RaDIATE: Graphite Status Report

Kavin Ammigan[1], Nikolaos Simos[2], Frederique Pellemoine[3]

[1] Fermi National Accelerator Laboratory
Batavia, IL 60510, USA

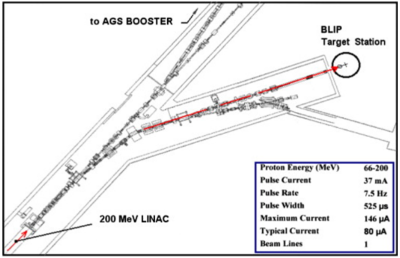
[2] Brookhaven National Laboratory
Upton, NY 11973, USA

[3] Michigan State University, FRIB
East Lansing, MI 48824, USA

RaDIATE Collaboration Meeting
19 May 2014

BNL BLIP irradiation experiment

- Proton energy $\sim$ 180 MeV
- Irradiation performed in tandem with isotope production
- 9 weeks of irradiation of various graphite grade and C/C composite specimens
- PIE at BLIP target processing laboratory



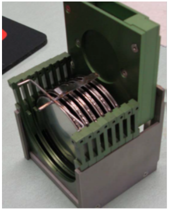
Irradiated graphite grades

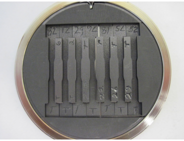
Graphite grades

- POCO
- IG-430
- SGL R7650
- C2020
- 3D C/C composite

Irradiation parameters

- Proton energy $\sim$ 180 MeV
- $\sigma_x \sim$ 10 mm, $\sigma_y \sim$ 7 mm
- Peak DPA $\sim$ 0.1
- Peak temperature $\sim$ 200 $^{\circ}$C

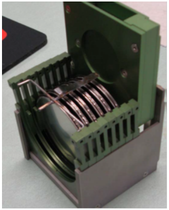




Irradiated graphite grades

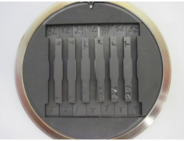
Graphite grades

- POCO
- IG-430
- SGL R7650
- C2020
- 3D C/C composite

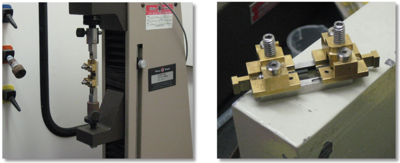


Irradiation parameters

- Proton energy $\sim$ 180 MeV
- $\sigma_x \sim$ 10 mm, $\sigma_y \sim$ 7 mm
- Peak DPA $\sim$ 0.1
- Peak temperature $\sim$ 200 °C

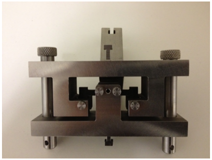


PIE equipment

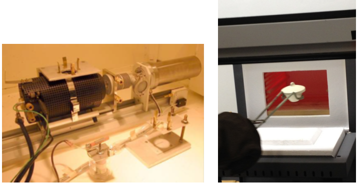


Tensile tester and specimen fixture

Ultrasonic system



3/4 point flexural test fixture



Dilatometer and high temperature furnace

PIE status and recent results

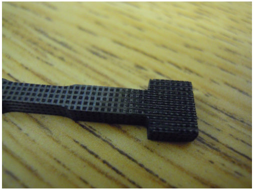
✓ Tensile and thermal tests already performed on various graphite grades[1]

✓ Additional testing:

- ☞ Mechanical (flexural) tests on 3D C/C specimens
- ☞ Electrical resistivity measurements to evaluate thermal conductivity

[1] LBNE target material radiation damage from energetic protons of the Brookhaven Linear Isotope Production (BLIP) facility, http://www-radiate.fnal.gov/downloads.html

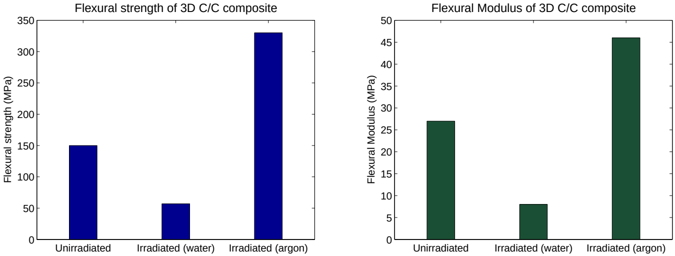
Initial 3D C/C flexural test results



✓ Un-irradiated specimens
  - ☞ Consistent failure at the middle of the gage with 3 point bending
  - ☞ Flexural strength dependent on number of longitudinal fibers along specimen gage

✓ Irradiated specimens

- ☞ Specimens irradiated in both water and argon environment
- ☞ 0.05 DPA
- ☞ $\sim$ 4 fibers along gage



☞ More specimen tests to compare for DPA effects

EDXRD experiment at NSLS

Objective:

- ☞ EDXRD studies of irradiated novel materials and composites under consideration in next generation fusion/fission reactors and high-power accelerators

Irradiated array of materials relevant to LBNE and RaDIATE:

- ☞ Graphite: POCO, IG-430, SGL R7650
- ☞ Carbon composite: 3D C/C, 2D C/C
- ☞ Other: Ti-6Al-4V

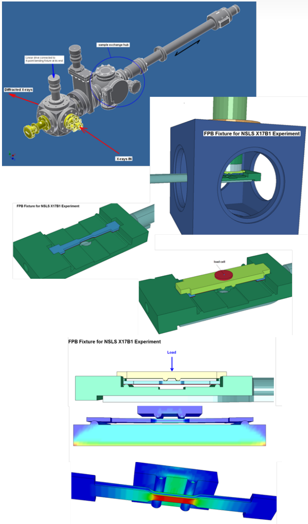
Experiment performed:

- ☞ April 29 - May 6

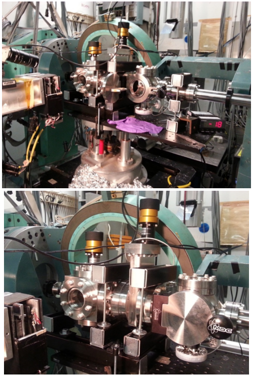
Other potentially relevant materials:

- ☞ Gum metal, Super Invar, Tungsten

EDXRD experimental stage

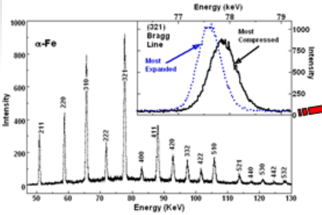


Multi-functional stage capable of handling real size irradiated specimens, under vacuum, four point bending state of stress and eventually heating/annealing via a portable, collimated laser beam



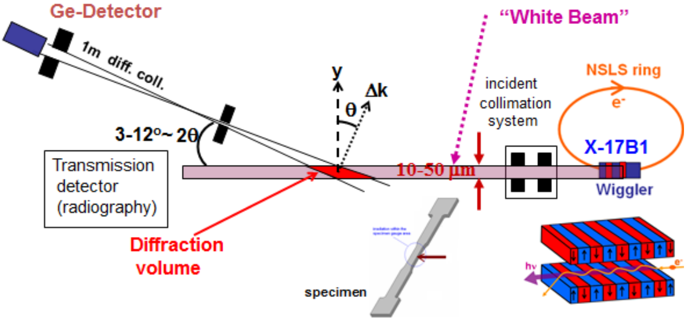
Experimental stage at X17B1 beamline at NSLS

STRAIN MAPPING
Energy Dispersive Diffraction Mode

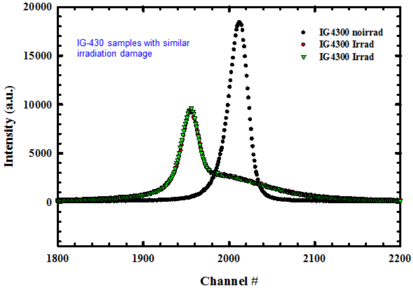


$$E_{hkl}\,[\text{in keV}] = \frac{6.199}{d_{hkl}\sin\theta} \Rightarrow \varepsilon = \frac{\Delta d}{d_0}$$

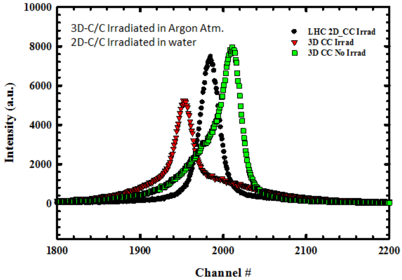
Like having imbedded inter-atomic strain gauges !!!!



Preliminary results



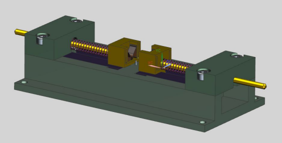
Irradiated graphite (IG-430)

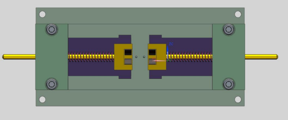


Irradiated C/C composite

Future tests

- Further flexural tests on 3D C/C composite
- Thermal conductivity measurements

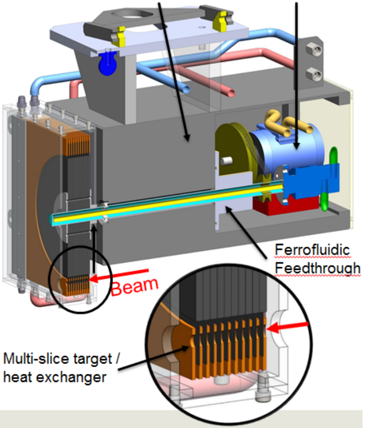




- Next EDXRD experimental window (summer 2014)
  - Monochromatic high energy X-rays
  - Laser induced annealing of specimens

FRIB Production Target
Rotating Multi-slice Graphite Target Design

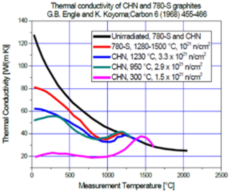
- Rotating multi-slice graphite target chosen for FRIB baseline
  - Multiple rotating target slices
  - Thermal radiation cooling
- Target requirements
  - Up to 100 kW power deposition in 1 mm diameter beam spot
  - Target lifetime 2 weeks to meet experimental program requirements
- Target parameters defined by thermo-mechanical simulations
  - 5000 RPM and 30 cm diameter to limit maximum temperature and amplitude of temperature changes
    - » High temperature: ~ 1900ºC
      - Evaporation of graphite mitigated



FRIB Facility for Rare Isotope Beams
U.S. Department of Energy Office of Science
Michigan State University

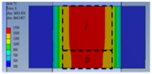
Radiation Damage Studies in Graphite
For Better Lifetime Predictions

- Irradiations by charged heavy ion induce changes of physical properties ⇨ decrease target performance
  - Thermo-mechanical properties (thermal conductivity, tensile and flexural strength)
  - Electronic properties (Resistivity)
  - Structural properties (microstructure and dimensional changes, Swelling)
- Most of the studies were done with neutron and proton irradiation but not a lot of data for heavy ion beams
- How much will annealing help?



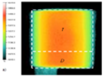
- Two type of polycrystalline graphite (5 and 13 μm grain size) irradiated with Au-beam 8.6 MeV/u
  - Up to $5.6 \cdot 10^{10}$ $cm^{-2}.s^{-1}$, Fluence up to 10^{15} cm^{-2}
  - Samples heated to different temperature

I = 35 A + beam

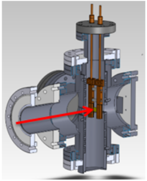


T_{max} = 1600 °C

I = 35 A



T_{max} = 1480 (± 30 °C)

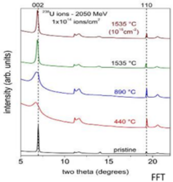


FRIB Facility for Rare Isotope Beams
U.S. Department of Energy Office of Science
Michigan State University

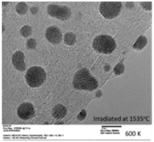
Radiation Damage Studies in Graphite

Annealing of Damage at High Temperature (> 1300ºC)

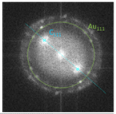
X-Ray Diffraction analyses

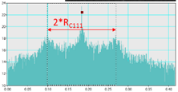


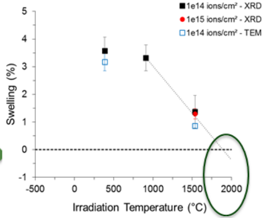
TEM analyses



FFT





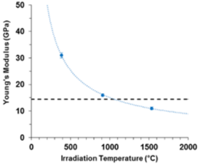


Swelling is completely recovered at 1900ºC

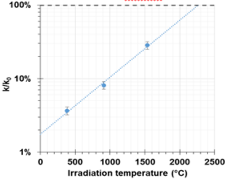
Radiation Damage Studies in Graphite
Annealing of Damage at High Temperature (> 1300°C)

- Additional analyses (Young's modulus, thermal diffusivity, lattice parameter, electrical resistance) of irradiated samples all confirm annealing at high temperature
- Results of material property changes were used as input in thermo-mechanical studies
  - Swelling is completely recovered at 1900°C
  - 30% of thermal conductivity value will be recovered but lead to insignificant change in average temperature of the production target. Main heat transfer in target is thermal radiation at high temperature
  - Electrical resistivity change has no impact on thermo-mechanical behavior
  - Decrease of CTE (coefficient of thermal expansion) has no impact on thermo-mechanical behavior
- Annealing promises sufficient lifetime for FRIB beam production targets

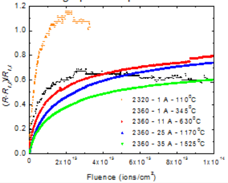
Young's Modulus of irradiated graphite samples - ^{197}Au – fluence 10^{14} ions/cm²



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



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



Future plans

- New irradiation runs to achieve higher DPA and gas production
- PIE of old/failed graphite targets [2], e.g. NuMI target fins

- Challenges
  - Irradiation cost and time
  - Irradiation facilities: BLIP, FETS?

[2] Irradiated Materials Table, http://www-radiate.fnal.gov/downloads.html

Future plans

- ☞ New irradiation runs to achieve higher DPA and gas production
- ☞ PIE of old/failed graphite targets [2], e.g. NuMI target fins

- ☞ Challenges
  - ➥ Irradiation cost and time
  - ➥ Irradiation facilities: BLIP, FETS?

[2] Irradiated Materials Table, http://www-radiate.fnal.gov/downloads.html

Future plans

- ☞ New irradiation runs to achieve higher DPA and gas production
- ☞ PIE of old/failed graphite targets [2], e.g. NuMI target fins

- ☞ Challenges
  - ➥ Irradiation cost and time
  - ➥ Irradiation facilities: BLIP, FETS?

[2] Irradiated Materials Table, http://www-radiate.fnal.gov/downloads.html