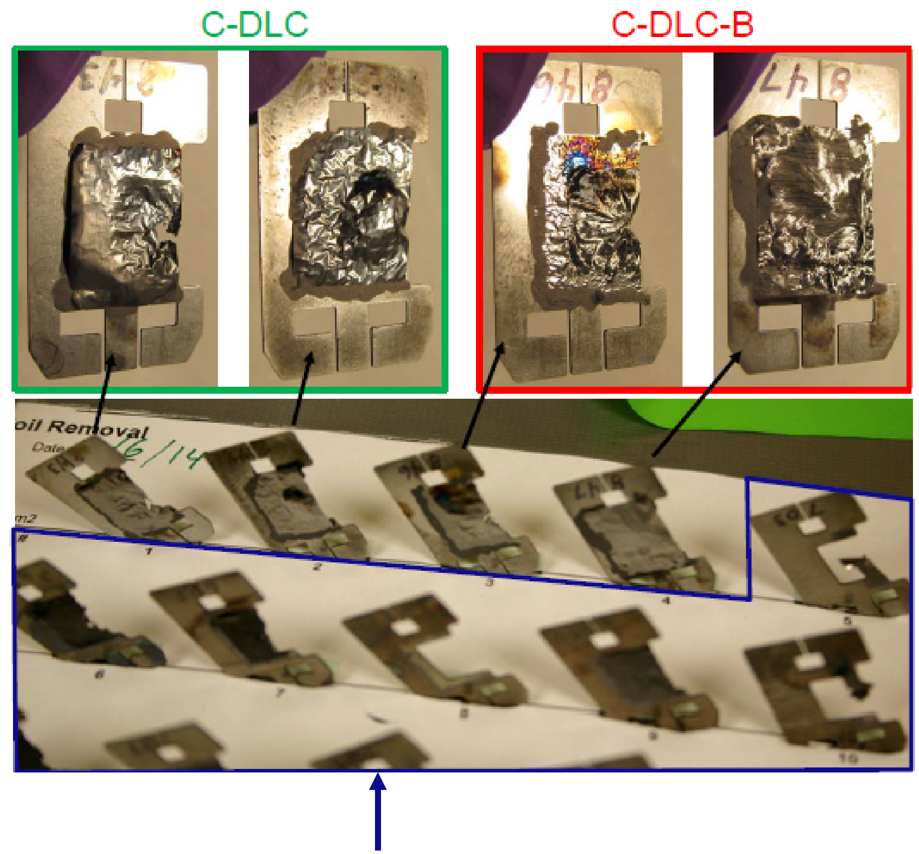
Young's Modulus of irradiated graphite samples - ^{197}Au fluence 10^{14} ions/cm²



- Annealing at high temperature confirmed by 3 other analyses

NSCL-FRIB Strippers
Irradiated Strippers at NSCL

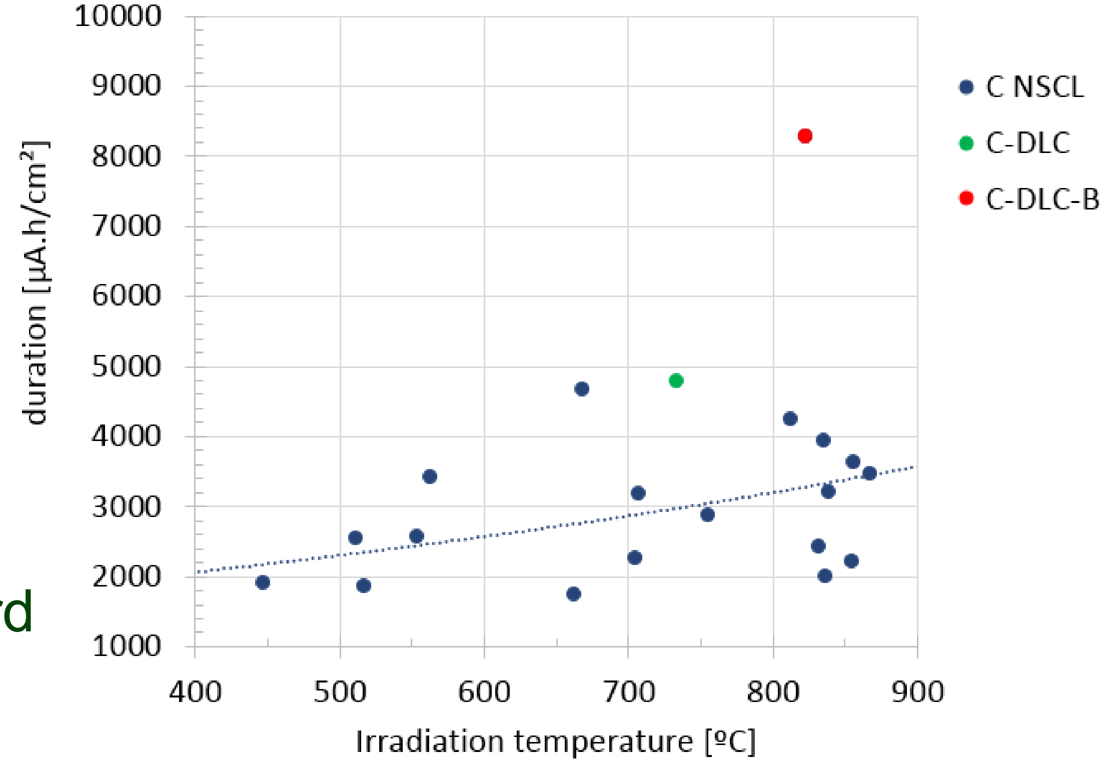
- 3 types of foils with a thickness of ~ 500 µg/cm² were used for this current study:
  - C-DLC: alternate layers of nano-crystalline carbon deposited by pulse arc deposition and nano-crystalline diamond like carbon deposited by pulse laser deposition
  - C-DLC-B: ordered mixture of carbon, diamond and boron nano-layers
  - Standard carbon foil used currently at NSCL
- All foils were irradiated in the NSCL at MSU with $^{78}Kr^{14+}$ at 13 MeV/u at the stripping injection of the K1200 cyclotron.
- Images show more damage on the standard carbon foils compare to the new multi-layer foils.



Current carbon strippers used at NSCL

Improvement of the lifetime

- Previous studies [3] showed annealing effects of radiation damage at high temperature.
- A clear tendency of increased lifetime with irradiation temperature was observed.
- The lifetime of the 10 multilayer foil C-DLC-B was significantly higher (factor 3) than the standard C-NSCL foils. The 10 multilayer foil C-DLC was somewhat superior (about a factor 2) as compared to the standard foils.



Lifetime time (μA·h/cm²) as a function of the irradiation temperature and the microstructure of graphite stripper foils.

[3] S. Fernandes et al., "In-Situ Electric Resistance Measurements and Annealing Effects of Graphite Exposed to Swift Heavy Ions", Nucl. Instrum. Methods Phys. Res. B 314 (2013) 125-129.

Beryllium

Slava Kuksenko, Oxford University

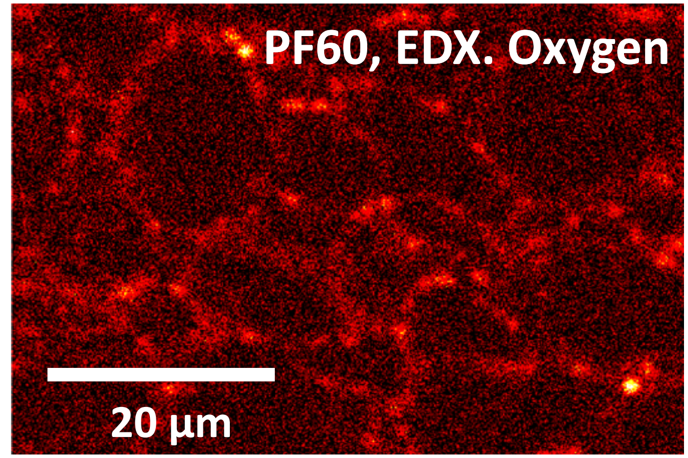
Status Report:
Experimental investigation of beryllium

Viacheslav Kuksenko[1], Chris Densham[2], Patrick Hurh[3], Steve Roberts[1]

[1] *University of Oxford, UK*
[2] *Rutherford Appleton Laboratory, UK*
[3] *Fermi National Accelerator Laboratory, USA*

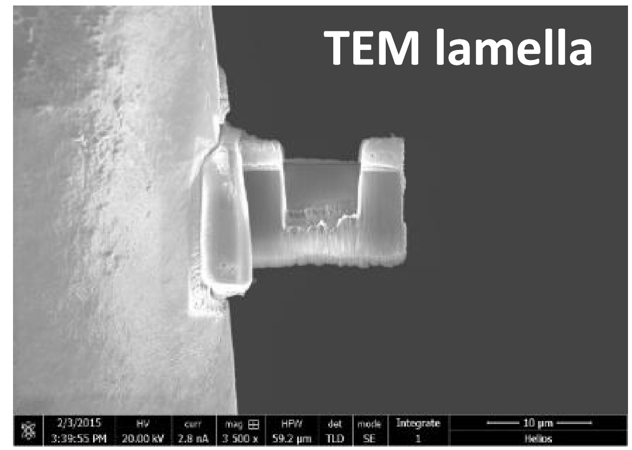
- **Investigation of the as-received Be**
- **Investigation of the proton Be window (NuMI)**
- **Ion irradiation experiments**

Characterisation of as-received Be



Samples preparation

- Mechanical polishing of samples from the PF60 and S200F: samples are “clean” and of “EBSD quality”.
- Still have S65 samples that should be polished;
- TEM lamellas and APT needles are produced by FIB lift-out procedure at CCFE

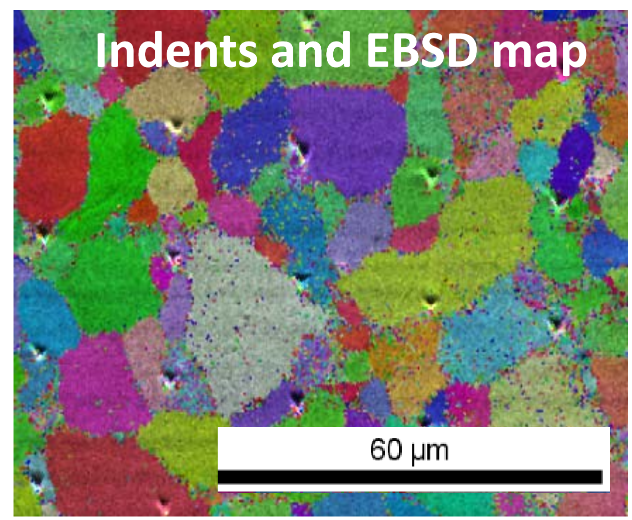


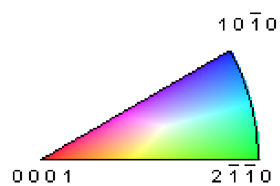
SEM+EDX+EBSD

- PF60 and S200F – have been analysed;
- 2 grades have similar mean gran size (~9 µm), no significant texture and wide variety of impurity precipitates

TEM + APT

- TEM of PF60 – still ongoing. Preliminary – low dislocation density, GB has different chemistry – Fe segregation, BeO particles + impurities.
- APT of PF60 is done (intragranular) – Fe, Ni, Cu impurities are in the matrix, no segregations





NANOINDENTATION

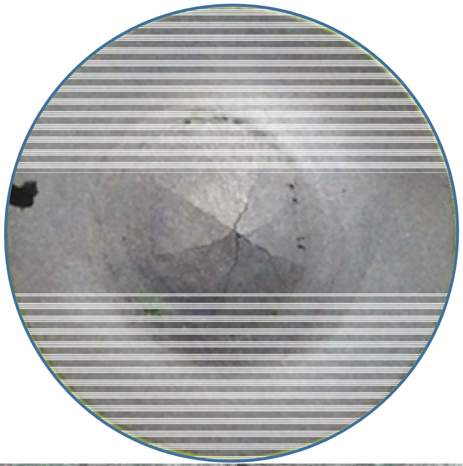
- PF60 is characterised.
- PF60 has very high hardness anisotropy

Investigation of the NuMI beam window

- 120GeV proton beam
- 1.57×10^{21} protons during its lifetime (up to 0.5 dpa)
- 1.1mm beam sigma
- T ≈ 50ºC

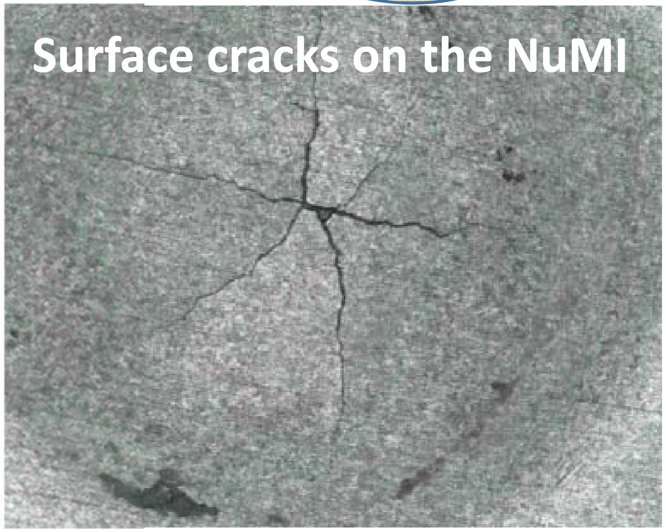
Profilometry:

- o the window has been heavily deformed during the removal process;
- o cracks have been developed on the surface;
- o local thickness increase in the central part – probably swelling



EDX analysis:

- o non-homogeneous Ni- and O-enriched flakes on the surface;
- o Ca, S, C, Cl impurities on the surface – most probably originate from drainage water



Sample has been shipped to CCFE for FIB lift-out

APT samples – June, 2015

TEM samples – summer 2015.

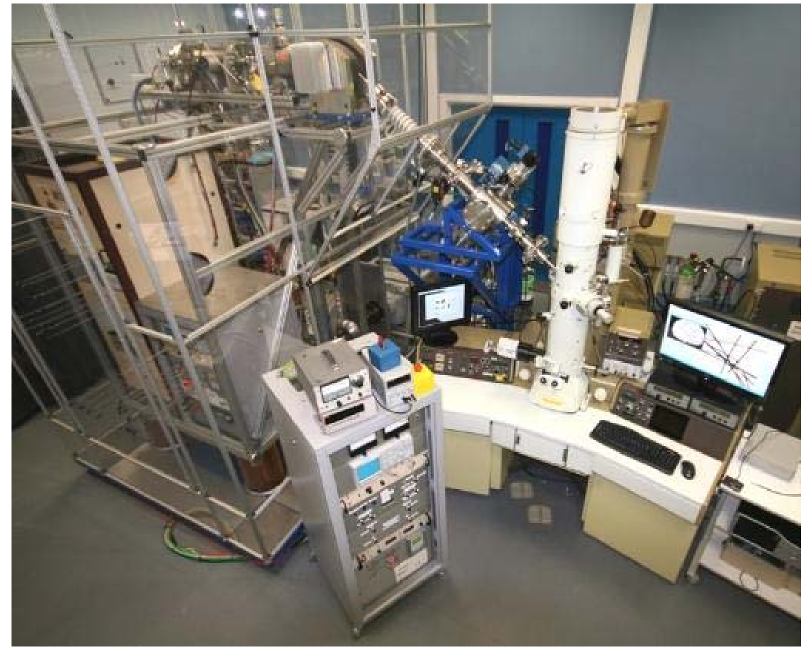
Micromechanical tests can be done only after the window cutting. The end of the year at CCFE?

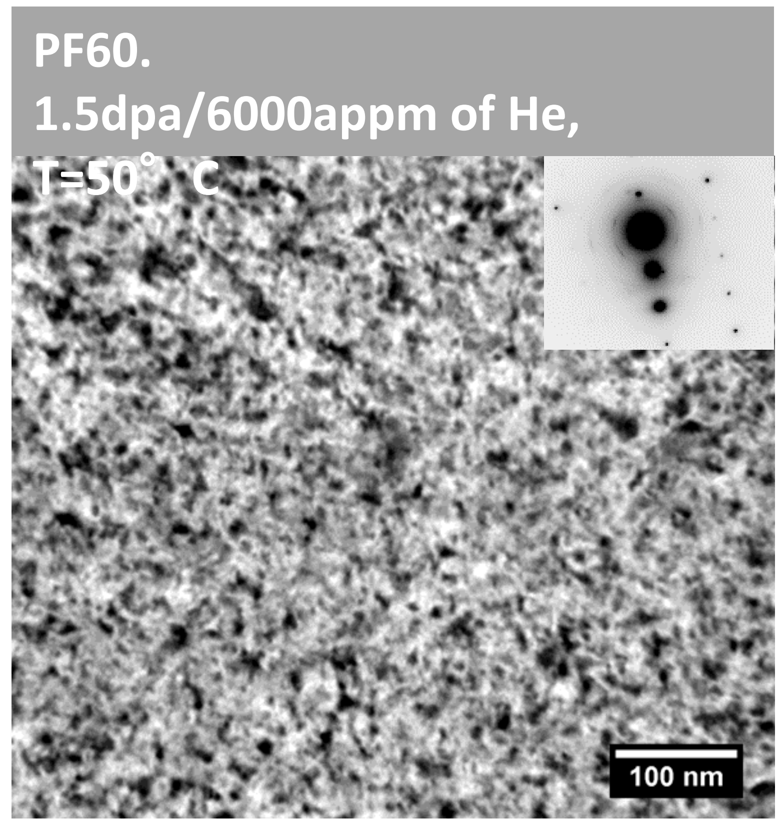
Ion irradiation experiments

Low energy ion irradiation (MIAMI, University of Huddersfield):

- 2 lamellas have been in-situ implanted at 50°C up to 2 dpa and 8000 appm of He;
- In-situ TEM didn't reveal bubbles or loops creation…
- …but the ex-situ analysis revealed a lot of tiny bubbles (1.5 nm in diameter) and "black dots"
- post-irradiation TEM analysis is ongoing

Further steps (this year): repeat implantation at 50°C but only to 0.5 dpa (to mimic the NuMI conditions). Then we will shift to 200°C.





Ion irradiation experiments

High energy ion irradiation (University of Surrey):

- Al coating for preventing of beryllium sputtering is tested.
  1 µm layer of Al also works as degrader and will be removed by mechanical polishing after the He implantation;
- Sample holder for the implantation are manufactured and tested at Surrey;
- Irradiation condition were revised due to the unrealistic implantation time (caused by the need to restrict the beam current for reducing of the induced heat)
- **Further steps (this year)**: perform an implantation at 50°C to 0.5 dpa (PF60 and S200F). Then we will shift to 200°C. Nanoindentation, TEM and APT are in plans

More details on the results will be given during the further presentations: Tue 11:45 and Wed 9:00

Tungsten: ISIS targets – heavily proton-irradiated W

Tristram Davenne, Rutherford Appleton Lab.

TS1 core FLUKA geometry

Geometry includes 12 tantalum clad tungsten plates and heavy water channels in between. Does not include stainless steel water manifolds on side of target.

Figure 1 Simplified ISIS geometry used for FLUKA model

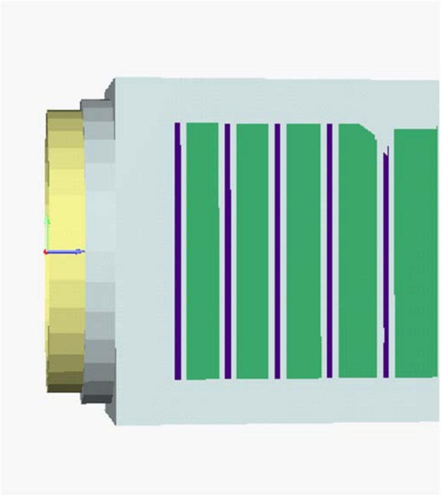
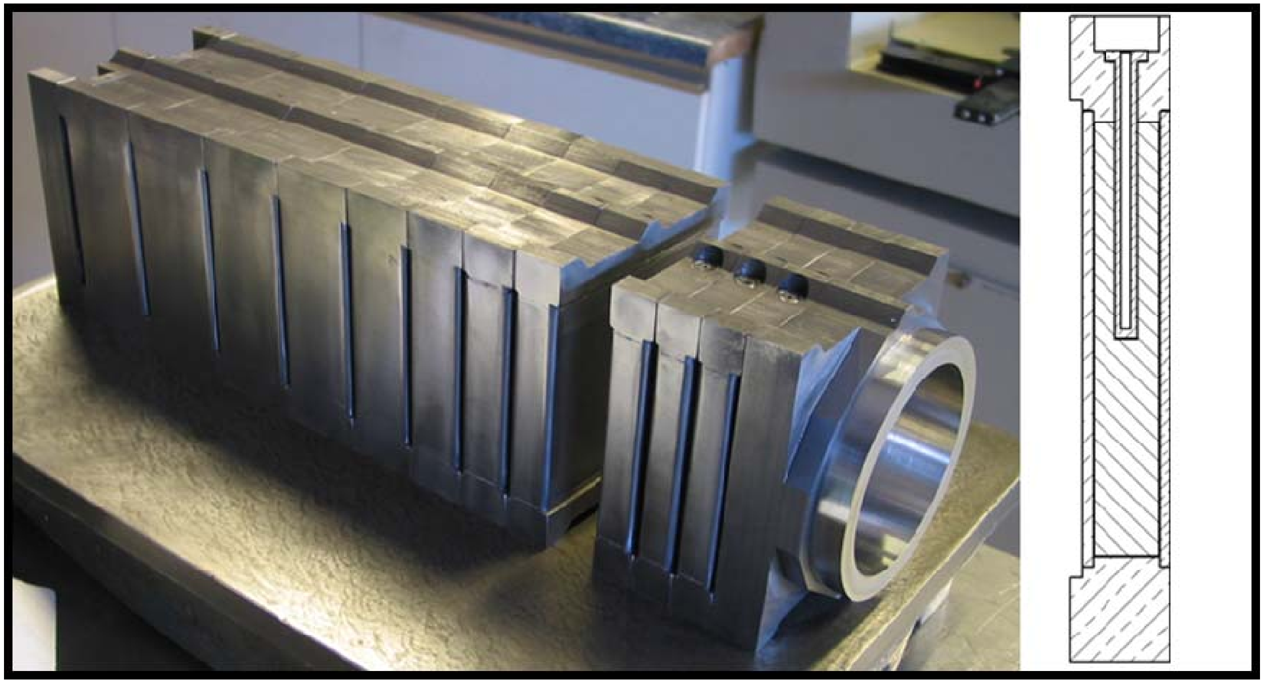
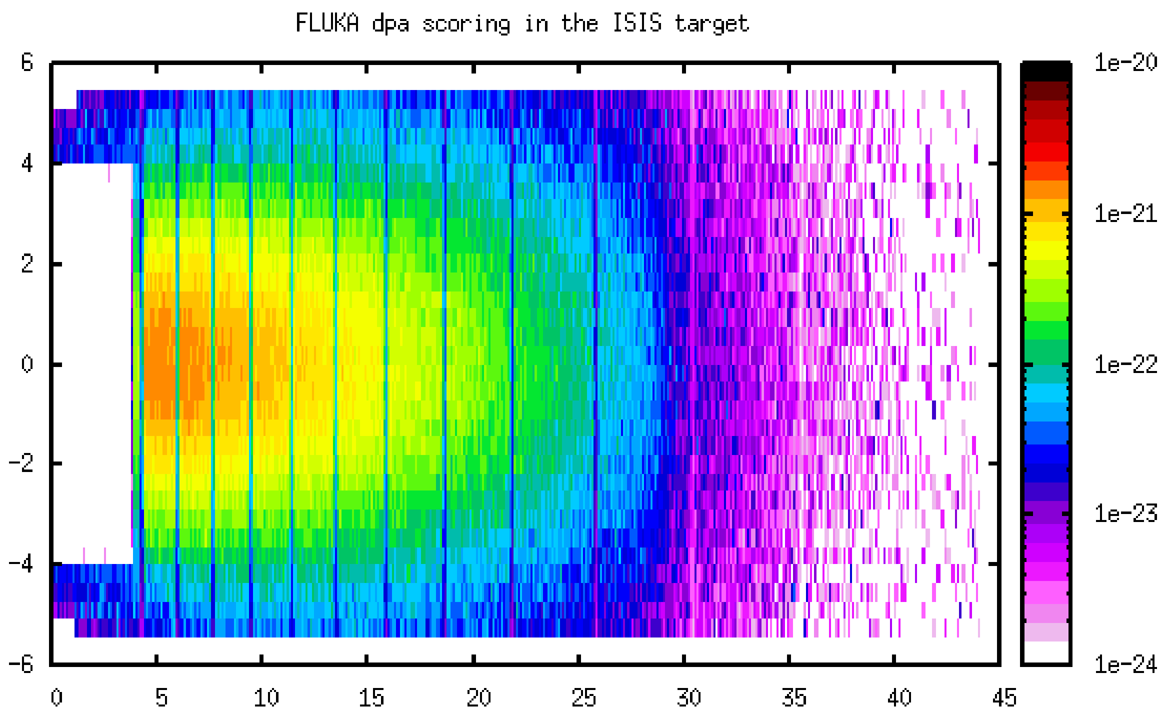


Figure 2 Simplified ISIS geometry used for FLUKA model

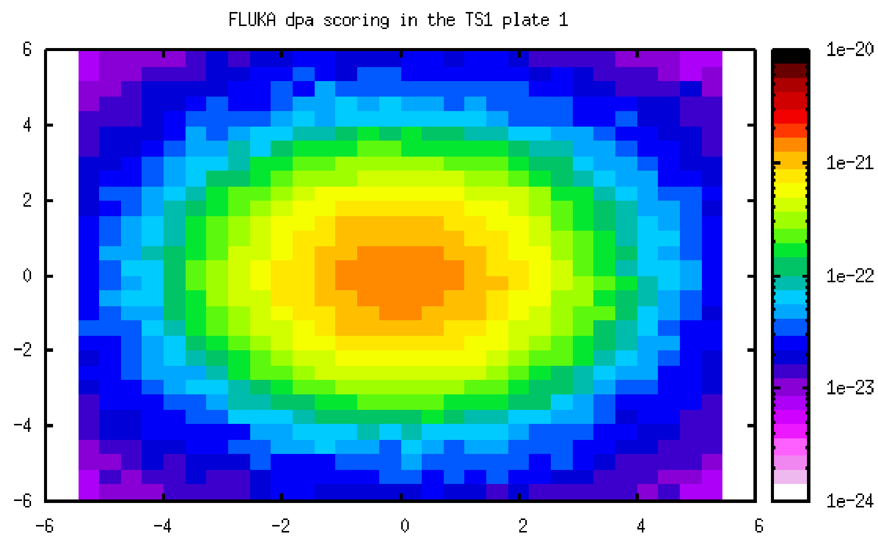


TS1 energy deposition and FLUKA dpa

Target Plate [800MeV sigx=16.3mm sigy=16.3mm]	max dpa/proton	dpa/s at 210µamps (equivalent to 1.31e15protons/s)	dpa per year 2e7s	Total Power deposited at 210µamps [kW]	Peak energy density at 210µamps [W/m3]	max temp calculated with CFX at 210µamps [°C]
1	1.90E-21	2.49E-06	49.8	11.76	4.79E+08	207
2	1.67E-21	2.19E-06	43.8	12.14	4.64E+08	205
3	1.26E-21	1.65E-06	33.0	12.18	4.11E+08	199
4	1.19E-21	1.56E-06	31.2	11.97	3.67E+08	200
5	9.40E-22	1.23E-06	24.6	11.3	3.21E+08	191
6	7.10E-22	9.30E-07	18.6	10.96	2.46E+08	179
7	5.20E-22	6.81E-07	13.6	9.99	1.86E+08	161
8	4.00E-22	5.24E-07	10.5	9.11	1.32E+08	151
9	3.00E-22	3.93E-07	7.9	8.32	9.01E+07	146
10	1.38E-22	1.81E-07	3.6	5.38	6.34E+07	109
11	2.30E-23	3.01E-08	0.6	0.24	5.15E+06	33
12	1.77E-23	2.32E-08	0.5	0.11	4.18E+06	31



Variation of dpa through out target



Variation of dpa across a target plate

Target Activity

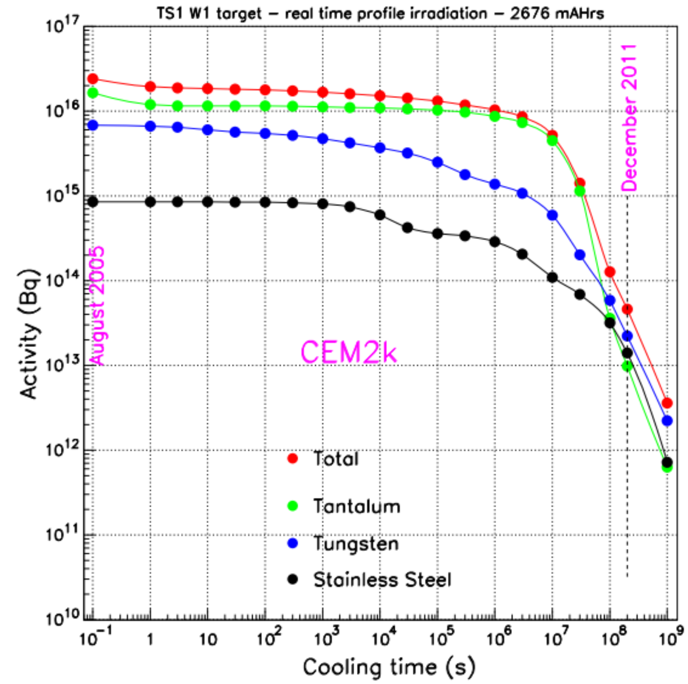
Irradiation profile of TS!-W1 from Goran Skoro's report

Table 1. Irradiation time profile for the TS1-W1 target.

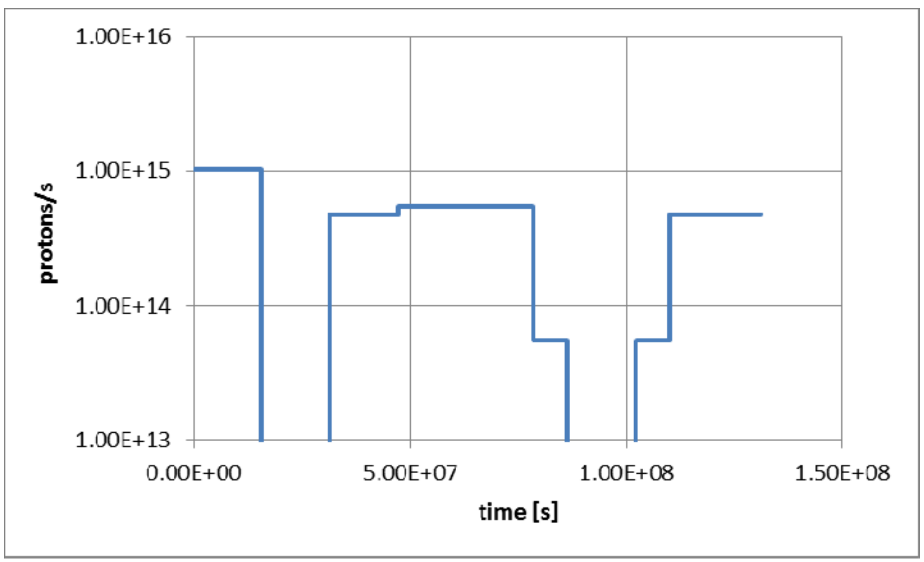
Time period	Protons on target (mAHrs)
May-Dec 2001	722.703
Jun-Dec 2002	338.293
2003	777.057
Jan-Mar; Oct-Dec 2004	387.844
Jan-Aug 2005	450.368

http://hepunx.rl.ac.uk/uknf/wp3/hidden/goran/ISIS_jobs/01_TrgtInven/ts1_w1_act.pdf

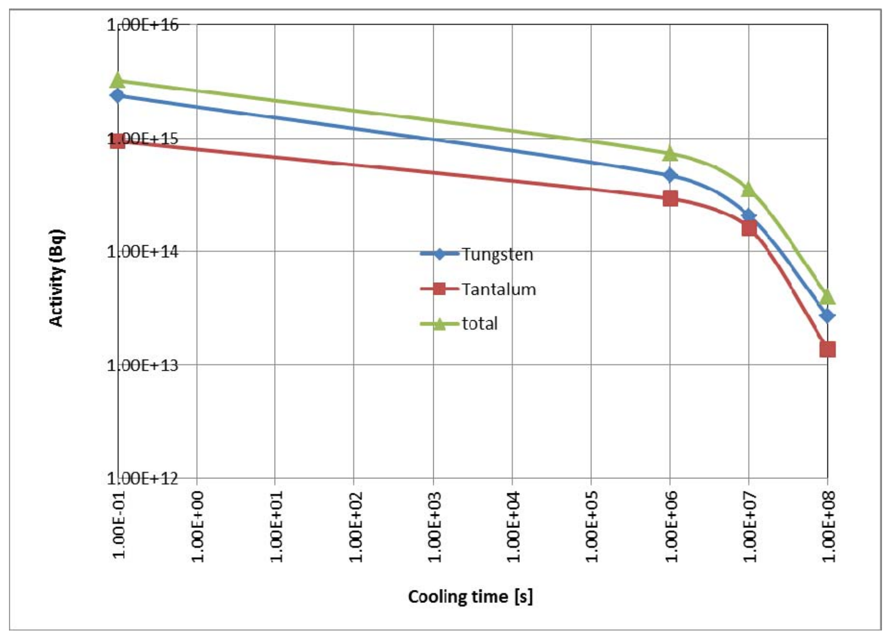
Total target activity from Goran Skoro's report



Irradiation profile interpreted for FLUKA



Total target activity calculated from simple FLUKA model



Peak Target Activity

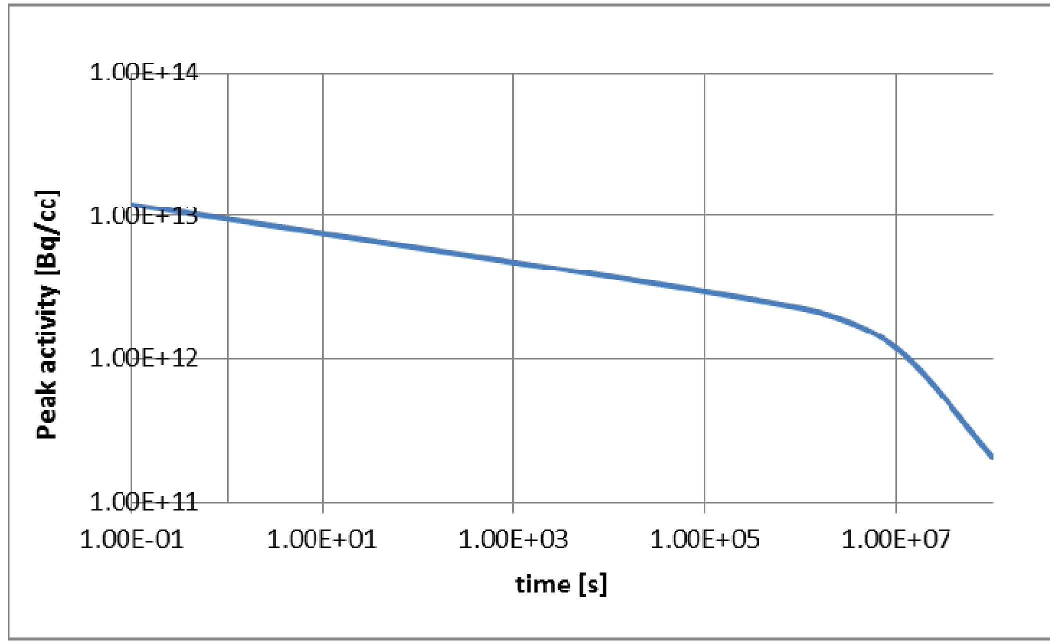
Maximum activity in target

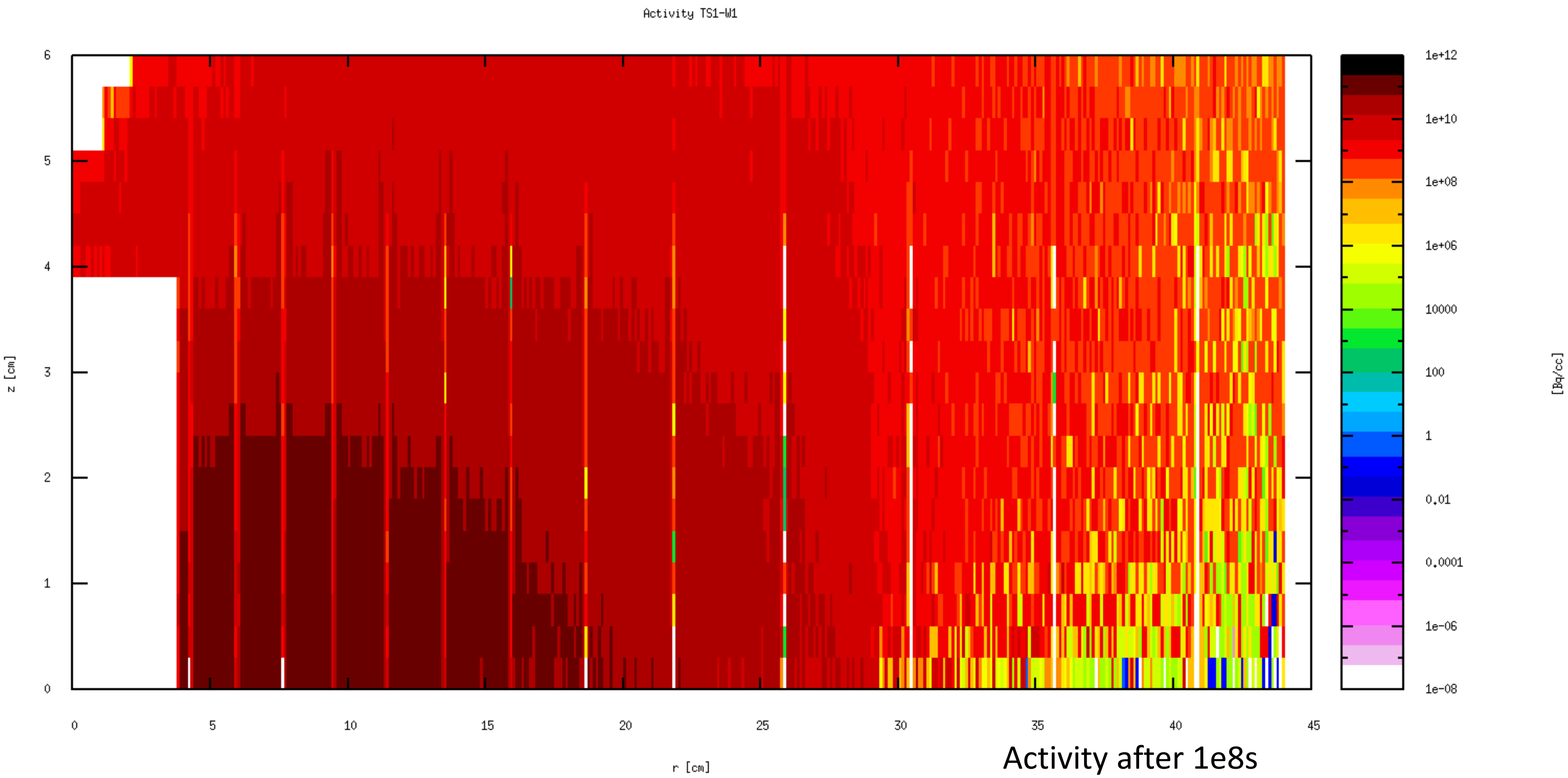
1.2e13 Bq/cc immediately after irradiation

2.1e11 Bq/cc after 1e8s

or for tungsten

1.1e10Bq/gram after 1e8s (i.e. 10GBq/gram)



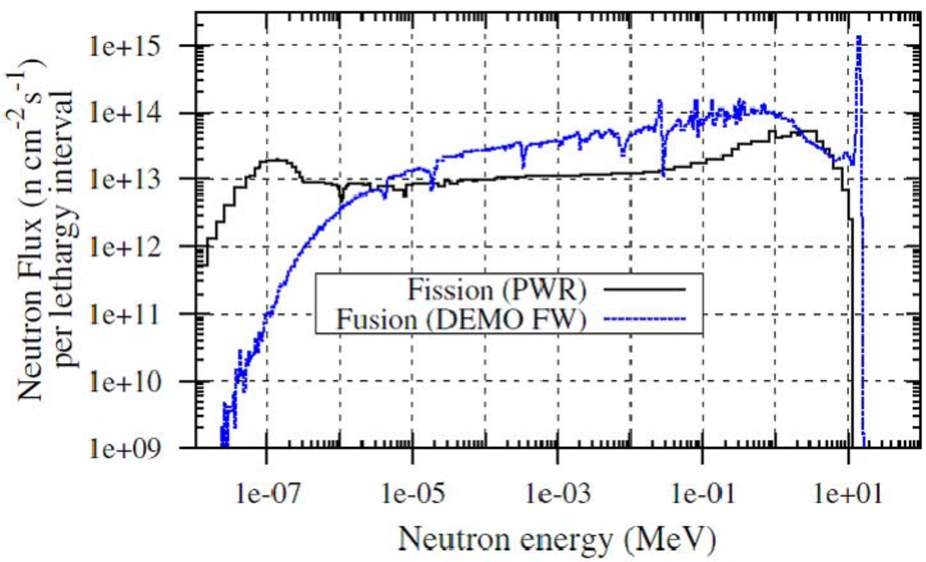


Activity after 1e8s

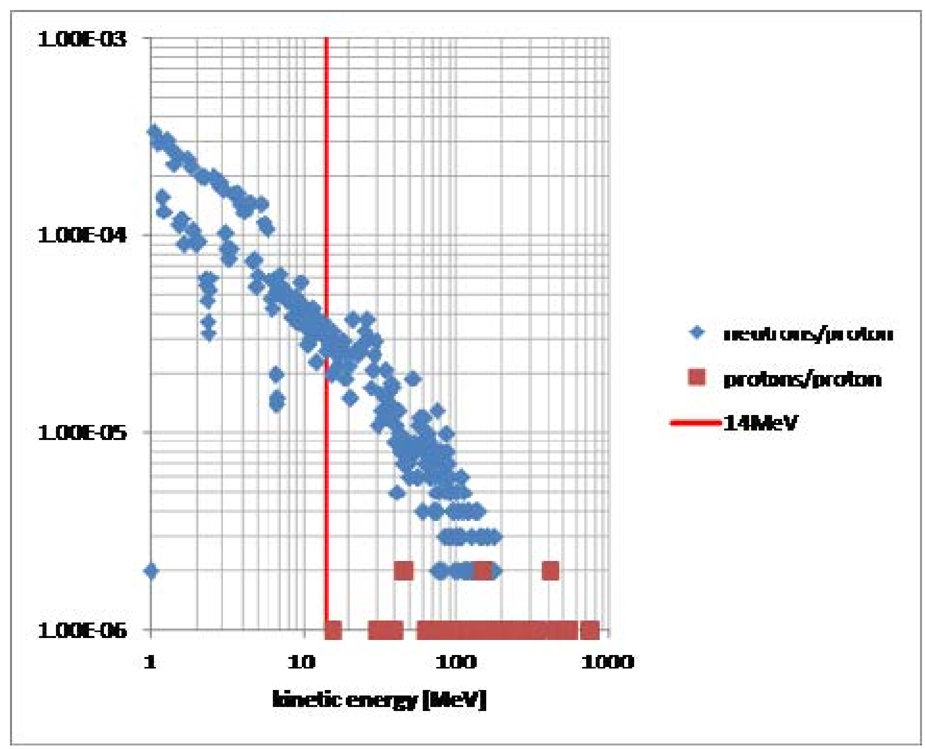
Applicability to fusion materials research?

Typical fusion neutron spectrum

Nucl. Fusion **52** (2012) 083019



Fluka calculation indicates ≈1dpa per fpy in sample corner and the following neutron and proton flux



Consider a sample corner of a TS1 target plate