

Characterization of radiation damage effects in high-energy neutrino target graphite using low-energy ion irradiation

Abe Burleigh

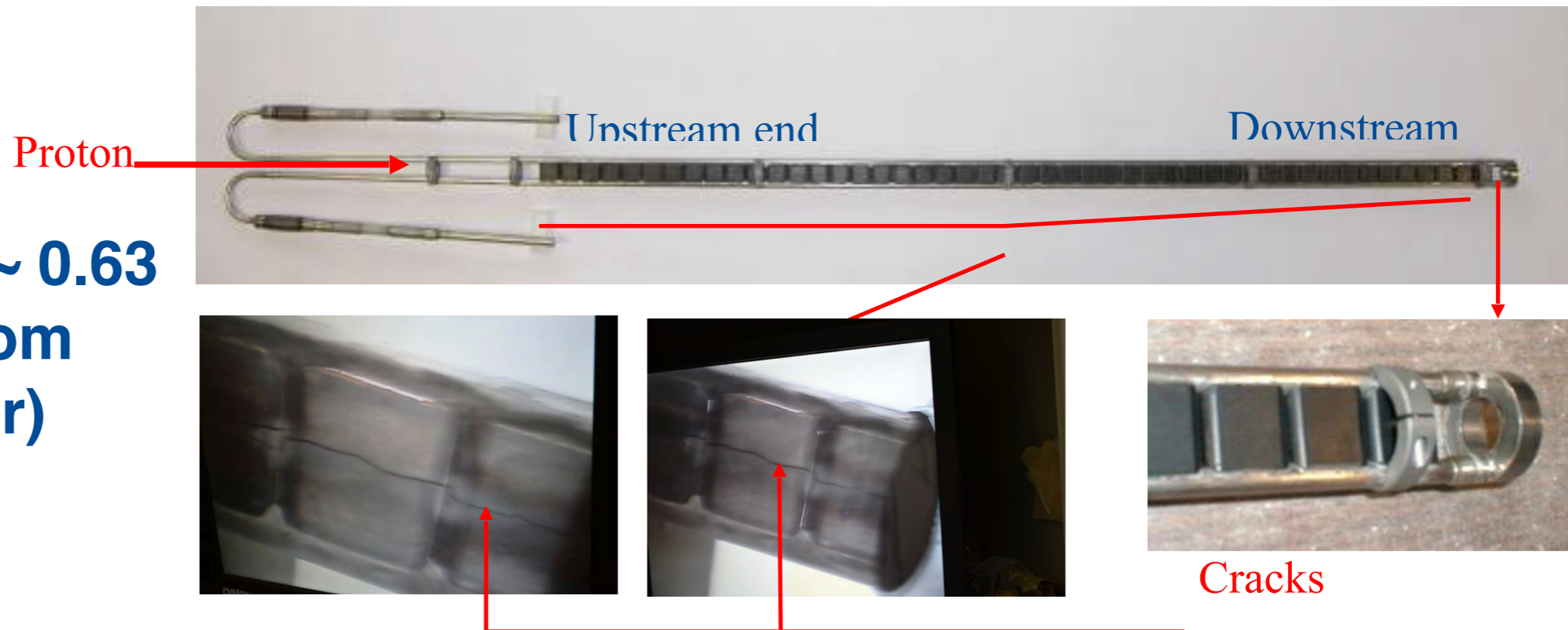
Illinois Institute of Technology

Fermi National Accelerator Laboratory

Motivation

- Determination of the effects of increasing beam power and fluence on target materials is essential to avoid component failure.
- High energy proton irradiation presents many difficulties: sample activation, high costs, long irradiation times, rigorous planning and sample preparation.
- Low-energy ion irradiations suggest a method of testing new beam designs without the costly and time consuming high-energy irradiations commonly undertaken while also allowing for faster experiment iteration without sample activation.

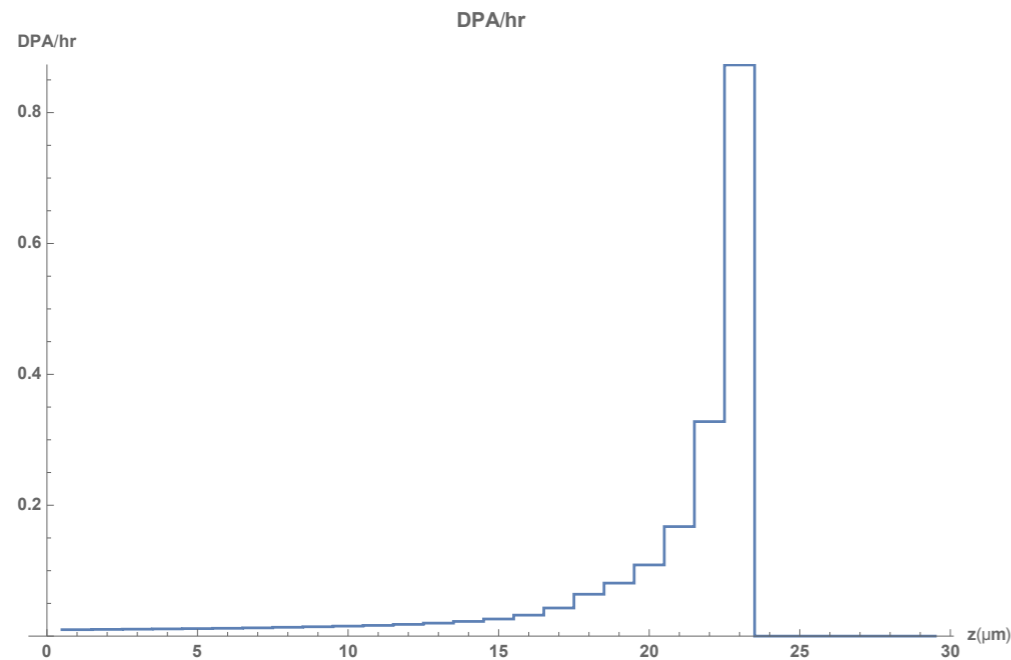
NT-02 Target:
Estimated DPA ~ 0.63
(More on this from
Sujit Bidhar later)



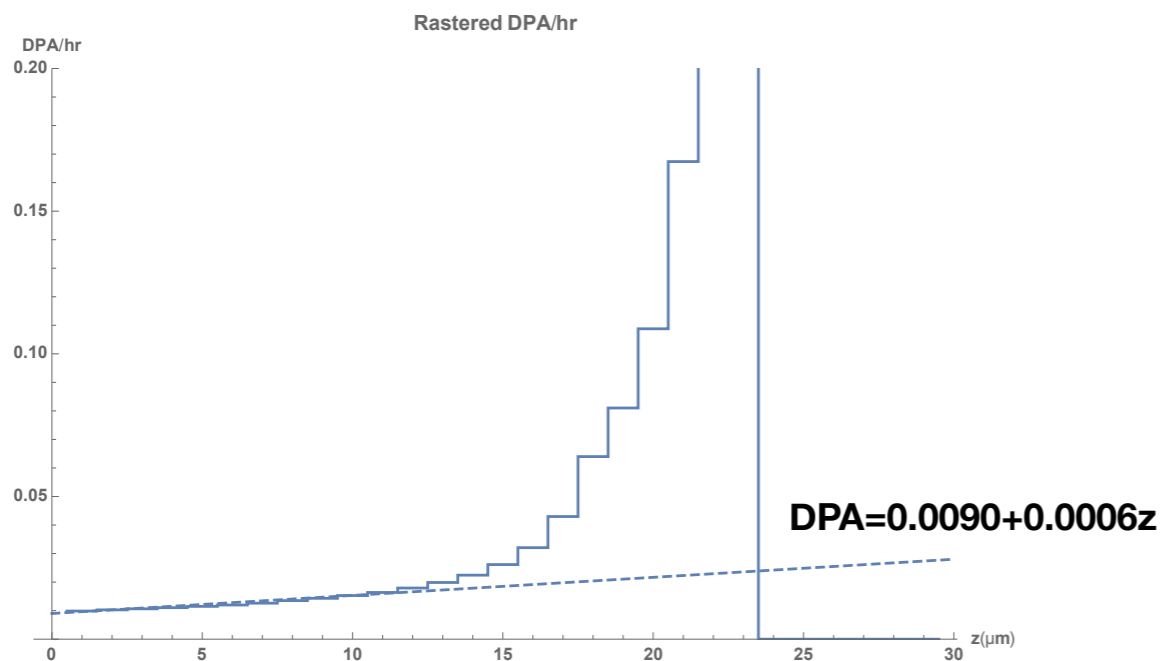
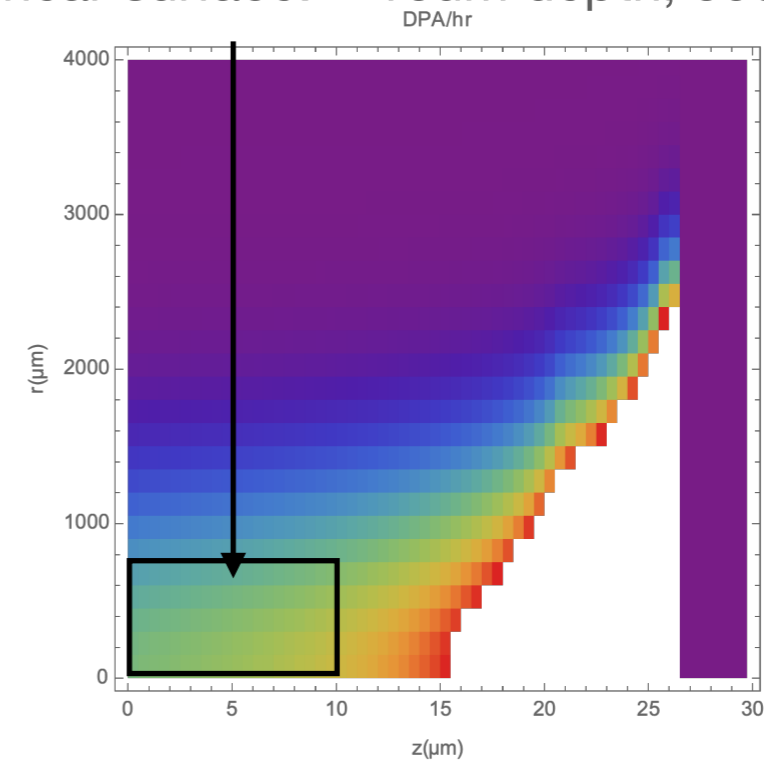
Ion irradiation advantages and limitations

4.5MeV He⁺⁺

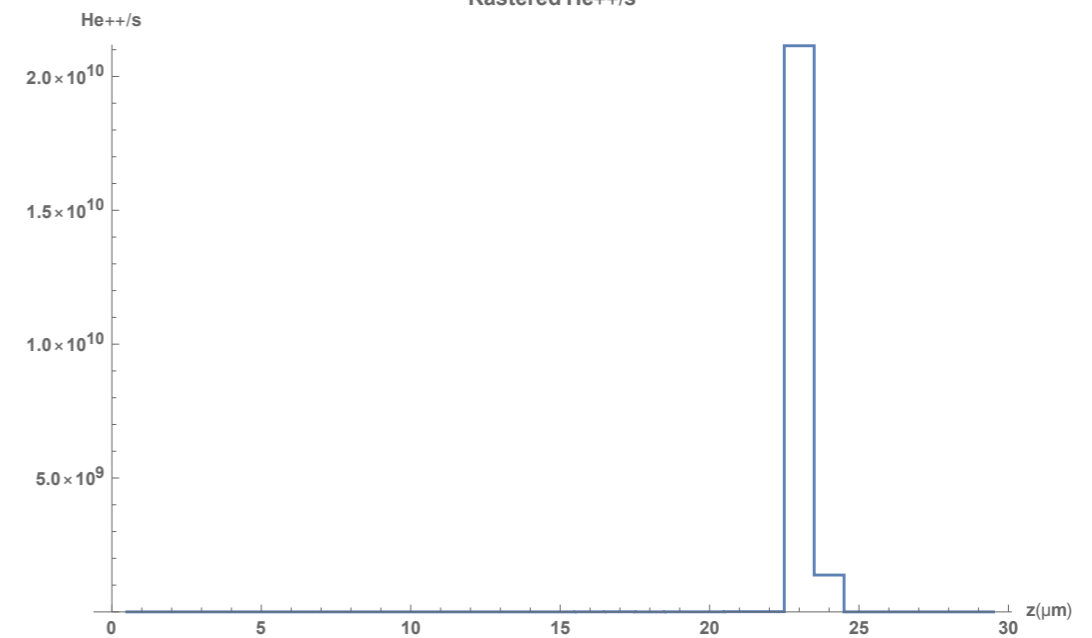
Very small irradiated volumes compared to HE protons:
linearity near surface: ~ 10μm depth, 800μm radius



High peak DPA achievable in short times



Highly linear DPA rate near the surface



Near zero ions come to rest near the surface

HE vs LE irradiations

HE protons

- \$420/DPA/mm³
- Very long irradiation times (months).
- BLIP: 0.11 DPA/55days
- Rigorous sample preparation and testing: many people/resources required
- Sample activation requires special shipping & handling
- Hot cell PIE work

LE ions

- \$20000/DPA/mm³
- Short irradiations (days)
- MIBL trial: 1 DPA/90hrs
- Simple and fast sample preparation
- Transport samples in my backpack on Amtrak
- PIE work at standard laboratories

Trial irradiation objectives

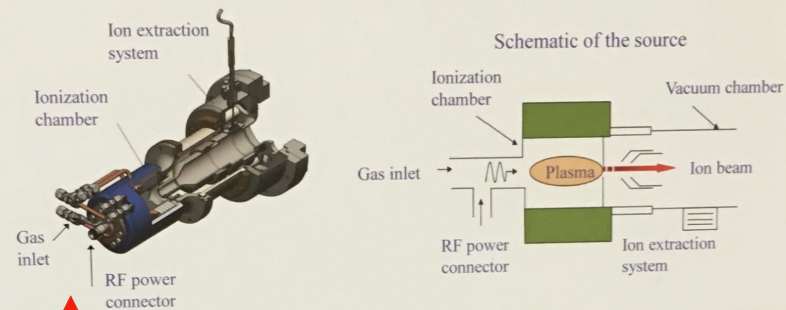
- Attain 3 ion irradiated samples across a range of DPA values corresponding to data from existing or upcoming high energy studies for preliminary characterization of damage effects.
- Familiarization with ion lab capabilities and procedures: sample mounting, temperature control, beam profiling and monitoring, measurement of current on target.



Implementation

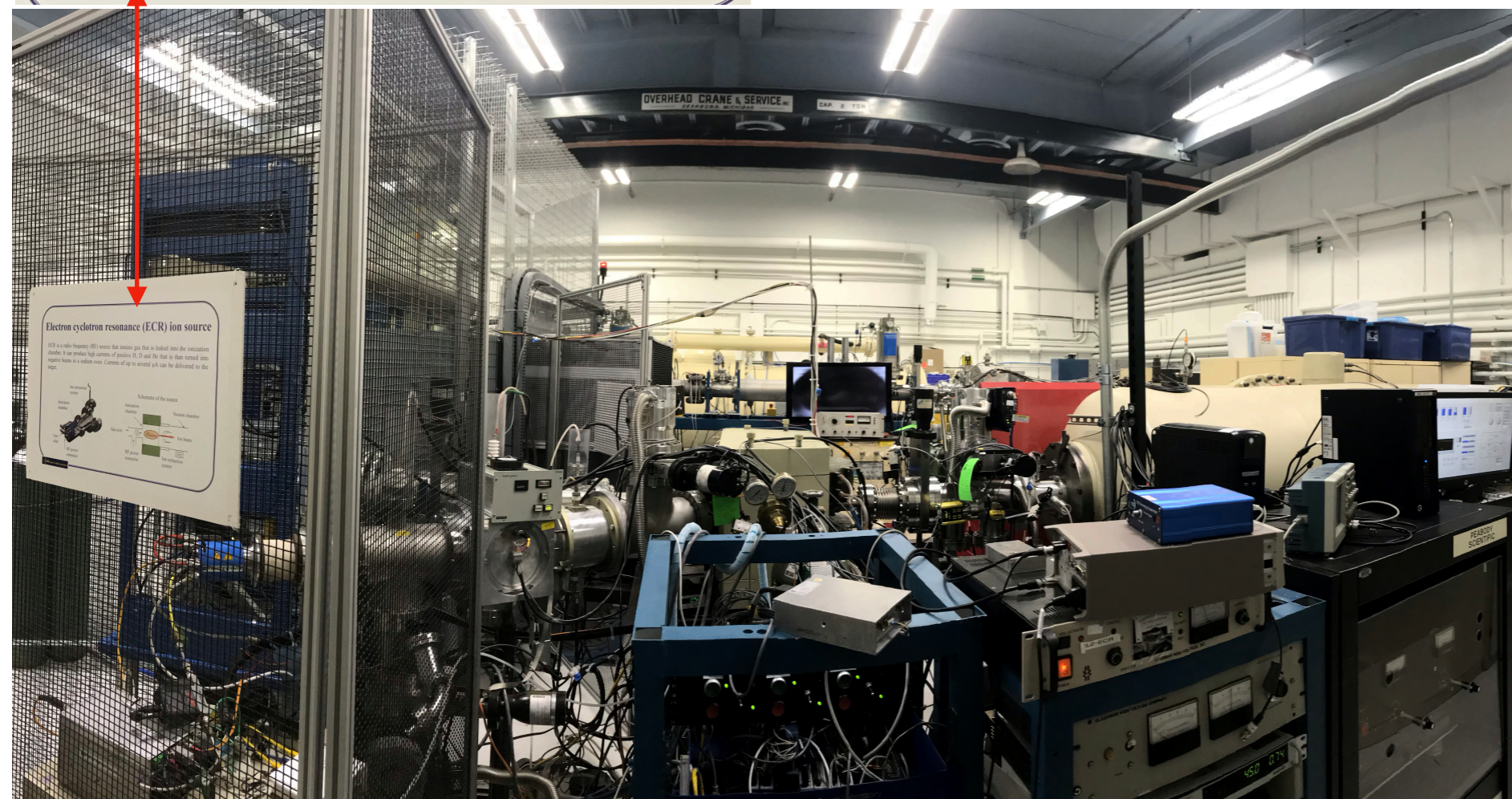
Electron cyclotron resonance (ECR) ion source

ECR is a radio frequency (RF) source that ionizes gas that is leaked into the ionization chamber. It can produce high currents of positive H, D and He that is then turned into negative beams in a sodium oven. Currents of up to several μA can be delivered to the target.



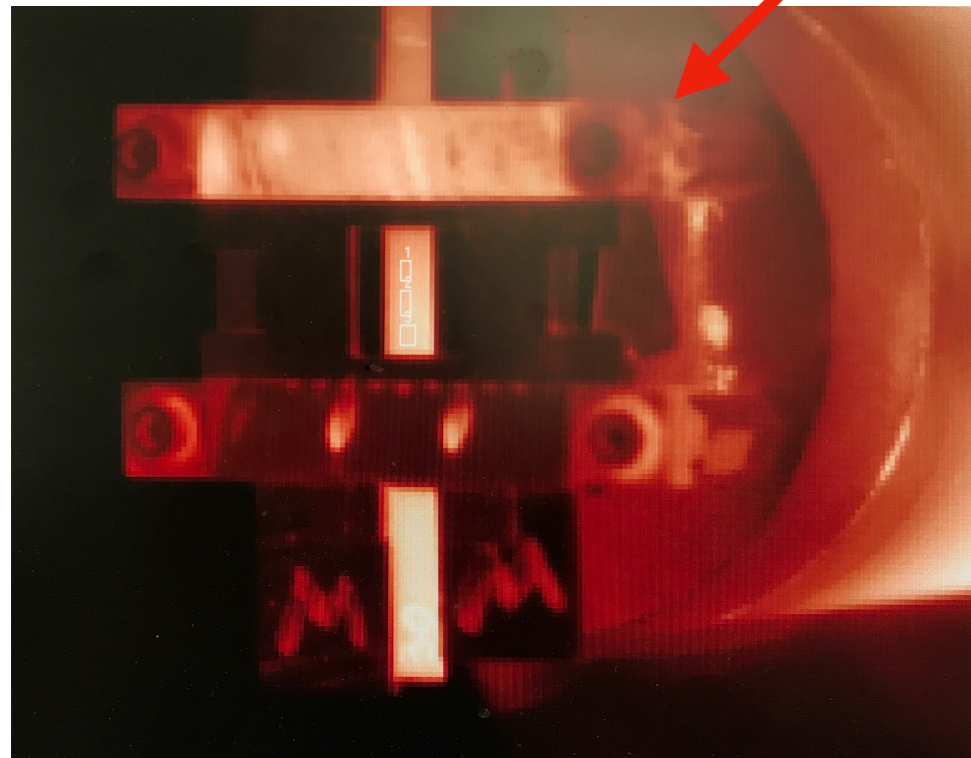
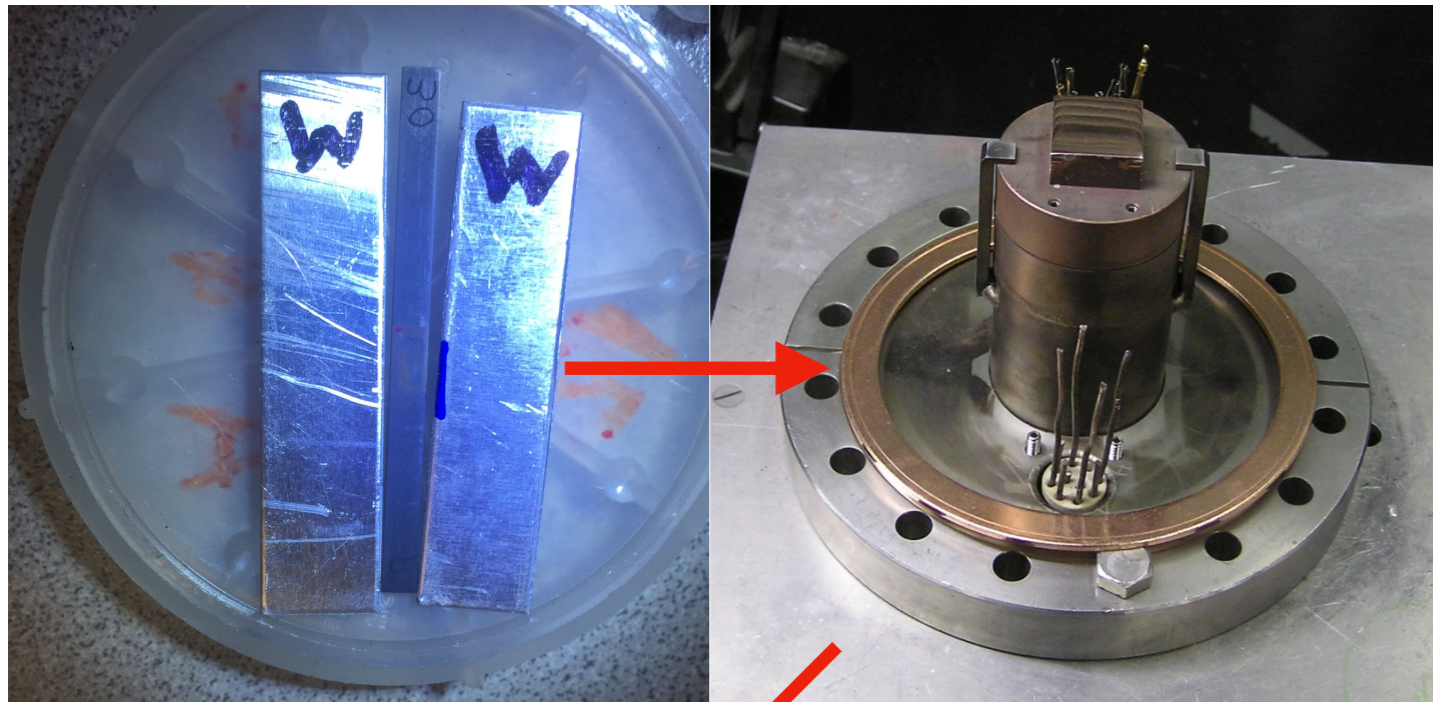
Facility: Michigan Ion Beam Laboratory (MIBL)

- 1.7MV Pelletron accelerator with ECR ion source: 4.5MeV He^{++} with $\sim 1\text{-}2\mu\text{A}$ beam current
- 400kV Implanter for future dual beam irradiation
- In lab TEM

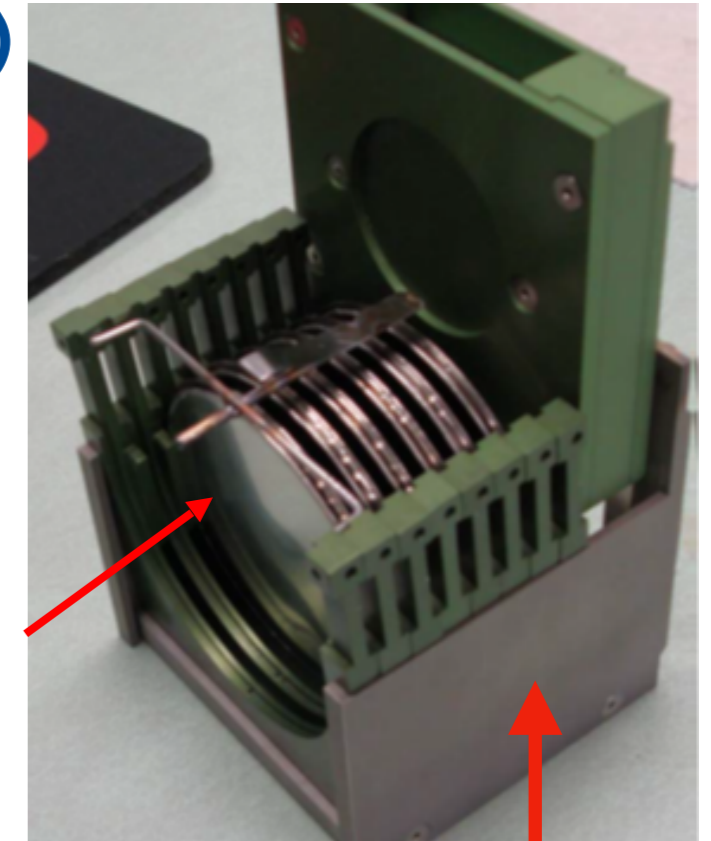


Sample preparation

MIBL Graphite Irradiation

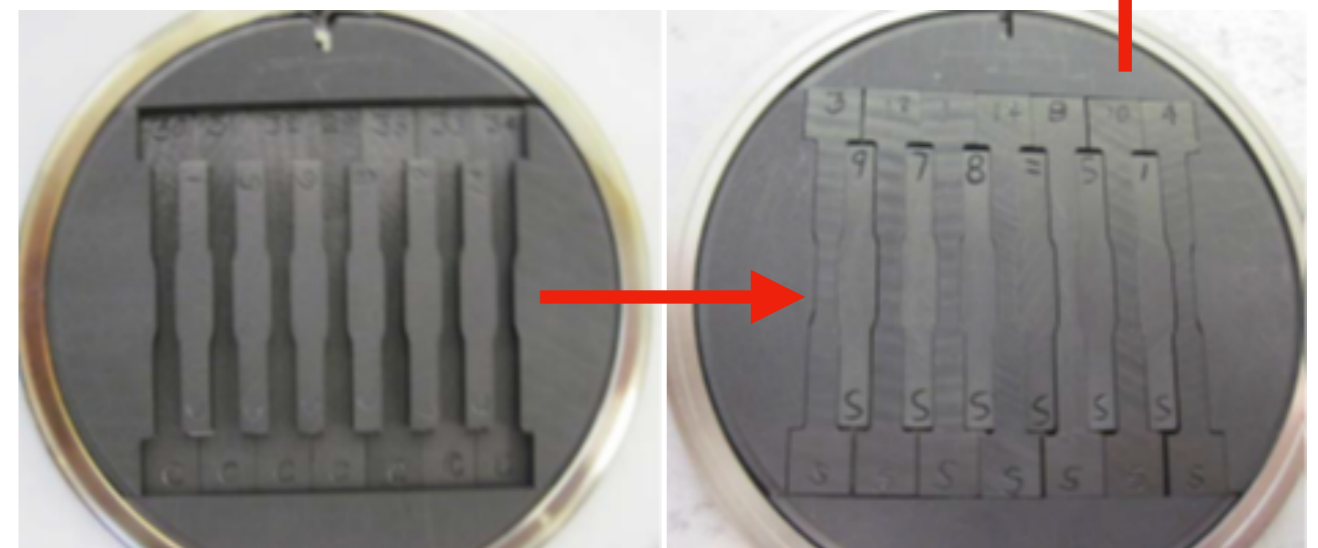


BLIP Graphite Irradiation (2010)



Proton beam

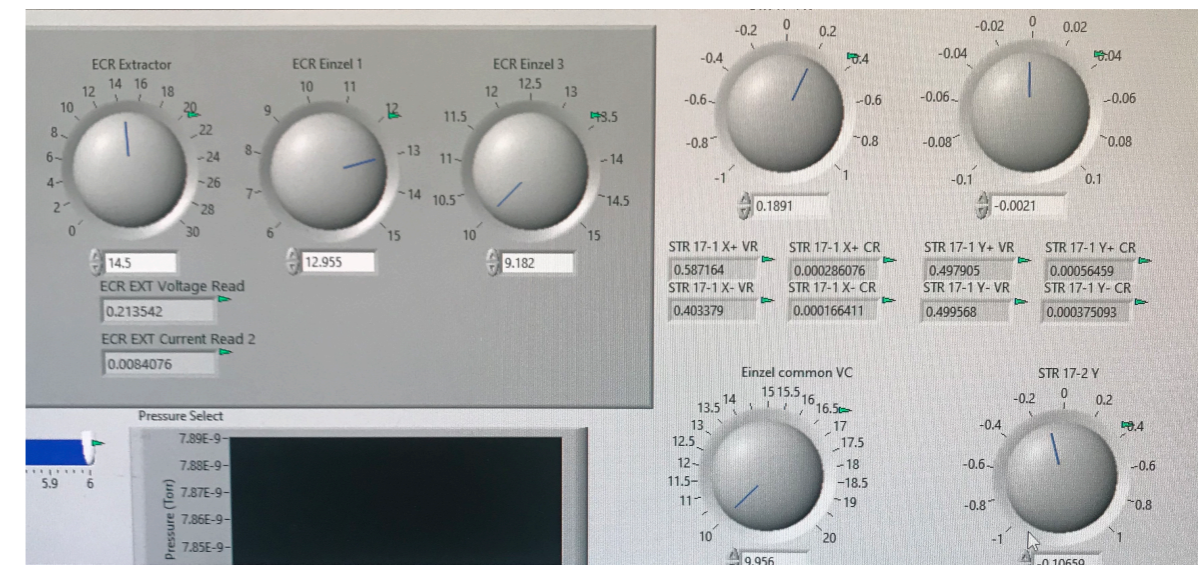
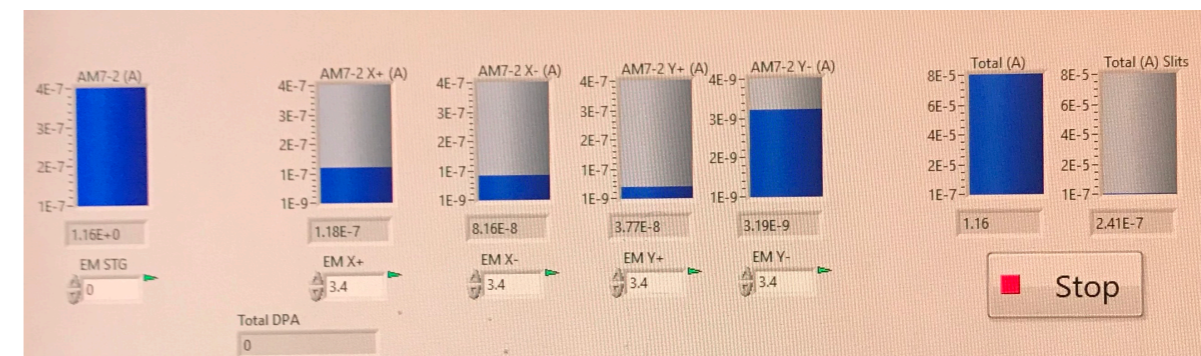
Encapsulated graphite specimens



Beam control and monitoring



**Digital monitors and controls:
Slit currents (TOP)
X steerers and ECR (BOTTOM)**



MIBL control room

Trial irradiation: beam parameters

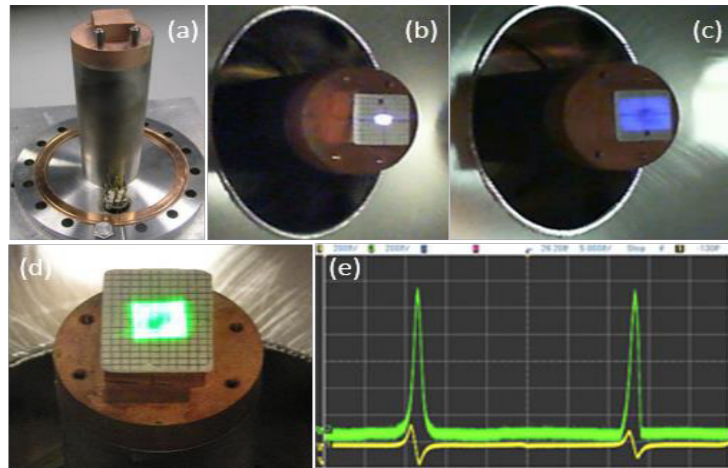
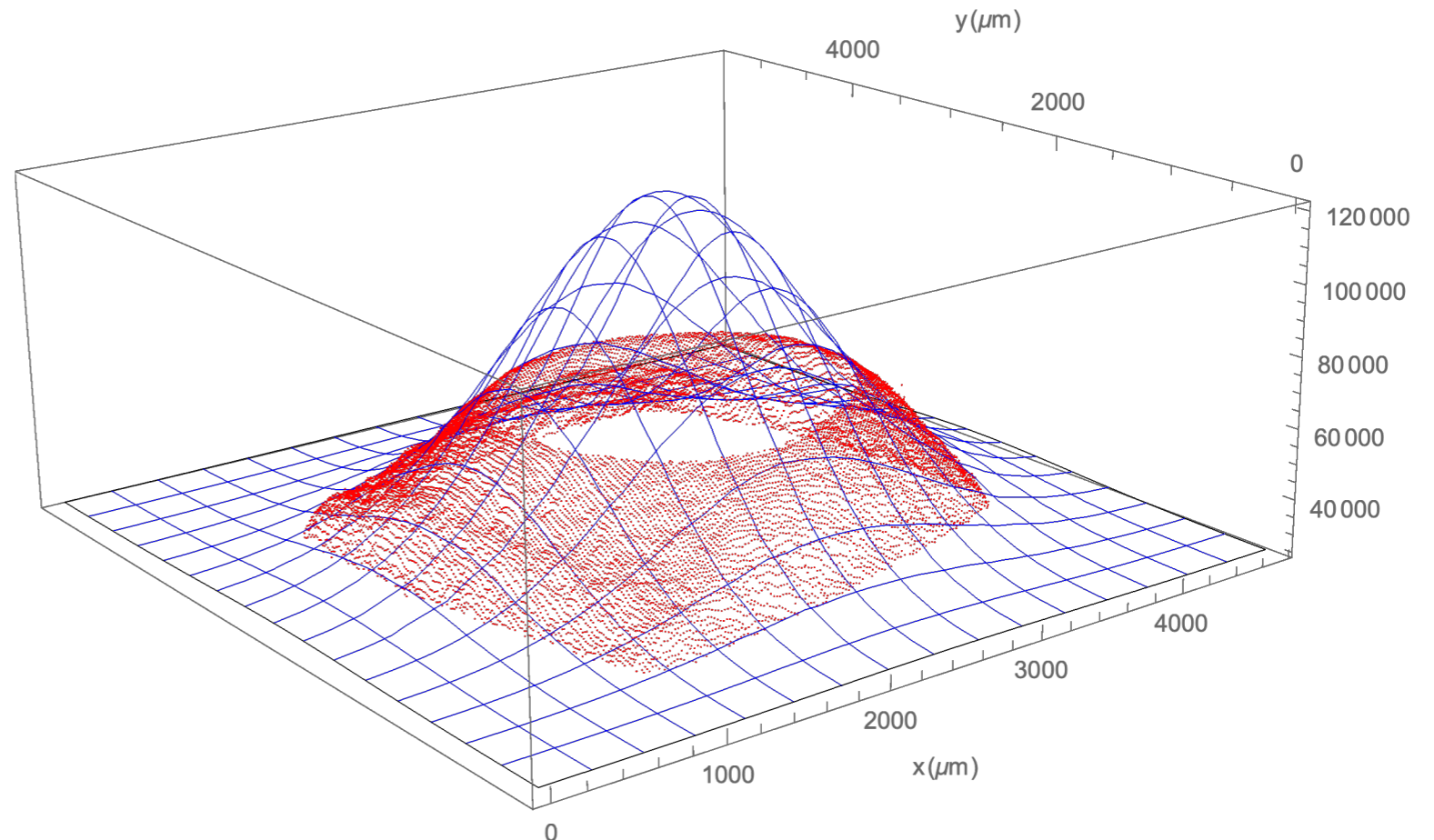
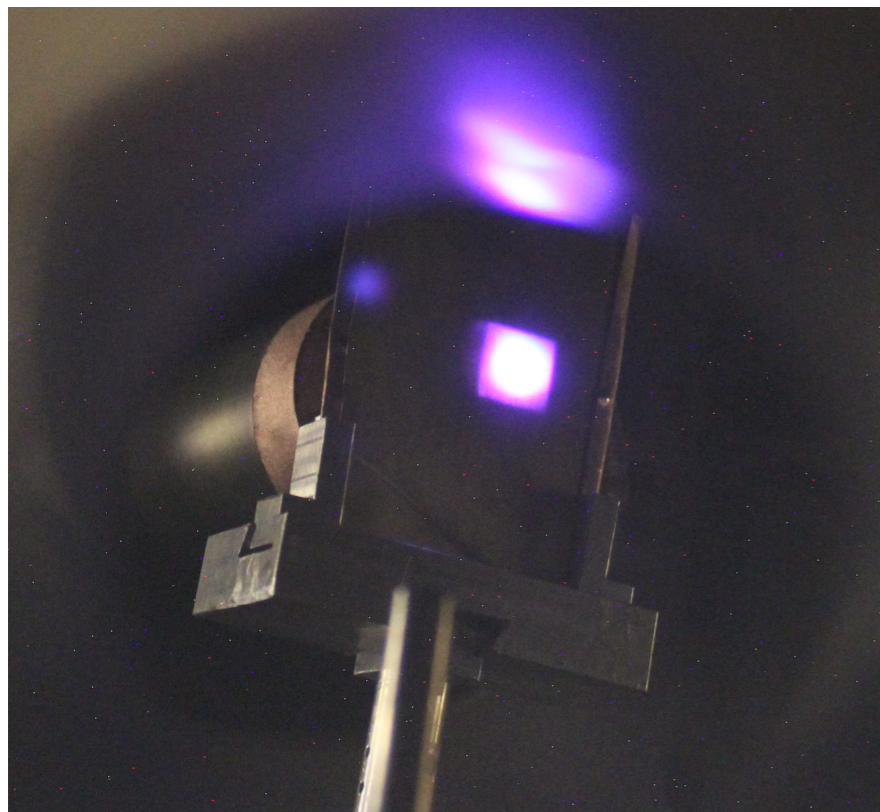


Figure 4: (a) stage, (b) focused beam on the stage, (c) rastered beam on stage, (d) laser light overlap on the ion beam and (e) BPM profile of the focused beam.

Ion energy: 4.5MeV
FWHM: ~2.2mm
Beam current: ~1.3 μ A

THE ALIGNMENT OF CONVERGENT BEMLINES AT A NEW TRIPLE ION BEAM FACILITY

O. F. Toader, T. Kubley, F.U. Naab, E. Uberseder, Michigan Ion Beam Laboratory, University of Michigan, Ann Arbor, Michigan, USA



Determination of beam FWHM accomplished by fitting fluorescence data from ceramic plate dropped into beam

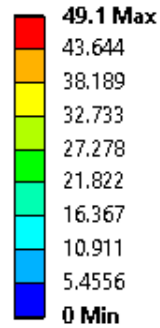
Trial irradiation: Temperature control

Gaussian Beam
Sigma : 1.1 mm

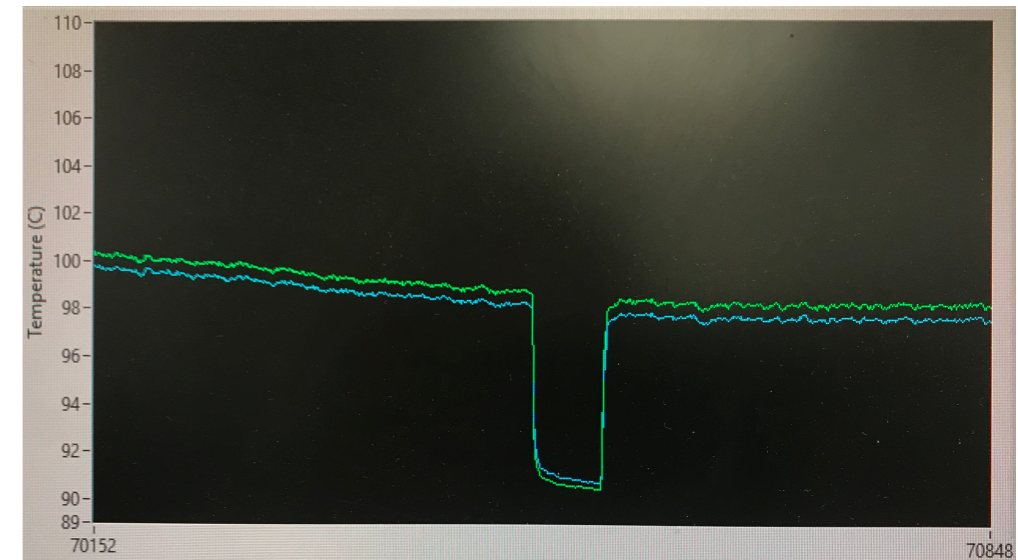
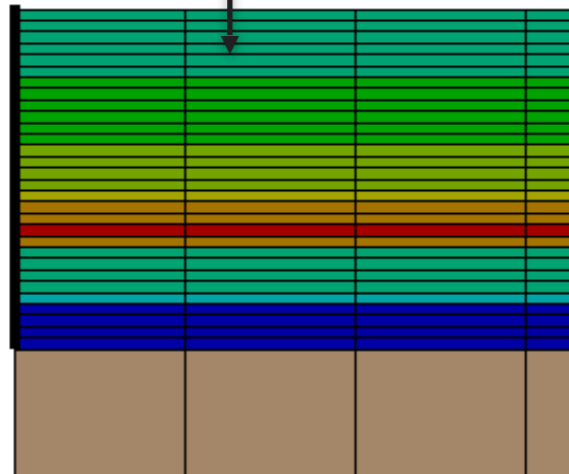
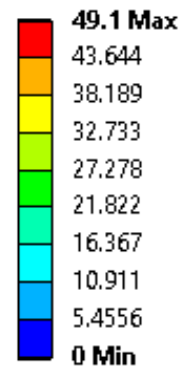


Heat deposition- axisymmetric model

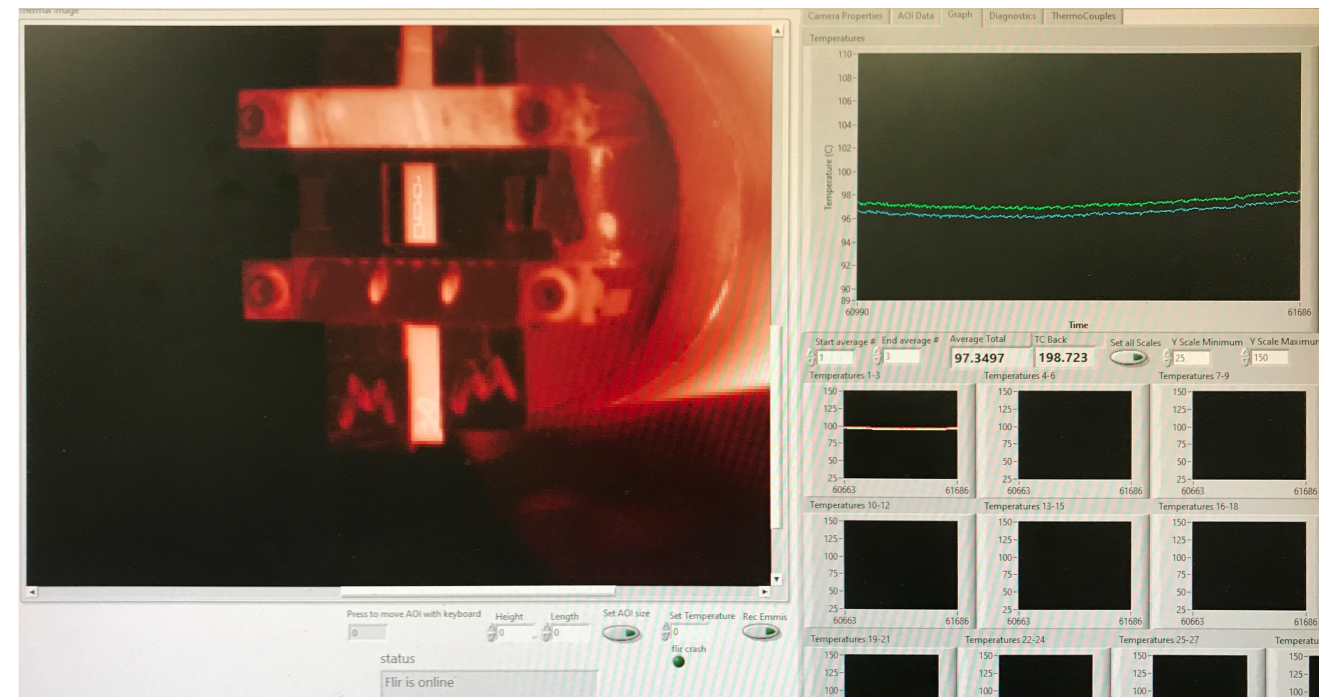
B: Steady-State Thermal
Imported Heat Generation
Time: 1. s
Unit: W/mm³
05/09/2019 09:43



B: Steady-State Thermal
Imported Heat Generation
Time: 1. s
Unit: W/mm³
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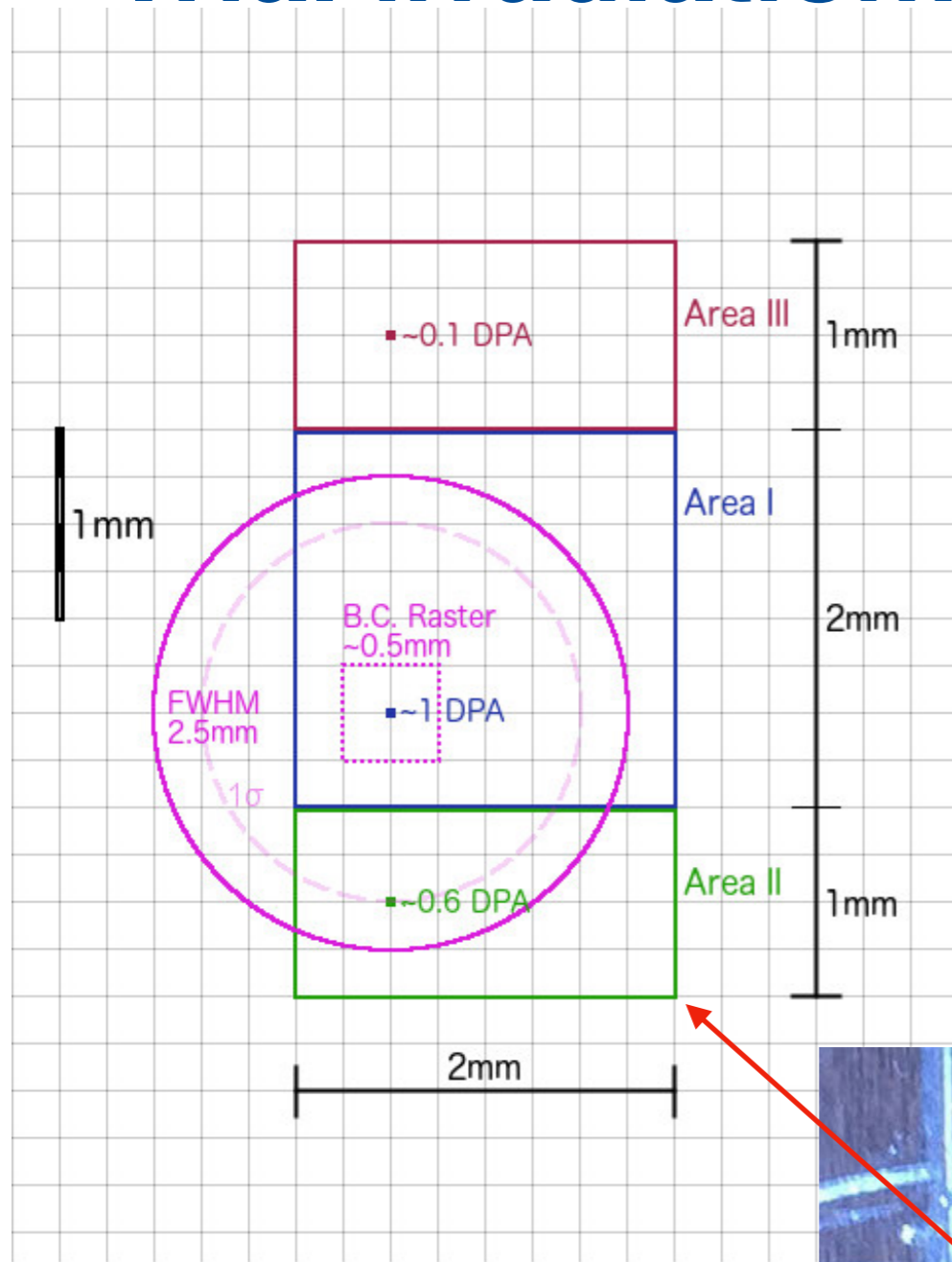


Temperature monitor showing T~100C with ~10C drop when measuring beam current



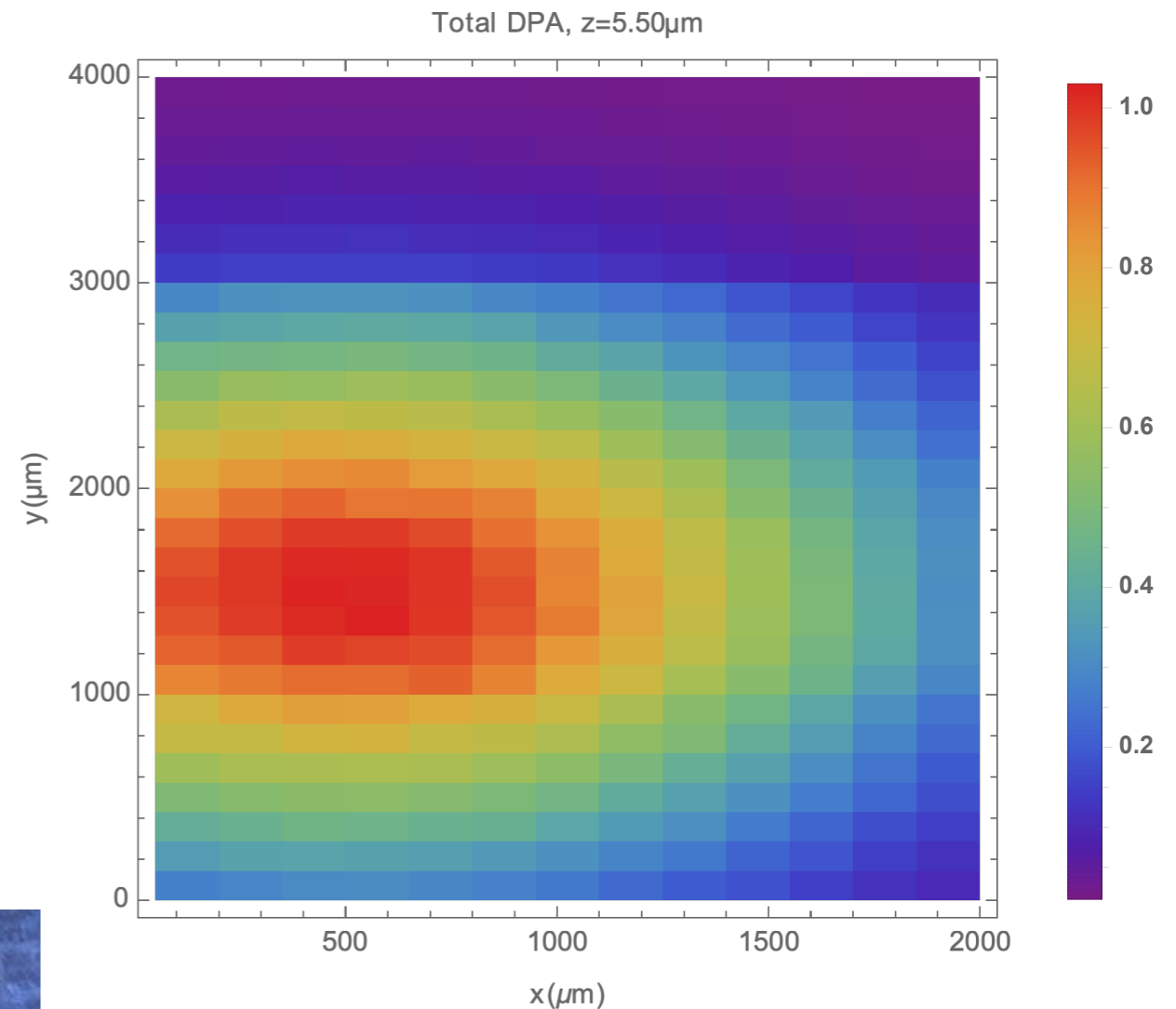
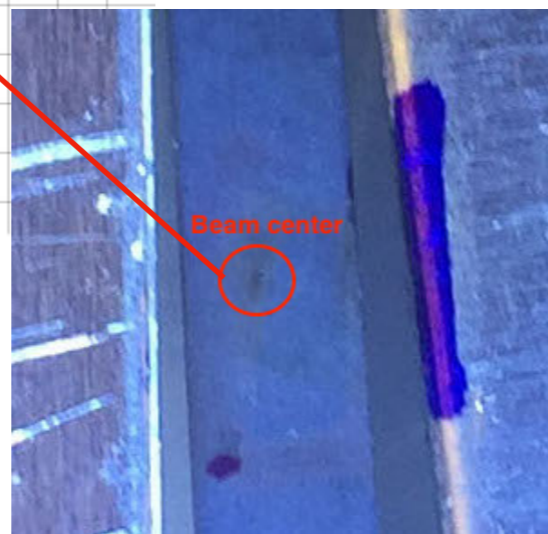
Courtesy: S. Bidhar

Trial irradiation: Footprint and DPA



Irradiation plan

- Irradiate Area I ~5hrs
- Open slits to Area II ~40hrs
- Open slits to Area III ~50hrs



MARS simulation of total DPA accumulation (NRT w/ Stoller correction)

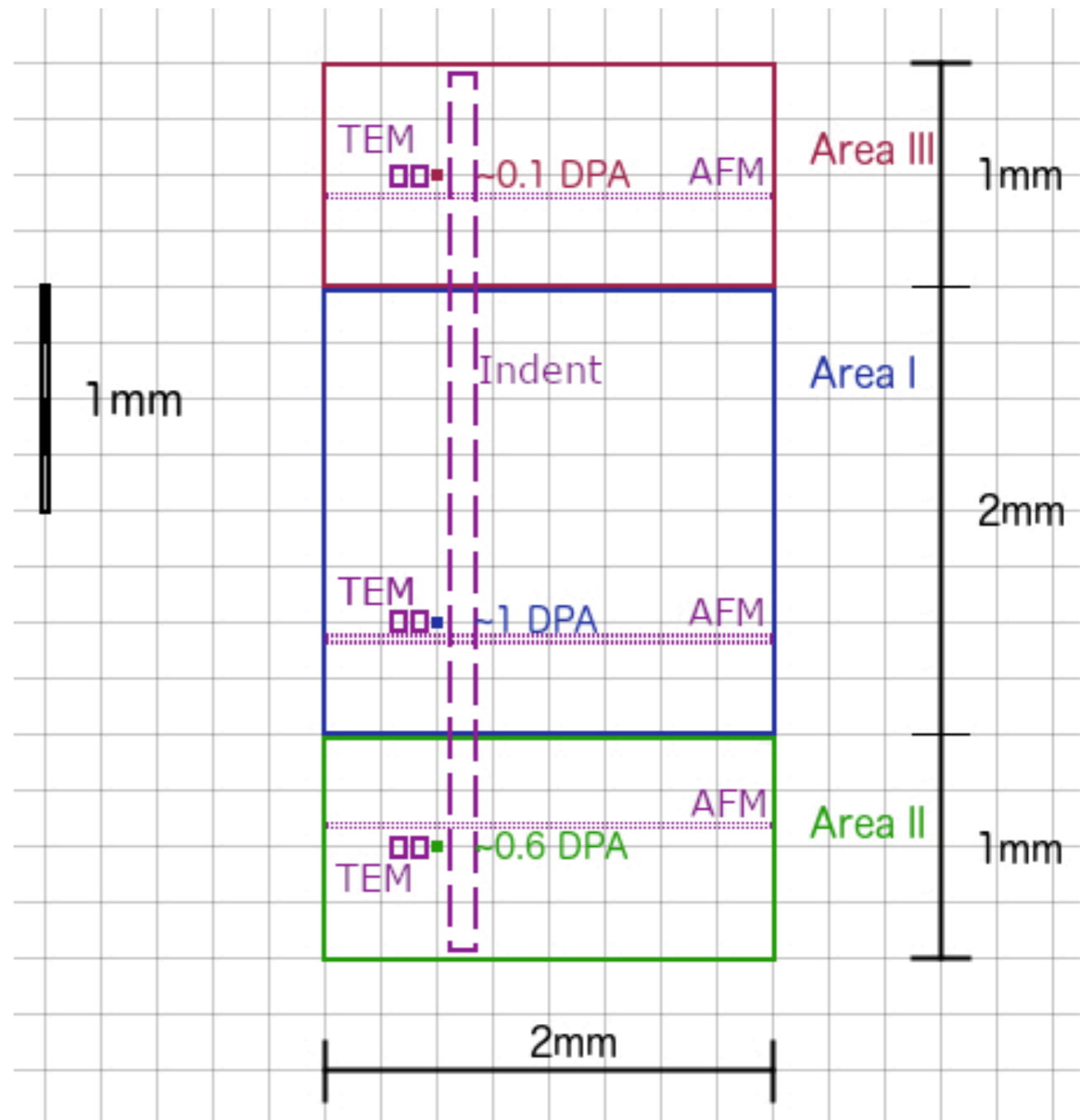
Trial irradiation: Damage rate

Irradiation Source	DPA rate (DPA/s)	He gas prod. (appm/DPA)	Irradiation Temp (°C)
Mixed spectrum fission reactor	3×10^{-7}	1×10^{-1}	200-600
Fusion reactor	1×10^{-6}	1×10^1	400-1000
High energy proton beam	6×10^{-3}	1×10^3	100-800
Low energy He ⁺⁺ beam	3.25×10^{-4} (Peak)	None (implantable)	<100-650

Courtesy: P. Huhr

Trial irradiation: Upcoming PIE work

- TEM at University of Michigan Center for Materials Characterization (MC)²
- AFM profilometry measurements of swelling above surface at IIT
- Nano(micro)indentation at IIT

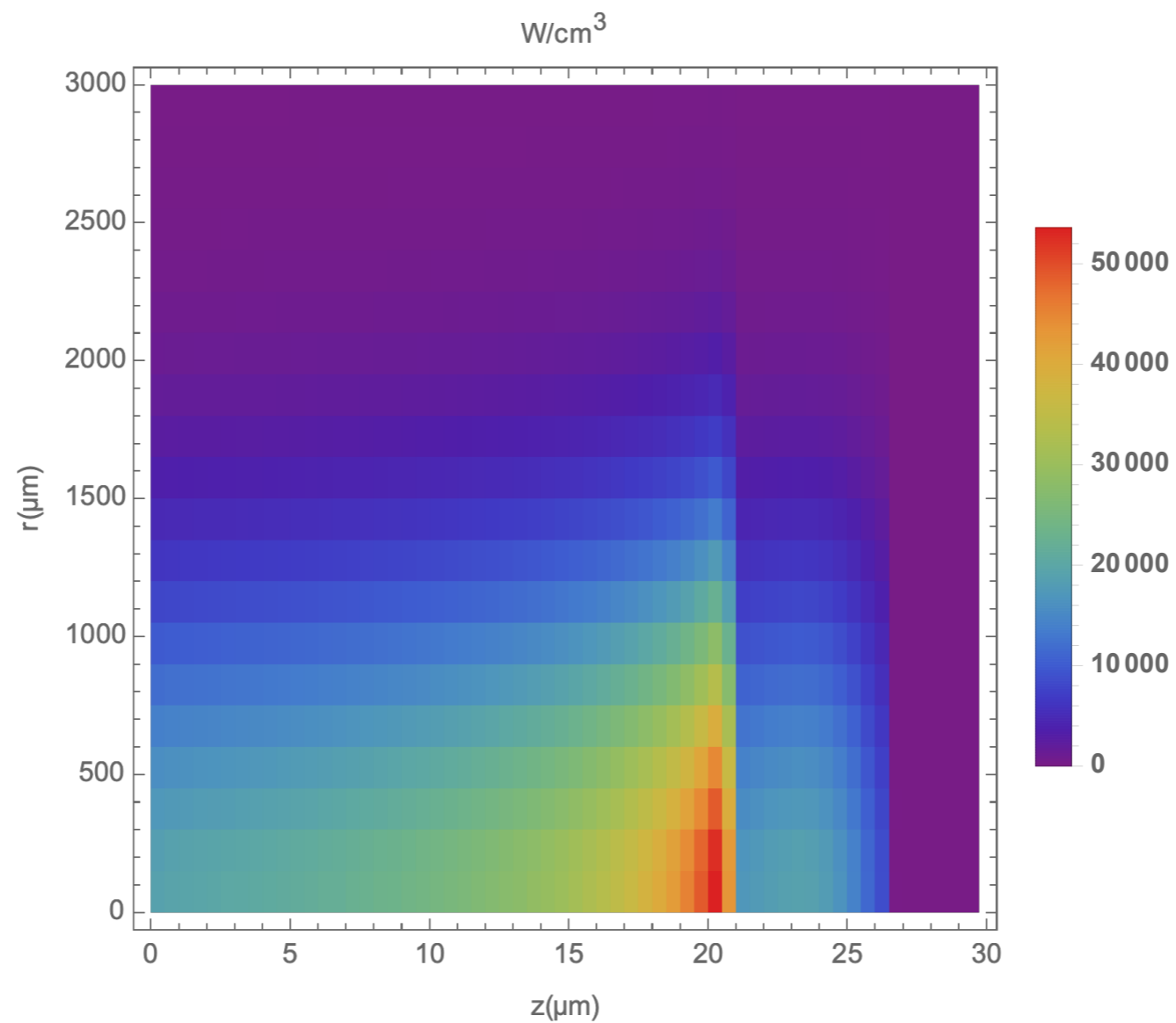


Future PIE work

- X-Ray Diffraction at Argonne National Laboratory Sector 10
- Microcantilever testing, facility TBD



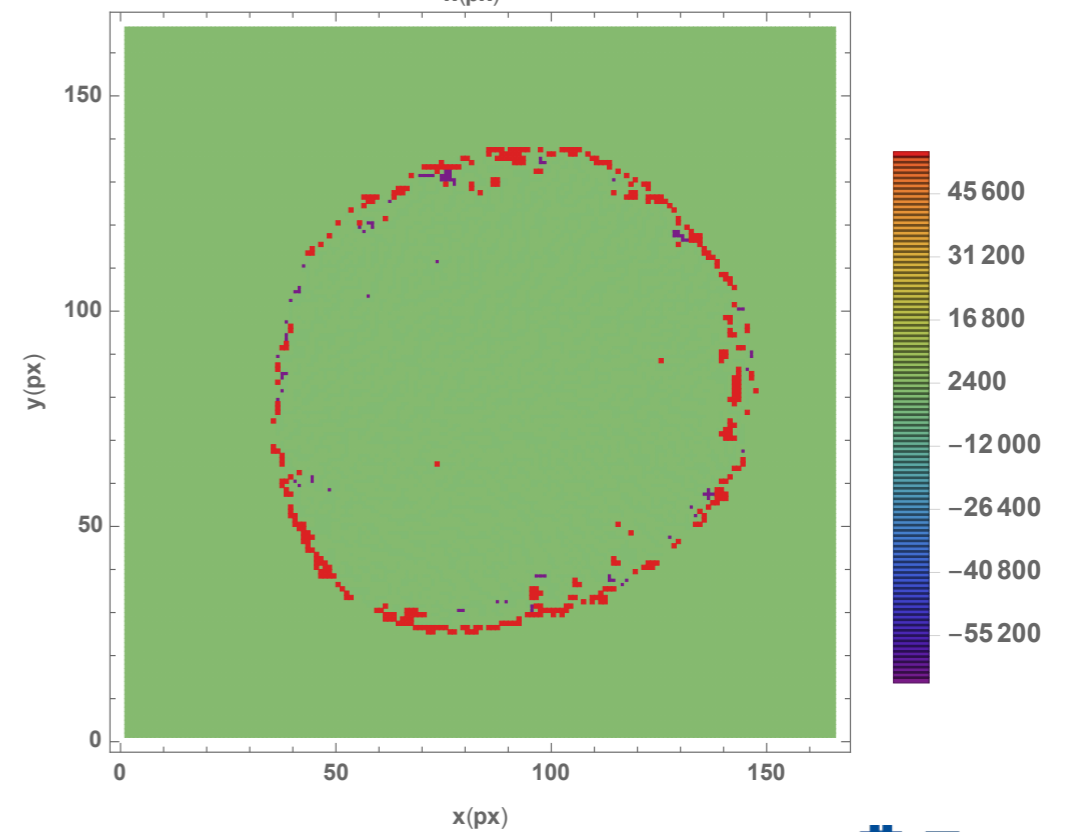
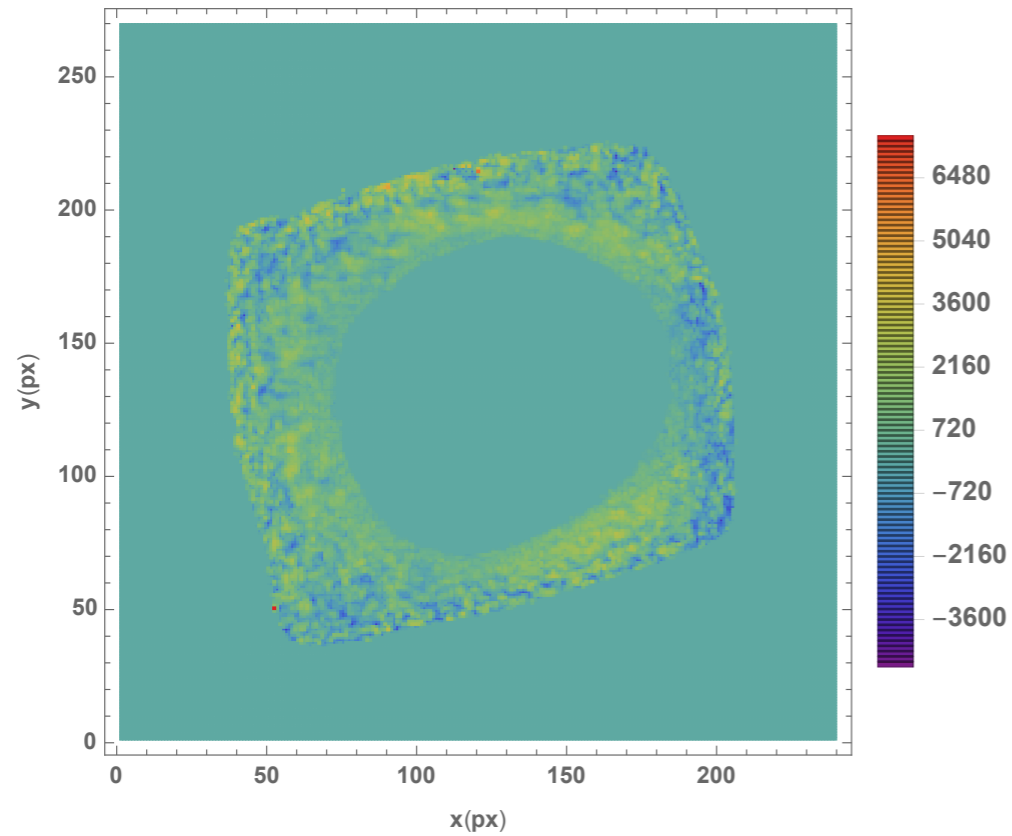
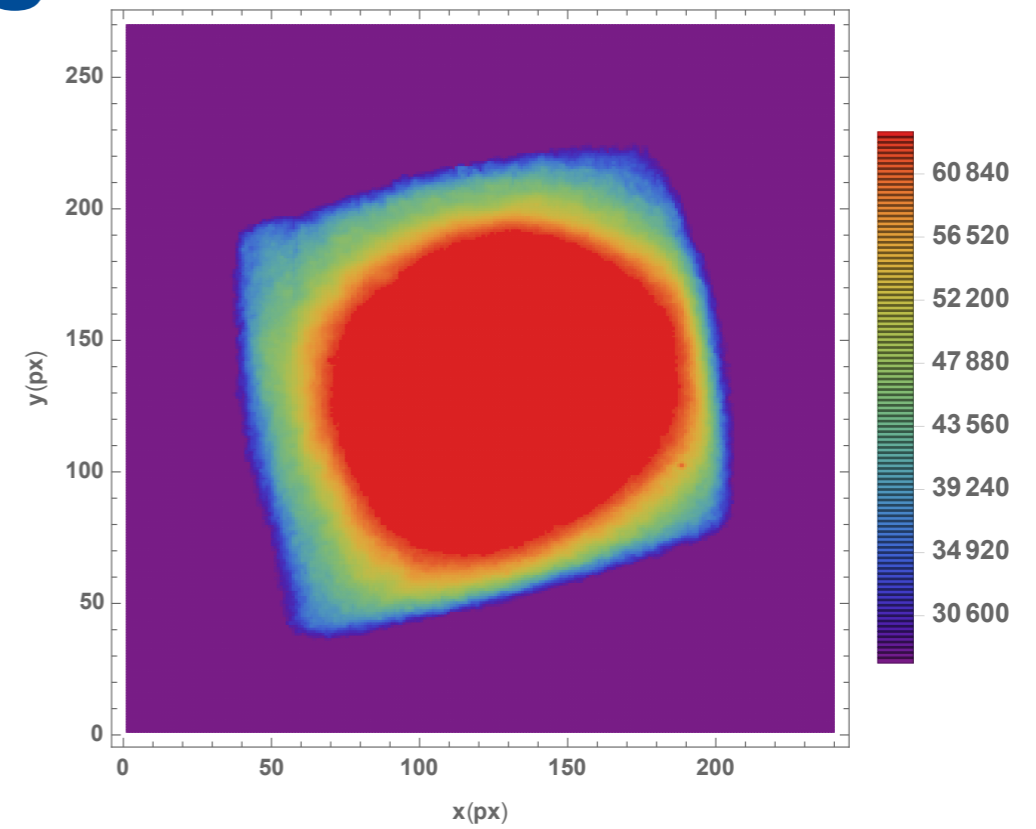
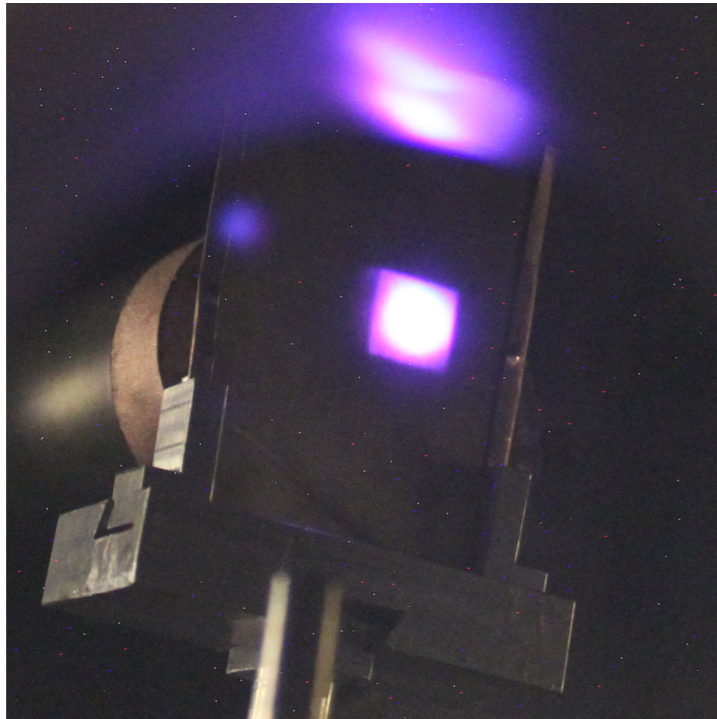
BONUS SLIDES



Current monitoring and logs

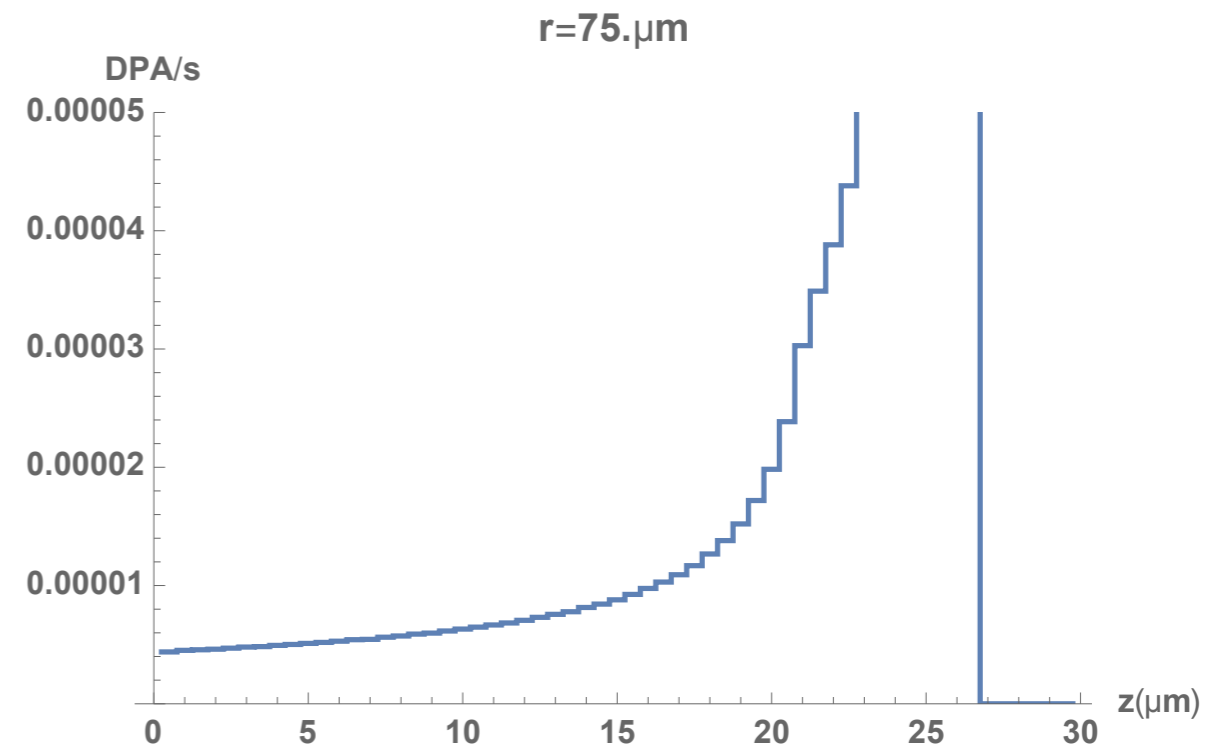
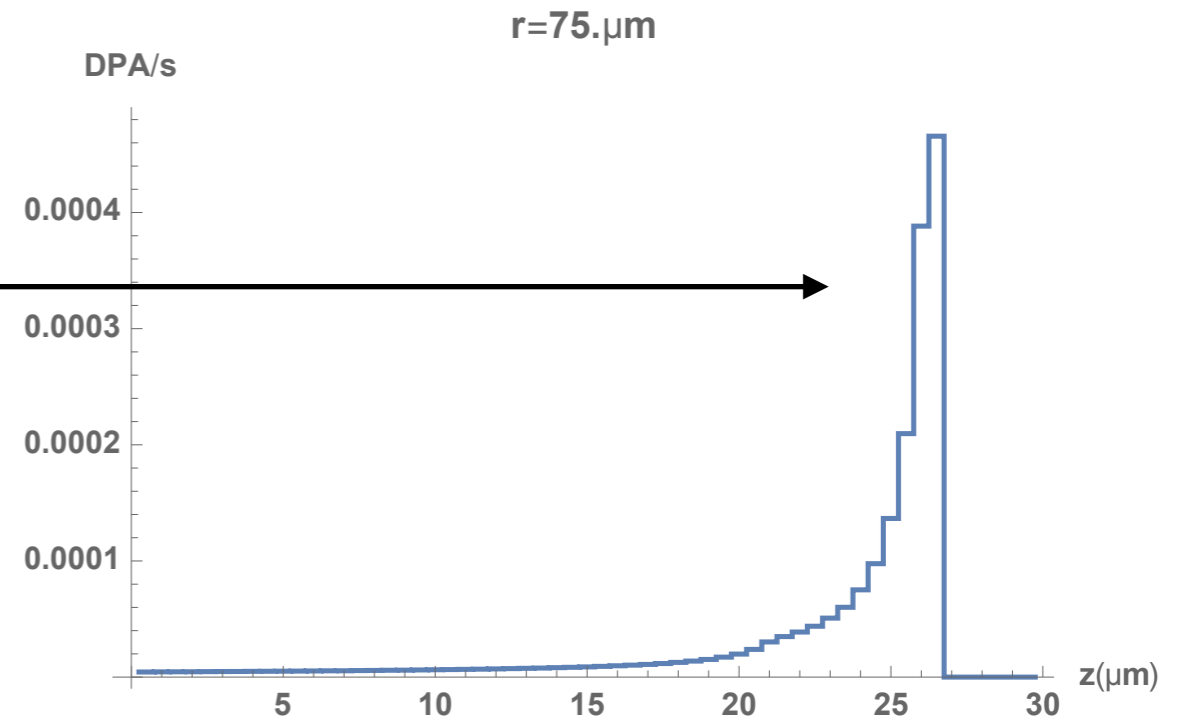
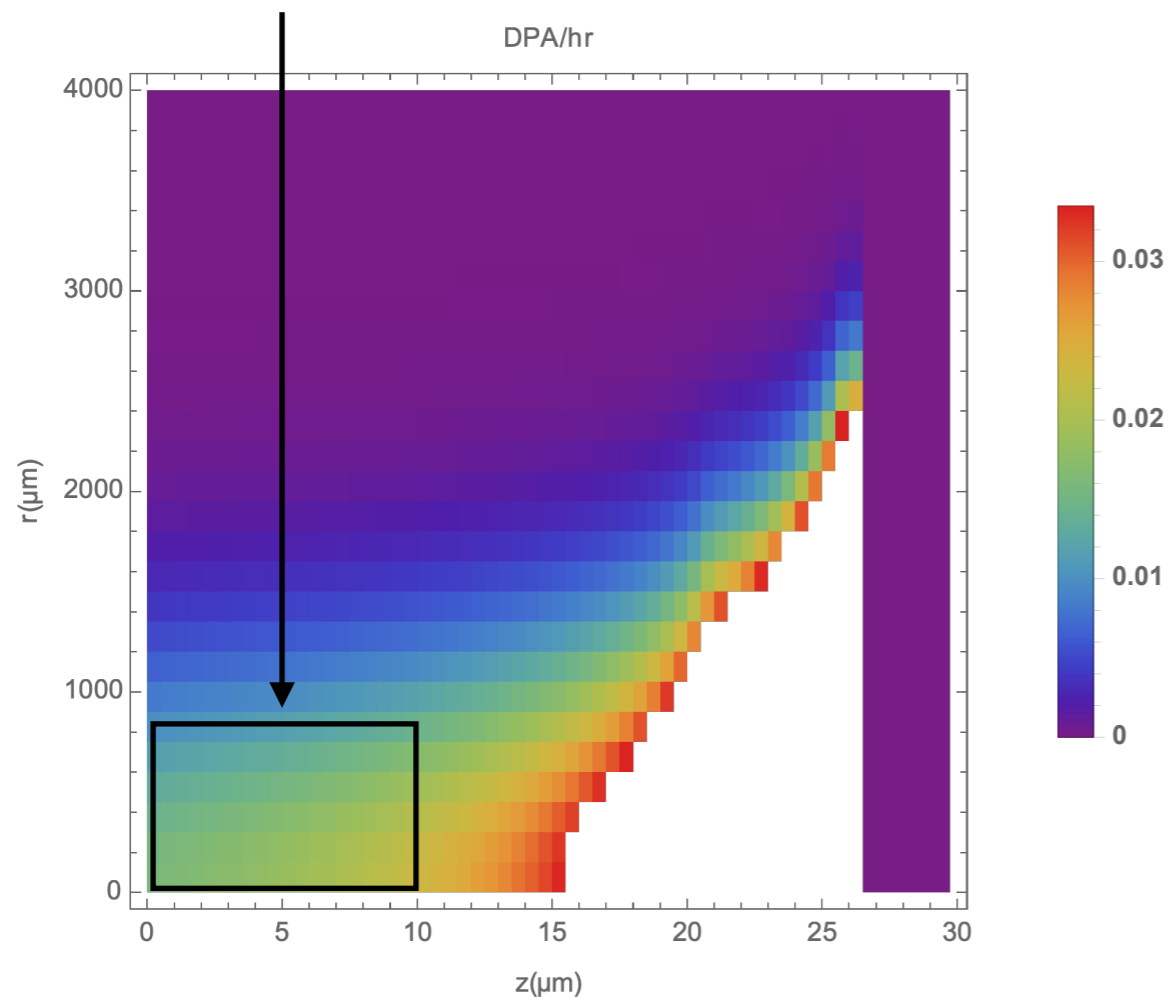
Name for experiment			Beam current samples					
Starting Beam Current	335	nA	Current (nA)	Time	Time Diff. (s)	Integrated Charge	hrs	43
Sample Area	0.06	cm ²	372	11/20/2019 14:45:00	0	0.00E+00	ions	8.75E+15
Charge State	2		372	11/20/2019 14:46:00	60.0	2.23E-05	total ions	
Desired Dose	3.50E+18	atoms/cm ²	350	11/20/2019 15:42:00	3360.0	1.21E-03		
Start Time	11/20/2019 14:45		300	11/20/2019 16:48:00	3960.0	1.29E-03		
			283	11/20/2019 17:28:00	2400.0	7.00E-04		
Coulombs Needed	6.73609E-02		268	11/20/2019 18:31:00	3780.0	1.04E-03		
Coulombs Remaining	-1.68564E-04		254	11/20/2019 19:41:00	4200.0	1.10E-03		
Last Current	640.00		245	11/20/2019 20:31:00	3000.0	7.48E-04		
Last Time	11/22/2019 9:45:00		240	11/20/2019 21:21:00	3000.0	7.27E-04		
Stop Time	11/22/2019 9:40:37		240	11/20/2019 22:33:00	4320.0	1.04E-03		
			236	11/20/2019 23:43:00	4200.0	1.00E-03		
RATIO OF Y UP TO DOWN	UP/DOWN	1.86E+01	225	11/21/2019 0:25:00	2520.0	5.81E-04		
			225	11/21/2019 0:54:00	1740.0	3.92E-04		
			220	11/21/2019 2:55:00	7260.0	1.62E-03		
			215	11/21/2019 4:59:00	7440.0	1.62E-03		
			157	11/21/2019 7:05:00	7560.0	1.41E-03		
			157	11/21/2019 8:57:00	6720.0	1.06E-03		
			360	11/21/2019 9:01:00	240.0	6.20E-05		
			360	11/21/2019 9:02:00	60.0	2.16E-05		
			420	11/21/2019 9:12:00	600.0	2.34E-04		
			480	11/21/2019 9:23:00	660.0	2.97E-04		
			516	11/21/2019 9:47:00	1440.0	7.17E-04		
			520	11/21/2019 10:12:00	1500.0	7.77E-04		
			520	11/21/2019 11:43:00	5460.0	2.84E-03		
			590	11/21/2019 12:53:00	4200.0	2.33E-03		
			590	11/21/2019 14:09:00	4560.0	2.69E-03		
			570	11/21/2019 15:33:00	5040.0	2.92E-03		
			568	11/21/2019 16:29:00	3360.0	1.91E-03		
			564	11/21/2019 17:55:00	5160.0	2.92E-03		
			560	11/21/2019 21:21:00	12360.0	6.95E-03		
			640	11/22/2019 6:02:00	31260.0	1.88E-02		
			640	11/22/2019 8:32:00	9000.0	5.76E-03		
			640	11/22/2019 9:45:00	4380.0	2.80E-03		

Rastered beam analysis



Ion Irradiation Simulations: DPA/activation

- Predicted total activity after 1d irradiation, 15min cooling = $3.91 \cdot 10^{-9}$ mSv/hr
- Peak DPA/s $\sim 5 \cdot 10^{-4}$
- Time to 1 DPA (near surface) ~ 65 hrs
- Linearity near surface: $\sim 10\mu\text{m}$ depth, $800\mu\text{m}$ radius

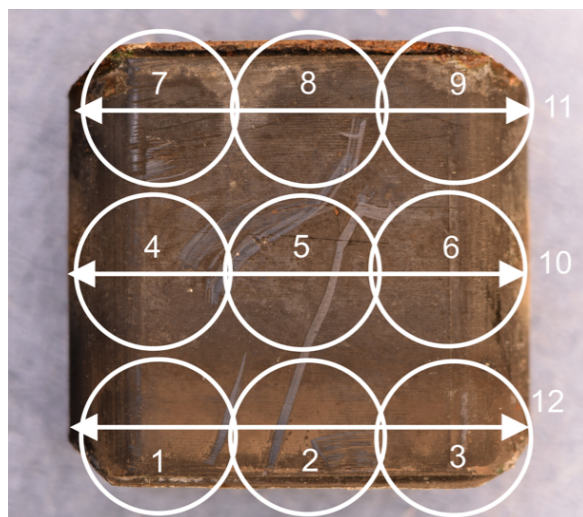
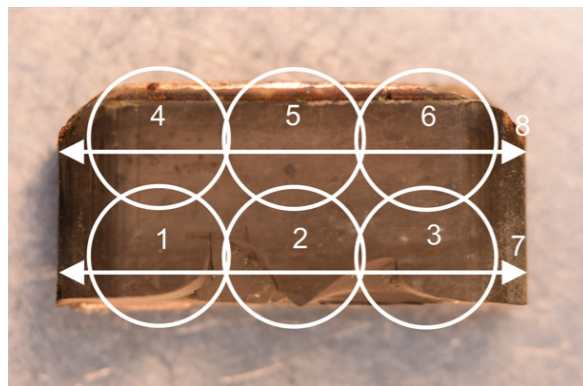


Post-Irradiation-Examination of Target Fins

To determine whether neutrino degradation was a result of radiation damage

- Measure bulk swelling
- Evaluate fracture surfaces
- Evaluate microstructural conditions and extent of radiation damage

Dimensional measurements



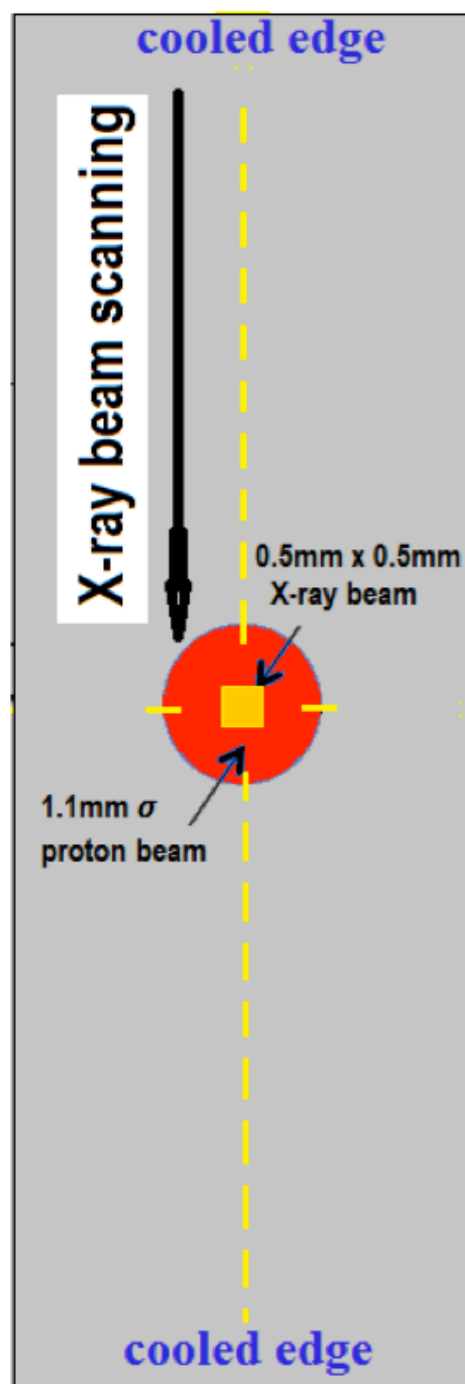
	US Half Fin	US Full Fin	DS Half Fin	DS Full Fin
Avg. End Thickness (mm)	6.54	6.57	6.55	6.55
Avg. Middle Thickness (mm)	6.67	6.64	6.60	6.57
Relative Swelling (%) (Middle-to-end)	2.0	1.1	0.7	0.2
Absolute Swelling (%) (Middle-to-ref*)	4.3	3.8	3.1	2.6

*Ref thickness = 6.4 mm

- Greater swelling in middle vs. ends
- Greater swelling upstream vs. downstream of target
- Greater swelling in half fins vs. full fins

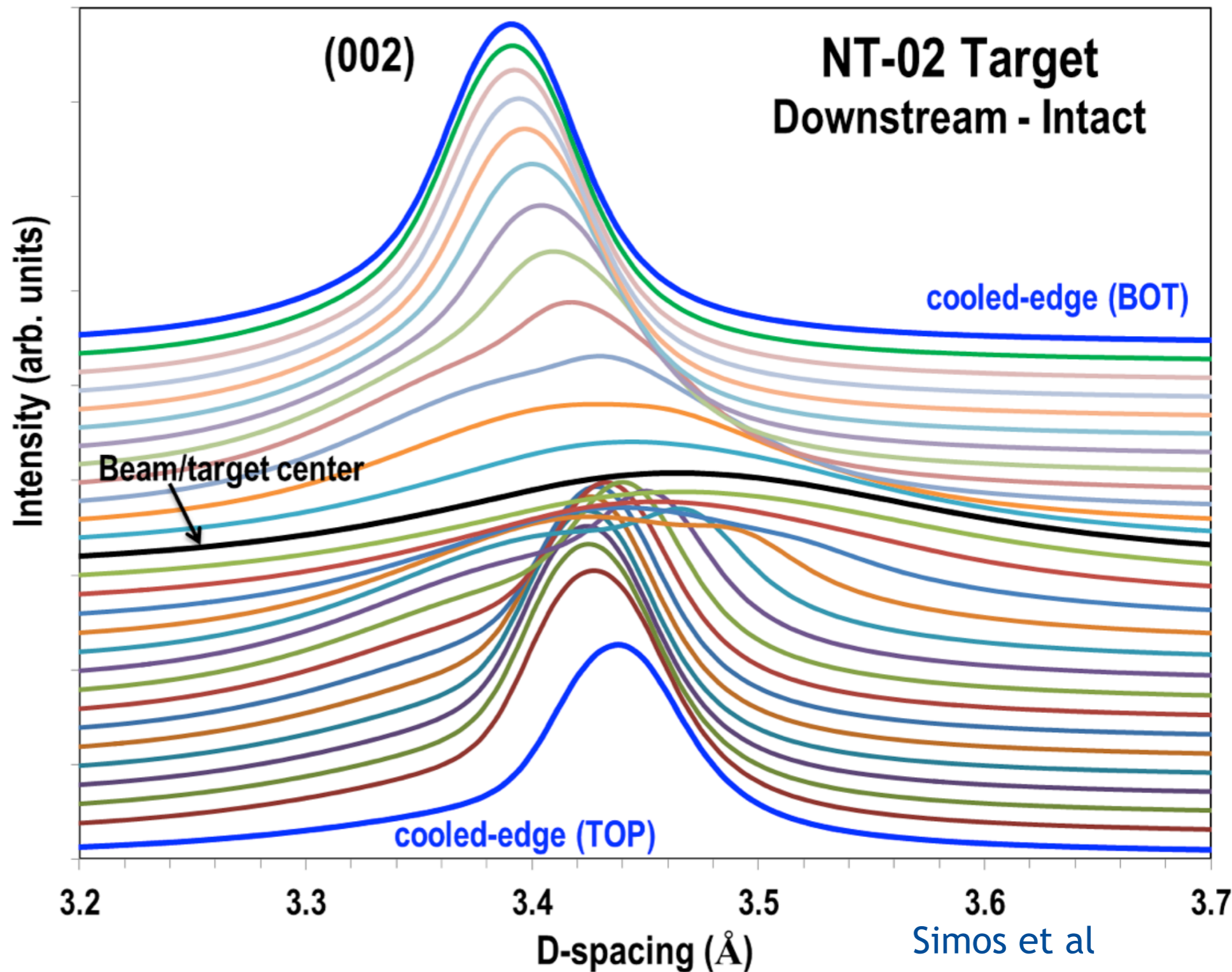
Results are self-consistent and provide indication of bulk swelling

Graphite Results – X-ray diffraction of NuMI graphite fin shows lattice growth and amorphitization at beam center



Upper (cooler) half

Lower (warmer) half



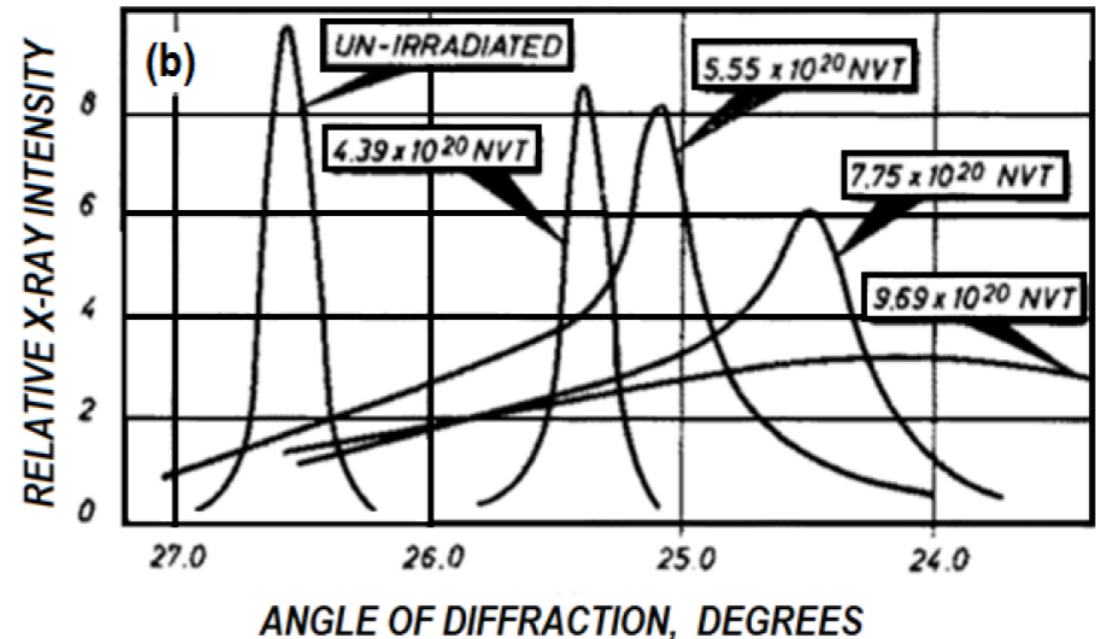
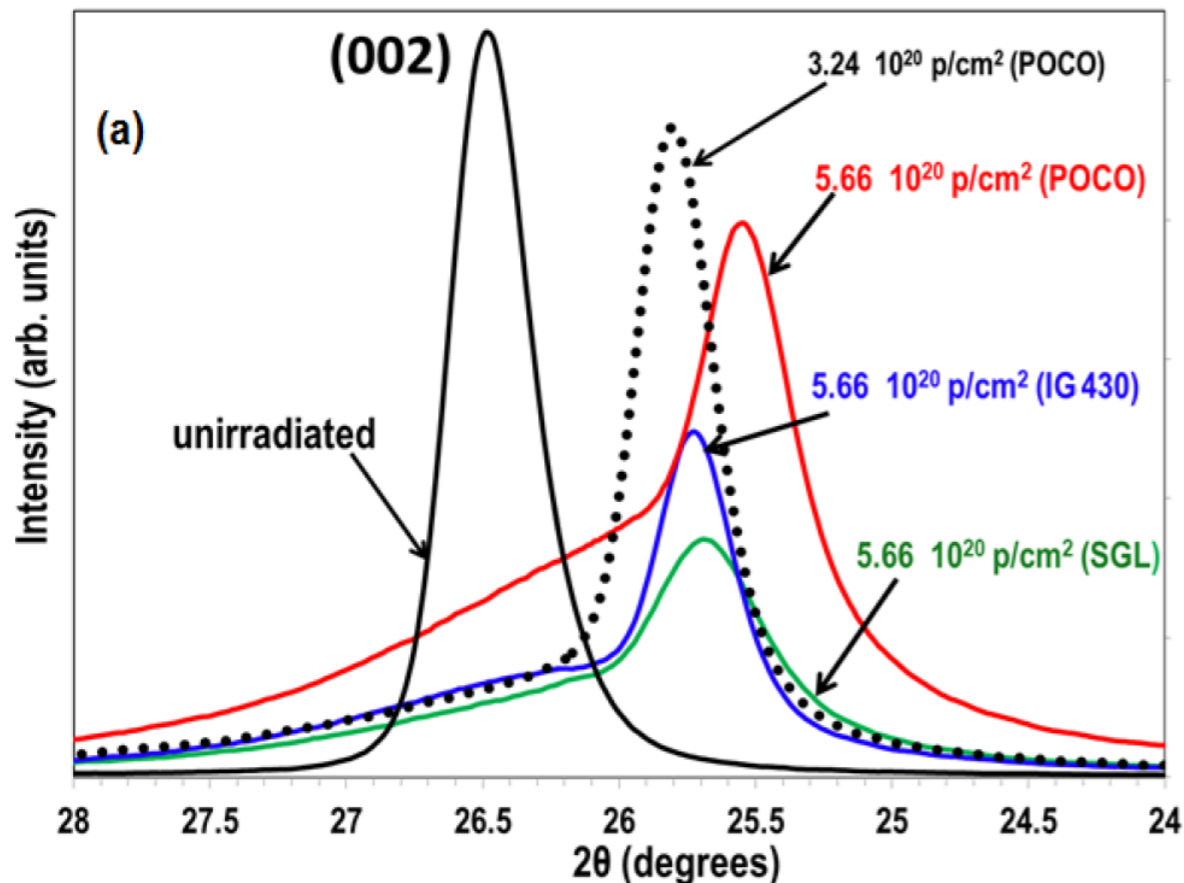
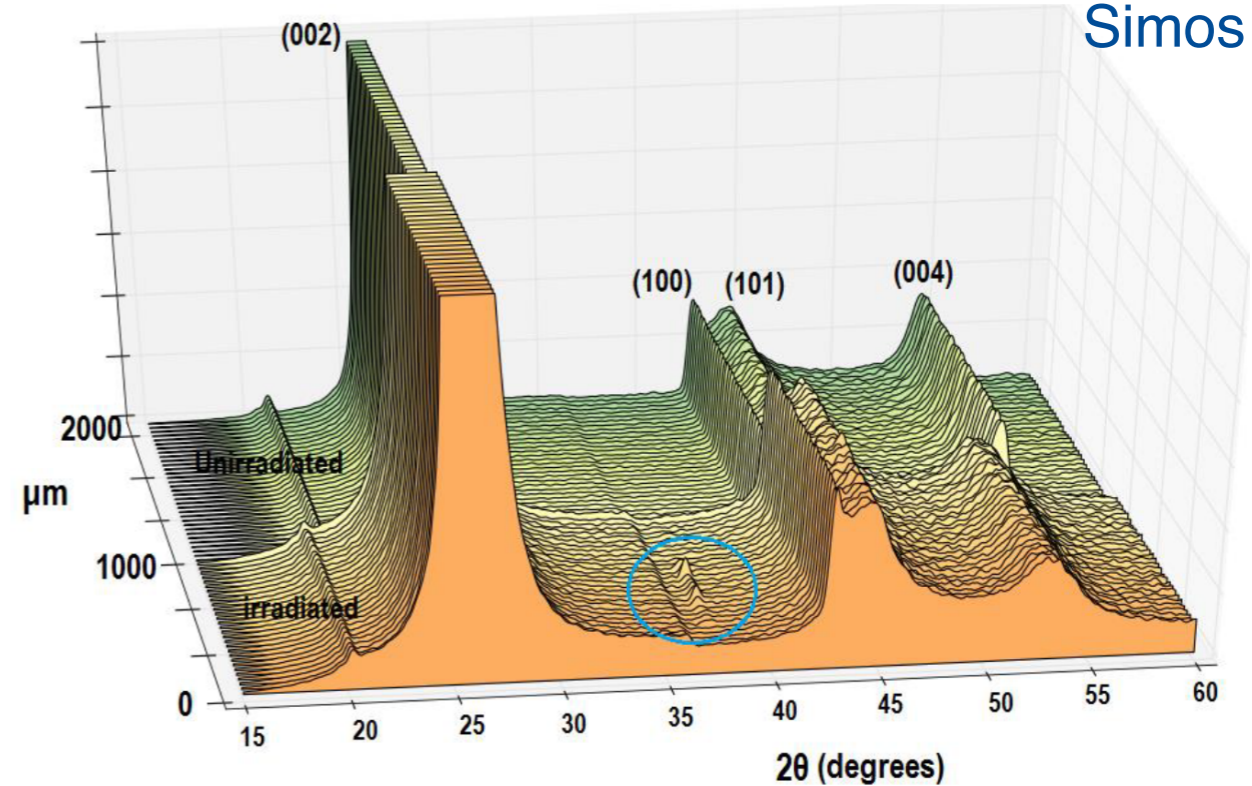
Simos et al



Graphite Results – X-ray diffraction

Simos et al

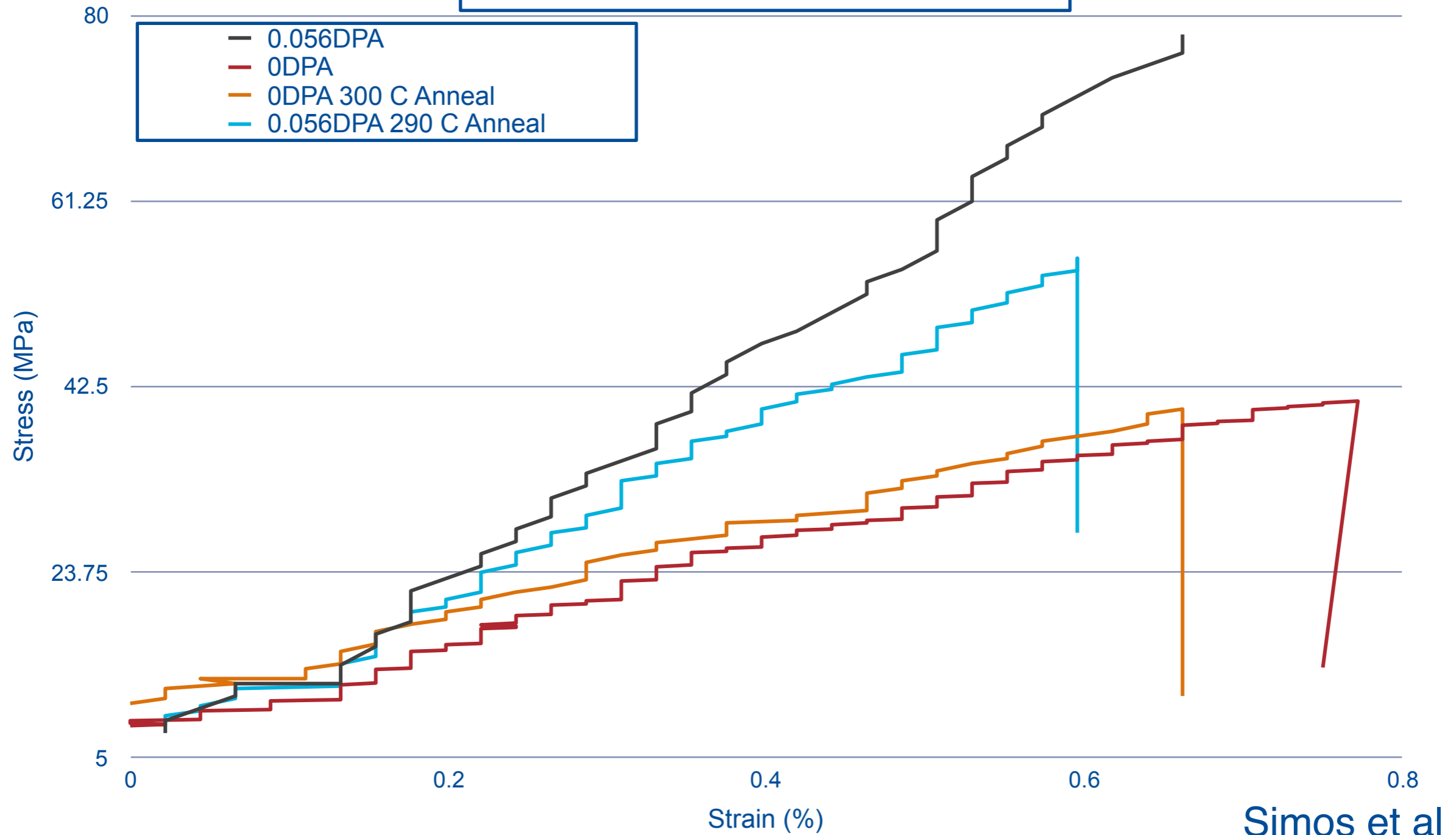
XRD on BLIP irradiated POCO graphite indicates agreement with c-axis lattice growth results from neutron irradiation



W. Bollmann. "Electron-microscopic observations on radiation damage in graphite" Phil. Mag., 5(54):621-624, June 1960.

Graphite Results – Tensile Properties Partially Recover When Annealed

Irradiation Temperature ~150 °C



Simos et al