



# Radiation damage of beryllium: MIAMI irradiation test plans

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Science & Technology Facilities Council  
Rutherford Appleton Laboratory



Collaboration with:

Graeme Greaves and Stephen Donnelly from the University of Huddersfield



Xiaou Yi *University of Oxford*

# What do we study and why?

## Beryllium - Grade PF-60

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

## 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	
<b>Beam window (vacuum to air)</b>	200	300	~ 0.23/yr	>2000	>2500	700 kW; 120 GeV; ~1 Hz; $\sigma_{\text{rms}} = 1.3$ mm
<b>Target</b>	375	450	~ 0.23/yr	>2000	>2500	700 kW; 120 GeV; ~1 Hz; $\sigma_{\text{rms}} = 1.3$ mm

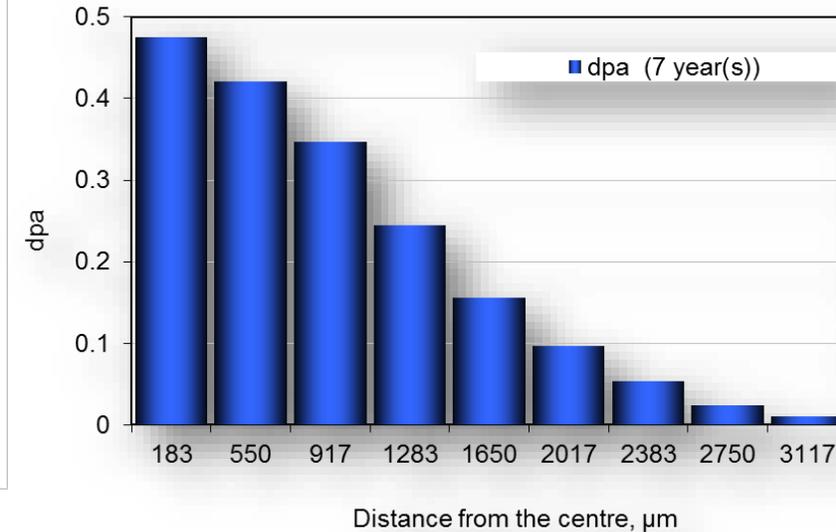
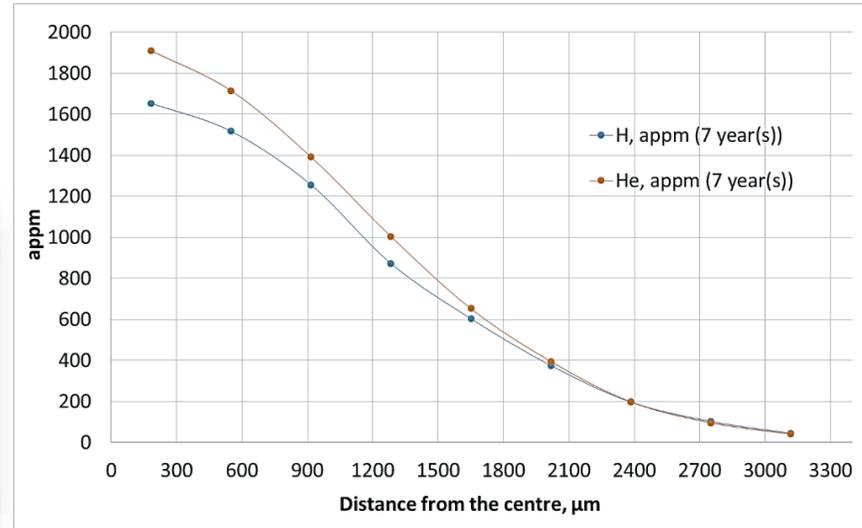
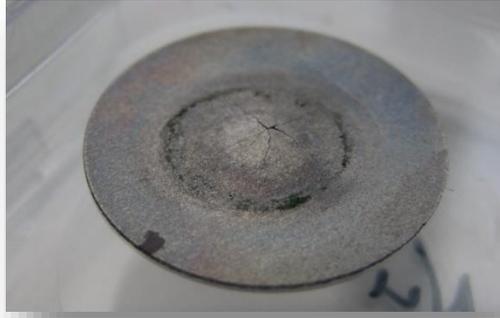
Environment: elevated temperature + radiation + pulsing loads

## 300 kW NuMI beam window

120GeV proton beam

about  $3 \times 10^{13}$  protons per pulse, 0.5 Hz

$T \approx 50^\circ\text{C}$



## Simulation with ion irradiation experiments

### Low energy in-situ irradiation:

- easy variation of irradiation parameters;
- reasonable correspondence of He/dpa ratio.
- observation of evolution of microstructure structure vs dose/appm;

### High-energy irradiation + PIE

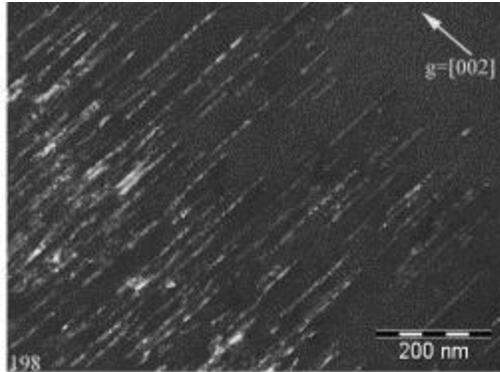
- microstructural and micromechanical tests data will be available

But: **validity of the simulation should be confirmed**

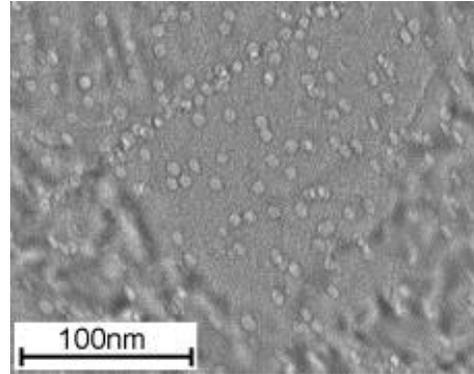
# Be under irradiation

## n-irradiation:

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

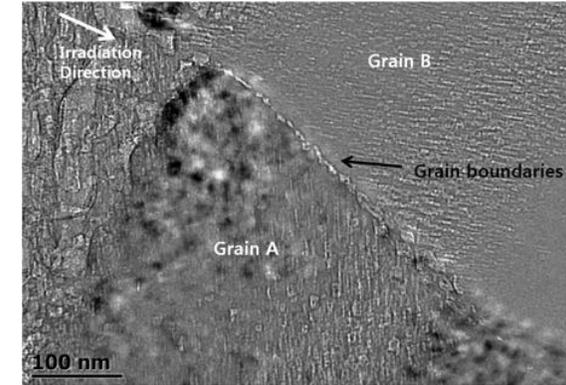


Irr. Be, TEM, DF, dislocation loops,  
 $T_{irr}=70^\circ\text{C}$ ,  $F = 6 \times 10^{22} \text{ cm}^{-2}$   
 $(E > 0.1 \text{ MeV})$  (Chakin et al. JNM 2009)



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)	$\sim 4000$
Ion implantation	$\sim 35000 \dots 50000$

*What can we expect from GeV protons?*

## Consequences of supersaturation of defects:

1. Enhancement of phase transformation

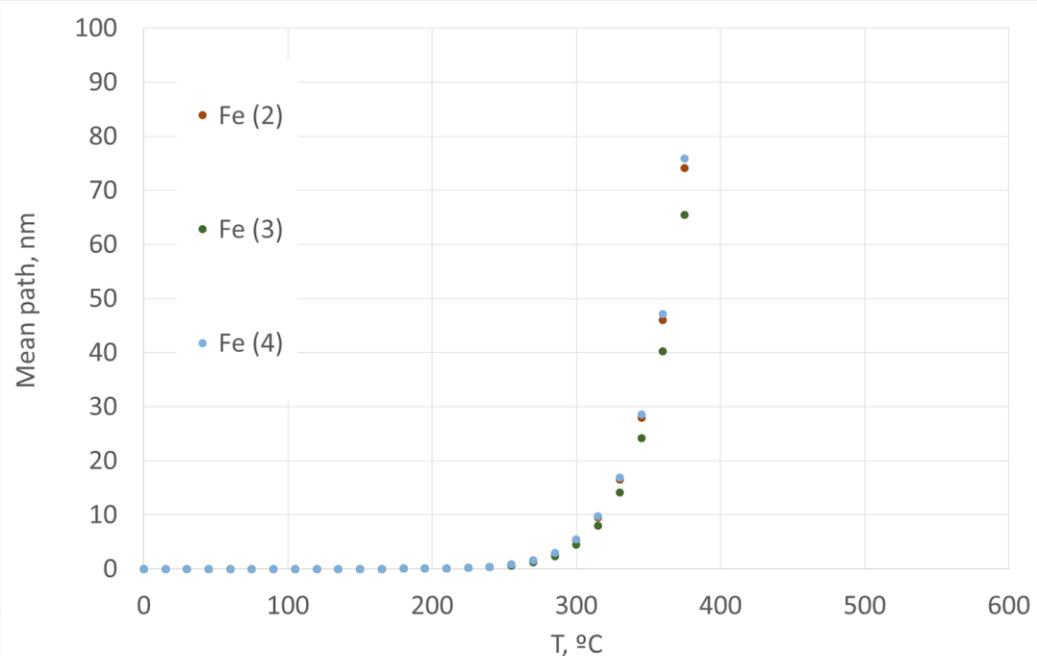
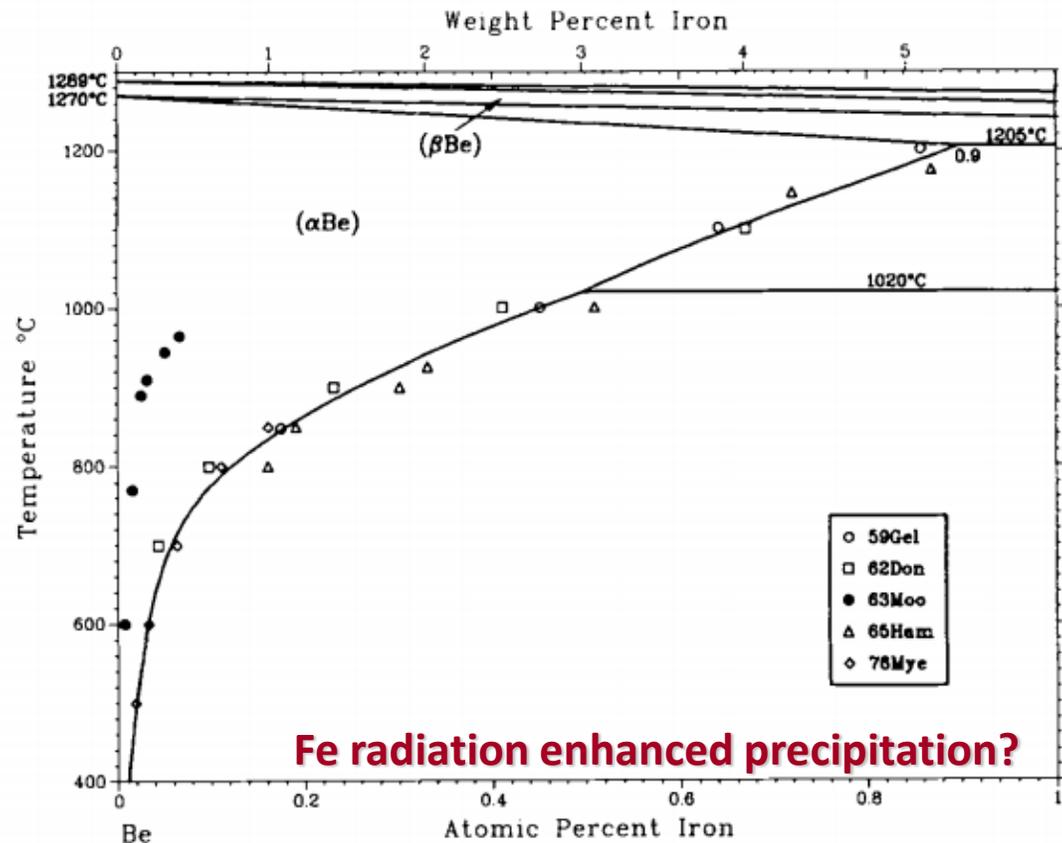
$$D^* = \alpha_V D_V C_V^* + \alpha_x D_x C_x^* \quad C_V^* > C_V^T$$

*X - self interstitial atom; clusters of point defects*

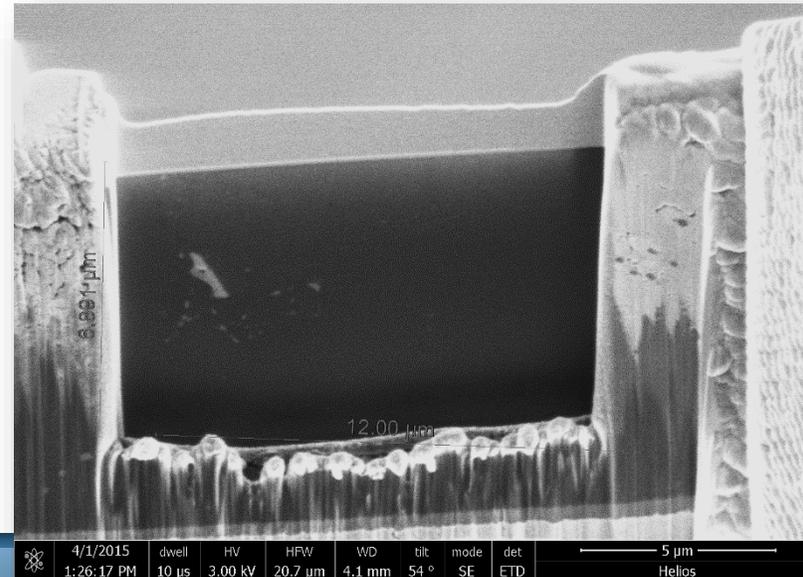
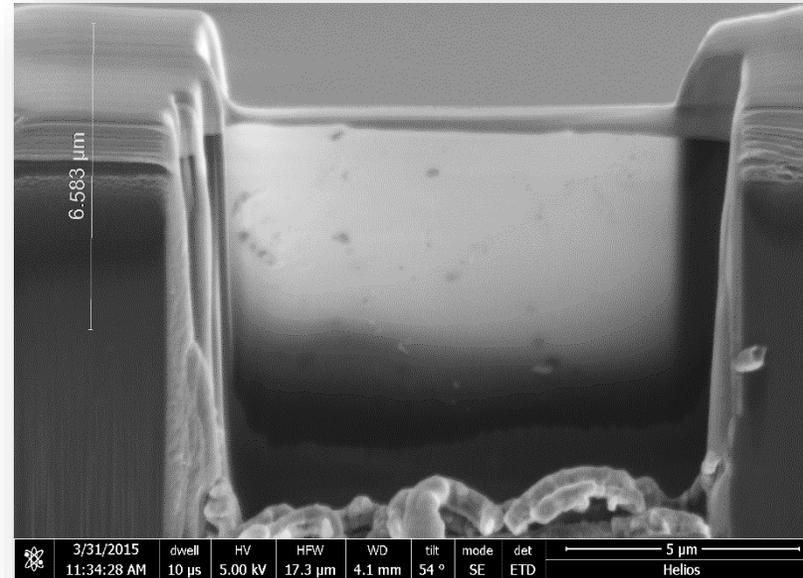
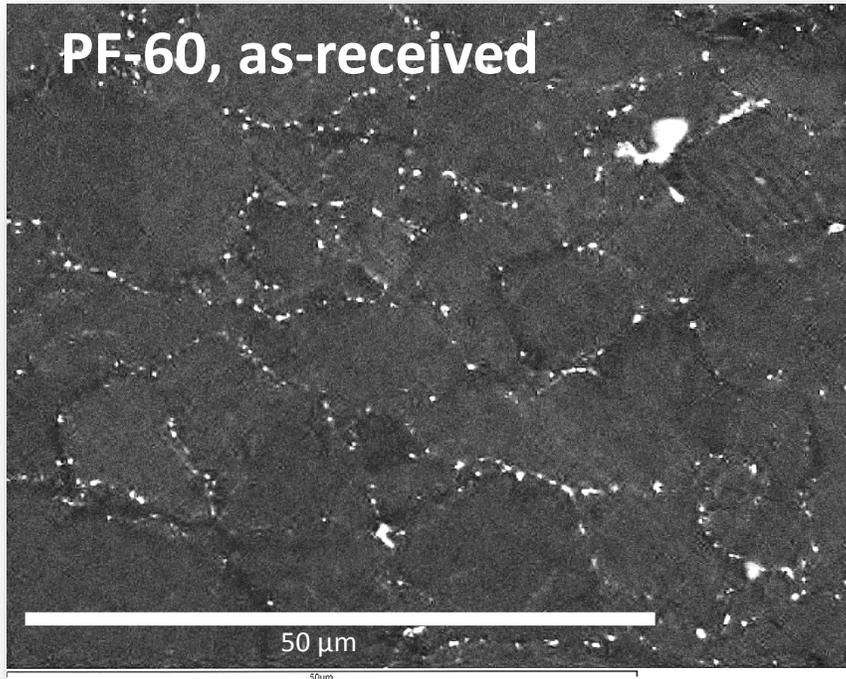
### Impurities in Beryllium matrix (preliminary)

Fe, appm	Cu, appm	Ni, appm	O, appm
520±10	80±10	140±10	330±10

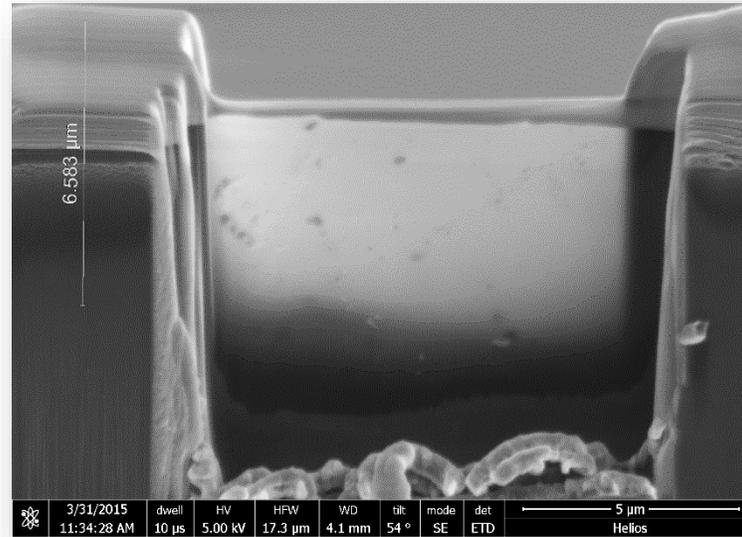
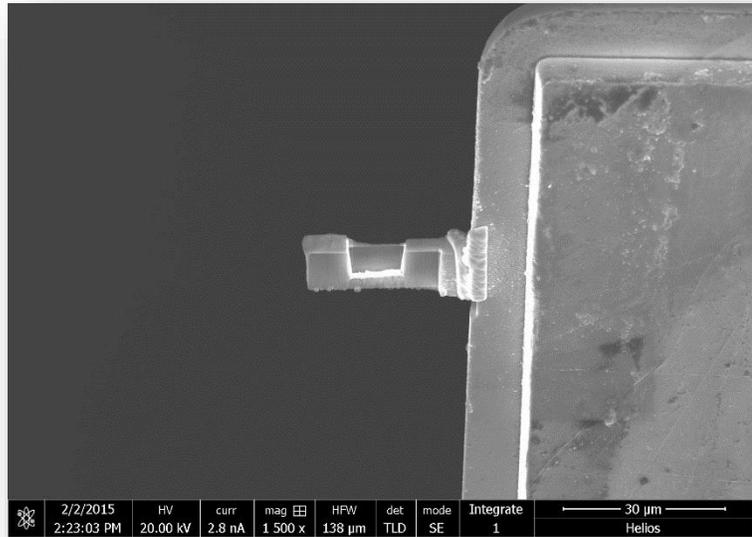
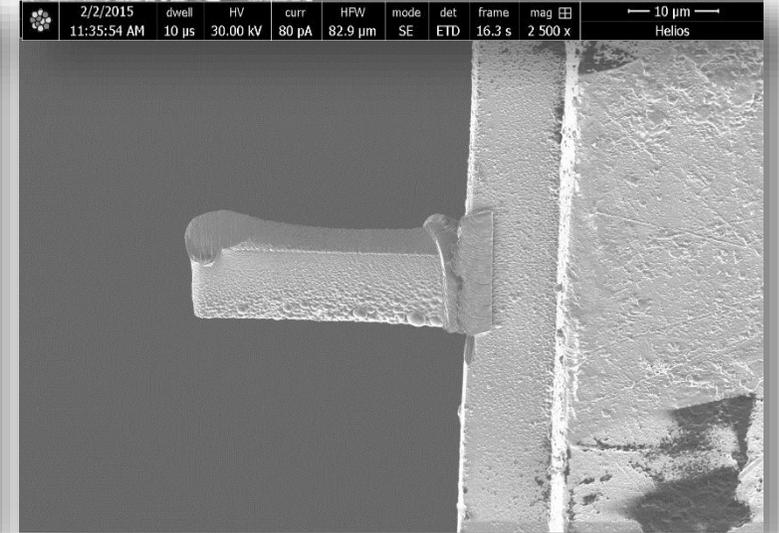
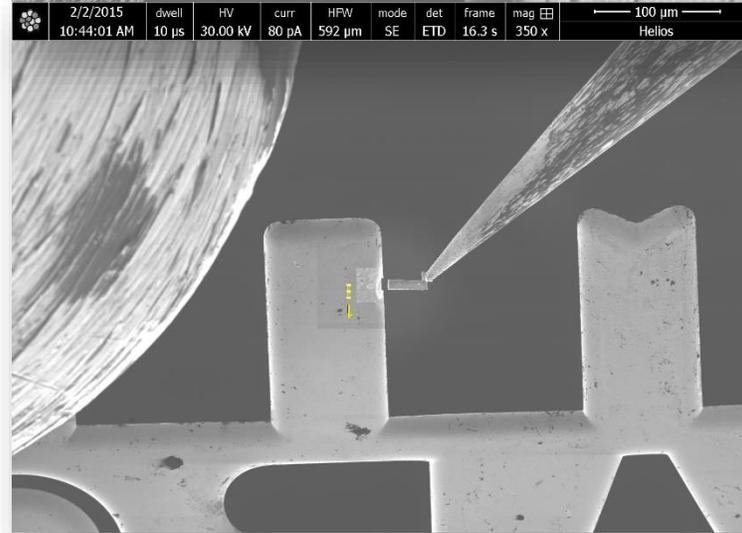
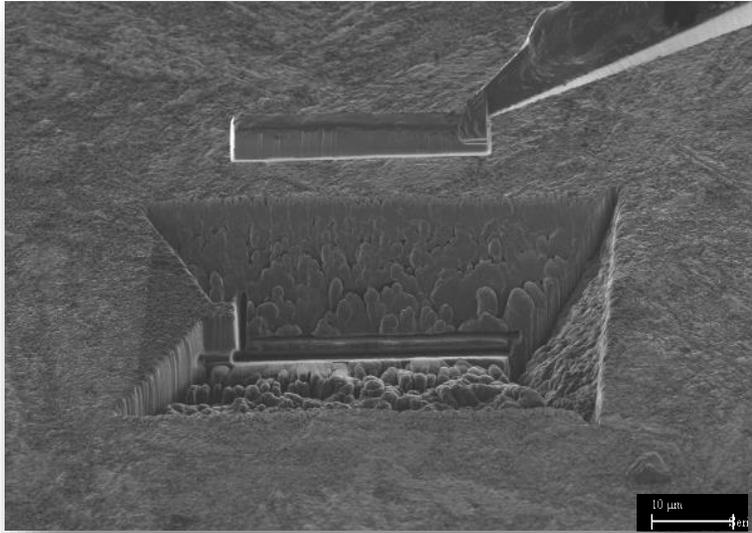
(αBe) Solvus Boundary of the Be-Fe Assessed Phase Diagram



# Samples preparation:



# preparation of TEM samples

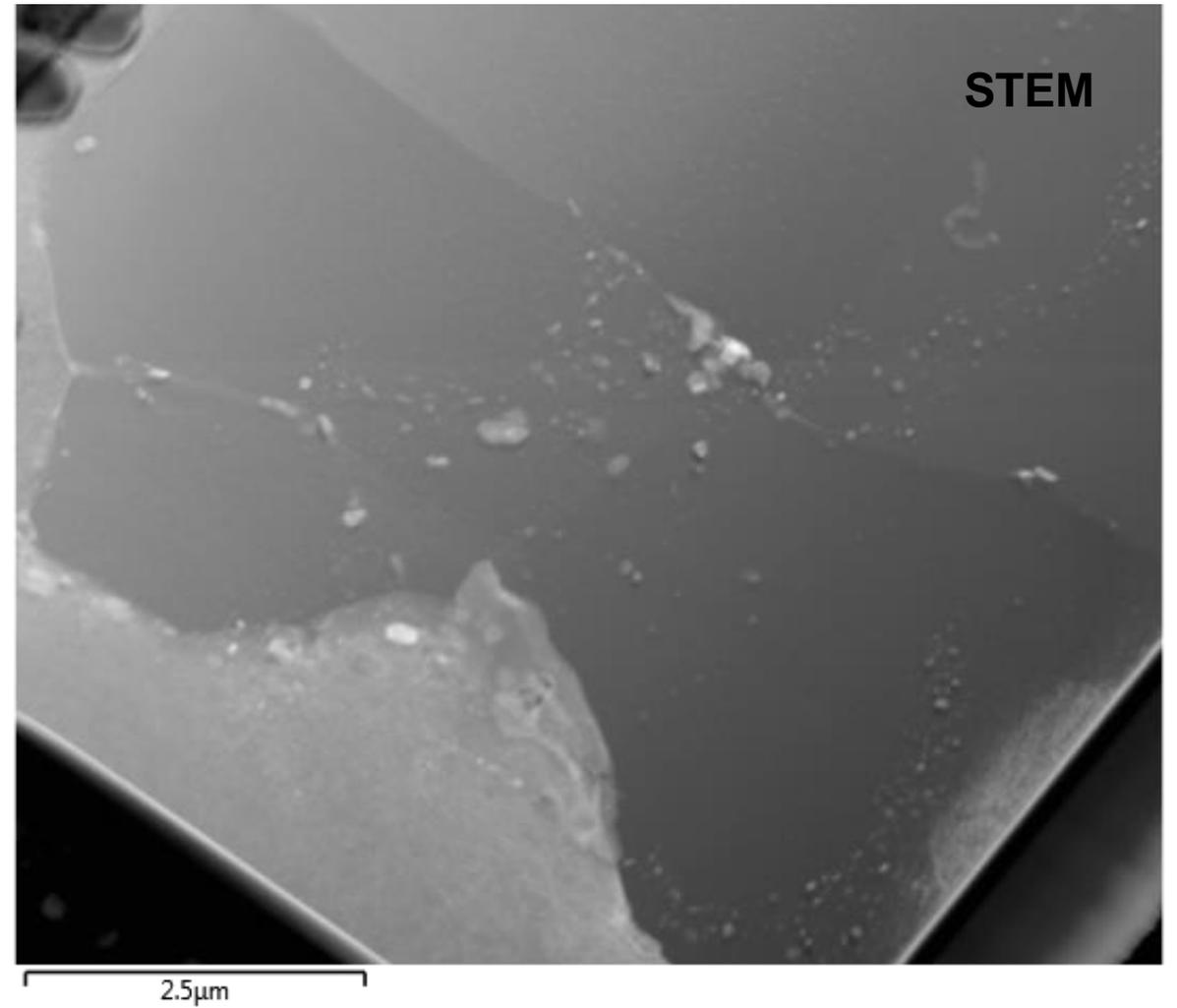
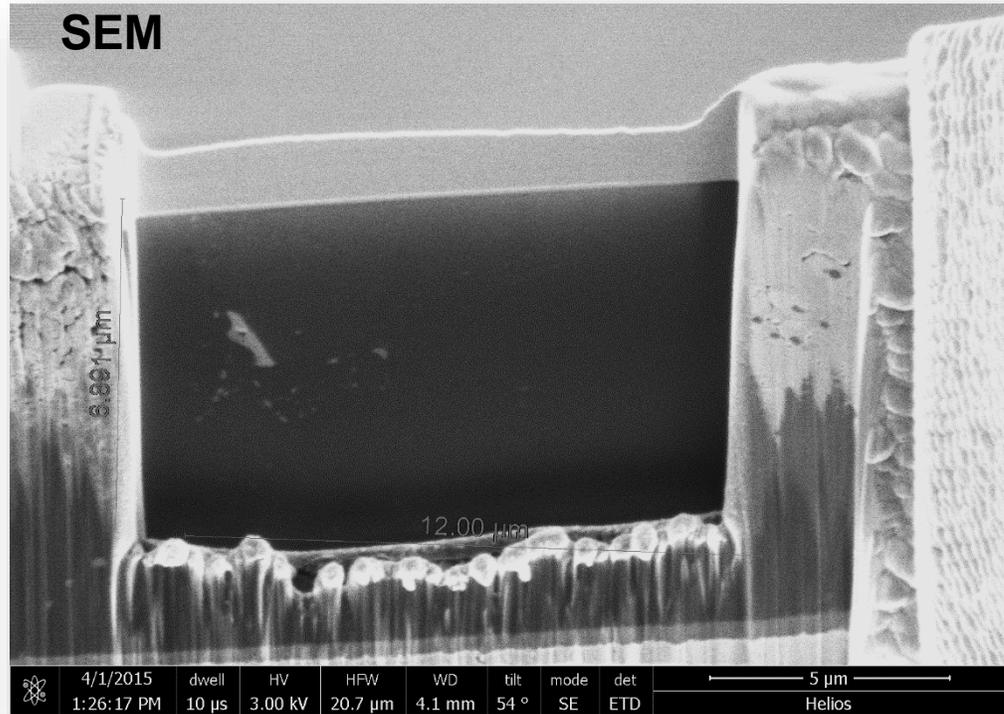


TEM lamella:

Size ~  $10 \times 7 \mu\text{m}$

Thickness ~ 100 nm

# Pre-examination of TEM samples

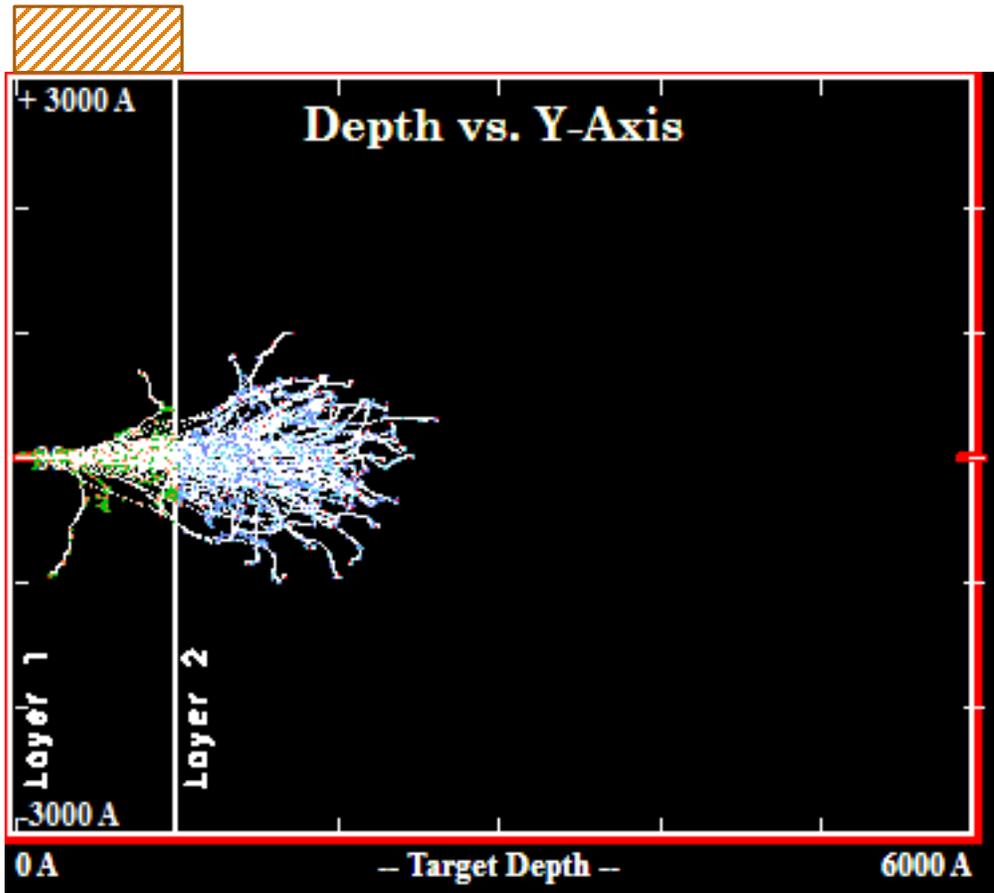


**Microscope and Ion Accelerator for Materials Investigations facility  
(MIAMI) University of Huddersfield , UK**



**Ions: He<sup>+</sup>**  
**Beam energy: ~ 18keV**  
**Temperature: 50°Cc**

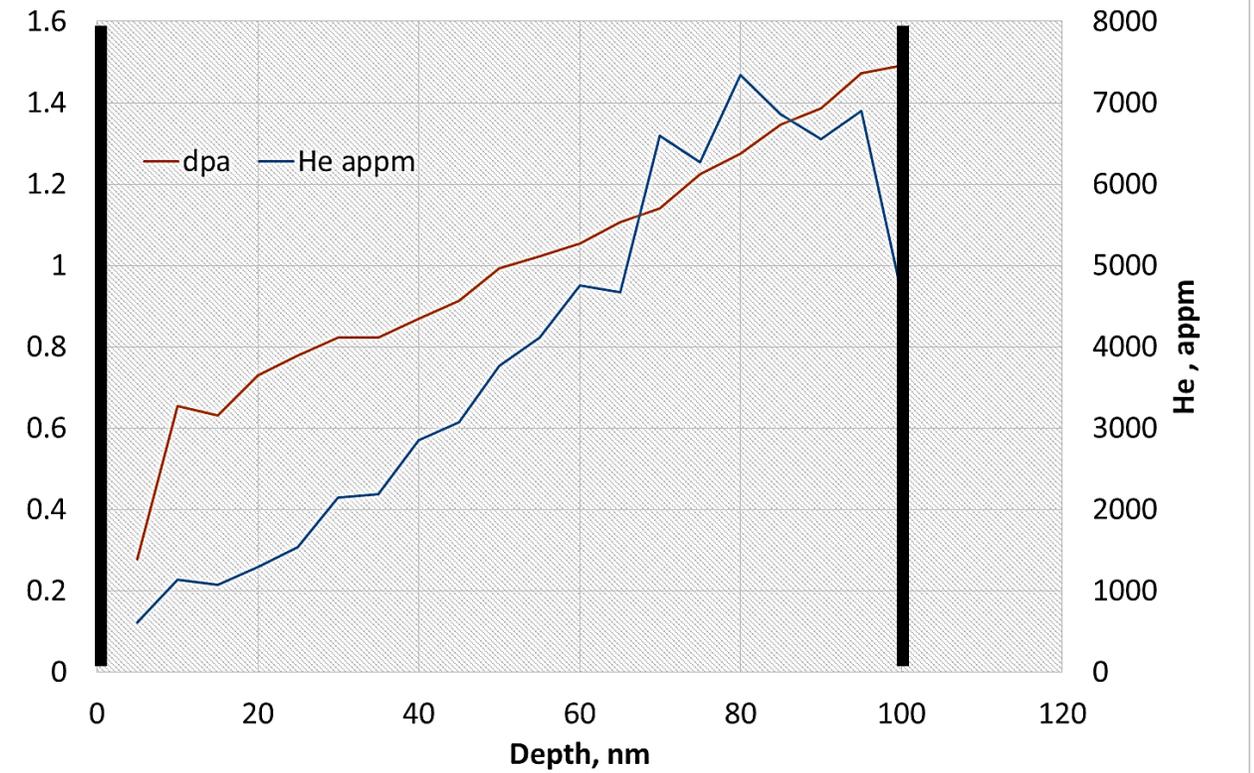
TEM foil



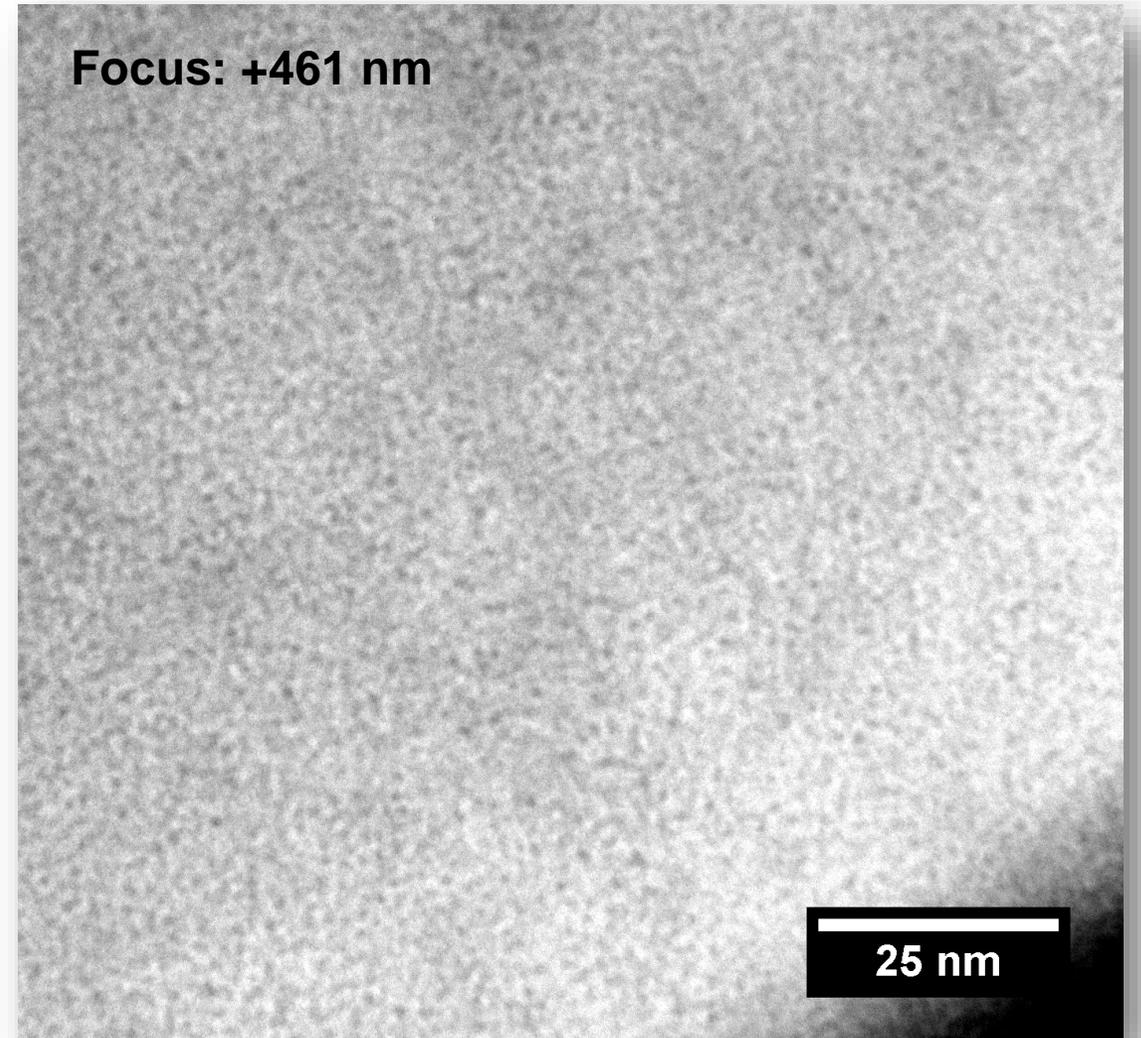
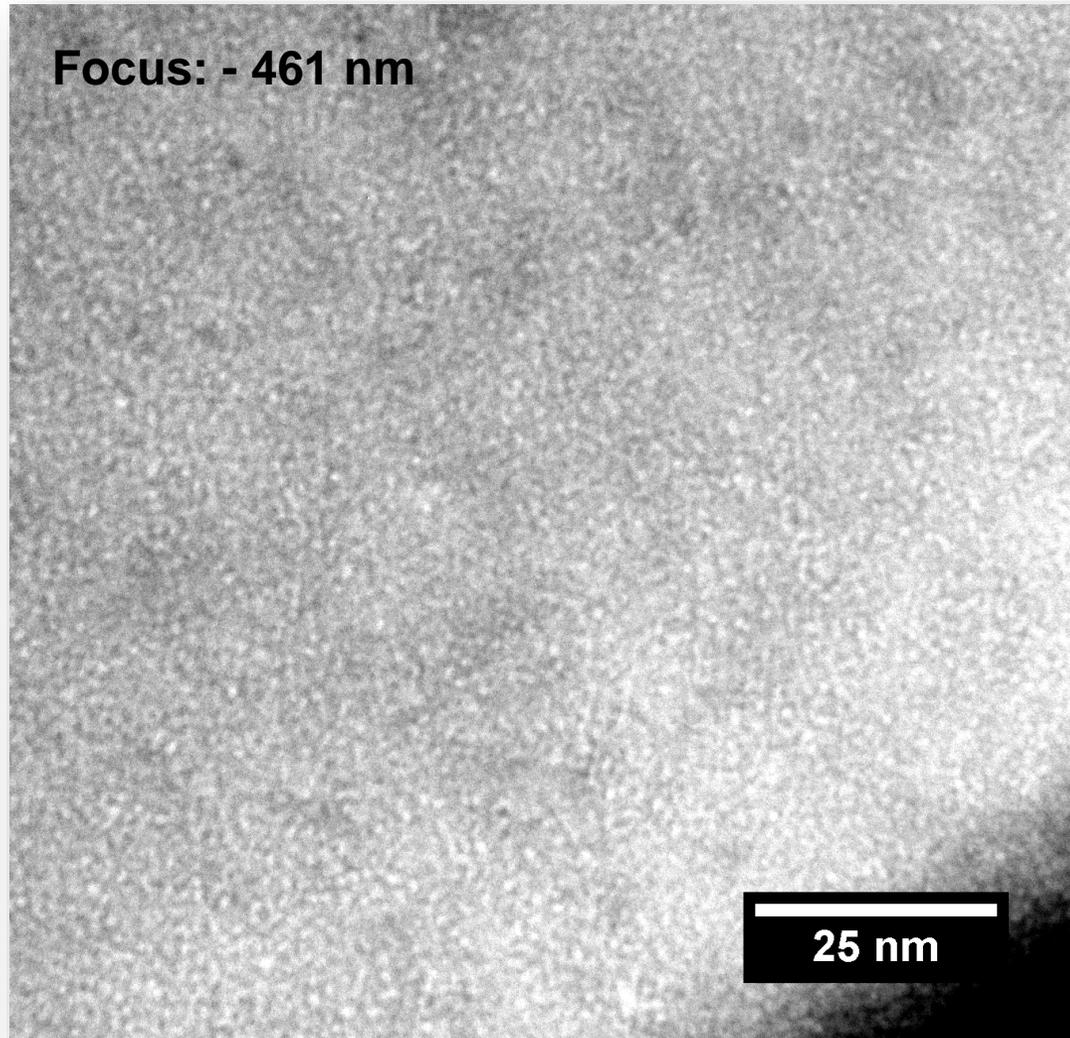
Ions: He<sup>+</sup>

Beam energy: ~ 18keV

Mean He/dpa = 4000



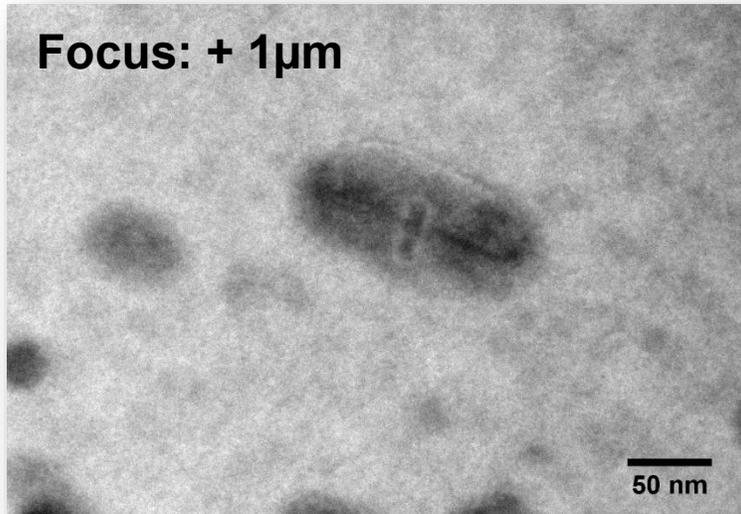
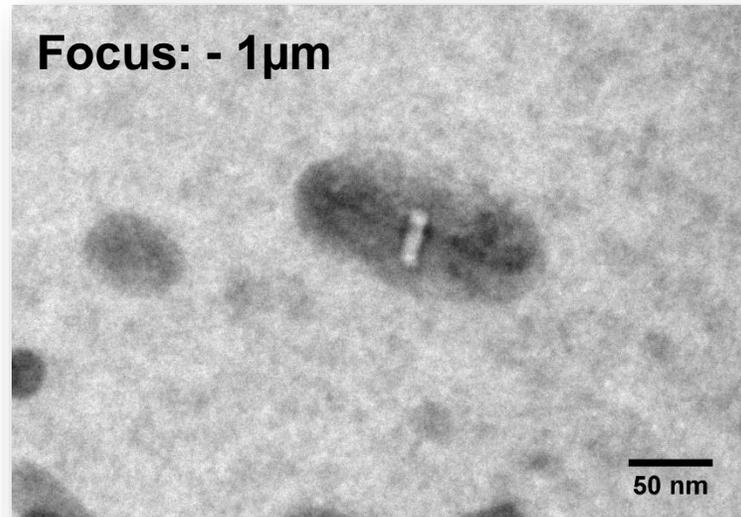
# Defocus contrast (preliminary)



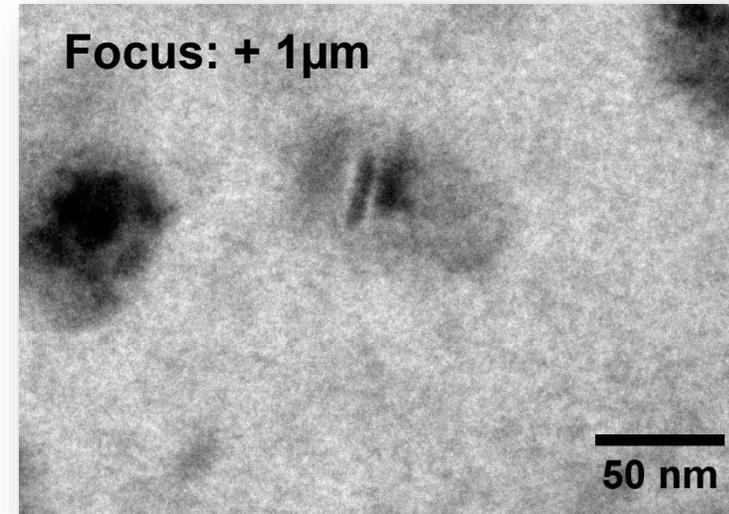
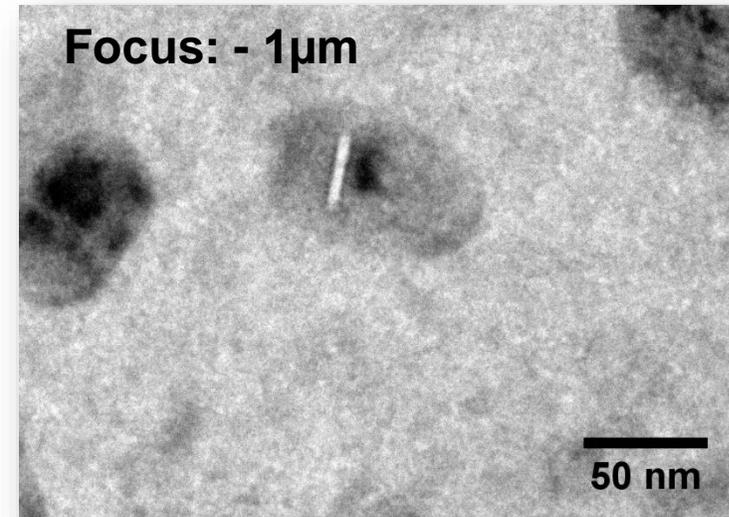
Size: up to 1...2 nm  
ND ~  $5 \cdot 10^{23}$  ...  $1 \cdot 10^{24} \text{ m}^{-3}$

# Defocus contrast (preliminary)

6000 appm / 1.5dpa



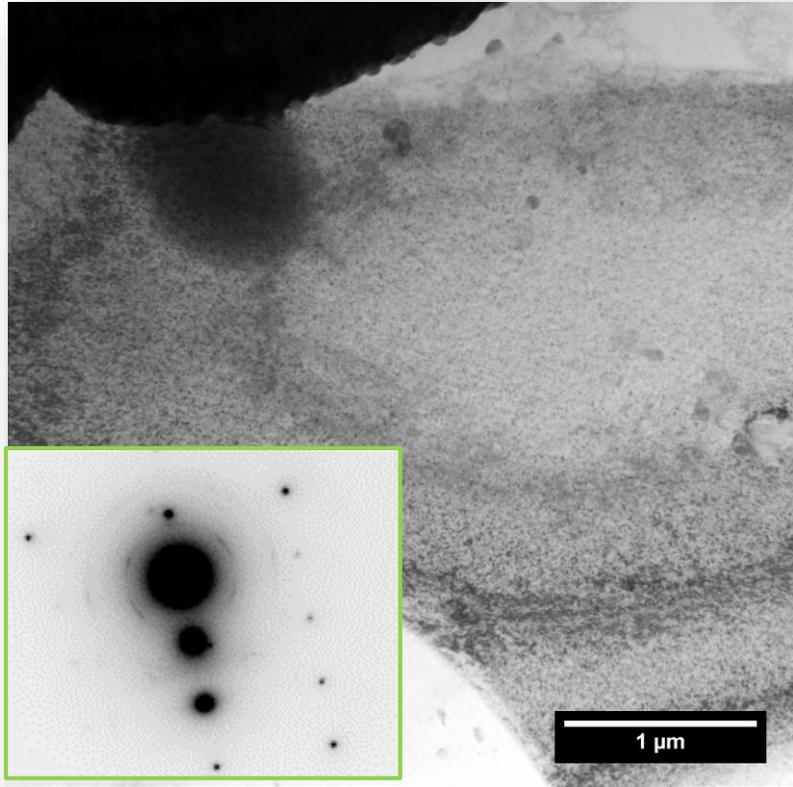
8000 appm / 2dpa



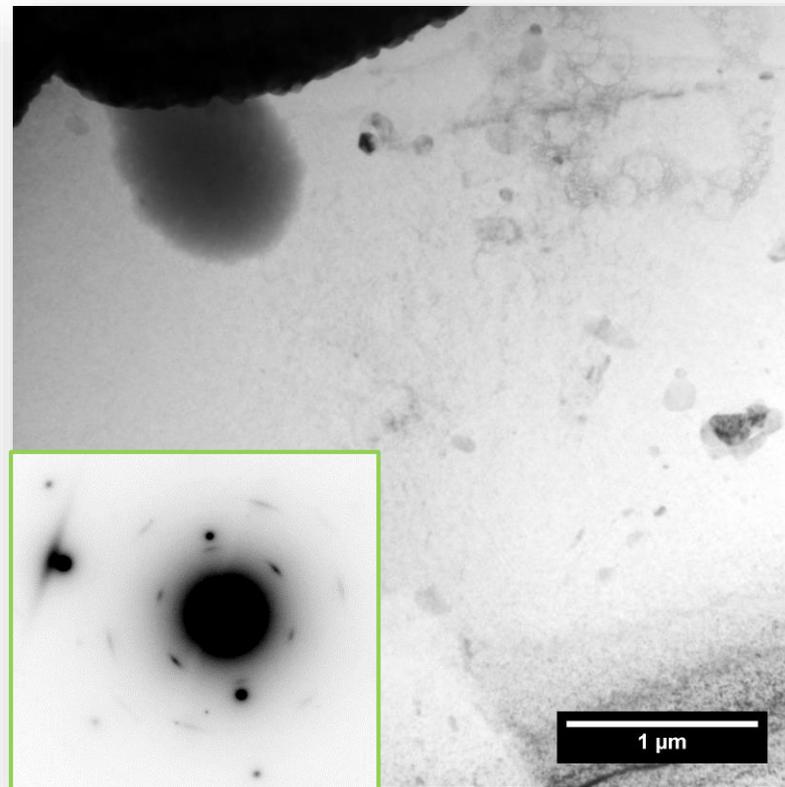
# Dislocation loops (preliminary)

PF60.

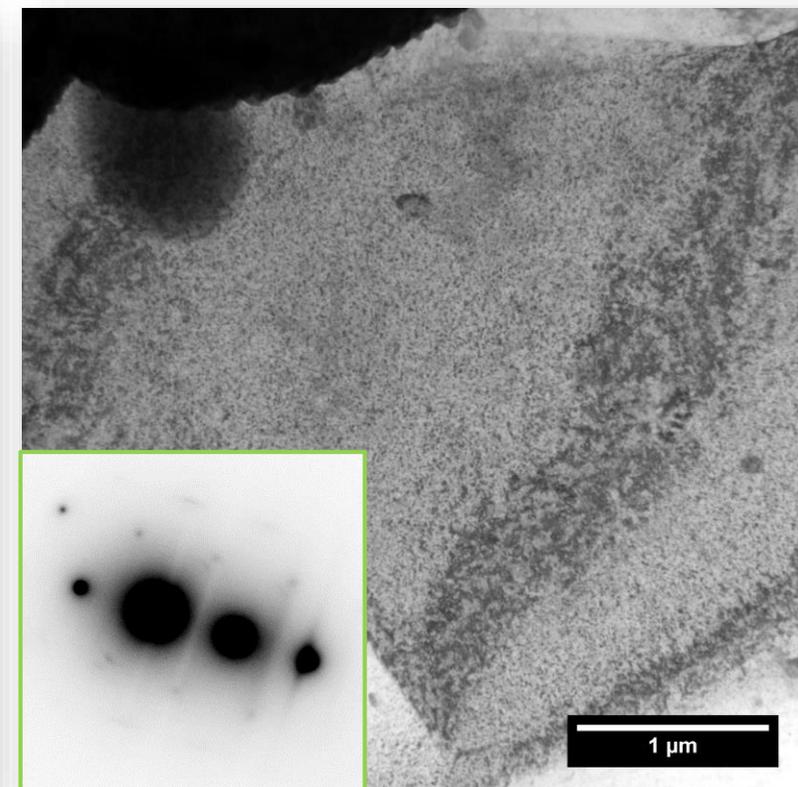
1.5dpa/6000appm of He, T=50°C



$g = [0\ 1\ -1\ 0]$



$g = [-2\ 2\ 0\ 1]$

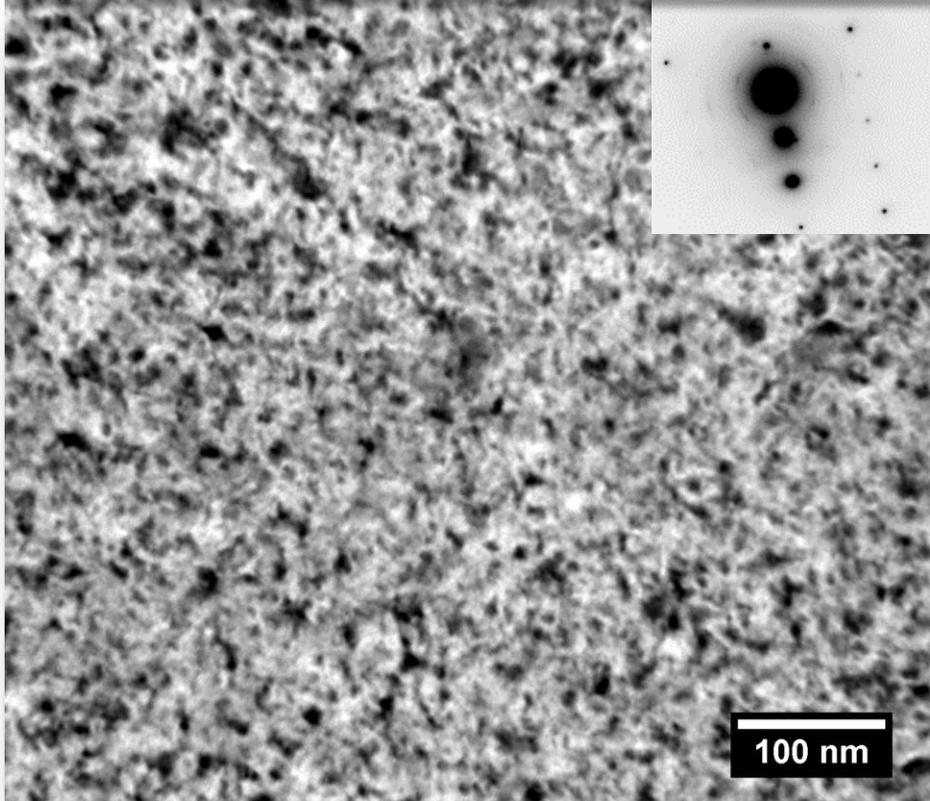


$g = [1\ -1\ 0\ 0]$

# Dislocation loops (preliminary)

PF60.

1.5dpa/6000appm of He, T=50°C



**Preliminary, only one lamella, 1.5 dpa**

**Size: up to 10 nm**

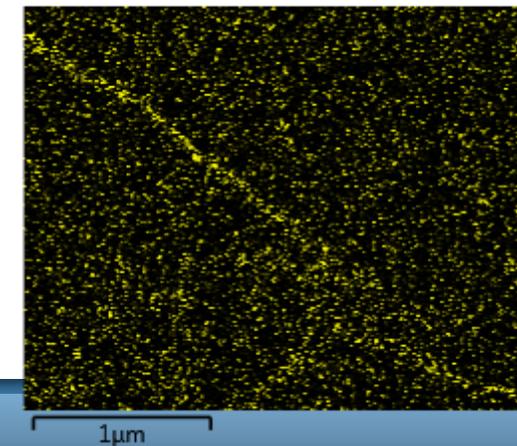
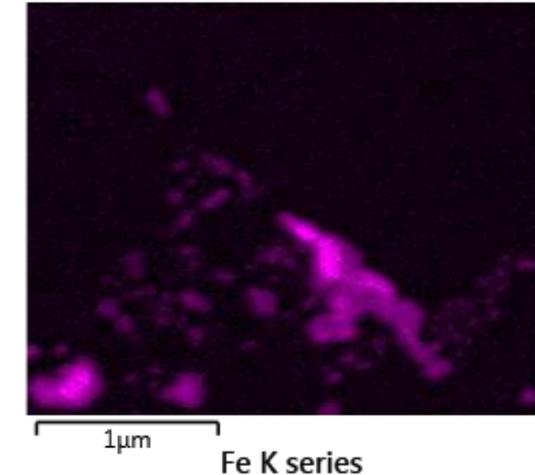
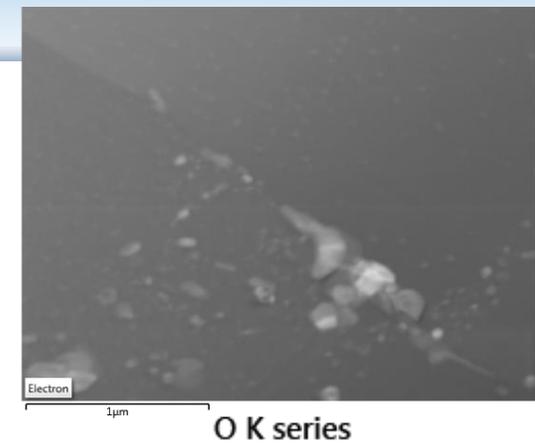
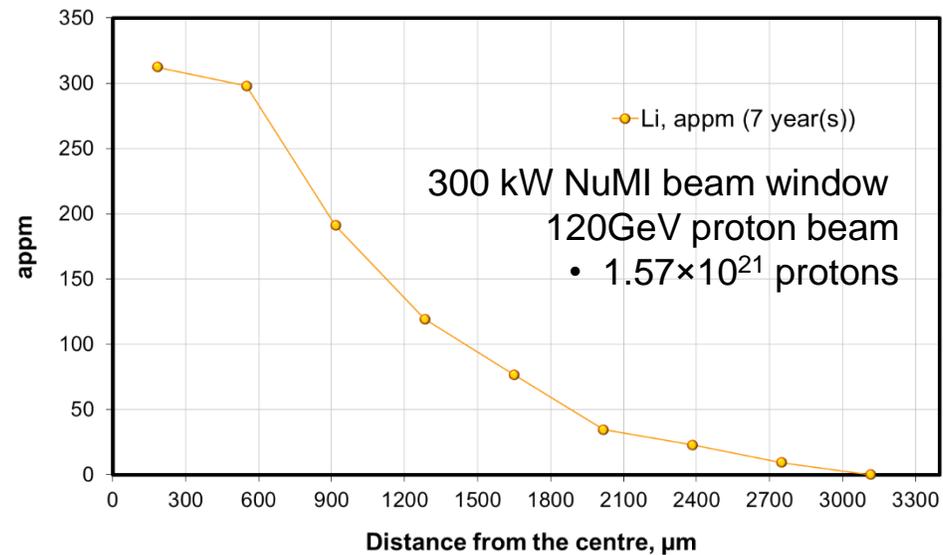
**ND  $\sim 5 \cdot 10^{23} \text{ m}^{-3}$**

**Dislocation density  $\sim 5 \cdot 10^{16} \text{ m}^{-2}$**

**$b = 1/3 \langle 1 \ 1 \ -2 \ 0 \rangle$**

## Future plans:

- Finish the ex-situ analysis (dislocations, bubbles, precipitates)
- Repeat 50°C irradiation up to 0.5 dpa (NuMI conditions)
- Perform irradiations at 200°C (future LBNF conditions)
- H and Li irradiations
- High-energy implantation



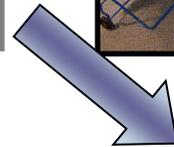
**Surrey Ion Beam Centre, UK**  
(collaboration with Prof. R.Gwilliam)

**Ions:** He<sup>+</sup>

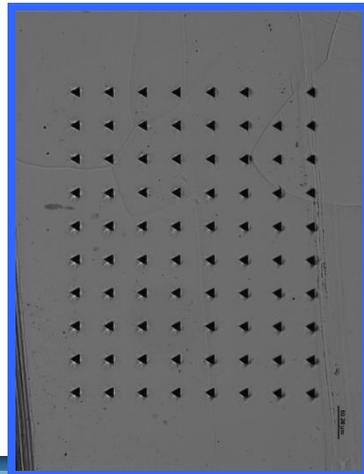
**Maximum beam energy:** 2 MeV => 7.5μm  
implantation depth (SRIM)

**Dose:** up to 1 dpa

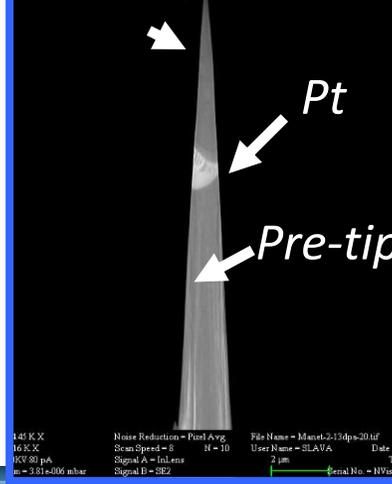
**Temperature:** 200°C (100°C, 500°C)



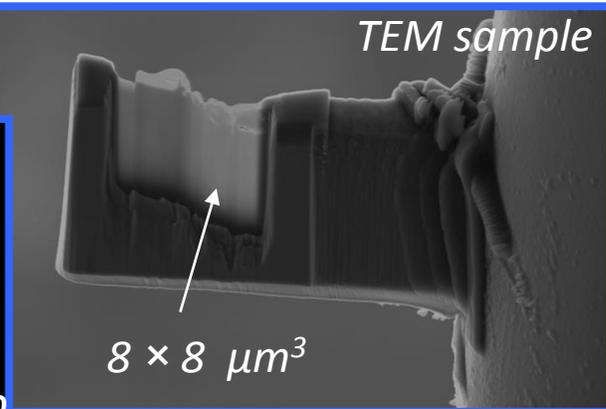
*Micromechanical tests*



*APT sample*

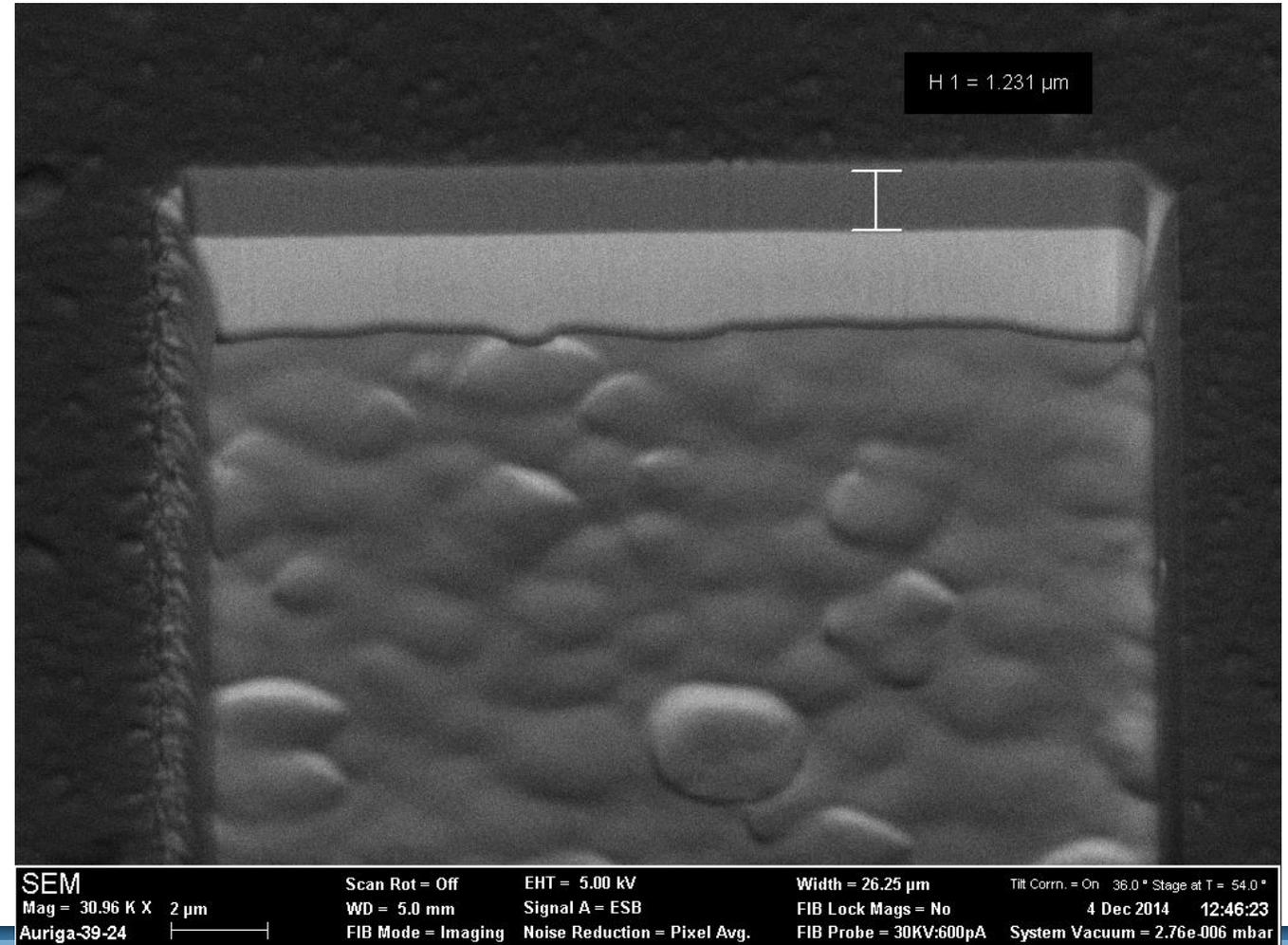


*TEM sample*



# Issues:

## Al coating – how to remove then?

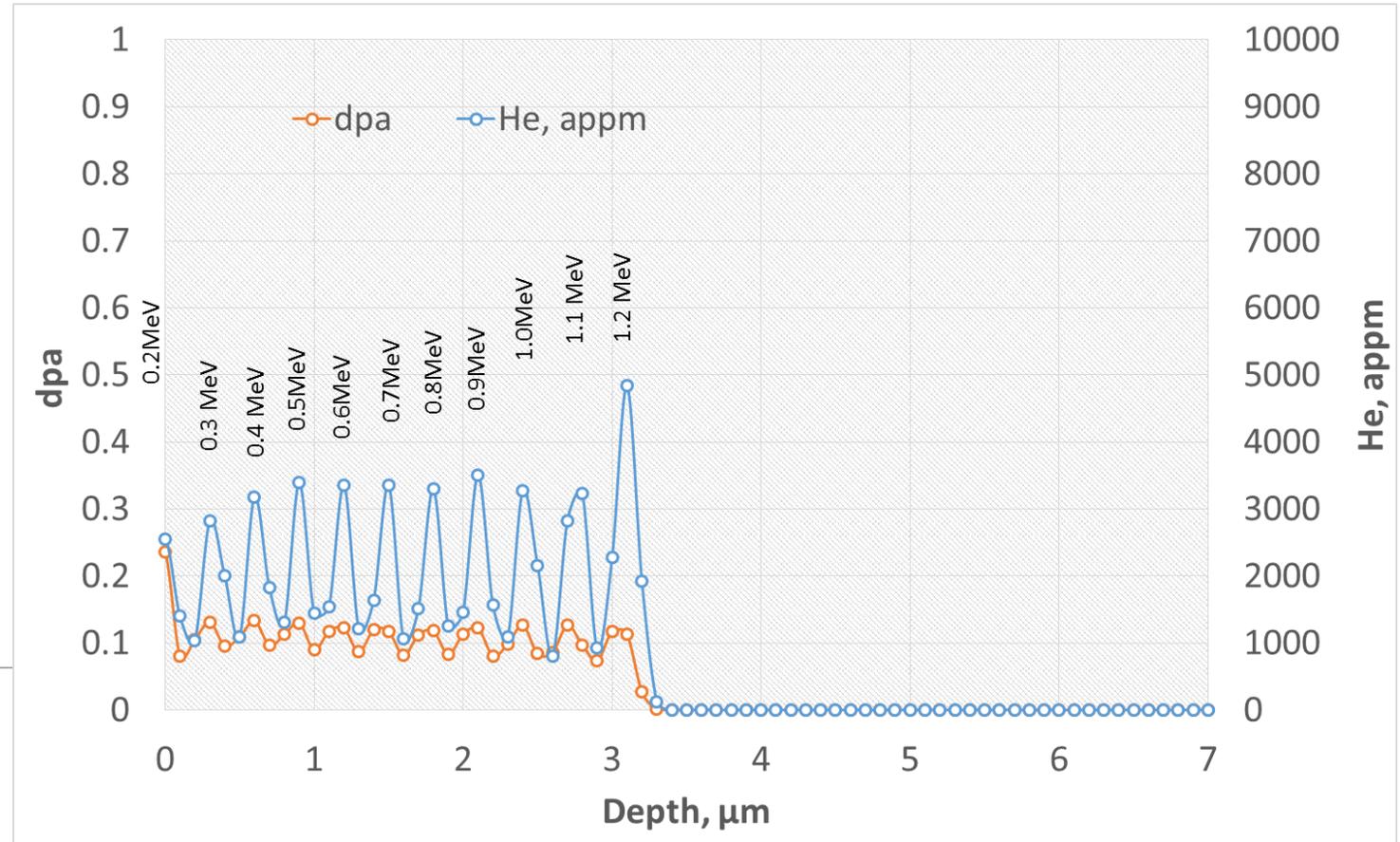


# Issues:

## He implantation in Be through Al degrader (1 $\mu$ m), high energy implantation

15000 appm/dpa

Ed(Al) = 25eV  
Ed(Be) = 31 eV



Steps	1	2	3	4	5	6	7	8	9	10	11
Energy, MeV	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1	1.1	1.2
He ions/cm <sup>2</sup> , $\times 10^{16}$	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.83	0.83	0.90	1.20

Thank you for your attention!