



Radiation damage of beryllium: NuMI beam window materials tests

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Science & Technology Facilities Council
Rutherford Appleton Laboratory
ΚΑΤΕΥΛΛΟΔΑ ΥΒΒΙΣΤΕΩΝ ΓΕΦΟΛΩΣΟΛΩ



19th of May, 2nd RaDIATE Collaboration Meeting

Beryllium

- is extensively used as a material of neutrino targets parts, for example – beam window
- is a promising candidate for future high-power neutrino sources

For example, the expected working conditions for some parts of the Long-Baseline Neutrino Facility (LBNF)

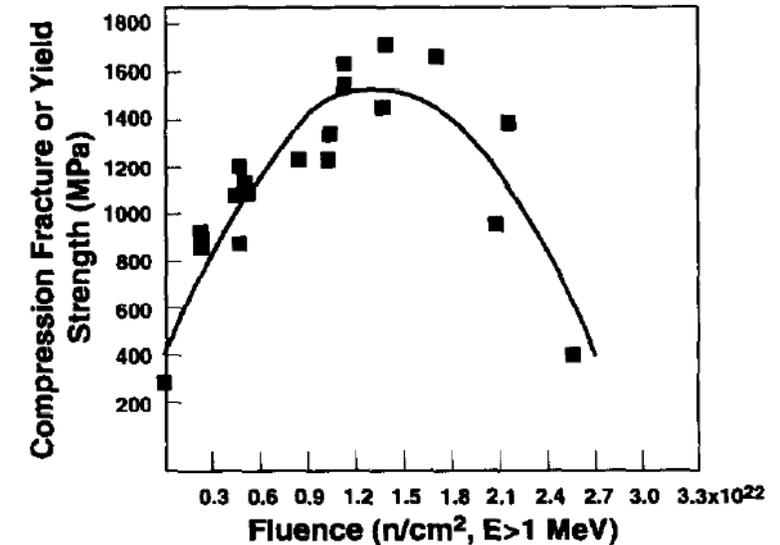
Application	Operating conditions					Proton beam parameters
	Avg. T (°C)	Peak T (°C)	Total DPA	Gas production (appm/DPA)		
				He	H	
Beam window (vacuum to air)	200	300	~ 0.23/yr	>1000	>2500	700 kW; 120 GeV; ~1 Hz; $\sigma_{rms} = 1.3$ mm
Target	375	450	~ 0.23/yr	>1000	>2500	700 kW; 120 GeV; ~1 Hz; $\sigma_{rms} = 1.3$ mm

Environment: elevated temperature + radiation + pulsing loads

What can we expect?

Examples form n-irradiation

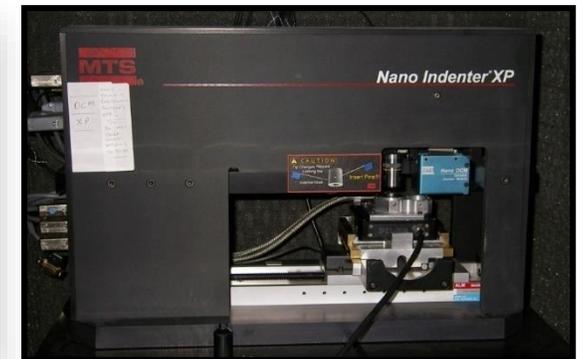
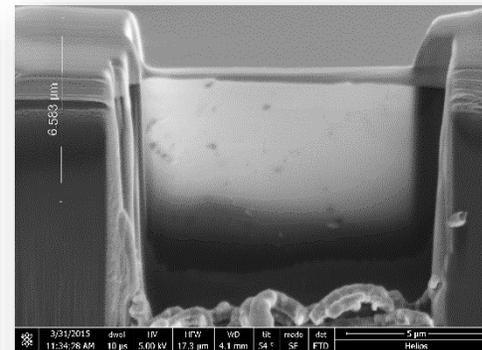
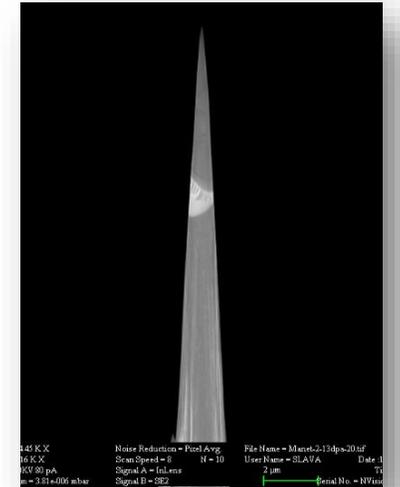
- reduction induced embrittlement
- irradiation induced hardening



Compressive fracture or yield strength as a function of fluence ($E > 1$ MeV) for beryllium specimens irradiated at 75-125°C.
 D.S. Gelles et al. /Journal of Nuclear Materials 212-215 (1994) 29-3X

Experimental investigation

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



Outline

- Material of the investigation
- Microstructure and properties of as-received beryllium
- NuMI beam window materials tests and future plans
- Conclusions

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

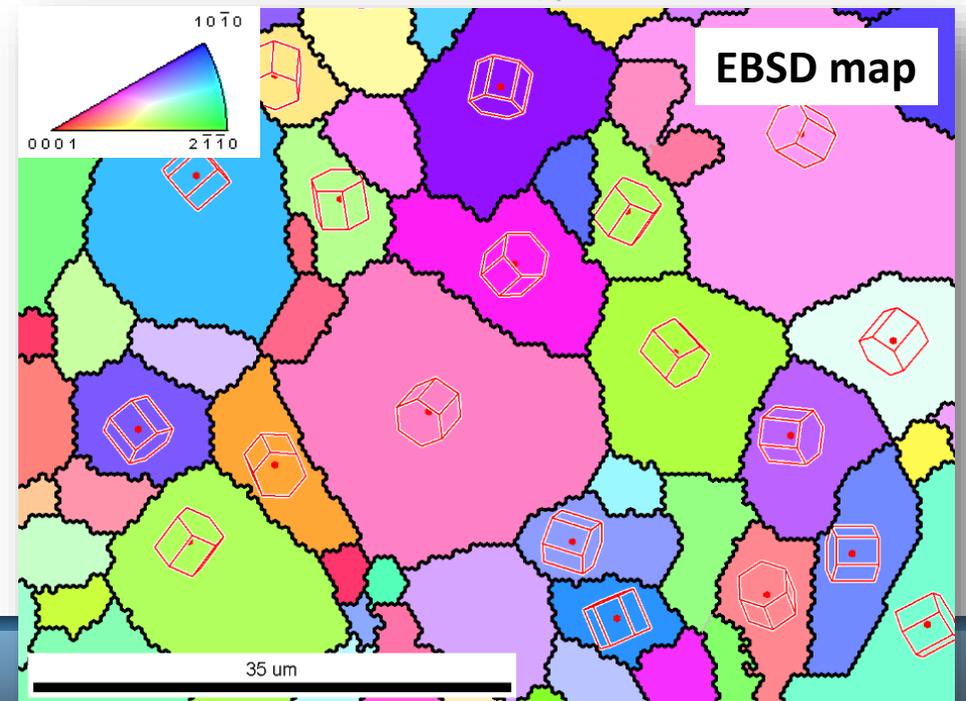
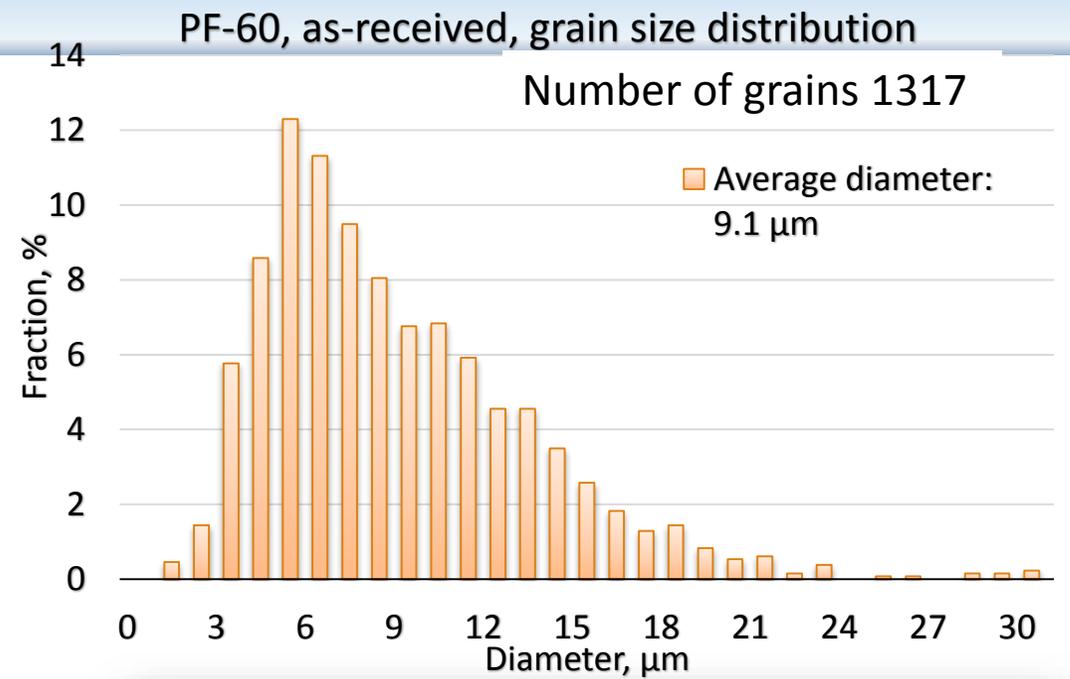
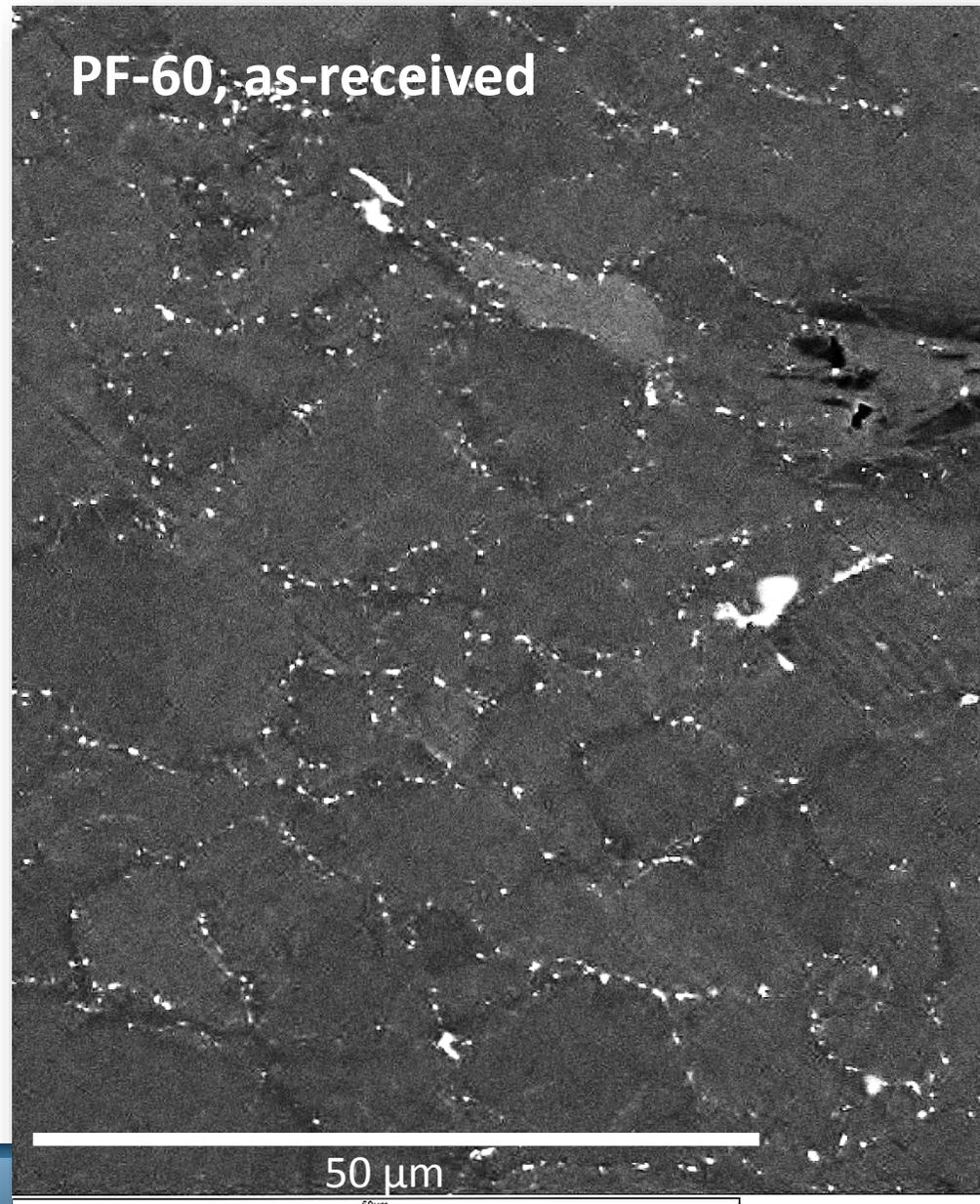
S-65

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

Method of manufacture: vacuum hot pressing

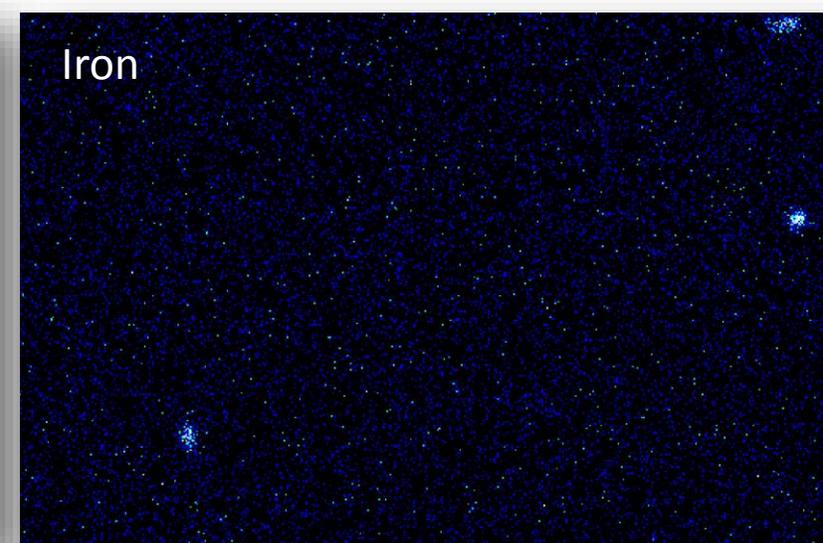
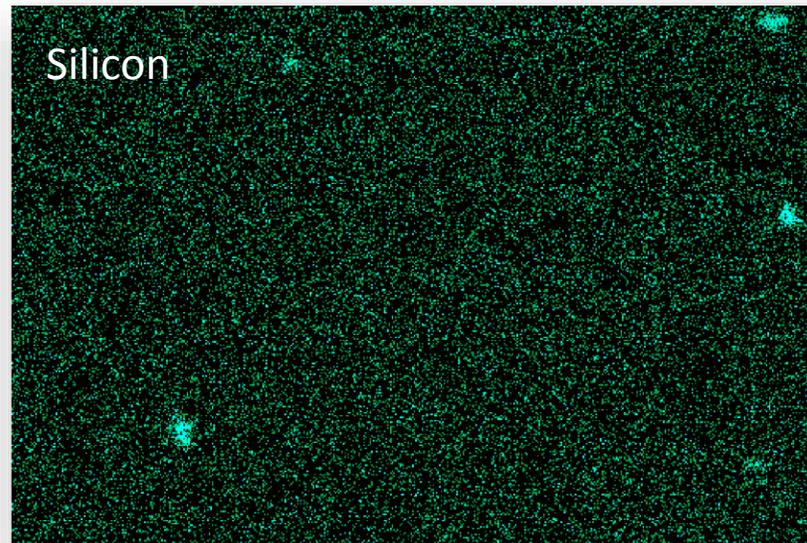
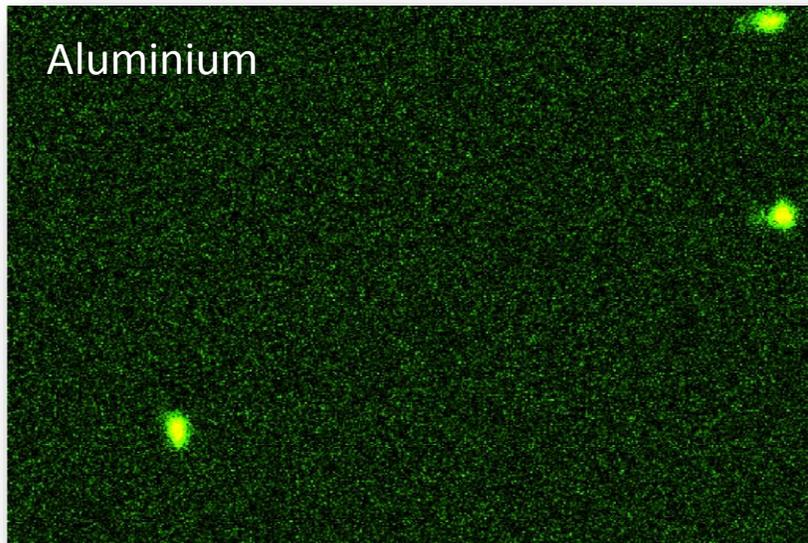
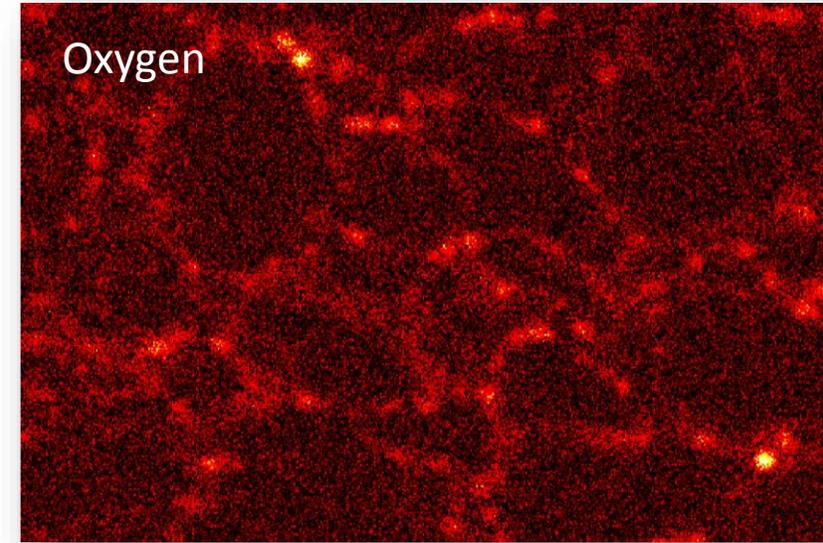
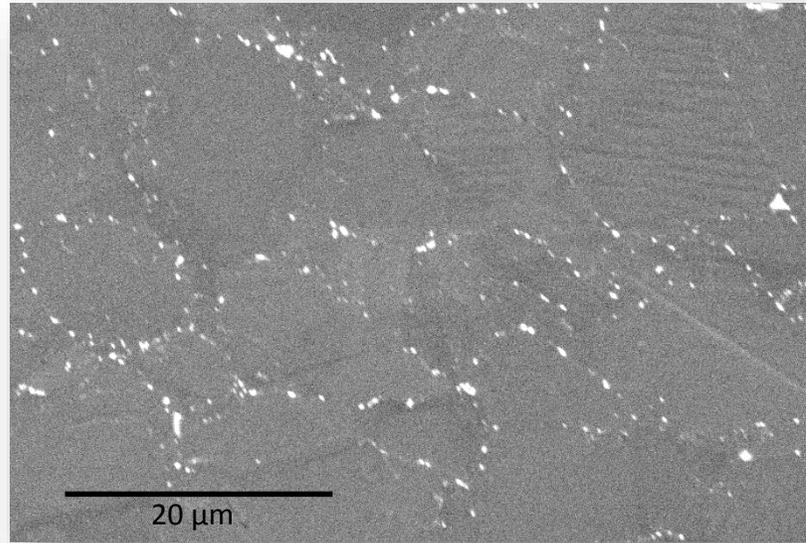
Beryllium is of industrial purity

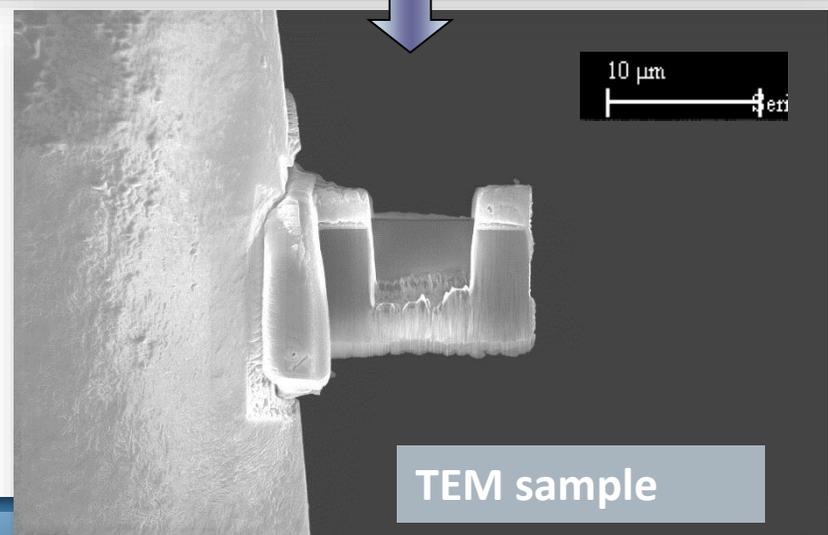
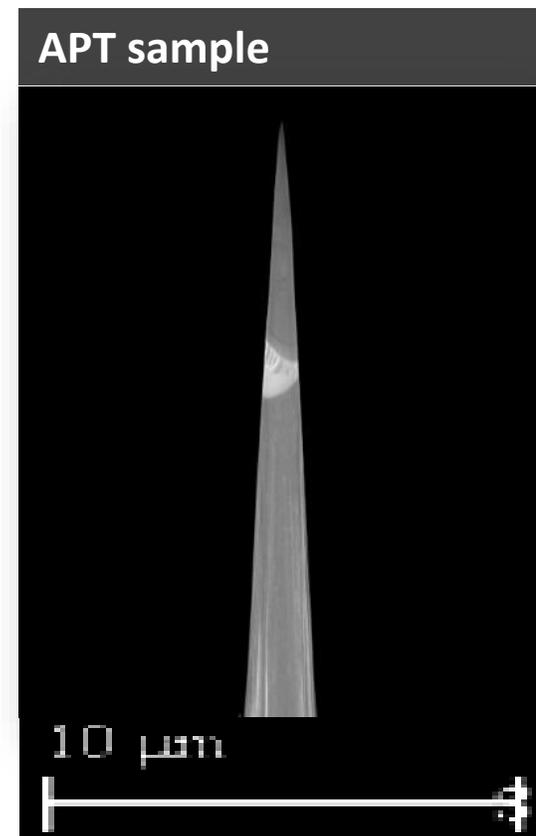
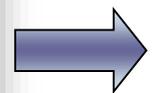
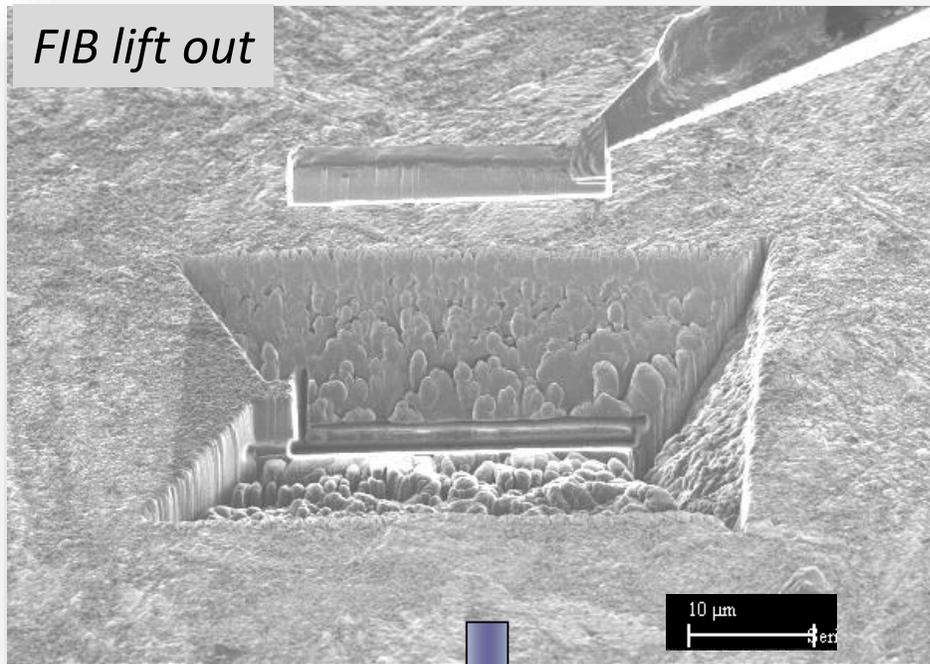
Microstructure and properties of as-received beryllium



Microstructure and properties of as-received beryllium

PF-60/VHP





Culham Materials Research Facility



FIB lift-out

- superpose the microstructural data with the dpa, appm and hardness data
- minimize the activity of samples
- minimize the toxicity of samples

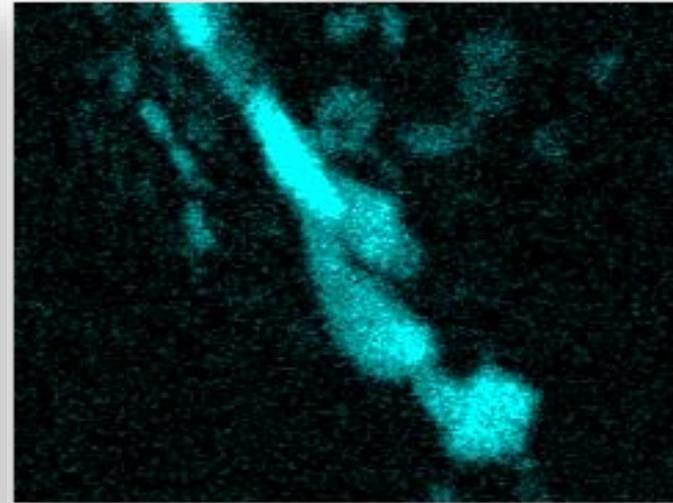
Pre- and post-irradiation experiments

PF-60
STEM

Map Data 1

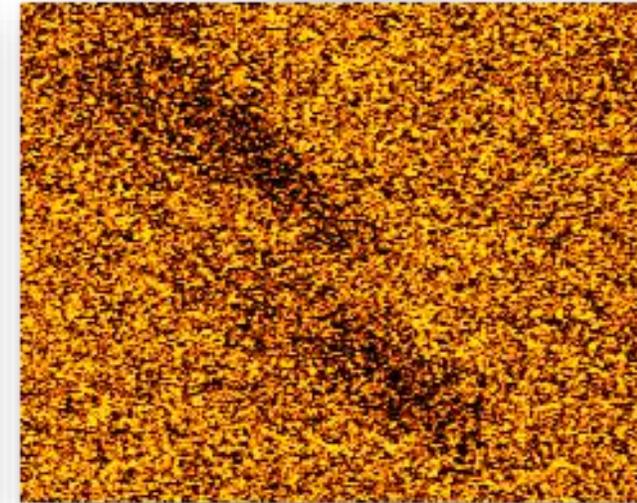
1 μ m

O K α 1



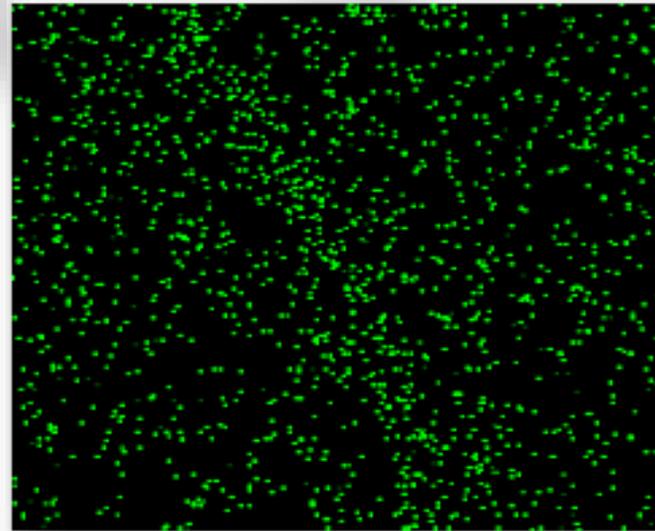
1 μ m

Be K α 1_2



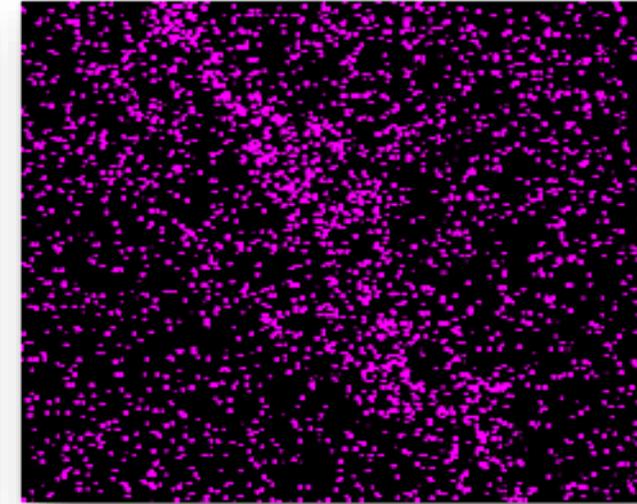
1 μ m

Fe K α 1



1 μ m

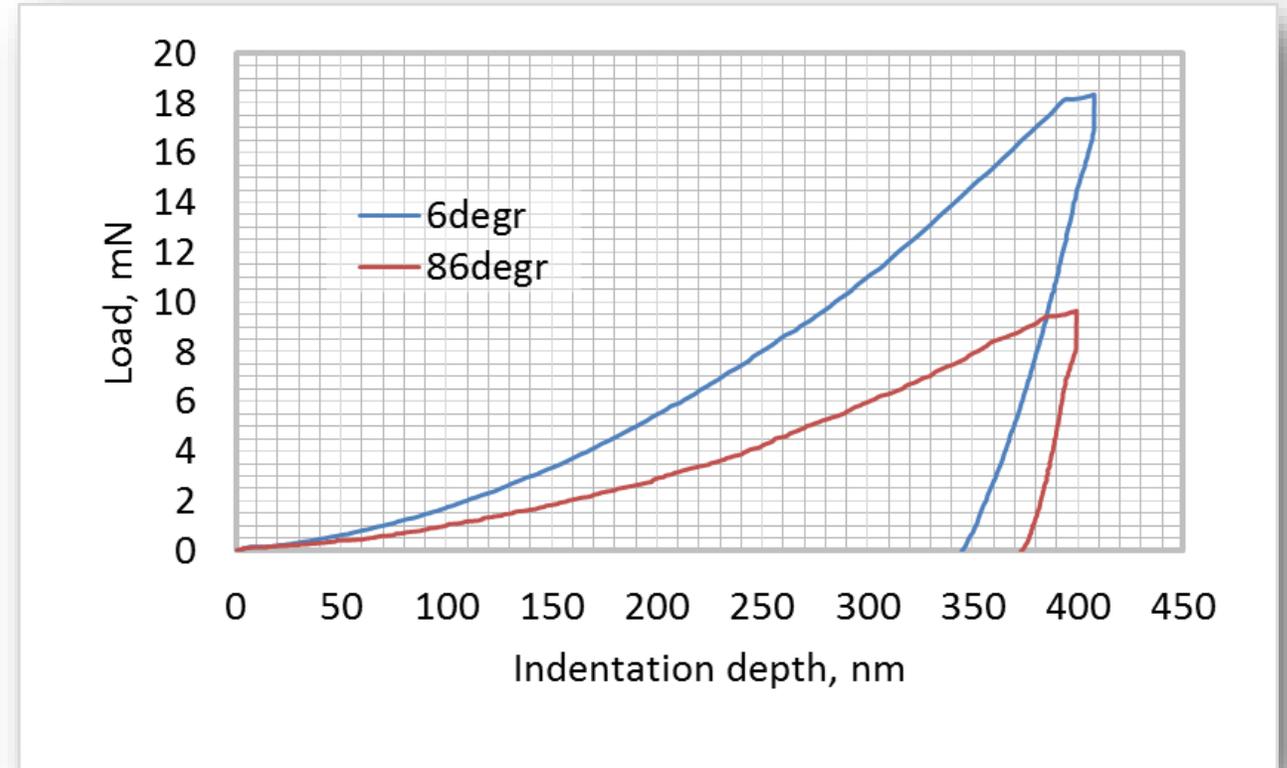
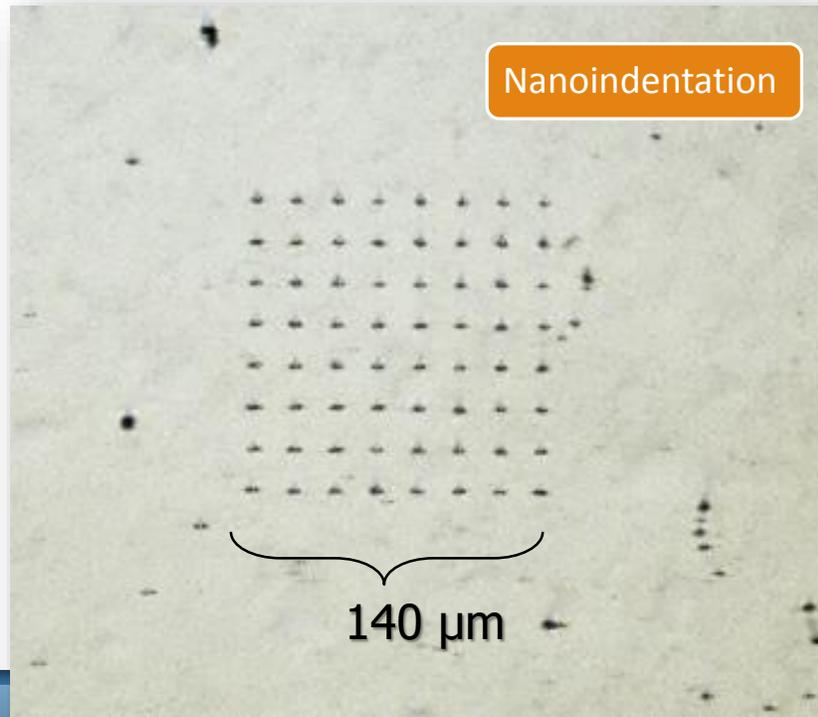
Si K α 1



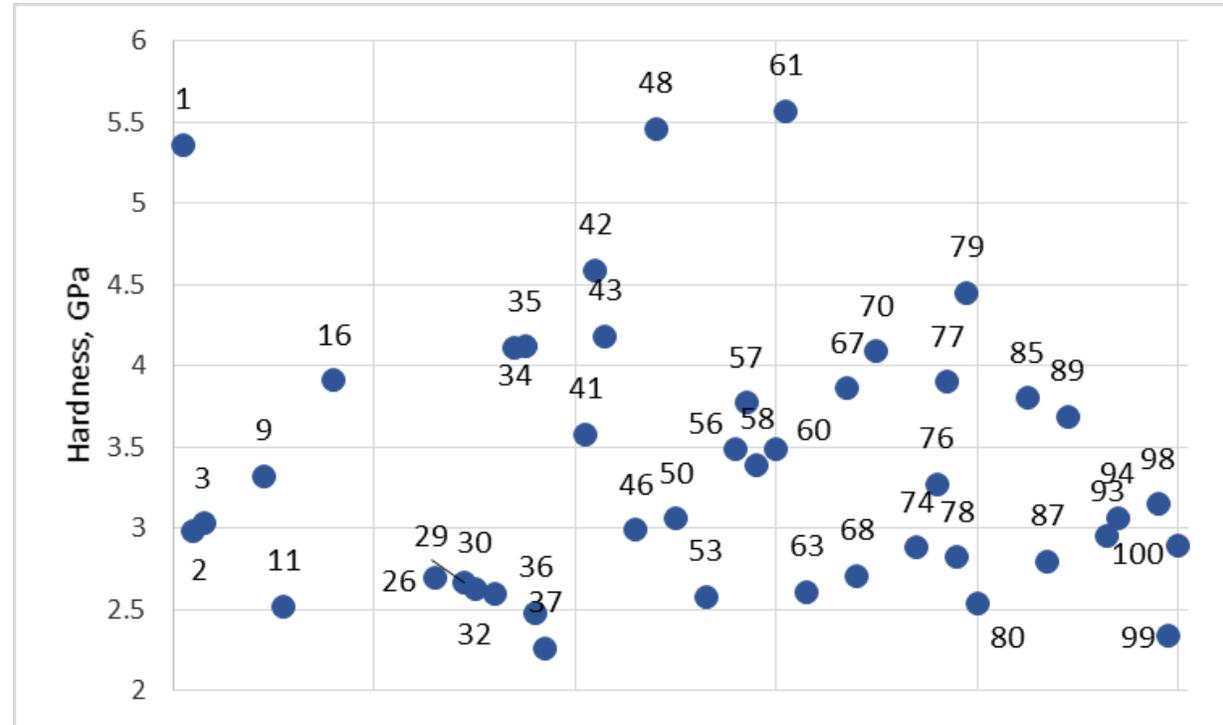
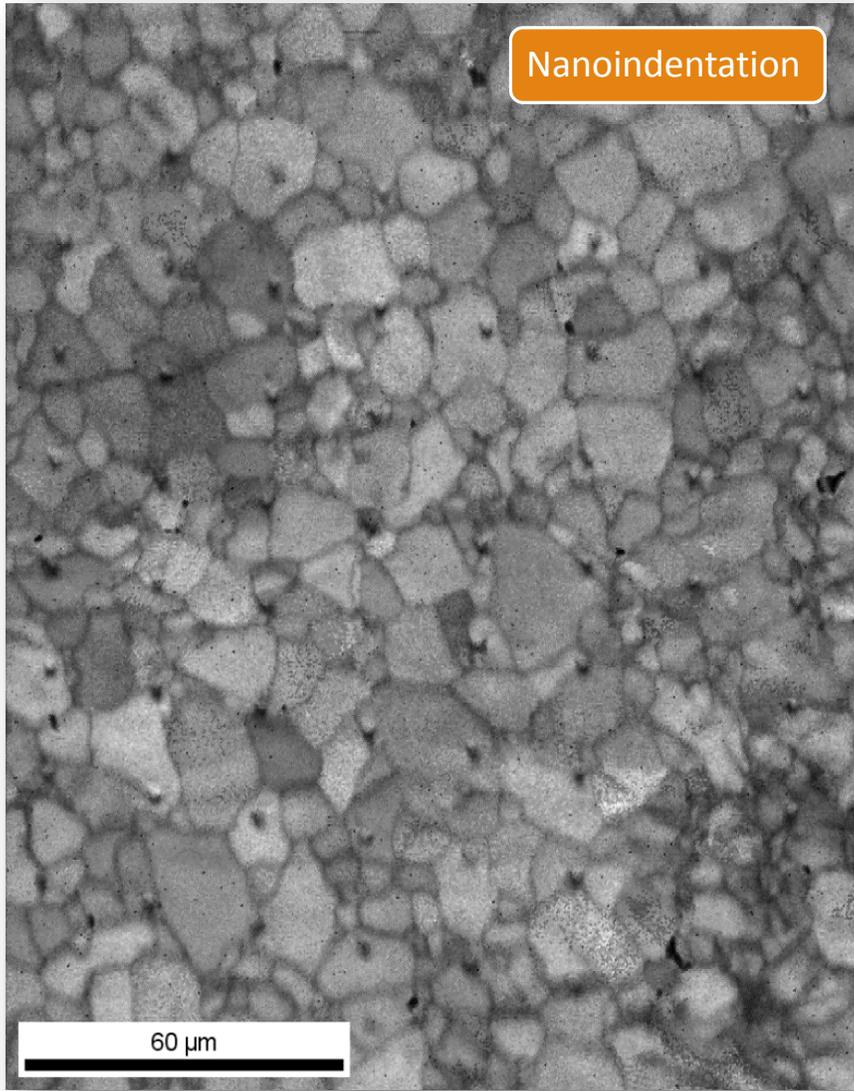
1 μ m

Preliminary: Fe and Si segregates to beryllium oxides

Nanoindentation

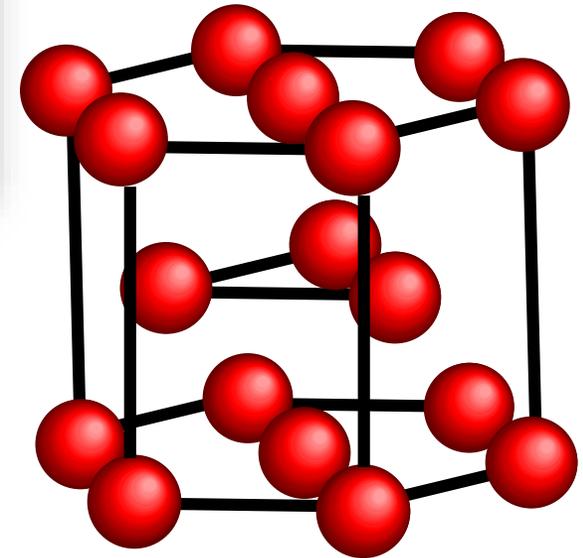
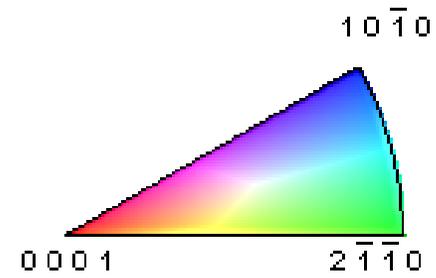
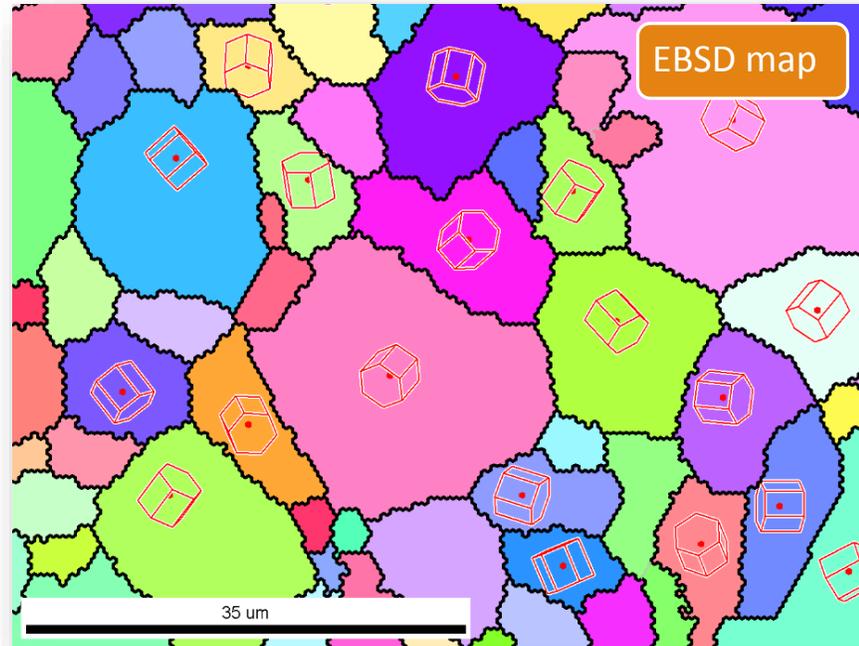
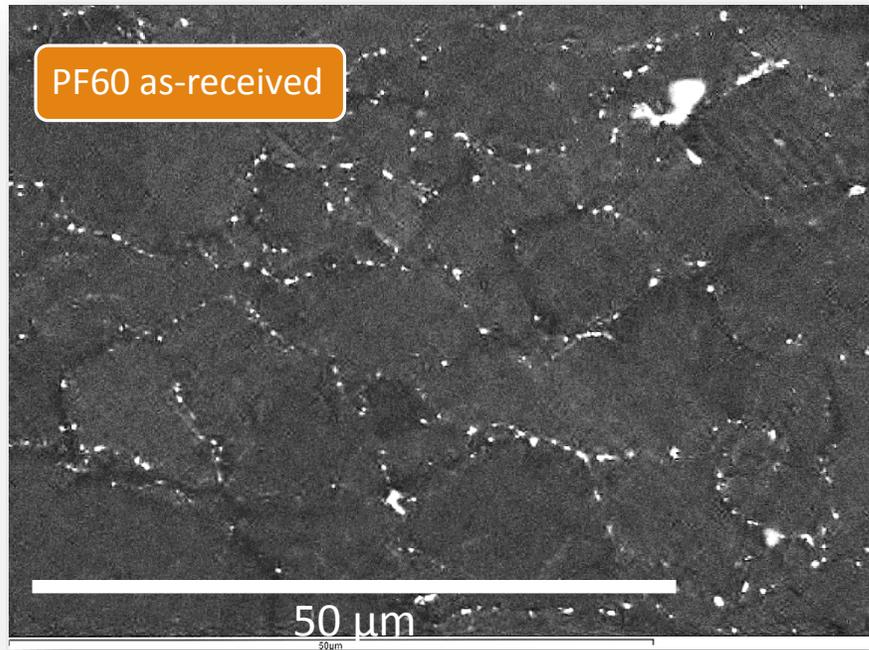


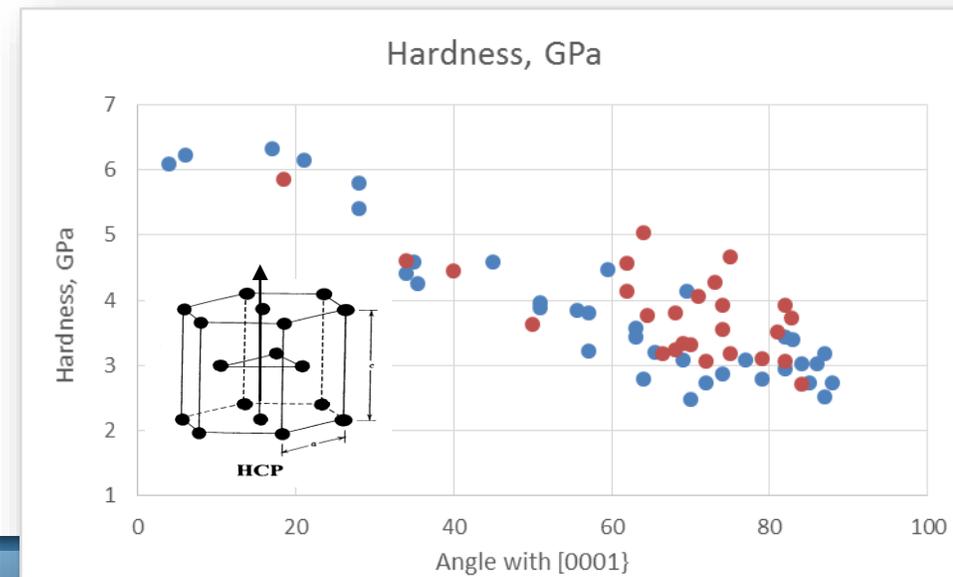
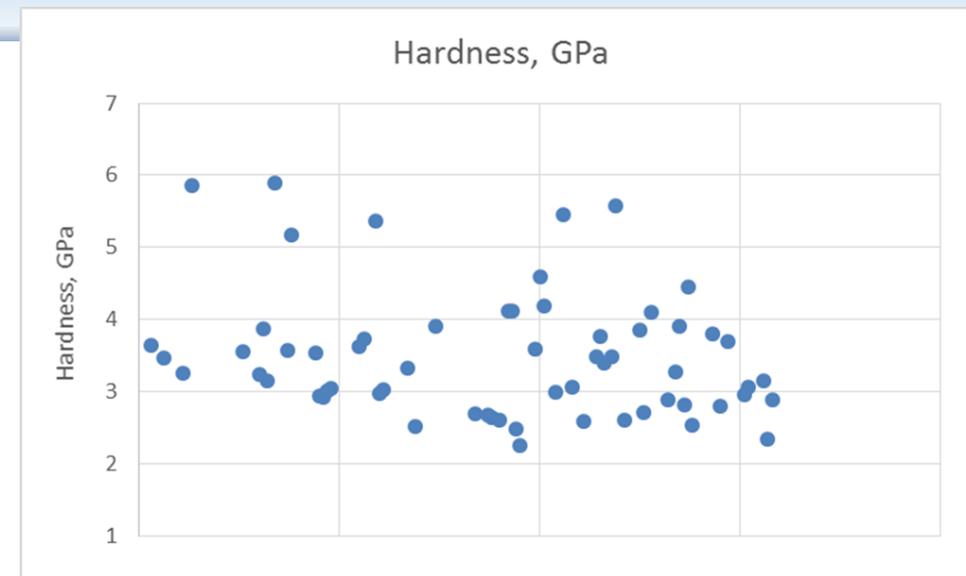
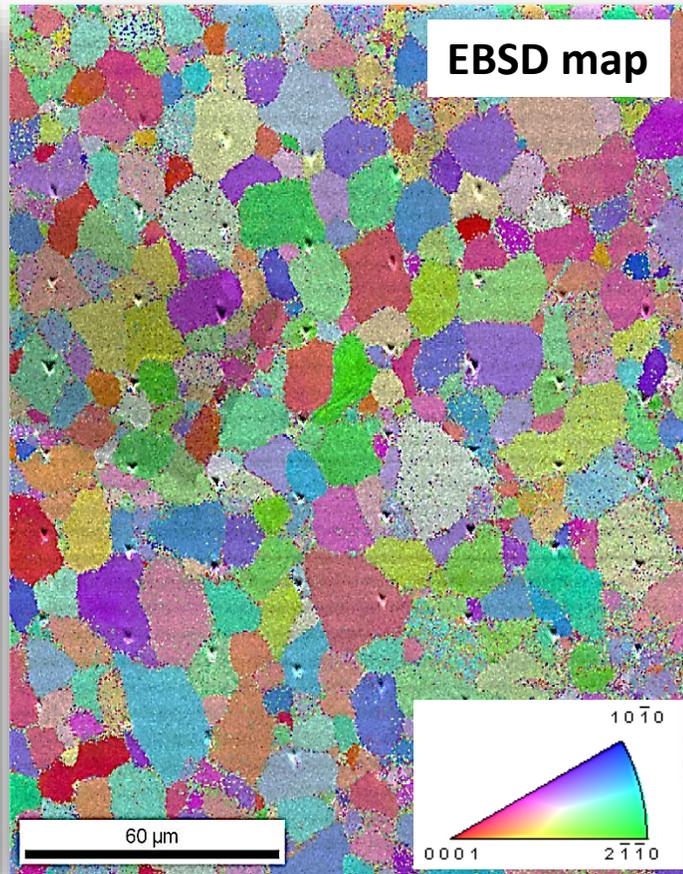
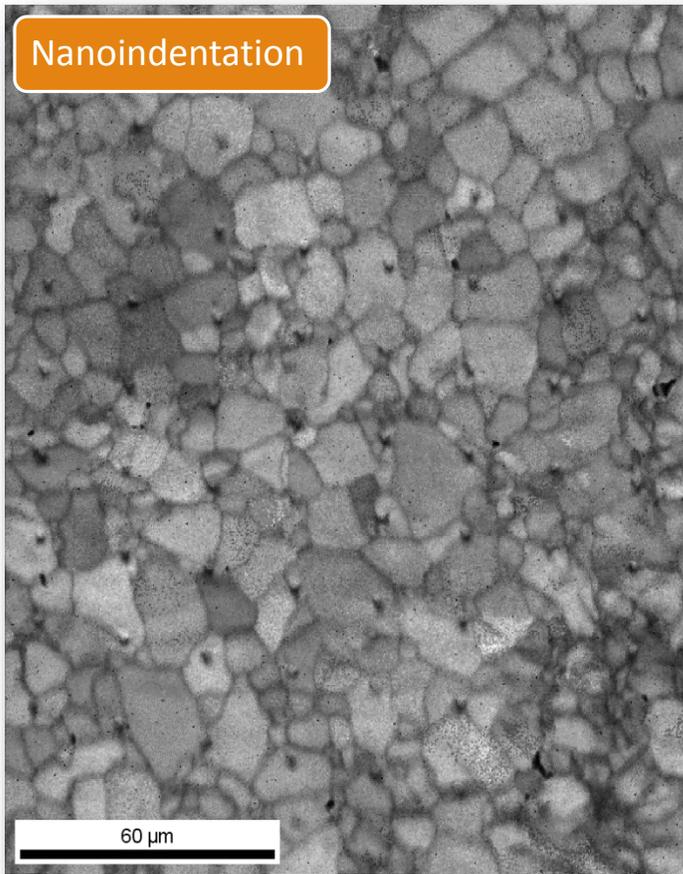
By knowing the contact area between sample and indenter hardness and modulus from unload can be calculated



Extremely wide scattering

Be lattice is hcp





The scattering makes sense... but the anisotropy is impressive

NuMI beam window experiments

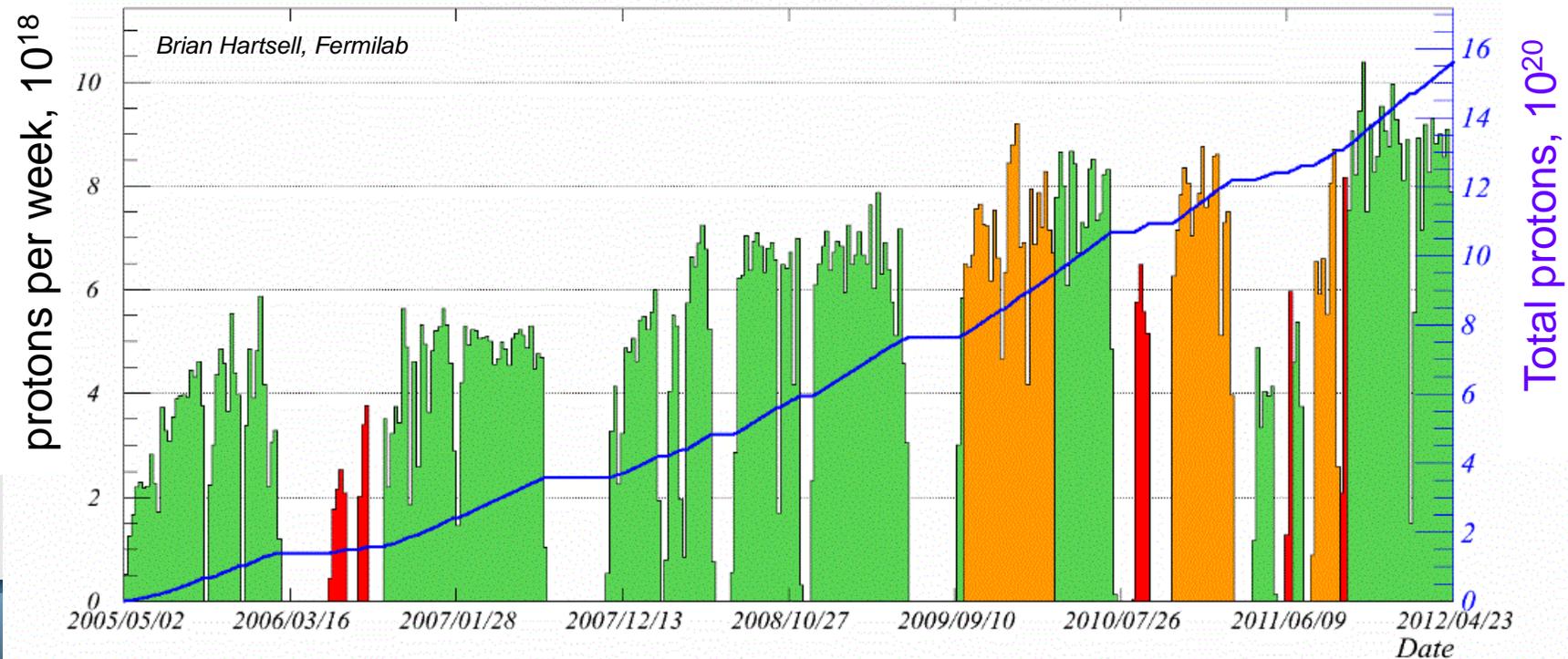
300 kW NuMI beam window

(MARS calculations of Brian Hartsell, Fermilab)

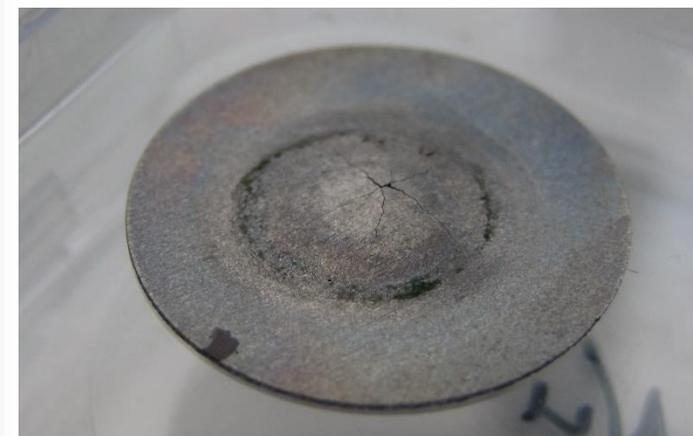
- 120 GeV proton beam
- about 3×10^{13} protons per pulse, 0.5 Hz
- 1.57×10^{21} protons during its lifetime
- 1.1 mm beam sigmas, X and Y
- $T \approx 50^\circ\text{C}$



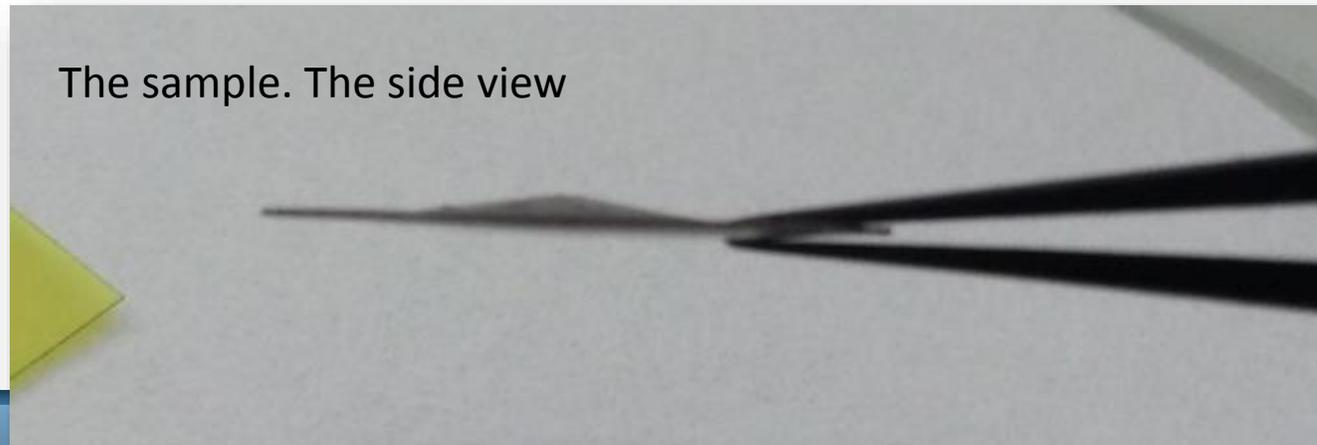
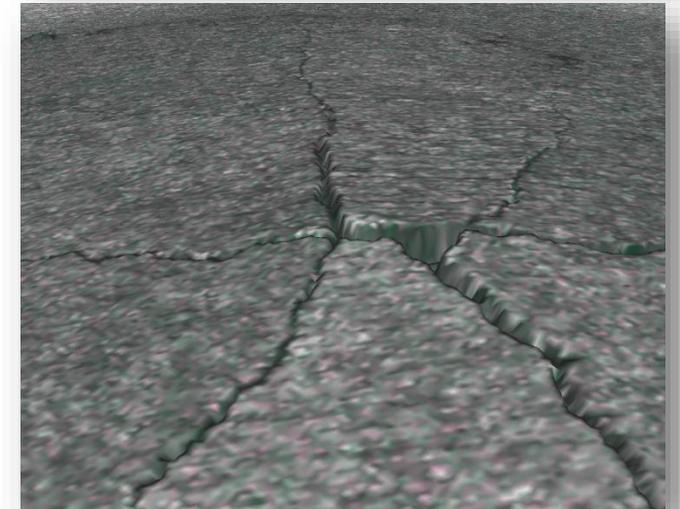
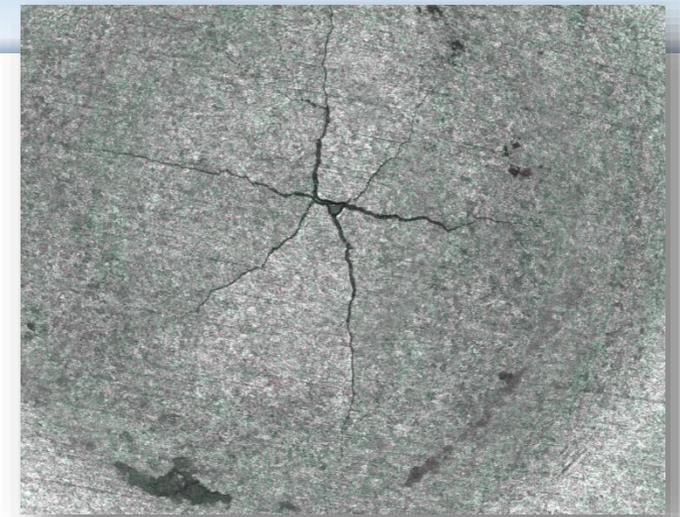
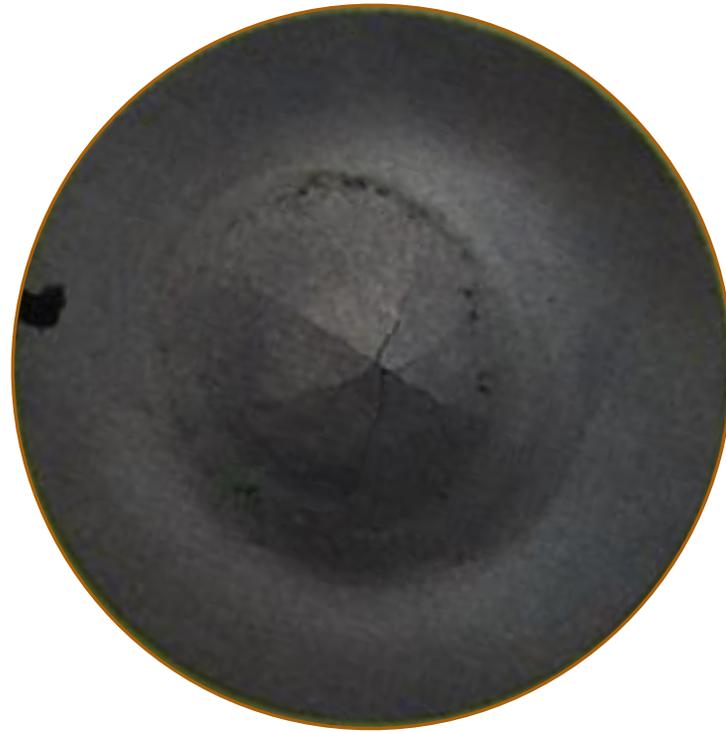
Total NuMI protons to 00:00 Monday 23 April 2012



Total protons, 10^{20}



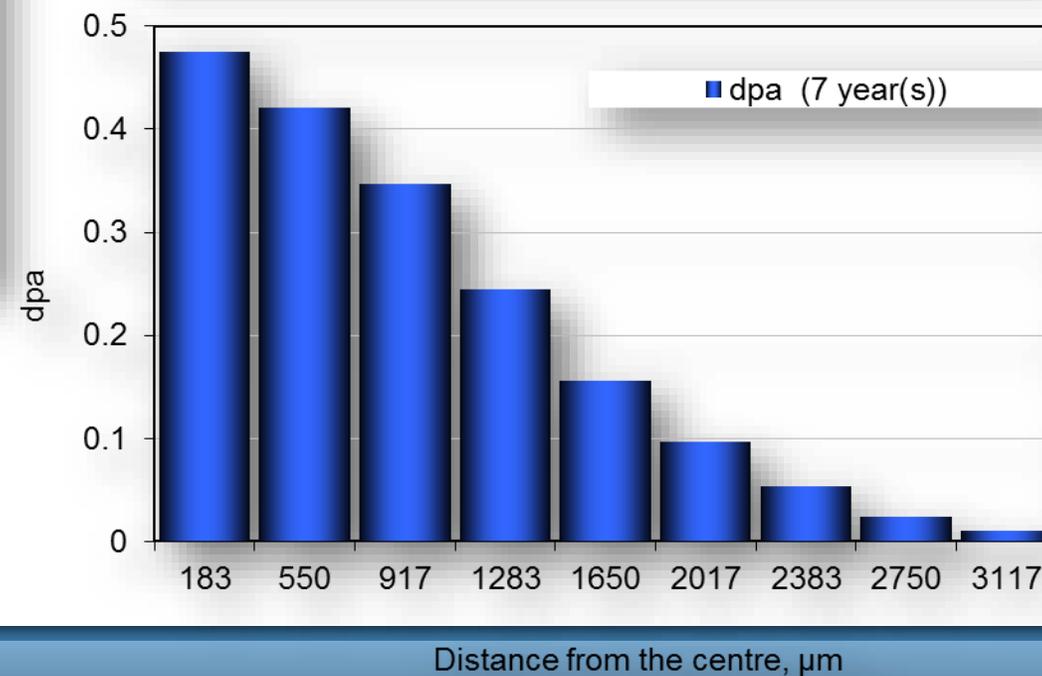
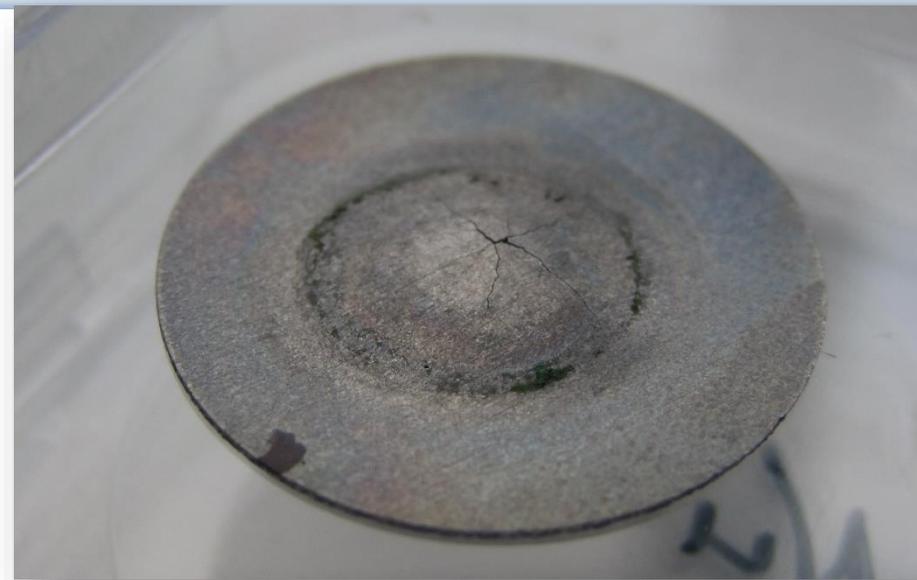
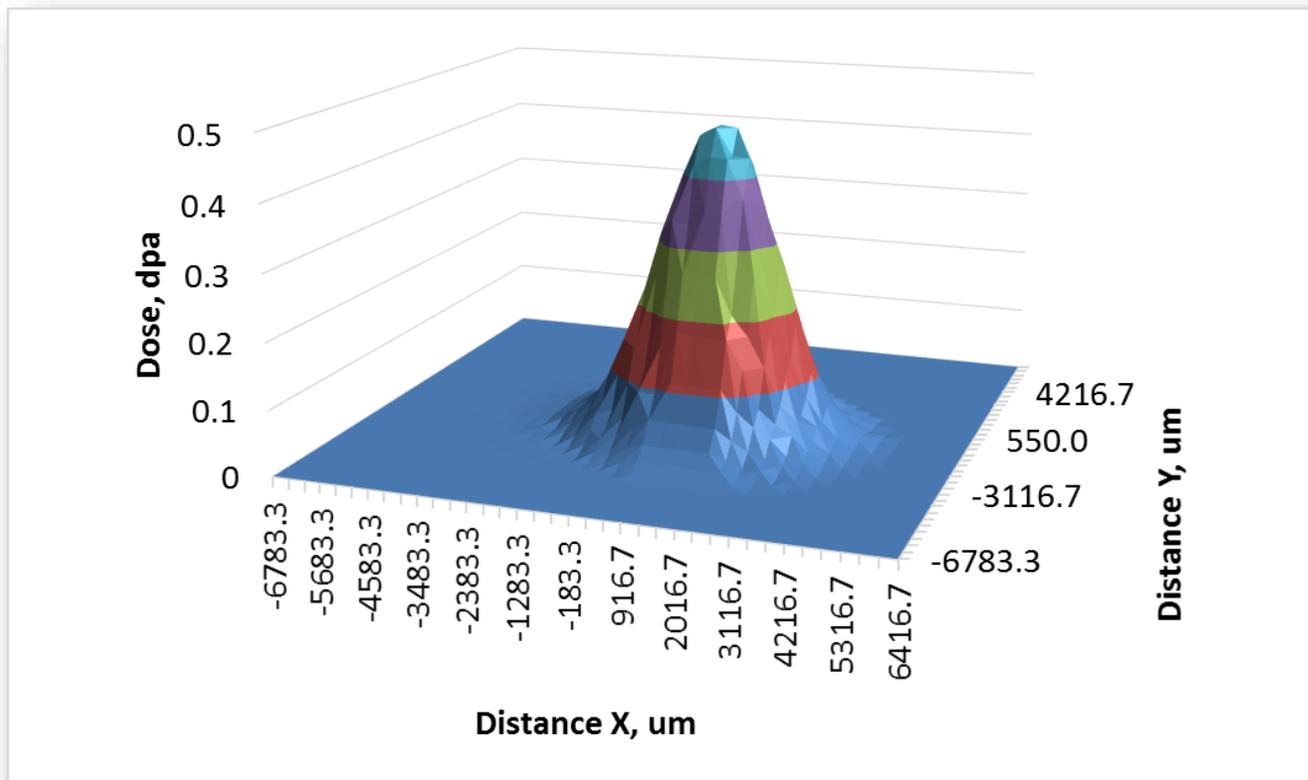
NuMI beam window experiments



The sample. The side view

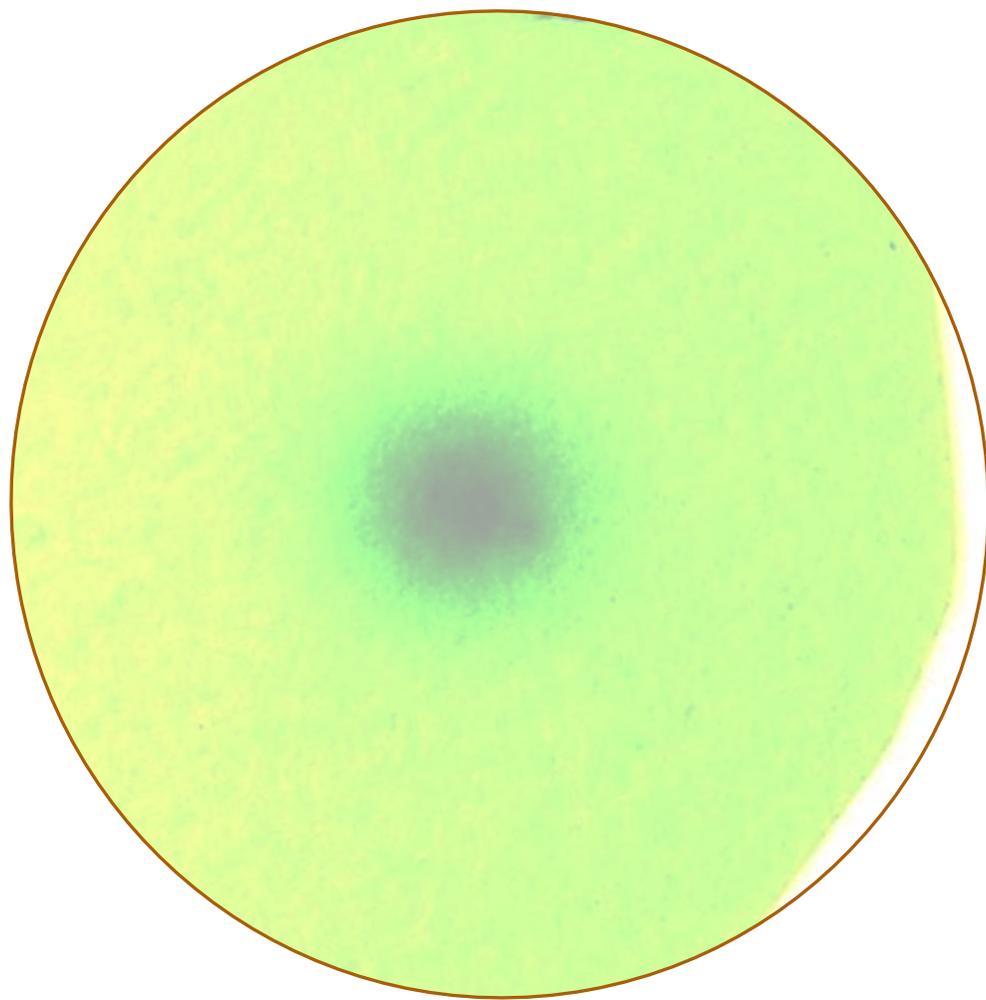
Radiation embrittlement?

Gaussian distribution of the beam

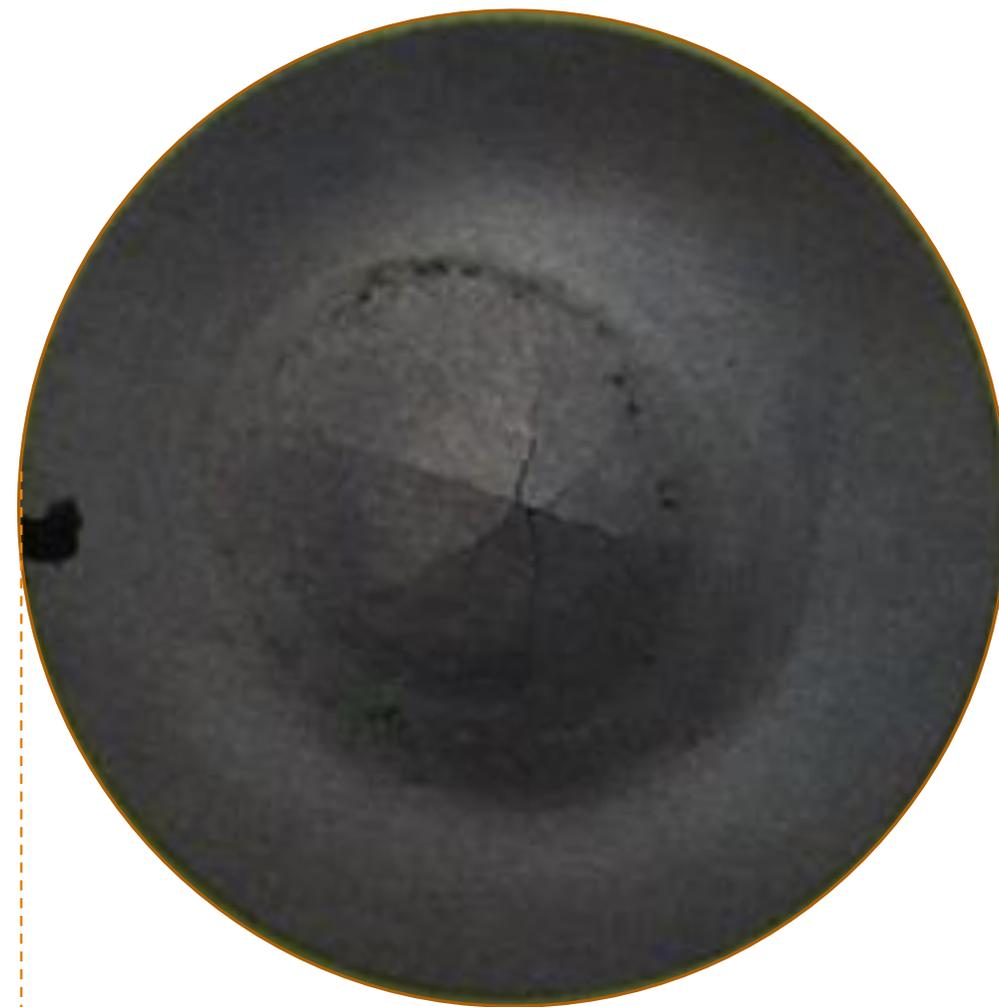


• Radiation damage distribution is not monotonic

Dosimetry film, 5 days of exposure

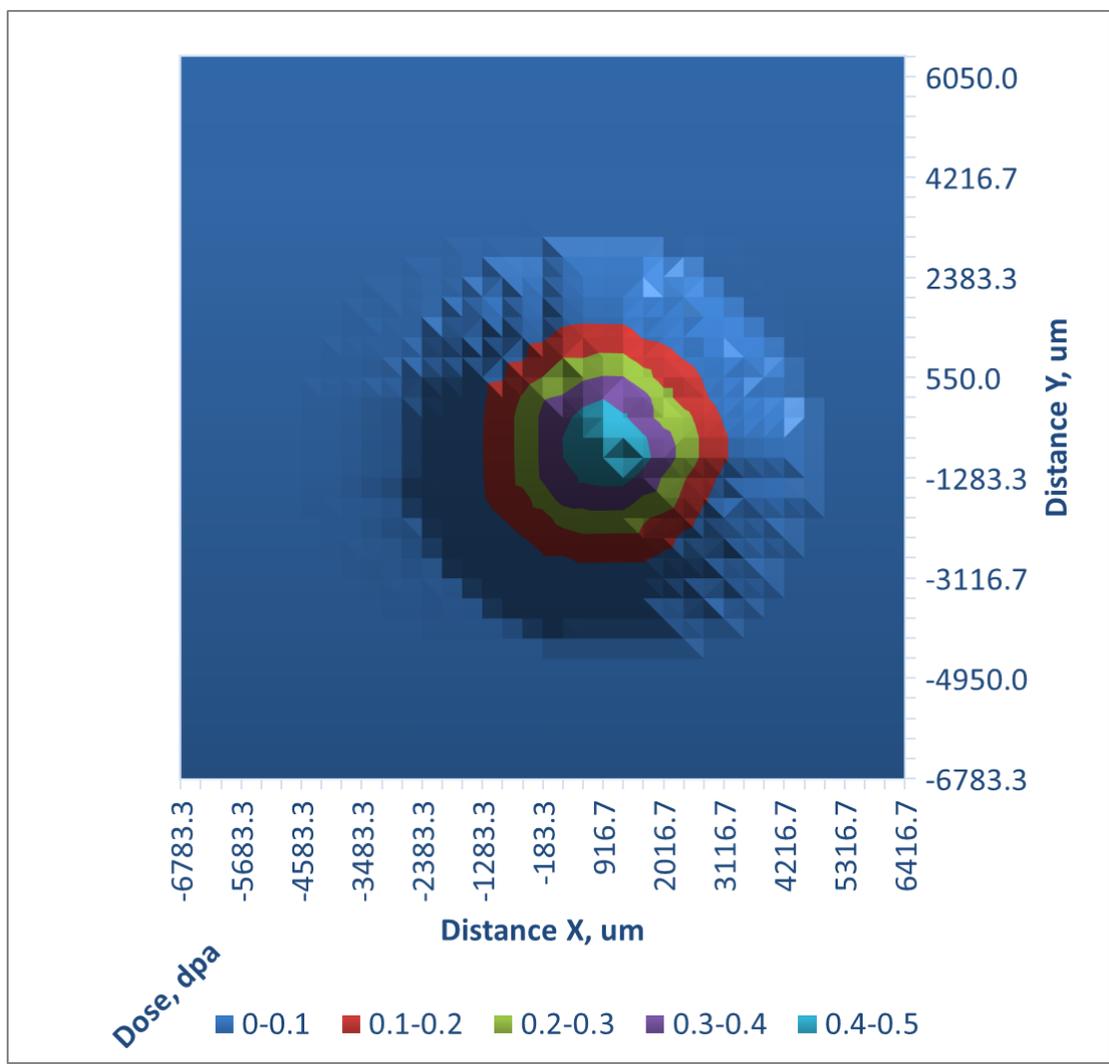


'Gafchromic™ HD-V2 dosimetry film'

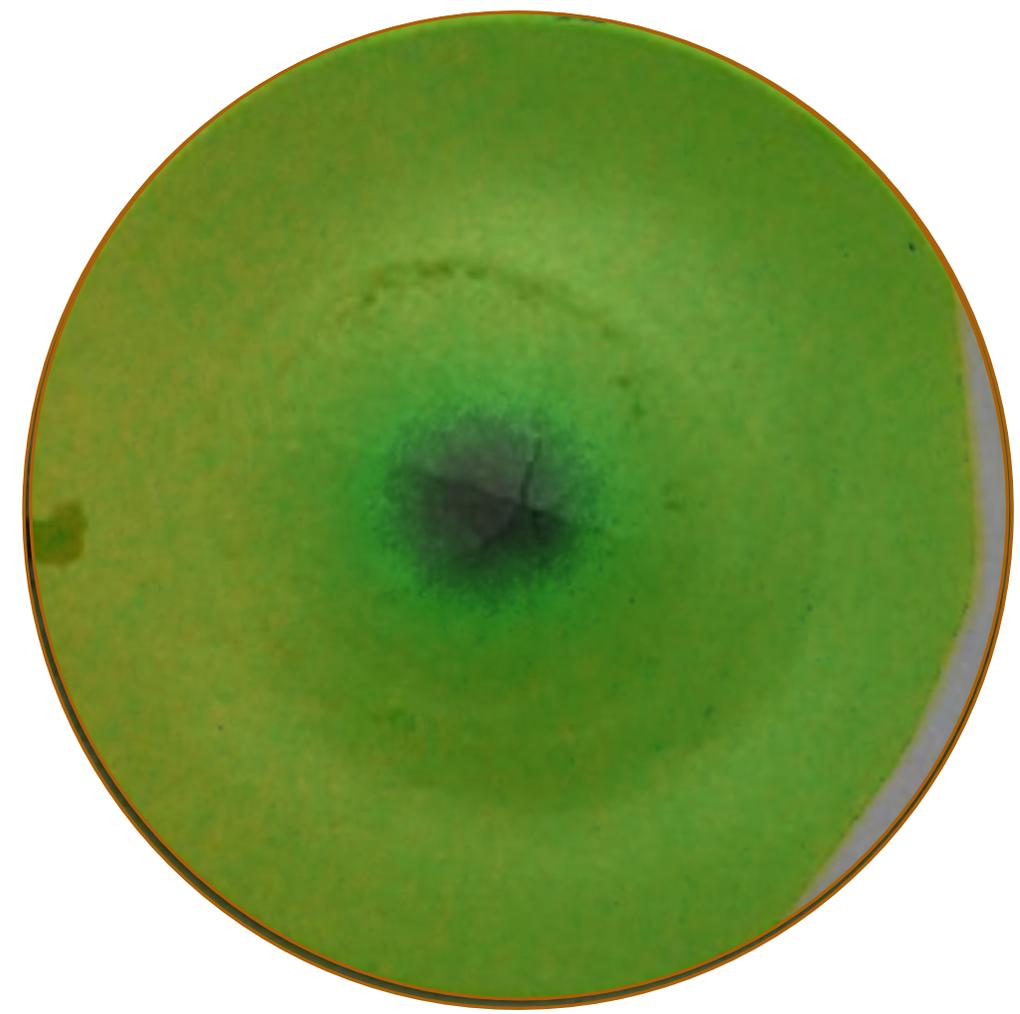


19 mm

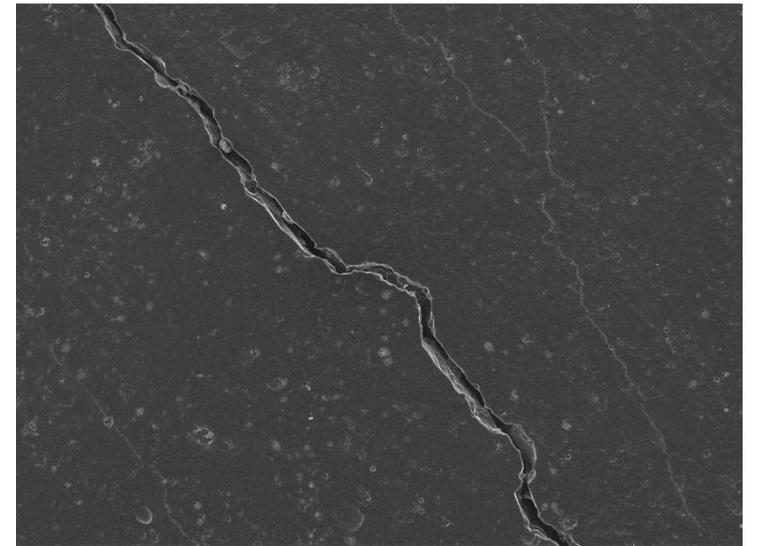
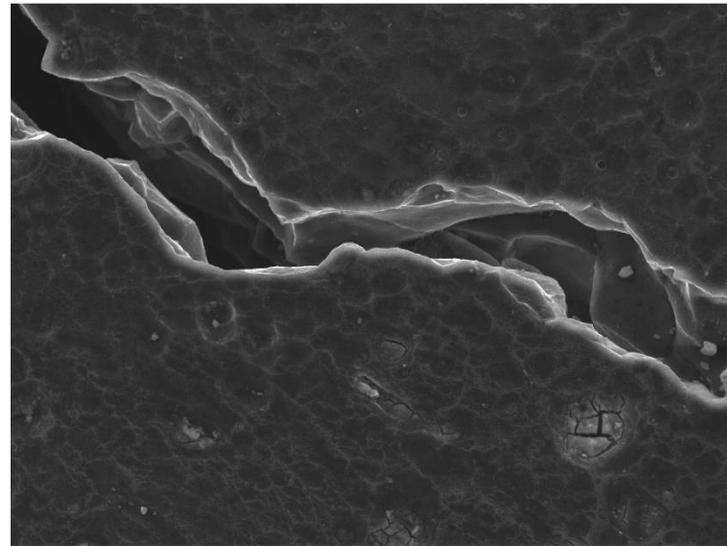
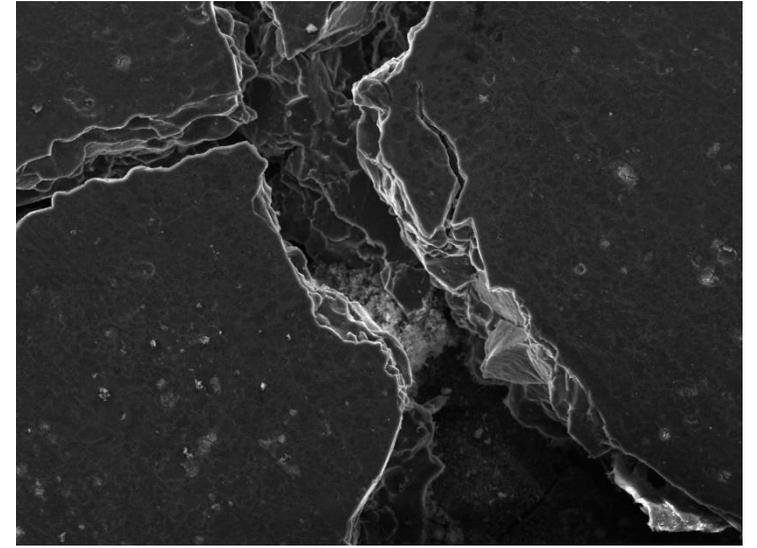
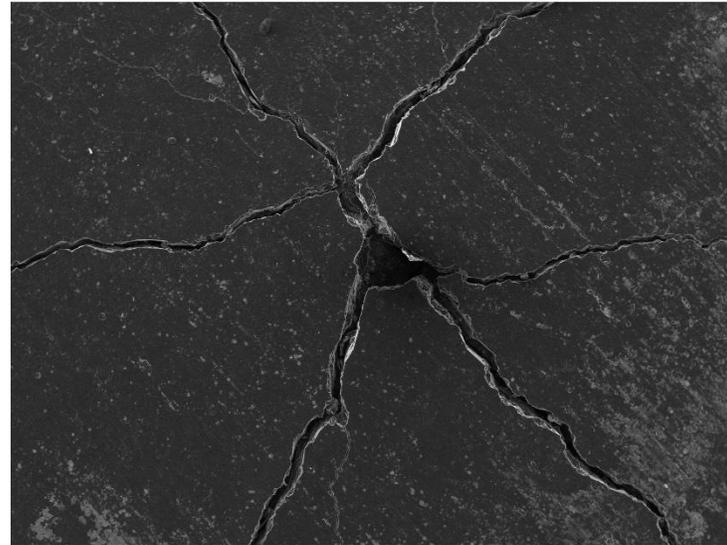
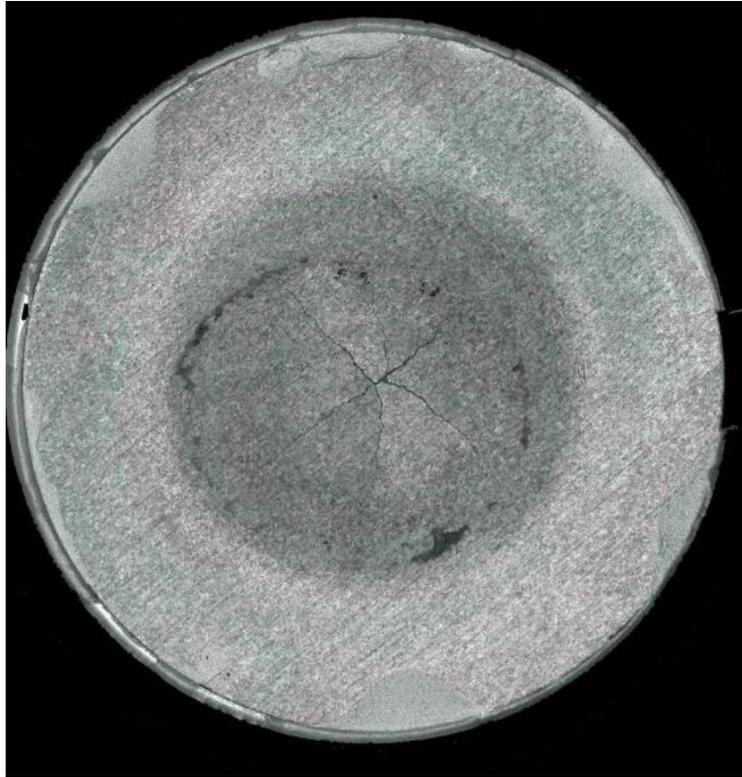
MARS calculations map



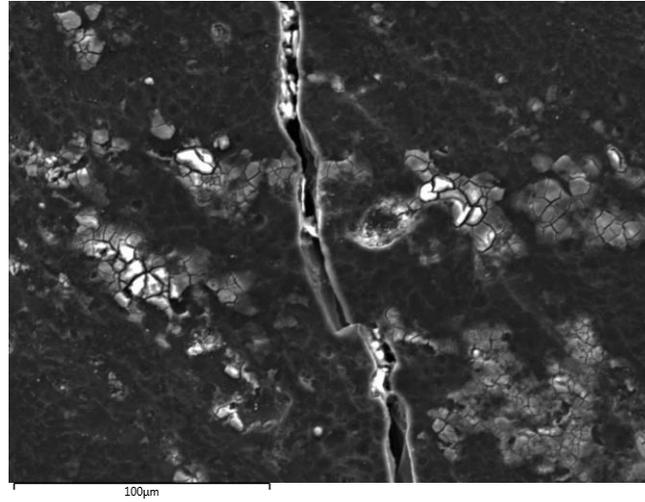
Dosimetry map



NuMI beam window experiments



Preliminary: The crack propagates by both intra- and intergranular mechanisms

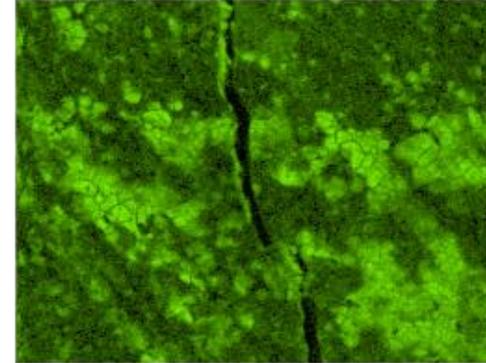


Surface contamination:

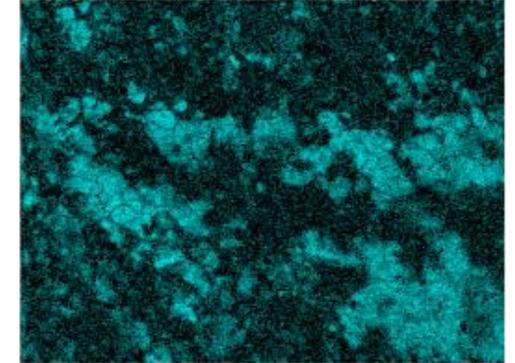
Ni - comes from plating?

Ca, S, Cl, C – drips of drainage water?

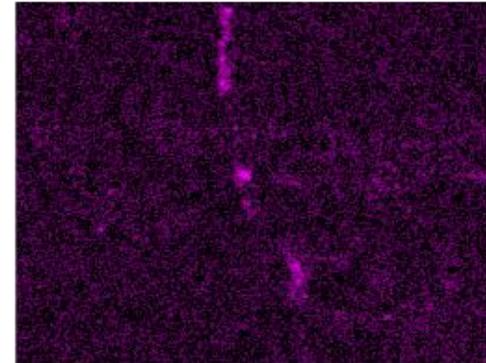
O K series



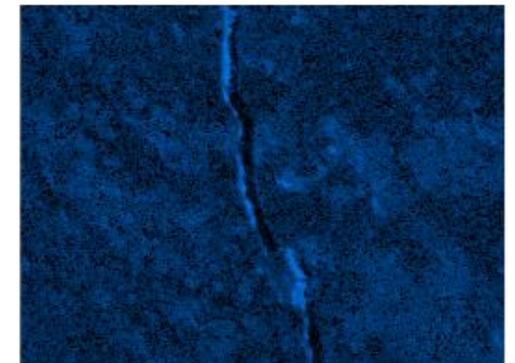
Ni K series



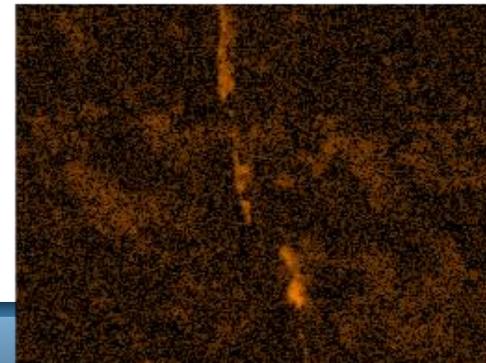
Ca K series



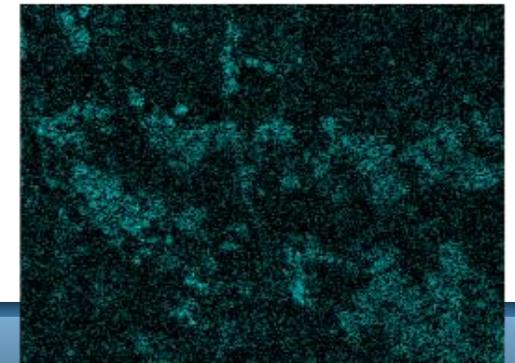
C K series



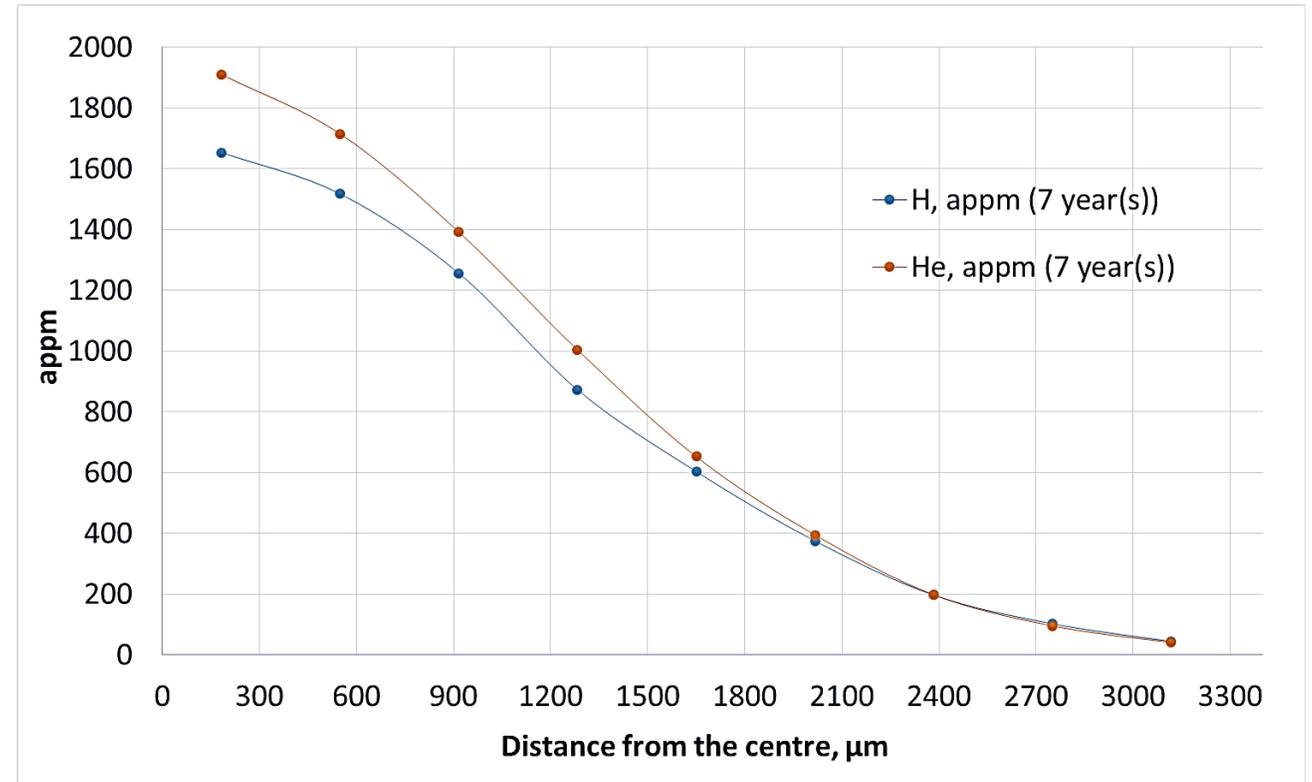
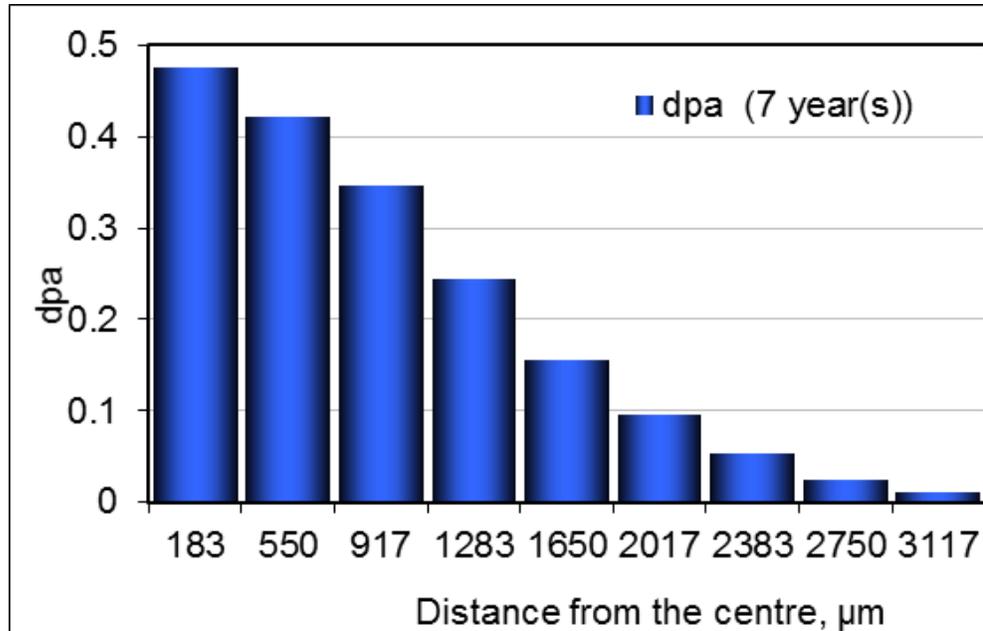
Cl K series



S K series

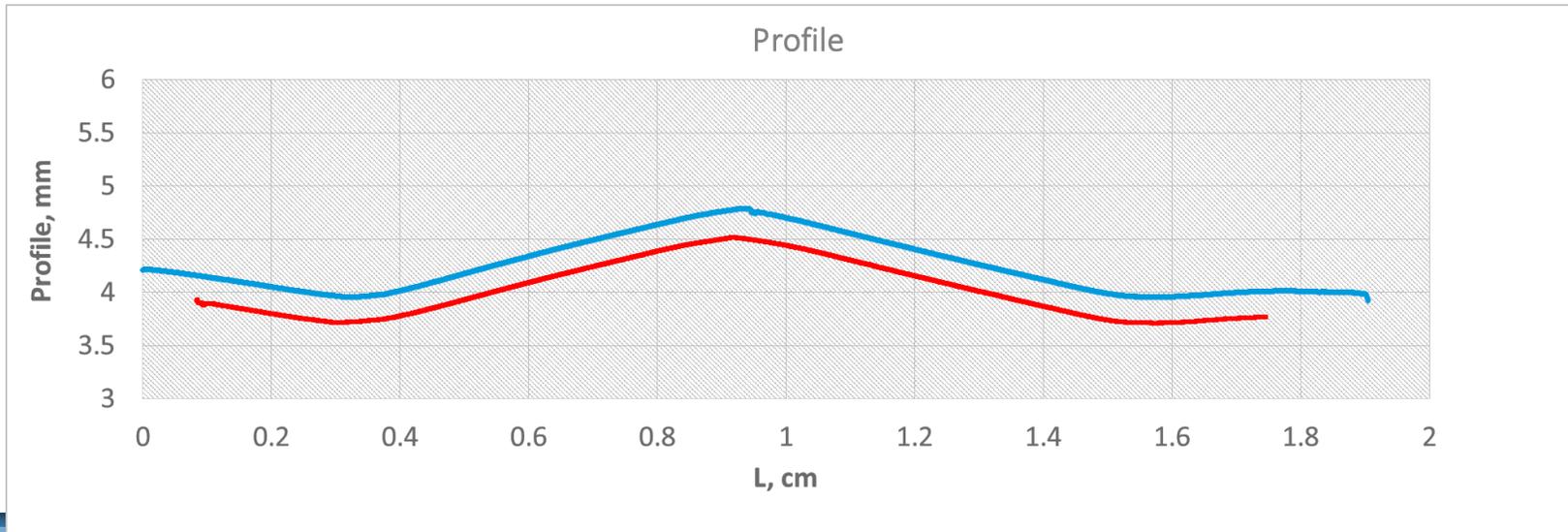
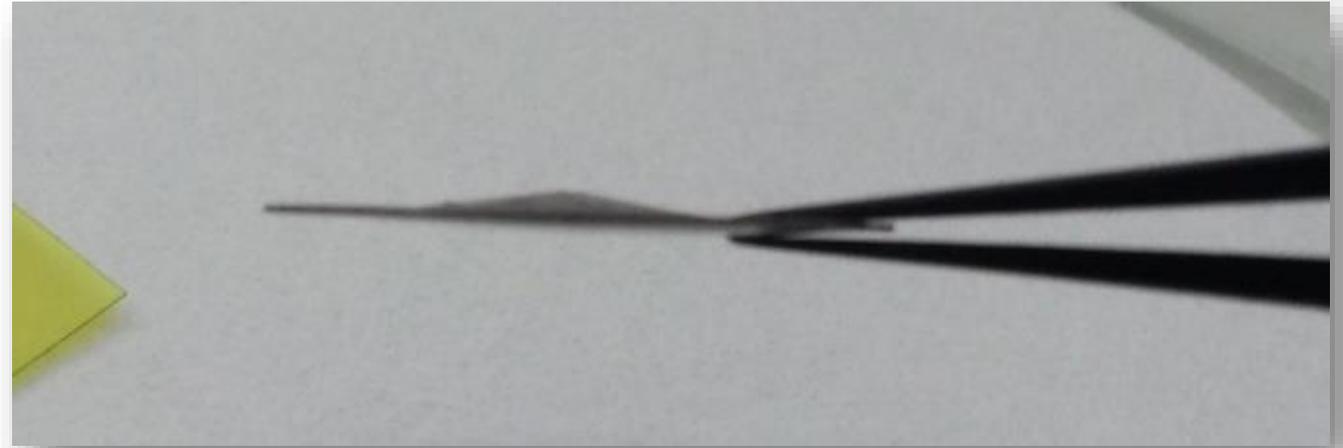
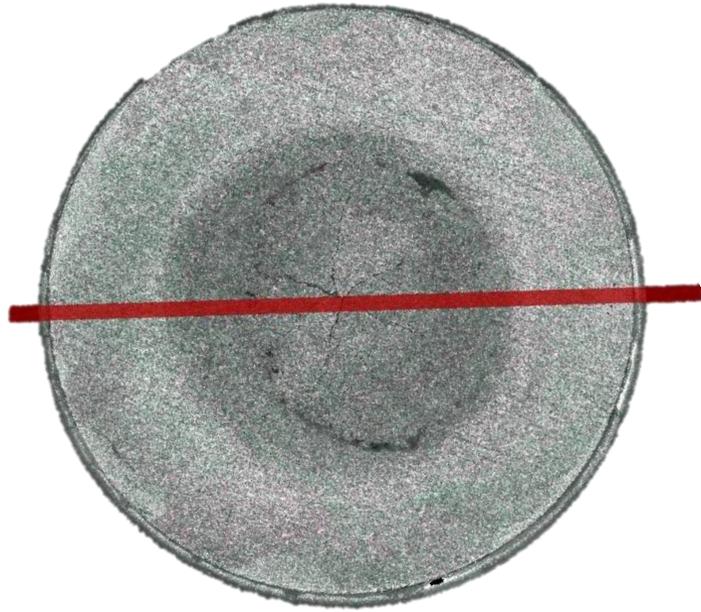


The main transmutation products are He and H

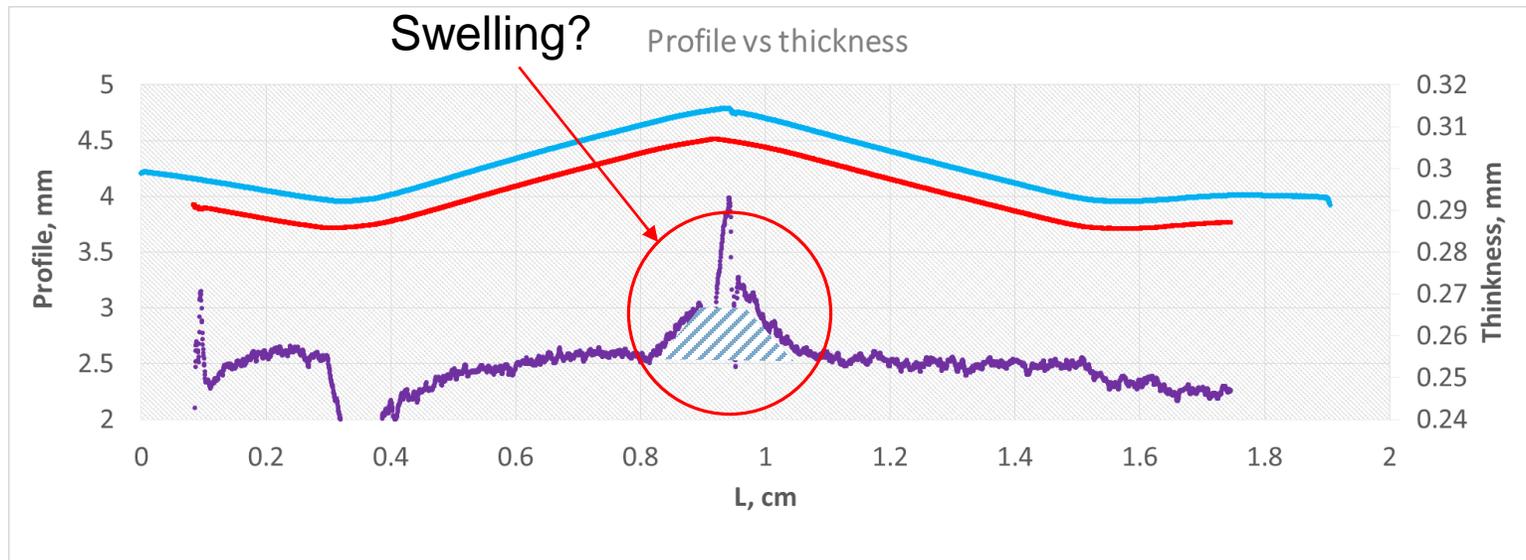
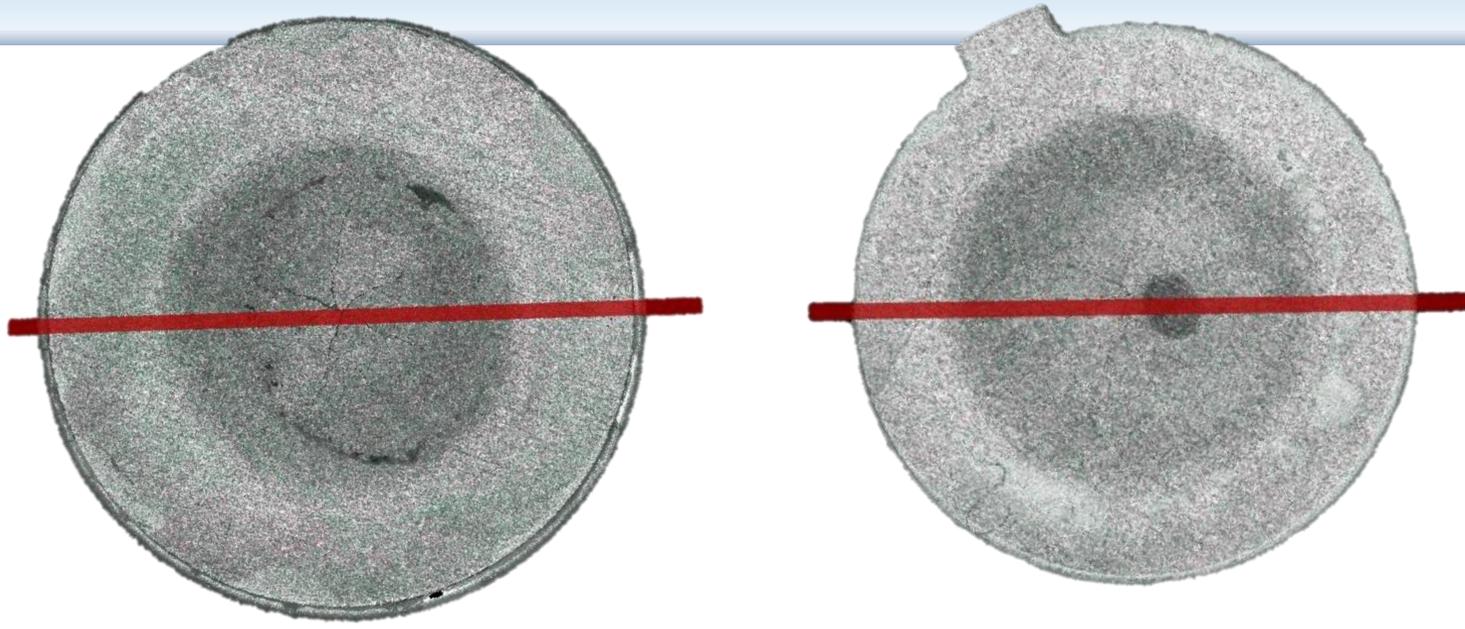


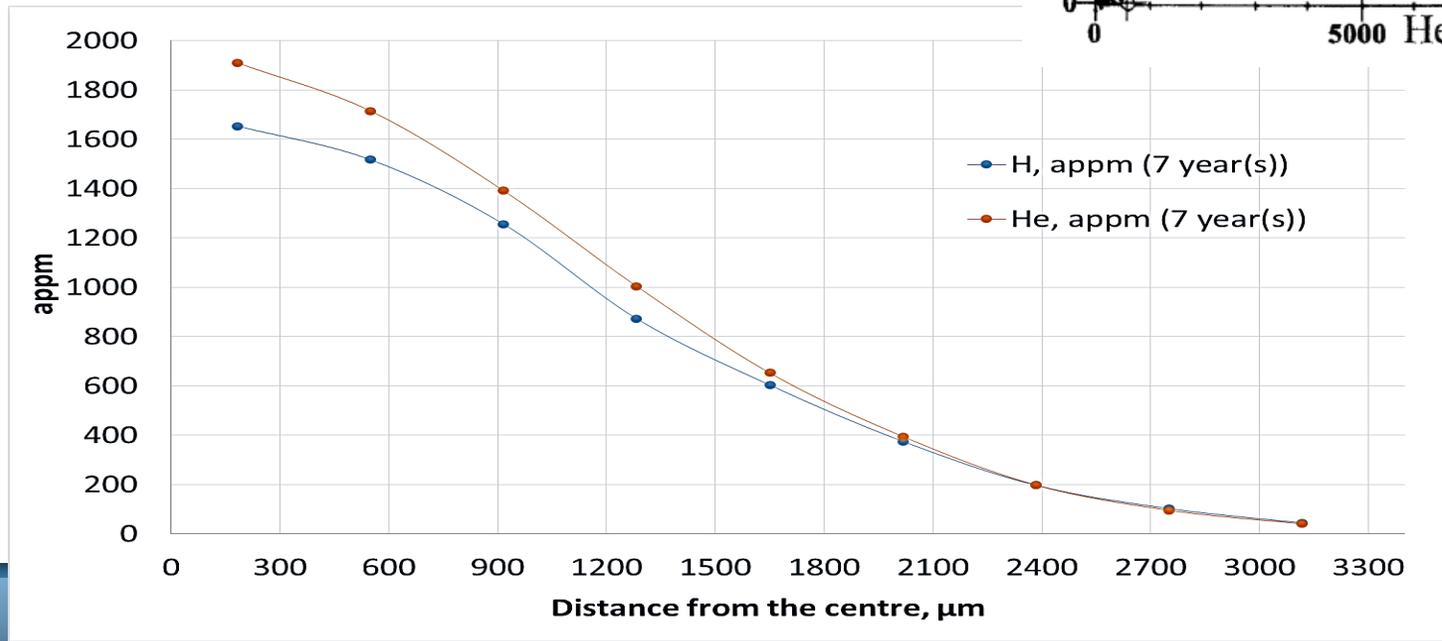
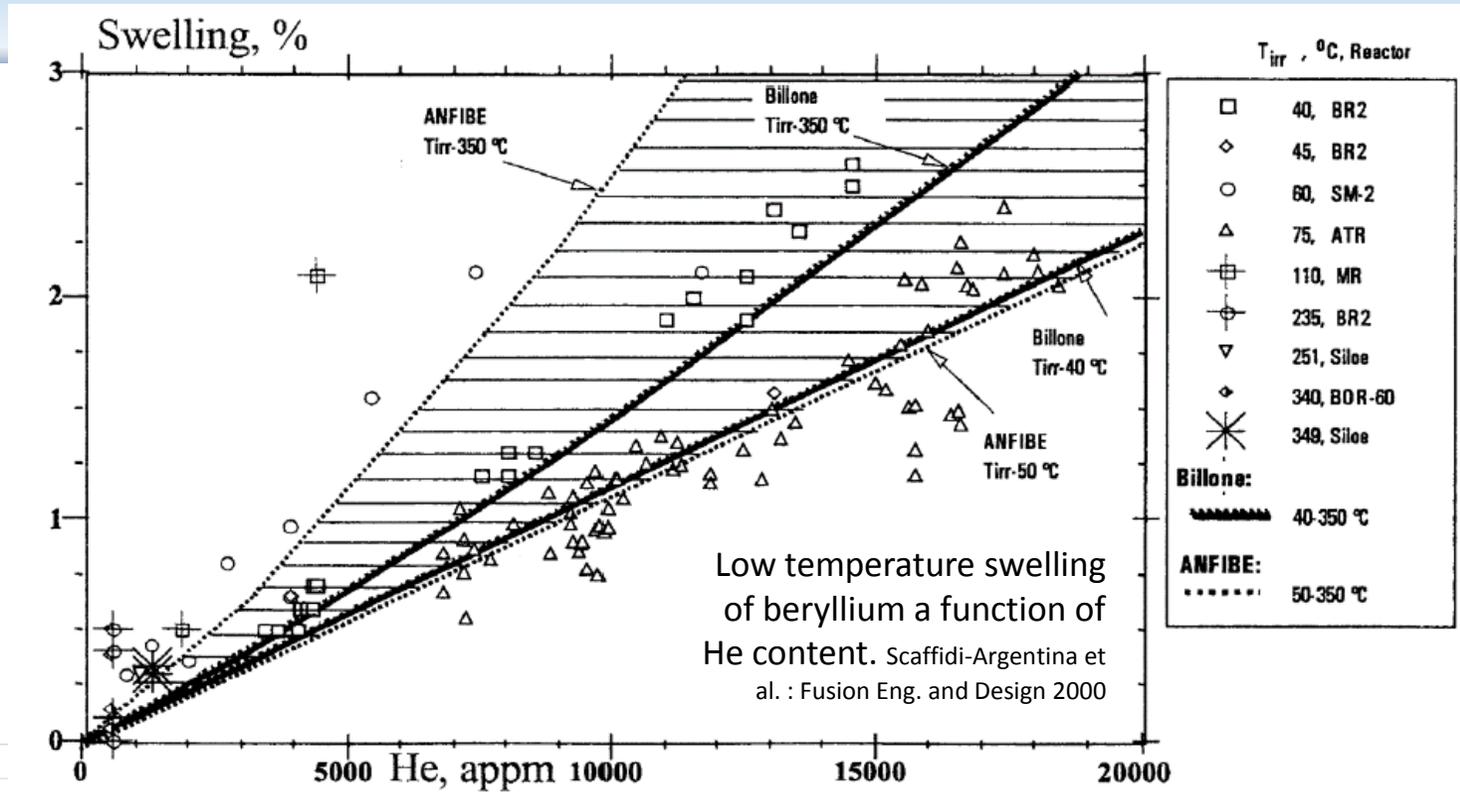
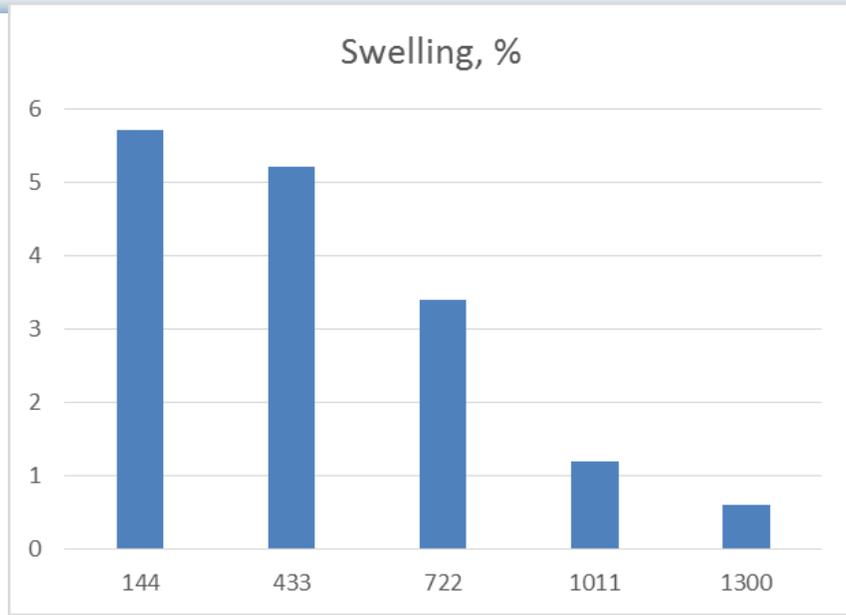
Large difference of dpa and transmutants production is likely to produce non-homogeneous changes across the surface of Be window.

NuMI beam window experiments



The window is deformed

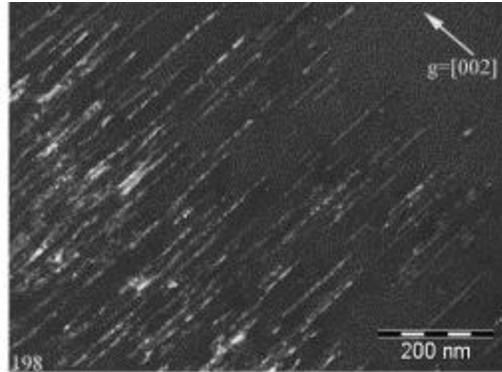




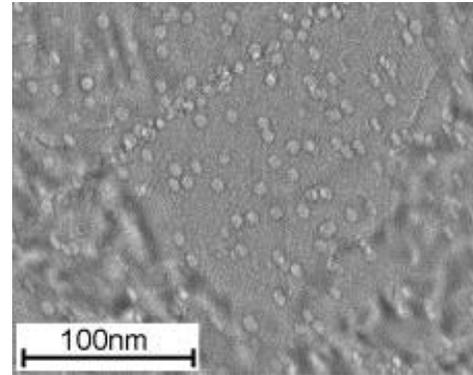
- High swelling at low T?
- But we have “only” about 2000 appm of He
- Too much, if the fission neutron data is applicable

n-irradiation:

- At low T_{irr} below $\sim 200^\circ\text{C}$ (Chakin et al. JNM 2009) or 400°C (Gelles et al. JNM 1994): “black dots” and dislocation loops.
- At higher T: mainly He babbles

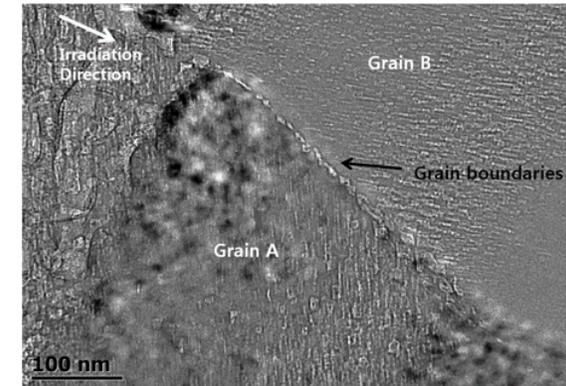


Irr. Be, TEM, DF, dislocation loops,
 $T_{irr.}=70^\circ\text{C}$, $F = 6 \times 10^{22} \text{ cm}^{-2}$
 ($E > 0.1 \text{ MeV}$) (Chakin et al. JNM
 2009)



Irr. Be, TEM, BF, He bubbles,
 $T_{irr.}=413^\circ\text{C}$, $F = 6.5 \times 10^{21} \text{ cm}^{-2}$
 ($E > 1 \text{ MeV}$) (Klimenkov et al. JNM
 2013)

Implantation of He and H: bubbles can dominate even at RT



S-200-F, H implantation (120keV, RT.
 $2 \times 10^{18} \text{ ions/cm}^2$), (from Kang et al. Journal
 of the Korean Physical Society, 63, 2013)

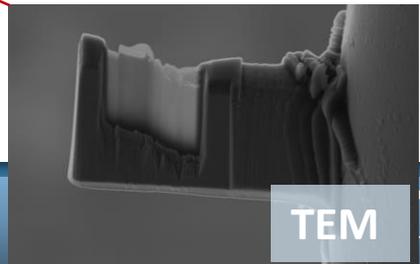
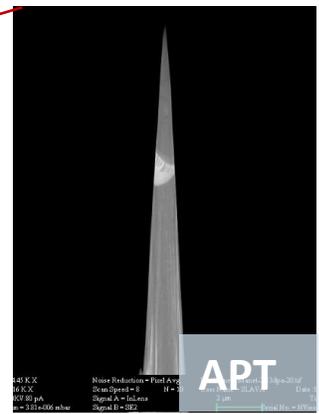
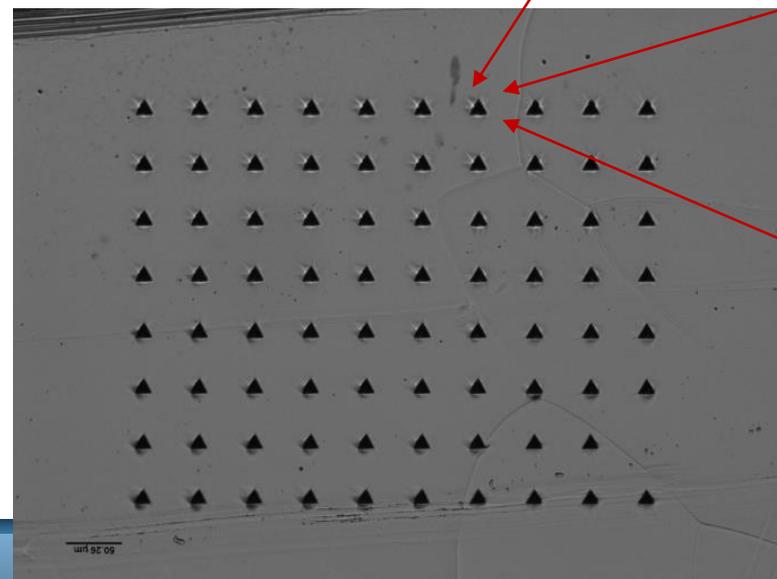
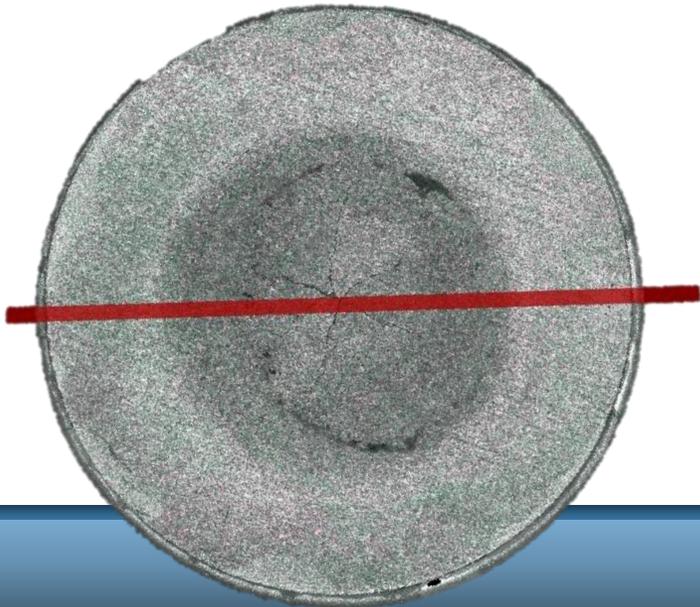
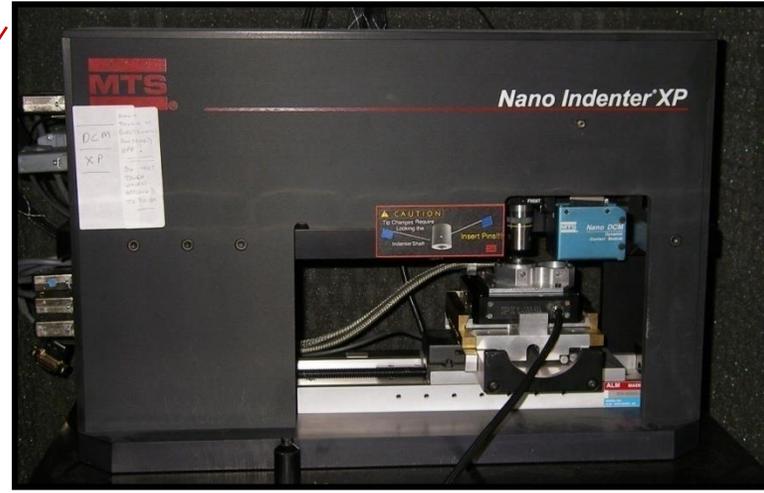
Irradiation Source	He gas production in Be (appm/DPA)
Mixed spectrum fission reactor	10-500
Fusion reactor	550
High energy proton beam (NuMI)	~ 4000

What can we expect from GeV protons?

Nano-hardness measurements:

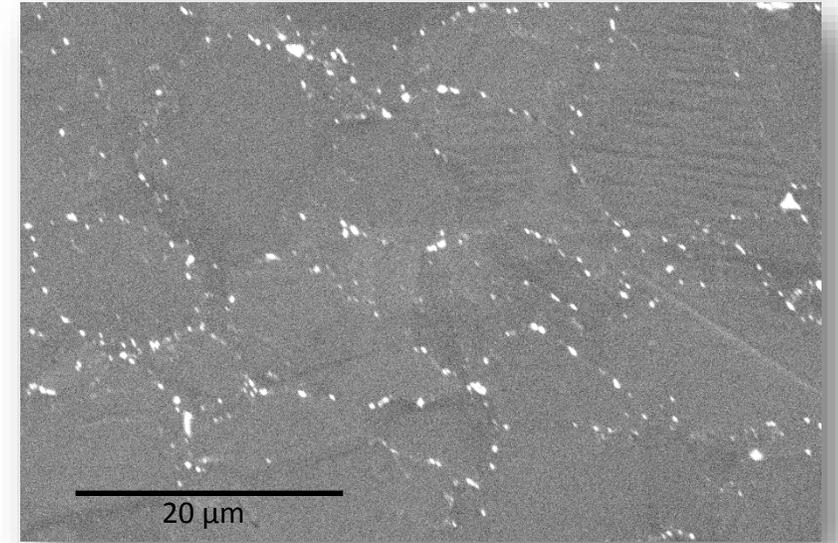
- to estimate the irradiation effect

Local microstructural investigations



Intermediate conclusion

- Industrial beryllium is an impure alloy: relatively pure grains and strongly "alloyed" GB
- Impurities strongly create GB precipitation and segregations, segregate to BeO
- Beryllium matrix has some quantity of homogeneously distributed iron, nickel and copper
- Mechanical properties of beryllium are highly anisotropic
- Proton irradiated beam windows show signs of relatively high swelling



Thank you for your attention!