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# RaDIATE thermal shock experiments at CERN's HiRadMat facility

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7th High Power Targetry Workshop

MSU – FRIB, East Lansing, MI

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# Outline

- Introduction
- BeGrid1 (HRMT24)
  - Experimental set-up
  - PIE work and results
- BeGrid2 (HRMT43)
  - Objectives
  - Experimental design
  - PIE activities
- Conclusion

# RaDIATE Collaboration

Radiation Damage In Accelerator Target Environments

radiate.fnal.gov

## Broad aims are threefold:

- to generate new and useful materials data for application within the **accelerator** and **fission/fusion** communities
- to recruit and develop new scientific and engineering experts who can **cross the boundaries** between these communities
- to initiate and coordinate a **continuing synergy** between research in these communities, benefitting both **proton accelerator applications** in science and industry and **carbon-free energy technologies**

### HRMT 24/43 contributors


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### RaDIATE Collaboration Members

Fermilab, Science & Technology Facilities Council, University of Oxford, Oak Ridge National Laboratory, Los Alamos National Laboratory, Brookhaven National Laboratory, Argonne National Laboratory, Pacific Northwest National Laboratory, Ciemat (Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas), European Spallation Source, CERN, FRIB

# Introduction

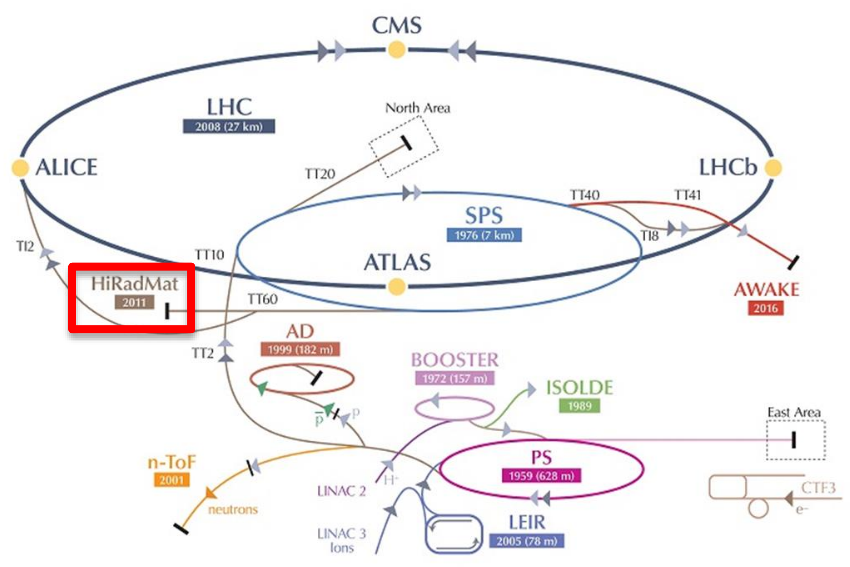
## Motivation

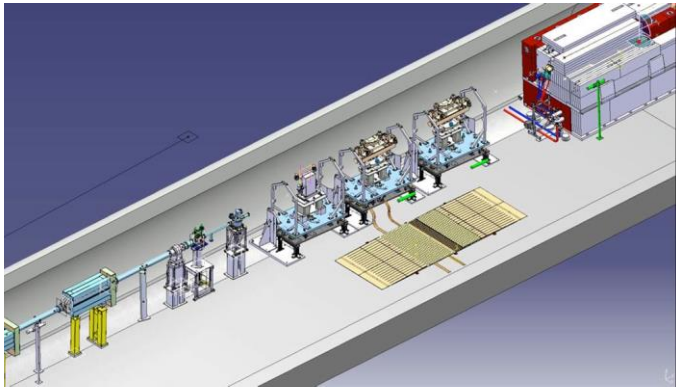
- To help reliably operate accelerator beam windows and secondary particle production targets without having to compromise particle production efficiency by limiting beam parameters in future multi-MW accelerator facilities
- Further understand the thermal shock response and identify material failure limits of conventional and novel materials used as beam windows/targets

## Objectives

- In-beam test to induce thermal shock in materials under controlled conditions at very high localized strain rates and temperatures
- Explore and identify onset of failure modes and potential material thermal shock limits
- Acquire real-time dynamic thermomechanical response of materials to help validate highly non-linear numerical simulations

# HiRadMat facility at CERN

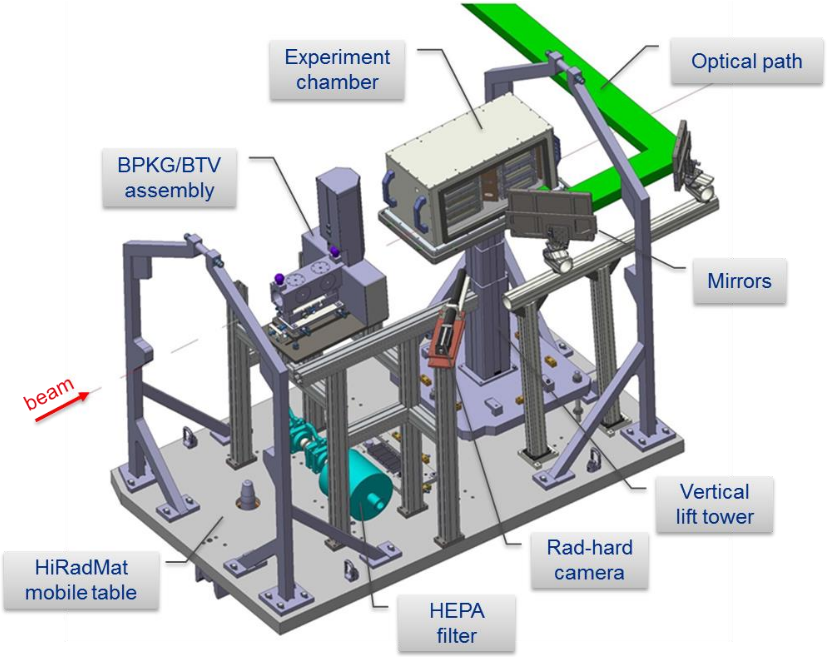


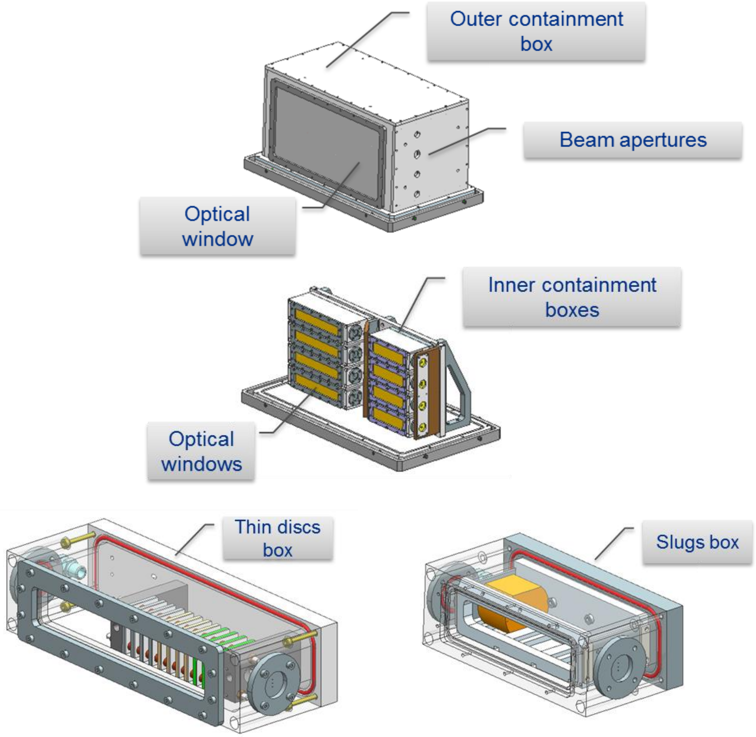




| Beam Parameters | |
|---|---|
| Beam energy | 440 GeV |
| Max. bunch intensity | 1.2 x $10^{11}$ |
| No. of bunches | 1 – 288 |
| Max. pulse intensity | 3.5 x $10^{13}$ ppp |
| Pulse length | 7.2 µs |
| Gaussian beam size | 1σ: 0.1 – 2 mm |

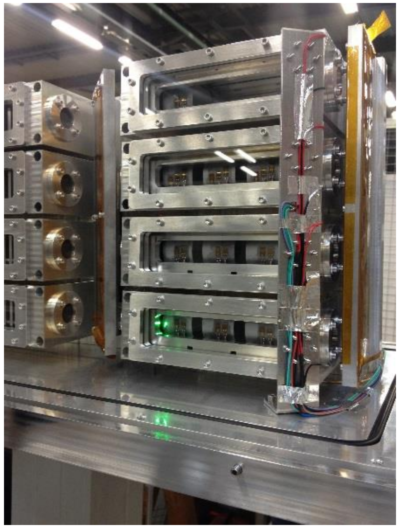
# BeGrid1 (HRMT24) – experimental set-up

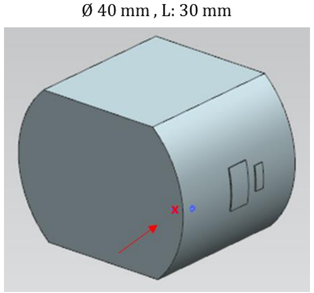
- Experiment successfully completed in September 2015
- Consisted of four specimen arrays of thin Beryllium discs and slugs
  - Various grades (S200F, S200FH, PF60, S65F) and thicknesses
  - Real time measurements of temperature, strain and displacement
  - PIE of thin disc specimen at the University of Oxford



# BeGrid1 (HRMT24) – online results



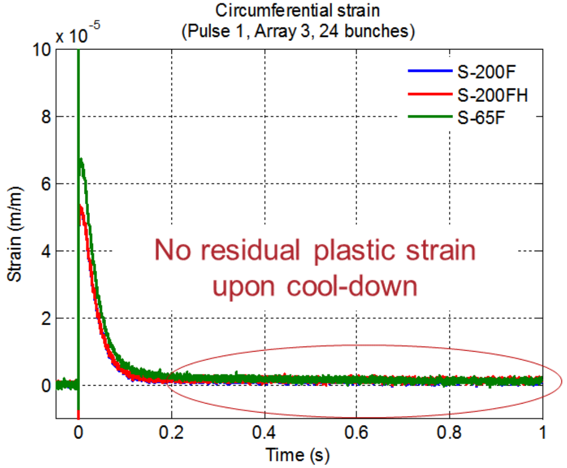



Real-time thermomechanical measurements

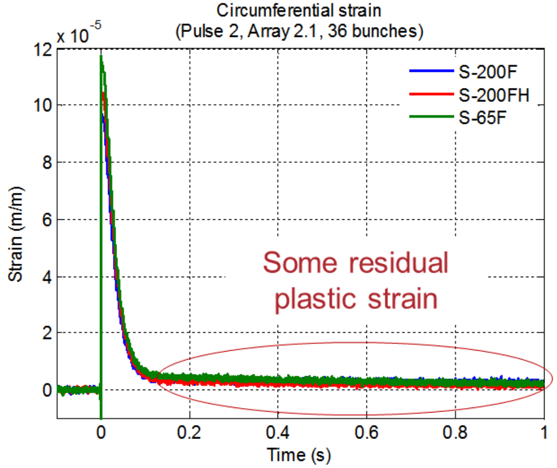
- Instrumented Be slugs in downstream containment boxes
- LDV for radial displacement measurements
- Strain and temperature gages

- ➢ Distinctive strain response for the three different Be grades
- ➢ Residual plastic strain observed upon cool-down
- ➢ Beam alignment/position effects on measurements not very clear

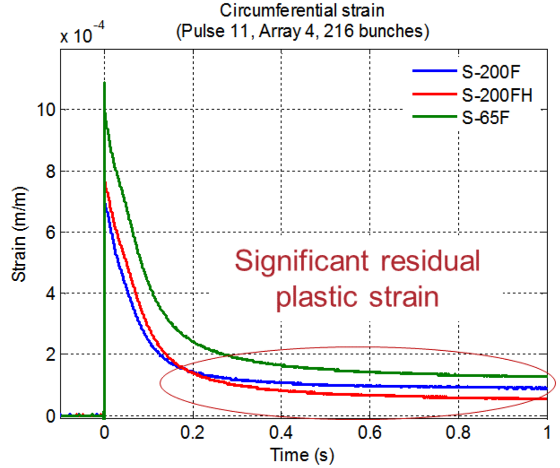
Array 3 – 24 bunches, 3.2e12 POT




Array 2 – 36 bunches, 4.7e12 POT

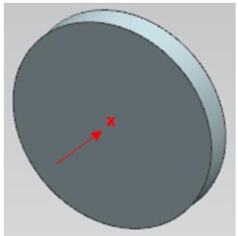



Array 4 – 216 bunches, 2.8e13 POT



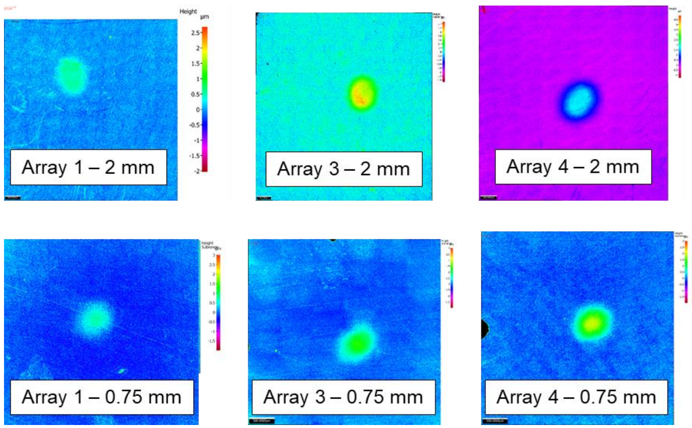
# BeGrid1 (HRMT24) – PIE results

Ø 15 mm, t: 0.25, 0.75, 2 mm



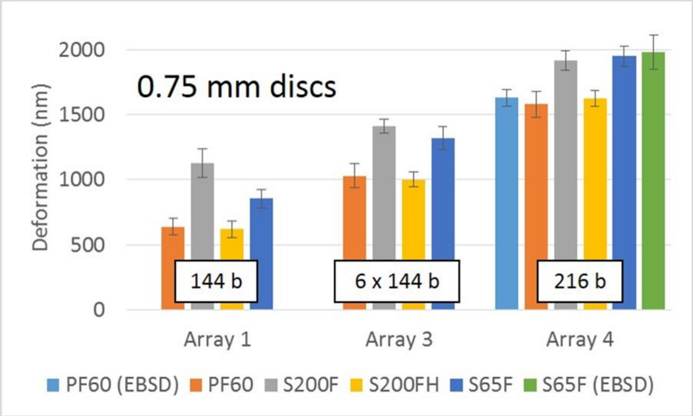

- Thin disc specimen PIE performed at University of Oxford
- Optical microscopy and profilometry to measure out-of-plane plastic deformations

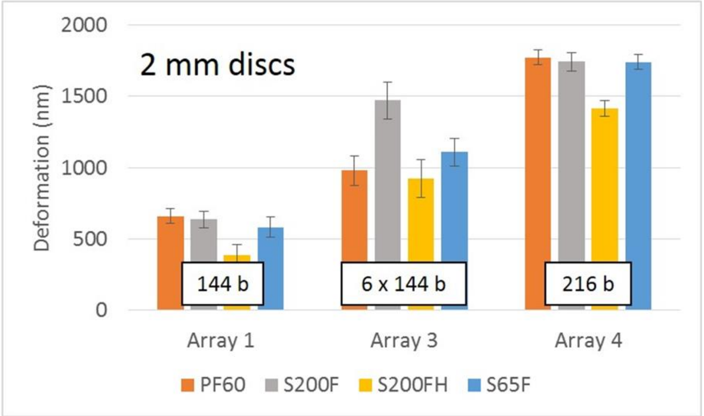
S-65F grade specimens




V. Kuksenko (University of Oxford)

- ➢ All Be grades showed less plastic deformation than predicted by generic strength models
- ➢ S200FH showed least plastic deformation, in agreement with empirical strength model
- ➢ Observed plastic strain ratcheting in Array 3
- ➢ Glassy carbon windows survived without signs of degradation



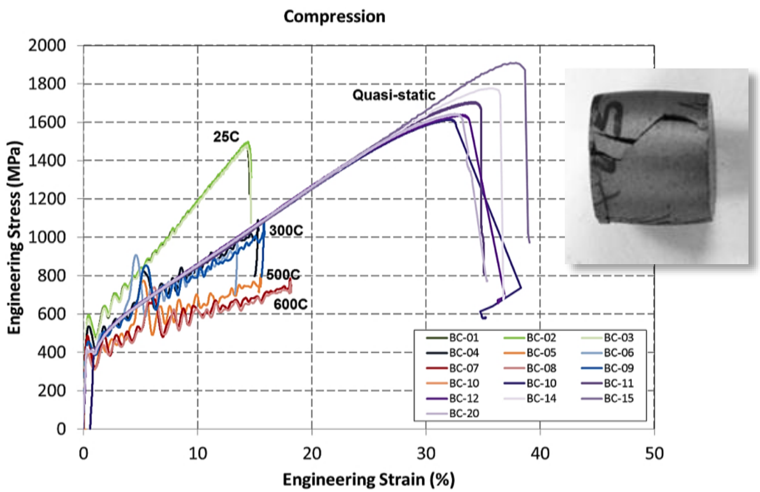



# BeGrid1 (HRMT24) – data analysis

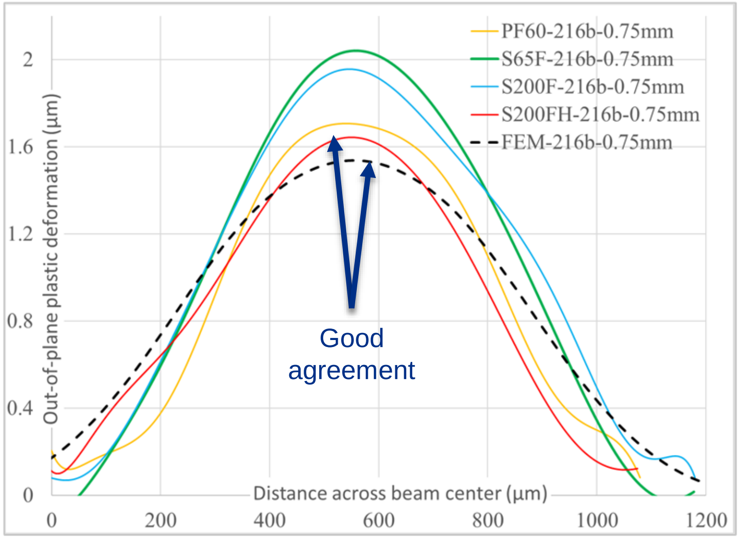
Beryllium S-200FH Johnson-Cook strength model

- Strength model parameters developed by Southwest Research Institute (SwRI)
- High strain rate and elevated temperature testing with Split-Hopkinson pressure bar

$$\sigma_Y = \left[A + B(\varepsilon_{eff}^{p})^n\right][1 + C ln\dot{\varepsilon}^*][1 - T_H^m]$$




S-200FH 0.75 mm thick specimen




S. Bidhar (FNAL)

- ➢ HRMT24 completed successfully and safely
  - ➢ No containment breach and safe containment box disassembly/shipment to Oxford
  - ➢ Valuable PIE results and validation of strength model

# BeGrid2 (HRMT43) objectives

Beam time allocated for Oct. 1st week (2018)

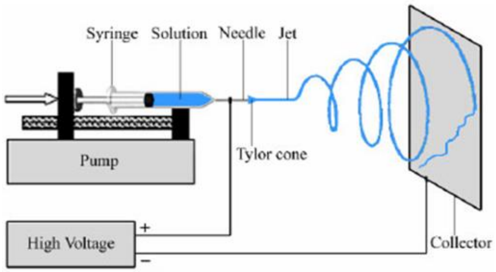
- Follow-up of BeGrid1 (HRMT24) experiment to expose Beryllium to even higher beam intensities than what was achieved in HRMT24
- Identify thermal shock response differences between non-irradiated and previously irradiated material specimens (Be, C, Ti, Si)
- Explore novel materials such as metal foams and electrospun fiber mats to evaluate their resistance to thermal shock and suitability as target materials
- Real-time measurement of dynamic thermomechanical response of graphite slugs in an effort to benchmark numerical simulations

# BeGrid2 (HRMT43) – experimental specifications

- Four specimen arrays exposed to varying beam intensities (σ: 0.25 mm, 1.3e11 ppb)

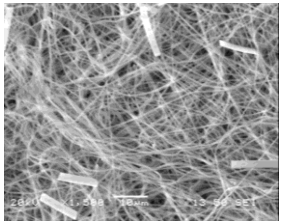
- Various material specimens and grades of varying thicknesses
  - Beryllium, graphite, glassy carbon, titanium alloys, silicon, SiC-coated graphite, foam materials (C, SiC) and electrospun fiber mats ($Al_2O_3$, $ZrO_2$)

Electrospinning concept



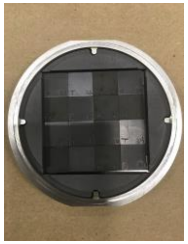

As-spun $Al_2O_3$-pvp mat

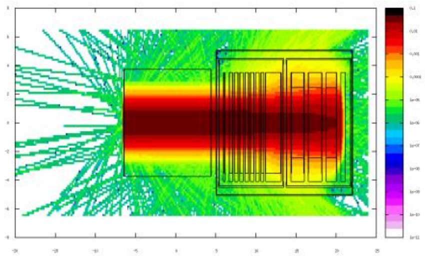
SEM: as-spun $Al_2O_3$



S. Bidhar (FNAL), "Improvised electrospinning set-up for thicker ceramic nanofiber mat for high power target", HPTW poster, June 5, 2018

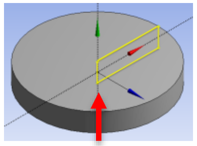
- Previously proton-irradiated specimens (BNL BLIP with 180 MeV protons)
  - Beryllium, graphite, glassy carbon, titanium alloys, silicon





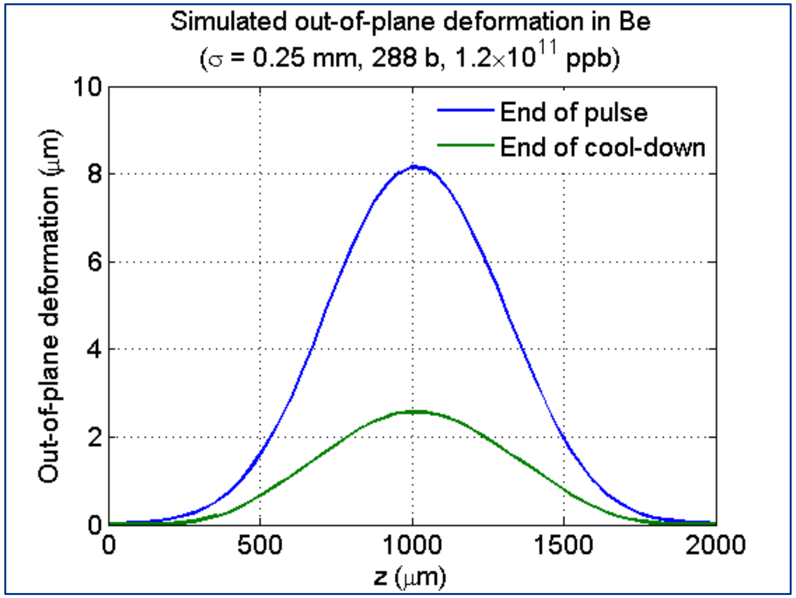
- Real-time measurements of temperature, strain and vibration of graphite slugs

# Numerical simulations of beam induced strain/stress




## Beryllium

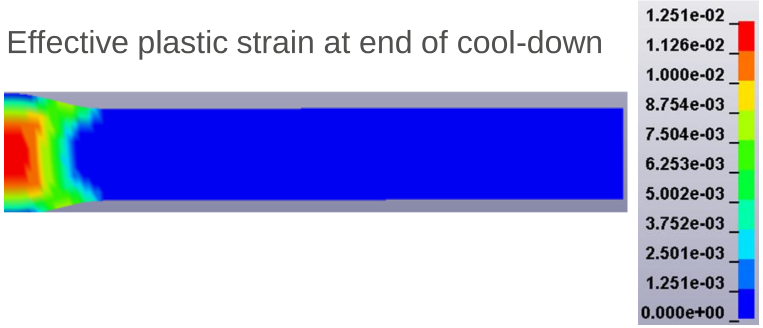
- Peak temperature jump ~1000 °C (MARS/FEA)




- Out-of-plane plastic deformation will be measured with profilometer during PIE work
- Peak effective plastic strain: 1.25 %
  - Failure plastic strain from literature: 1-2%

LS-DYNA (Johnson-Cook strength model)

Effective plastic strain at end of cool-down

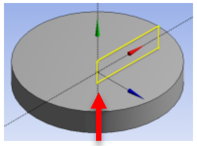



**Irradiated BLIP Be specimens: ~0.03 DPA**

- Explore effect of radiation damage on thermal shock response
- Reduced ductility expected (~100 appm He)

Specimen size
12 mm x 9 mm x 0.75 mm

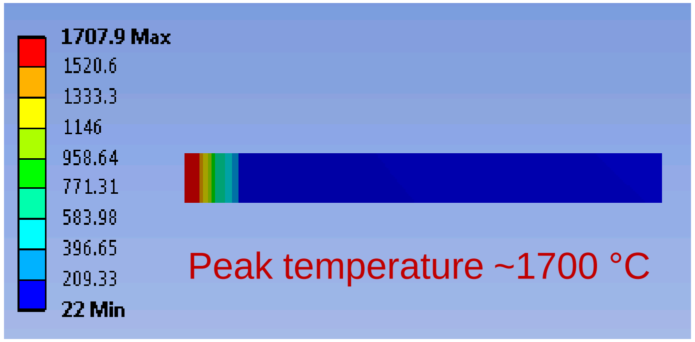
# Numerical simulations of beam induced strain/stress




Graphite

- Peak beam intensity: σ = 0.25 mm, 288 x 1.2e11 ppb (3.5e13 ppp)
- Temperature dependent thermal/mechanical properties
- UTS: 79 MPa, UCS: 175 MPa (POCO ZXF-5Q)

**2D axisymmetric ANSYS model**

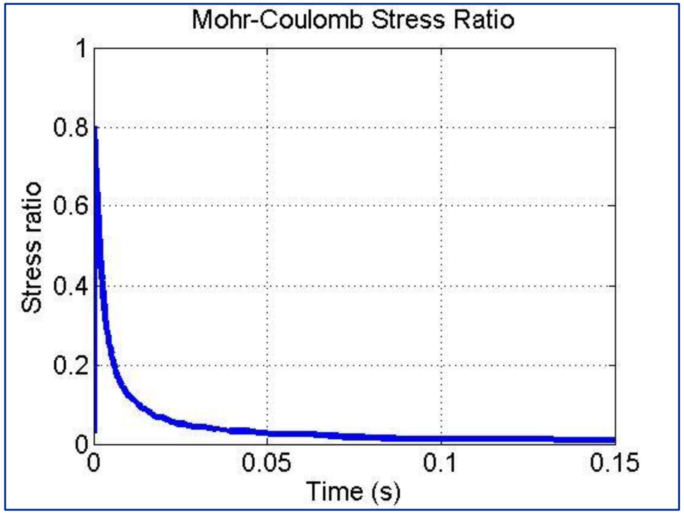



- Stress ratio (0.8) highest at end of beam pulse, then drops back to zero as specimen cools down

- **BLIP graphite specimens: ~0.05 DPA**
  - 10.5 mm x 10.5 mm x 0.5 mm

**Mohr-Coulomb failure criterion**

$$\frac{\sigma_1}{S_{tensile}} + \frac{\sigma_3}{S_{compressive}} < 1$$



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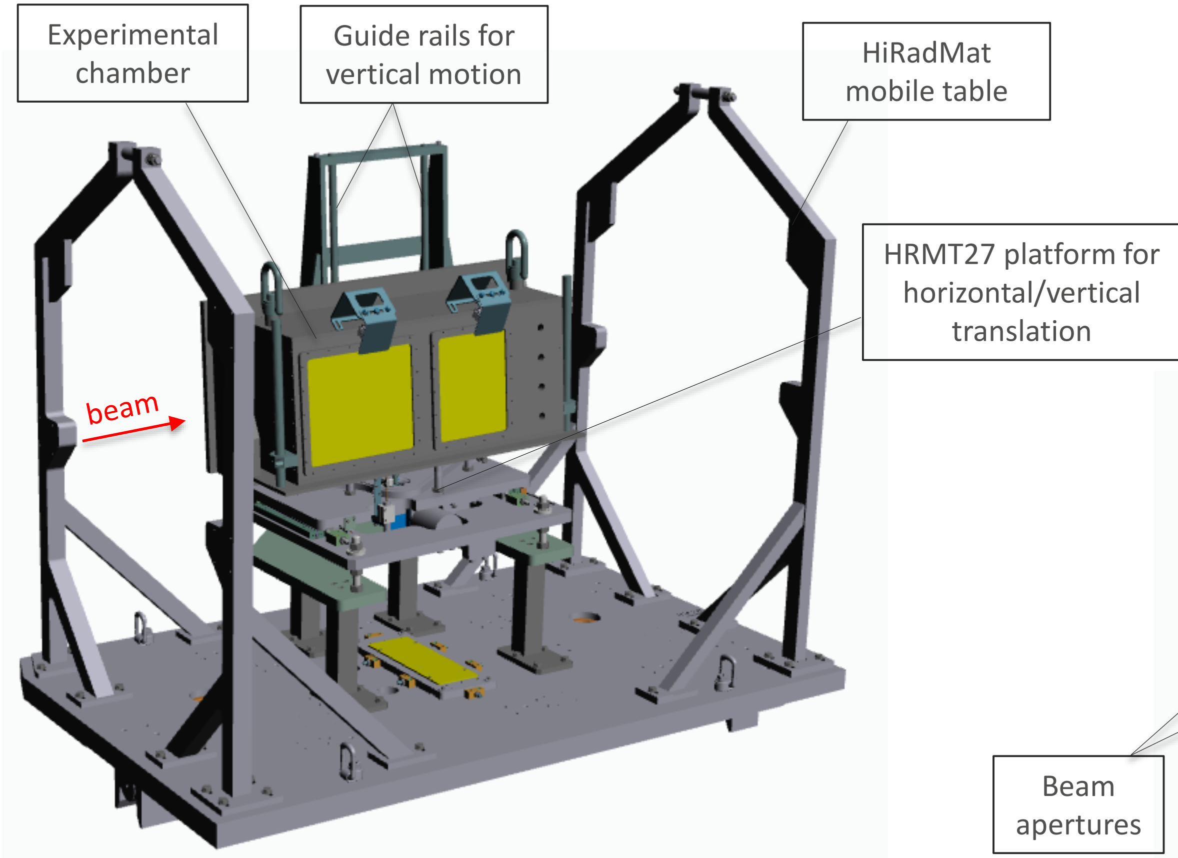
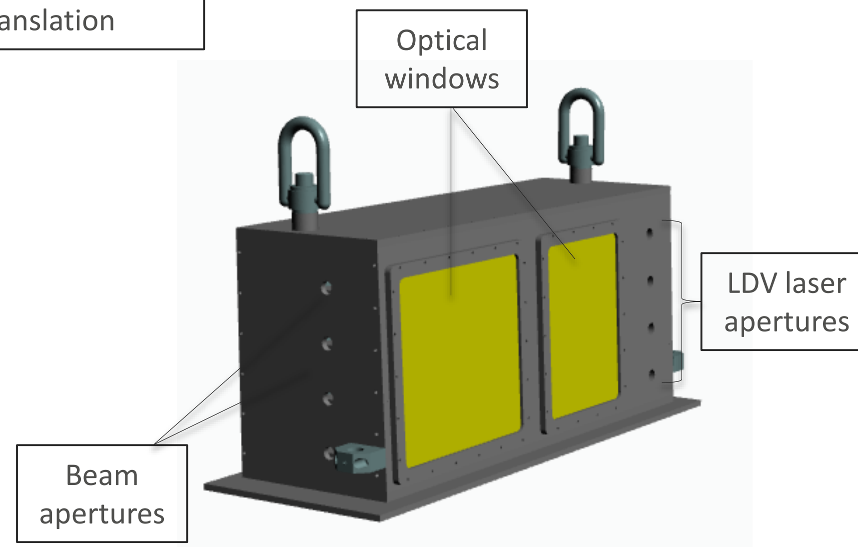
# BeGrid2 (HRMT43) – experimental set-up

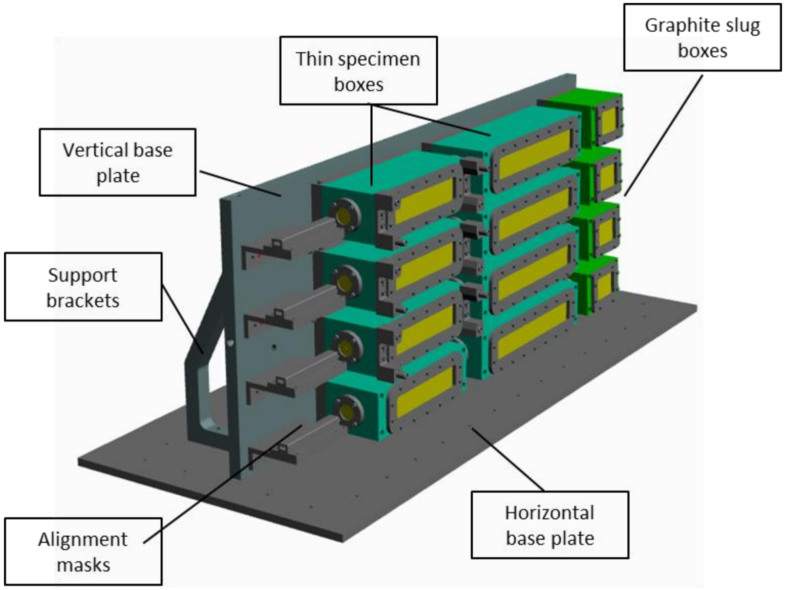



**Outer box dimensions**
Length: 83 cm
Width: 25 cm
Height: 36 cm
Mass: ~70 kg




- Re-use of HRMT27 experimental platform with some modifications
- Outer chamber kept at slightly negative pressure using vacuum cleaner equipped with HEPA filter
- Optical viewports for visual monitoring with rad-hard camera

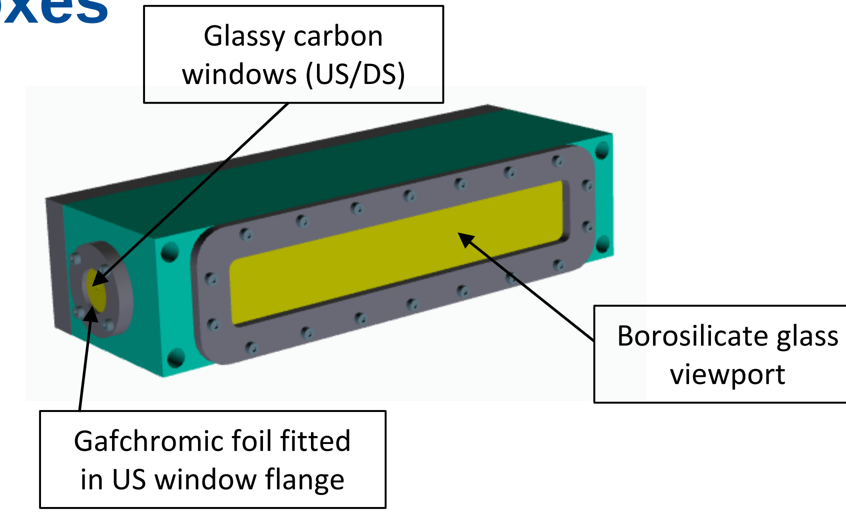
# BeGrid2 (HRMT43) – specimen arrays




- Four arrays separated vertically
  - Irradiated and non-irradiated specimens in separate inner boxes
  - Slug inner boxes at downstream end
- TZM alignment mask upstream of each array
  - Used to center beam on array (similar to the CuCrZr mask in HRMT18)
- Remote handling features for irradiated specimen boxes
- Optical viewports for visual and LDV access

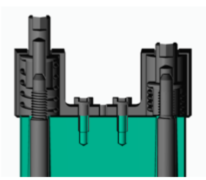
| Beam Pulse List | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **No** | Intensity | | | Beam spot [mm] | | Bunch spacing [ns] | Pulse length [us] | Target |
| | # bunches | p/bunch | Total | Sigma_x | Sigma_y | | | |
| **1** | 144 | 1.30E+11 | 1.87E+13 | 0.25 | 0.25 | 25 | 3.6 | Array 4 |
| **2** | 144 | 1.30E+11 | 1.87E+13 | 0.25 | 0.25 | 25 | 3.6 | Array 4 |
| **3** | 144 | 1.30E+11 | 1.87E+13 | 0.25 | 0.25 | 25 | 3.6 | Array 4 |
| **4** | 144 | 1.30E+11 | 1.87E+13 | 0.25 | 0.25 | 25 | 3.6 | Array 4 |
| **5** | 144 | 1.30E+11 | 1.87E+13 | 0.25 | 0.25 | 25 | 3.6 | Array 4 |
| **6** | 216 | 1.00E+11 | 2.16E+13 | 0.25 | 0.25 | 25 | 5.4 | Array 3 |
| **7** | 216 | 1.30E+11 | 2.81E+13 | 0.25 | 0.25 | 25 | 5.4 | Array 2 |
| **8** | 288 | 1.30E+11 | 3.74E+13 | 0.25 | 0.25 | 25 | 7.2 | Array 1 |

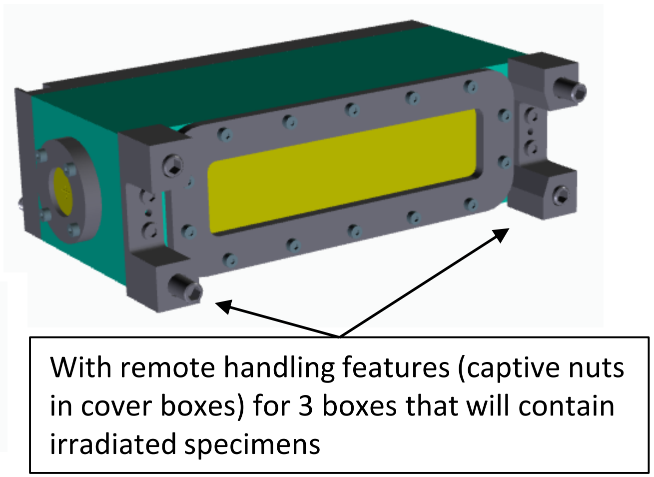
# BeGrid2 – inner containment boxes

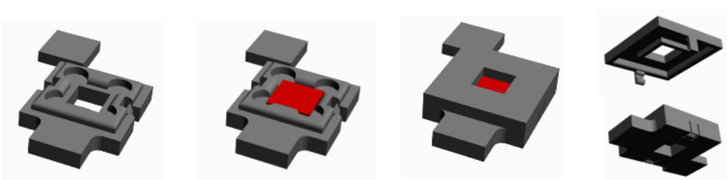


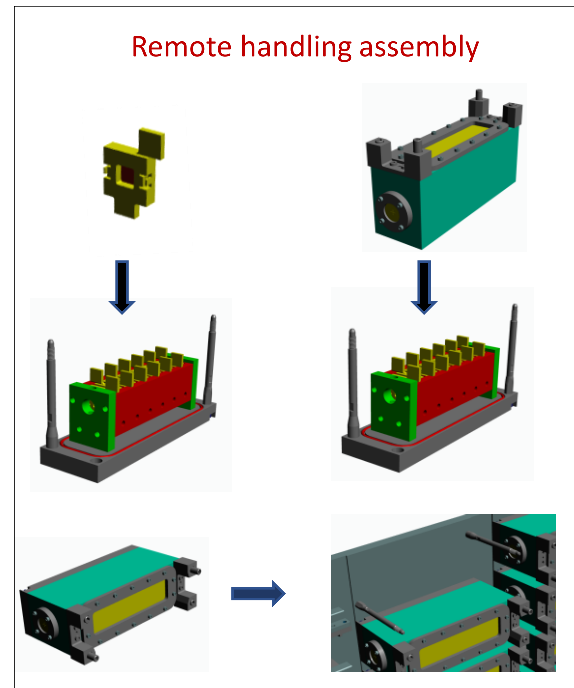

<u>Thin specimen inner containment box designs</u>

- Hermetically-sealed in air to fully contain specimens and avoid contamination release





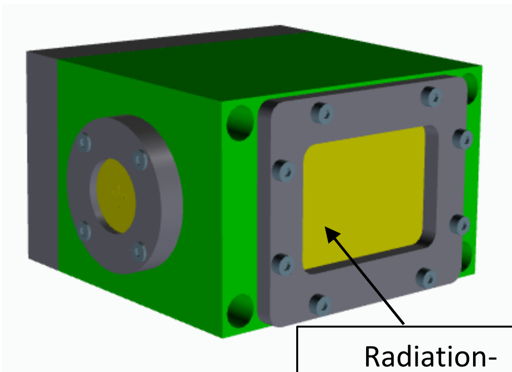



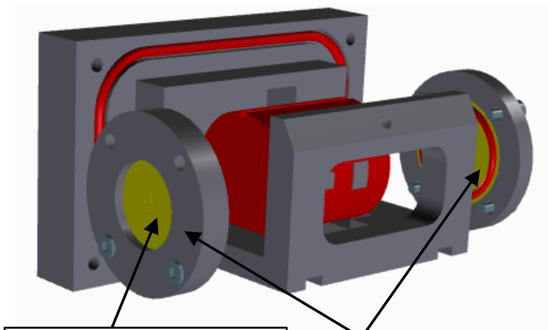


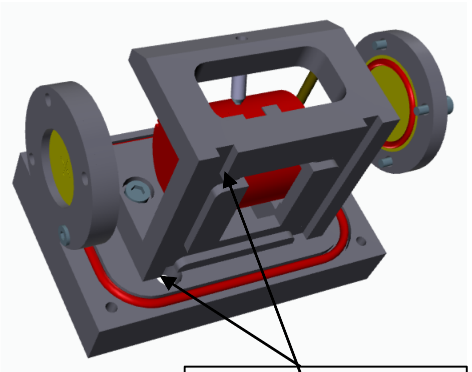

- Irradiated specimens currently at BNL
- Will be shipped to PNNL for assembly in holders and potentially inner containment boxes
- Shipment to CERN for final assembly to vertical base plate

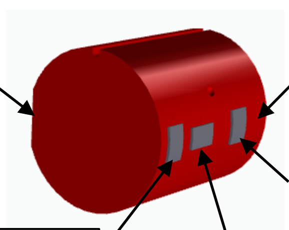
# BeGrid2 (HRMT43) – graphite slugs

## Graphite slug containment box



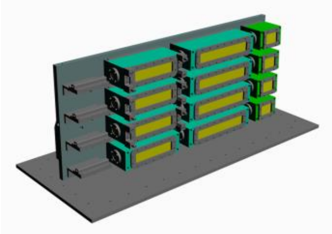




- POCO ZXF-5Q graphite slug
- Slug aligned so that beam is 5 mm away from cylindrical edge
- Strain, temperature and radial displacement measurements will be acquired
- Also useful as diagnostic tool during experiment to verify beam impact on targets

# Test matrix



## **Array 1 -** 1 x 288 bunches (3.74 × $10^{13}$ protons, 1.3 × $10^{11}$ ppb)

| ARRAY | Irradiated Specimens | | | | | | | | | | | | | Non-irradiated Specimens | | | | | | | | | | | | | | | | | | | Slug |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | PF-60 | S-65F | S-200F | S-200FH | GC20 | IG-430 | ~~ZXF-5Q~~ | ~~GC20 Lower dose~~ | ~~IG-430 Lower dose~~ | ~~ZXF-5Q Lower dose~~ | Silicon | Silicon | | PF-60 (1) | PF-60 (2) | S-65F (1) | S-65F (2) | S-200F (1) | S-200FH (1) | GC20 (1) | GC20 (2) | ZXF-5Q (1) | ZXF-5Q (2) | IG-430 (1) | IG-430 (2) | RVC (1) | RVC (2) | Silicon | Silicon | FREE | FREE | | ZXF-5Q |
| t (mm) | 0.75 | 0.75 | 0.75 | 0.75 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 1 | 1 | | 0.75 | 0.75 | 0.75 | 0.75 | 0.75 | 0.75 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 1 | 1 | 1 | 1 | | | | 35 |

## **Array 2 -** 1 x 216 bunches (2.81 × $10^{13}$ protons, 1.3 × $10^{11}$ ppb)

| ARRAY | Irradiated Specimens | | | | | | | | | | | | | Non-irradiated Specimens | | | | | | | | | | | | | | | | | | | Slug |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 | PF-60 | S-65F | S-200F | S-200FH | GC20 Lower dose | IG-430 Lower dose | ~~ZXF-5Q~~ | Silicon | Silicon | FREE | FREE | FREE | | PF-60 (1) | PF-60 (2) | S-65F (1) | S-65F (2) | S-200F (1) | S-200FH (1) | GC20 (1) | GC20 (2) | ZXF-5Q (1) | ZXF-5Q (2) | IG-430 (1) | IG-430 (2) | RVC (1) | RVC (2) | Silicon | Silicon | FREE | FREE | | ZXF-5Q |
| t (mm) | 0.75 | 0.75 | 0.75 | 0.75 | 0.5 | 0.5 | 0.5 | 1 | 1 | | | | | 0.75 | 0.75 | 0.75 | 0.75 | 0.75 | 0.75 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 1 | 1 | 1 | 1 | | | | 35 |

## **Array 3 -** 1 x 216 bunches (2.16 × $10^{13}$ protons, 1.0 × $10^{11}$ ppb)

| ARRAY | Irradiated Specimens | | | | | | | | | | | | | Solid, foam and novel Materials | | | | | | | | | | | | | | | | | | | Slug |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | Ti alloy | Ti alloy | SiC-graphite | FREE | FREE | FREE | FREE | FREE | FREE | FREE | FREE | FREE | | SiC solid (1) | SiC solid (2) | SiC foam (1) | SiC foam (2) | Al2O3 solid (1) | Al2O3 solid (2) | ZrO2 solid (1) | ZrO2 solid (2) | Al2O3 spun (1) | Al2O3 spun (2) | ZrO2 spun (1) | ZrO2 spun (2) | Ti alloy | Ti alloy | SiC-graphite | FREE | FREE | FREE | | ZXF-5Q |
| t (mm) | 0.5 | 0.5 | 1 | | | | | | | | | | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 0.5 | 0.5 | 1 | | | | | 35 |

## **Array 4 -** 5 x 144 bunches (9.36 × $10^{13}$ protons, 1.3 × $10^{11}$ ppb)

| ARRAY | Novel Materials | | | | | | | | | | | | | Solid & foam Materials | | | | | | | | | | | | | | | | | | | Slug |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | Al2O3 spun (1) | Al2O3 spun (2) | ZrO2 spun (1) | ZrO2 spun (2) | FREE | FREE | FREE | FREE | FREE | FREE | FREE | FREE | | SiC solid (1) | SiC solid (2) | SiC foam (1) | SiC foam (2) | Al2O3 solid (1) | Al2O3 solid (2) | ZrO2 solid (1) | ZrO2 solid (2) | Al-Be solid | Al-Be solid | FREE | FREE | FREE | FREE | FREE | FREE | FREE | FREE | | ZXF-5Q |
| t (mm) | 1 | 1 | 1 | 1 | | | | | | | | | | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | | | | | | | | 35 |

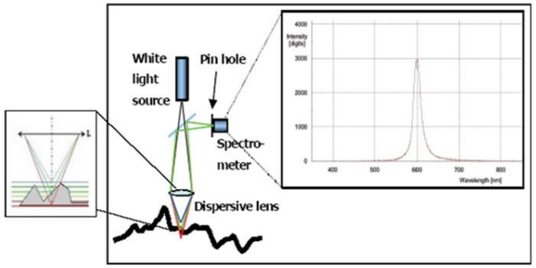
# BeGrid2 (HRMT24) – PIE work

## Thin specimens

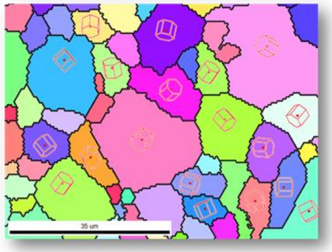
Inner containment boxes will be shipped, after sufficient cool-down time, to Culham Center for Fusion Energy (CCFE) laboratory in UK for PIE work

- Profilometry or Atomic Force Microscopy (AFM) to measure plastic out-of-plane deformations
- Scanning Electron Microscopy (SEM) for identifying local deformations/cracks
- Electron Backscatter Diffraction (EBSD) to map grain structure/orientation and crack analysis
- 3D FIB-SEM for 3D analysis of crack morphology if needed

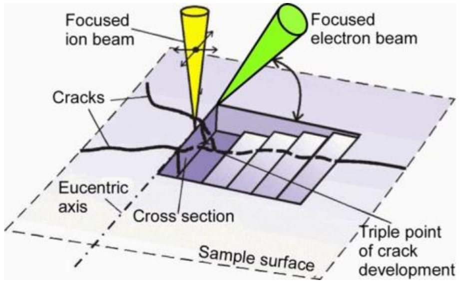
Profilometry




EBSD



FIB-SEM




## Graphite slug specimens

- Analysis of real-time recorded data
- Benchmark and validate POCO ZXF-5Q numerical simulations by comparing with experimental measurements

# Conclusions

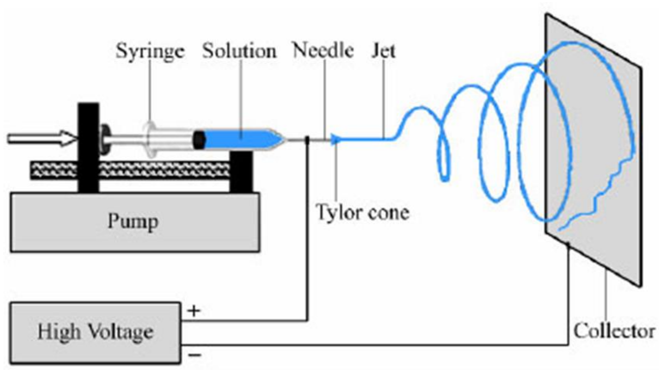
- BeGrid2 will build upon BeGrid1 experiment to expose specimens to even higher beam intensities and thermal shock conditions
- Direct comparison of thermal shock response between non-irradiated and irradiated material specimens
- Evaluate potential novel materials for secondary particle-production targets
- Benchmark and validate numerical simulation and material strength models

Many thanks to HiRadMat facility/team for providing us the opportunity to perform in-beam material/component tests to replicate actual operating conditions of high-energy high-power target facilities

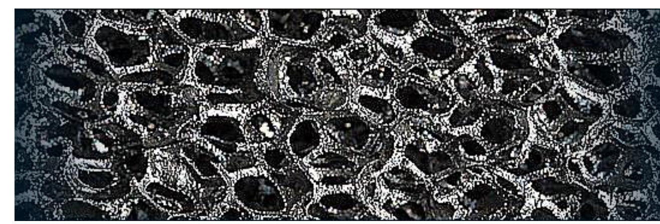
# Back-up slides

# Novel materials

- Electro-spun and foam materials
  - Local damage of fibers do not affect structural integrity as a whole
  - Reduced temperature gradient limited to fibers
  - High surface area and gaps: improved cooling by flowing gas
- In-beam test to evaluate thermal shock response of foam/fiber mats compared to solid material



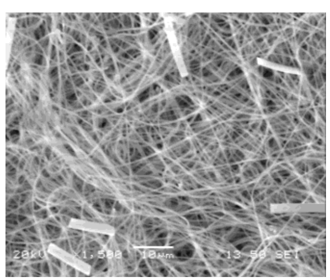

Reticulated vitreous carbon foam



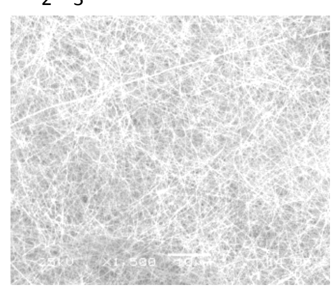
SiC foam

As-spun alumina-pvp mat

SEM: as-spun $Al_2O_3$

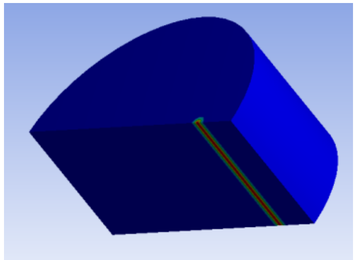


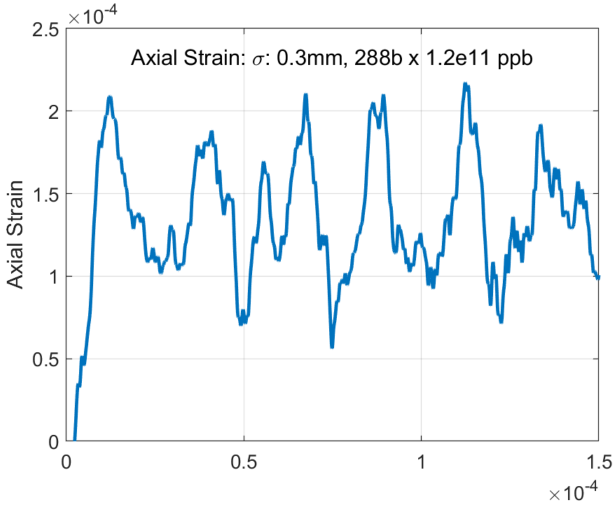
$Al_2O_3$ SEM after heat treatment

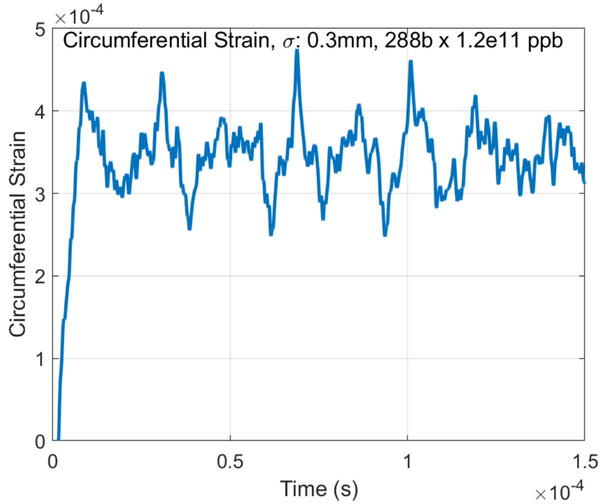


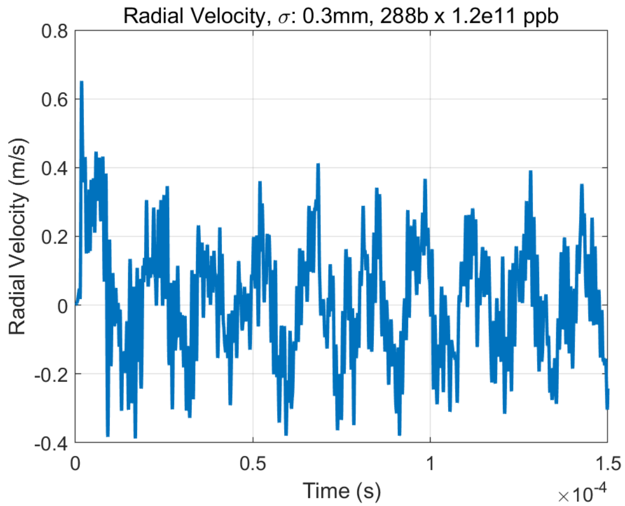
# Graphite slugs updated FEA results

- Graphite slug, D: 30 mm, L: 30 mm
- Beam spot 5 mm away from cylindrical surface
- σ = 0.3 mm, 288b x 1.2e11 ppb





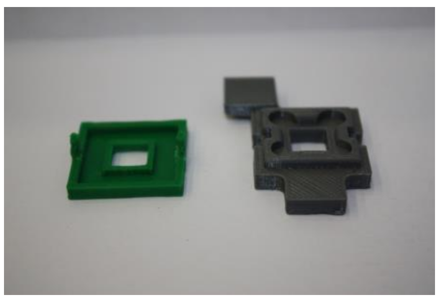



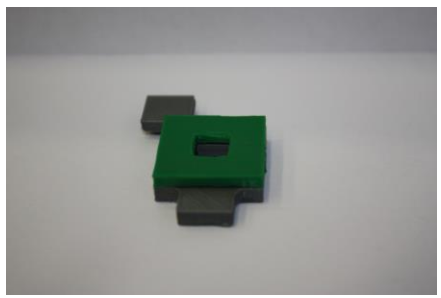

- Axial strain range: 50 – 220 με
- Circumferential strain: 250 – 475 με
- Radial velocity: -0.4 – 0.4 m/s

# ULTEM 9085 specimen holder

- Proposing to 3D print specimen holders in ULTEM 9085 (family of polyetherimide – PEI)
- Handle to enable easy handling
- Snap-fit to close/secure specimen in holder
- Separate fixture will be used to open holder by pressing down on handles

<u>3D-printed prototype</u>






- **CERN 98-01 report: compilation of radiation damage test data**
  - PEI fully safe up to 5 MGy
  - Mechanical properties relatively constant up to 100 MGy (ULTEM1000)

- **Glass transition temperature of PEI: 217 °C (heat deflection T: 150 °C)**
  - Maximum temperature rise due to heat conduction from specimens: 17°C (peak intensity pulse)
  - Peak temperature rise from direct alignment beam interaction (1.3e11 protons): 8 °C

# Instrumentation list

| Instrumentation | Purpose | Quantity |
|---|---|---|
| Resistive strain gages | Measurement of axial and circumferential strain on cylindrical surface of graphite slugs | 8 |
| Temperature sensors | Temperature measurement of cylindrical surface of graphite slugs | 4 |
| LDV | Radial velocity and displacement of cylindrical surface of graphite slugs | 1 |
| Radiation-hard camera | Visual observation of experimental test rig | 1 |
| LED lighting | Provide illumination for acquiring camera images | 2 |
| Vacuum cleaner equipped with HEPA filter | Pumping down on outer chamber to maintain slightly lower pressure than atmosphere | 1 |
| Horizontal/vertical translation stage | Align specimen arrays to beam | 1 |

# BLIP irradiated specimens dose rates

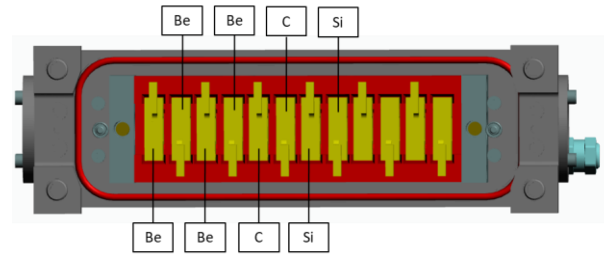
| Material | Dose rate/specimen (mSv/hr @ 1 ft) |
| --- | --- |
| Beryllium | 0.028 |
| Graphite & Glassy carbon | 0.036 |
| Titanium (DS Ti1 capsule) | 0.054 |
| Silicon | 0.115 |

Total dose rate from all 3 irradiated specimen boxes: **0.936 mSv/hr @ 1ft**

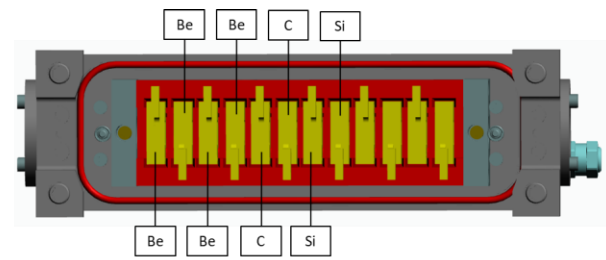
**Conservative MARS dose rate estimate**

- All specimens assumed to be lumped together
- No self shielding
- No aluminum boxes shielding

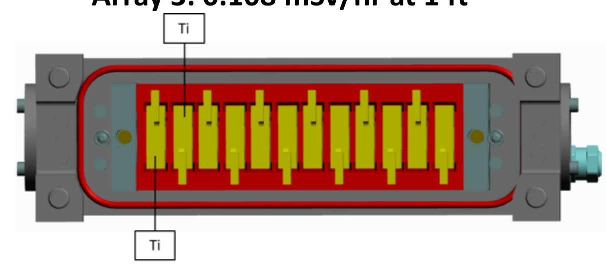
**Array 1: 0.414 mSv/hr at 1 ft**




**Array 2: 0.414 mSv/hr at 1 ft**




**Array 3: 0.108 mSv/hr at 1 ft**

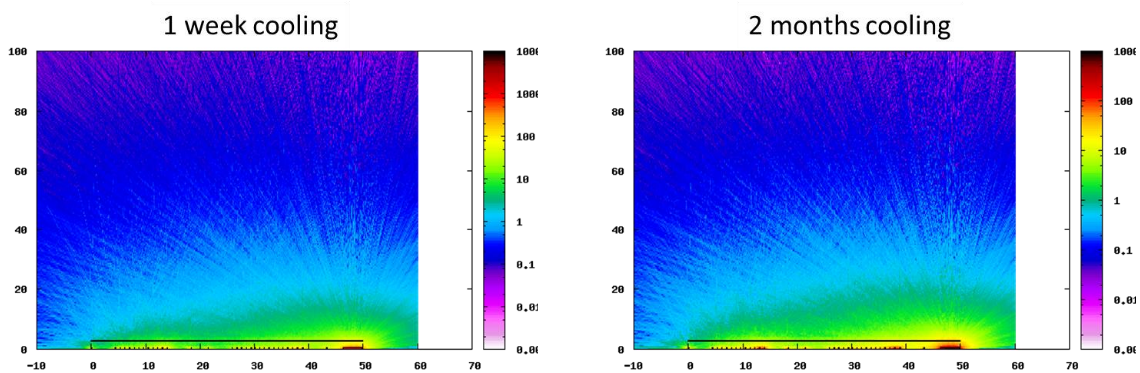


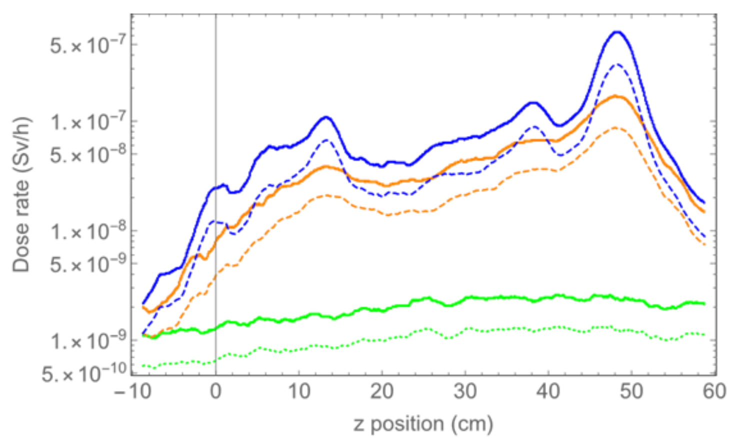
# Residual activity from HiRadMat beam

- FLUKA analysis of residual dose induced by HiRadMat beam pulses
- Analysis does not include dose rate from pre-irradiated specimens

<u>Array 1</u>

3.45e13 protons



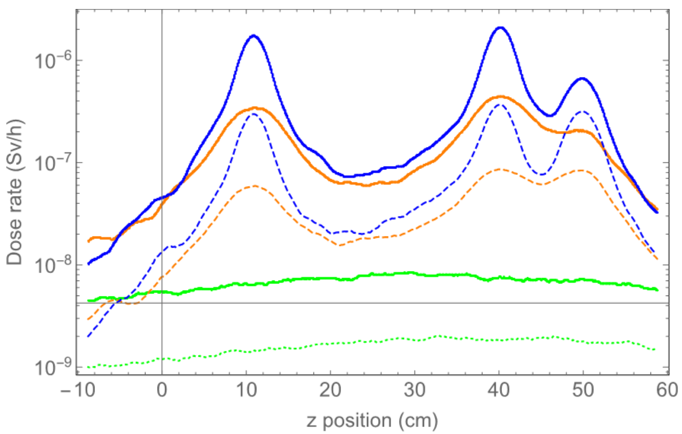




Blue: on contact with specimens
Orange: on contact with inner box
Green: 1 ft away from inner box surface

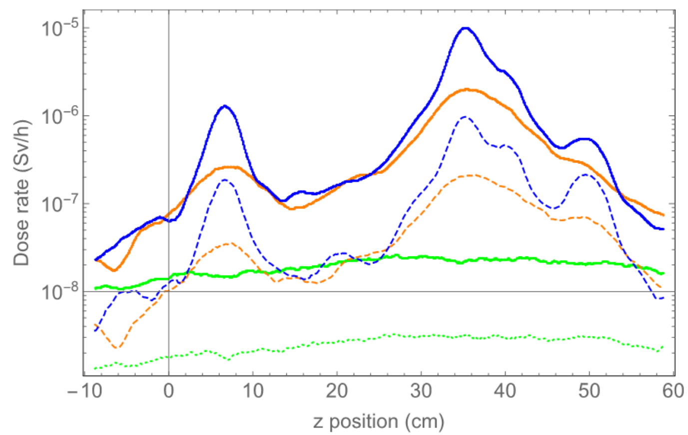
**Solid line**: 2 weeks cool-down
**Dashed line**: 2 months cool-down

# Residual activity from HiRadMat beam

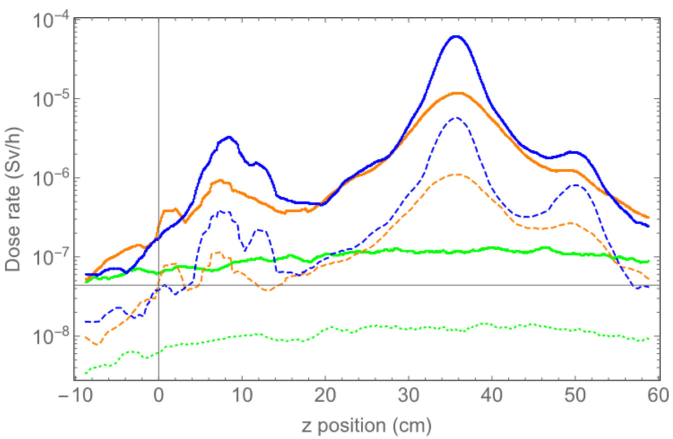
## Array 2
2.59e13 protons




## Array 3
2.02e13 protons




## Array 4
8.64e13 protons




Blue: on contact with specimens
Orange: on contact with inner box
Green: 1 ft away from inner box surface

**Solid line**: 2 weeks cool-down
**Dashed line**: 2 months cool-down

- More comprehensive FLUKA analysis including entire test rig geometry will be performed