Highlights of NSLS and NSLS-II work with Nick Simos: Comprehensive characterization of radiation damage in structural materials

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4) lots of co authors

NSLS-II: U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences

U.S. DEPARTMENT OF ENERGY Office of Science
CERN
RaDIATE Collaboration Radiation Damage In Accelerator Target Environments
Fermilab
Brookhaven National Laboratory

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Personal note – transition to SBU made smooth with highly productive collaboration

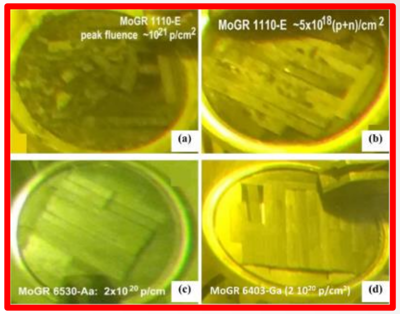
Stony Brook University

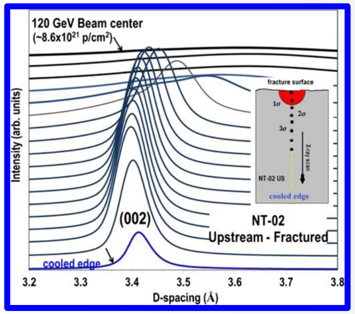
1. *120 GeV neutrino physics graphite target damage assessment using electron microscopy and high-energy x-ray diffraction* ***Phys. Rev. Accel. Beams***, 22, 041001 (2019)

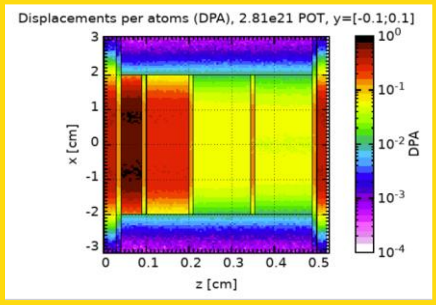
2. *Proton irradiation effects in Molybdenum-Carbide-Graphite composites* ***J. Nucl. Mater.*** 553, 153049 (2021)
3. *Radiation damage of a two-dimensional carbon fiber composite (CFC)”* ***Carbon Trends*** 3, 100028 (2021)

4. *Low-temperature proton irradiation damage of isotropic nuclear grade IG-430 graphite* ***J. Nucl. Mater.*** 542, 152438 (2020)
5. *Hexagonal boron nitride (h-BN) irradiated with 140 MeV protons* **NIMB**, 479, 110 (2020)
6. *200 MeV proton irradiation of the oxide-dispersion-strengthened copper alloy (GlidCop-Al15)* ***J. Nucl. Mater.***, 516, 360 (2019)

7. *Radiation damage from energetic particles at GRad-level of SiO_2 fibers of the Large Hadron Collider ATLAS Zero-Degree Calorimeter (ZDC)* **NIMA** 980, 164444 (2020)
8. *Fast Neutron Irradiation Embrittlement-ductilitization of an Iron-based Amorphous Alloy using In Situ Synchrotron X-ray Diffraction* ***International Journal of Metallurgy and Metal Physics***, 5, 052 (2020)
9. *Nuclear Material Characterization Using High Energy X-rays at BNL Synchrotrons: From Reactor Steels and Molten Salts to Large Hadron Collider Novel Materials,* ***Synchrotron Radiat. News****. 32, 50 (2019)*







Exposure of early career scientist to real world materials science

Presentation Outline

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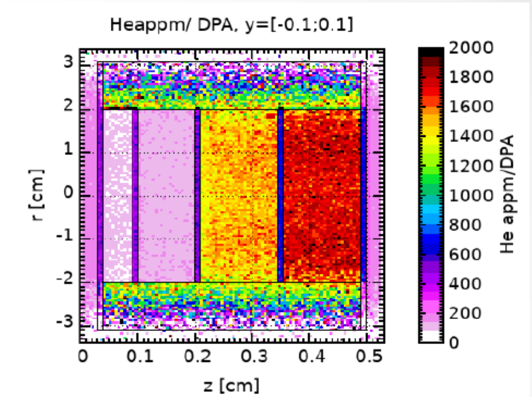
1) Suite of tools to understand radiation damage and link to microstructural and physical property degradation.

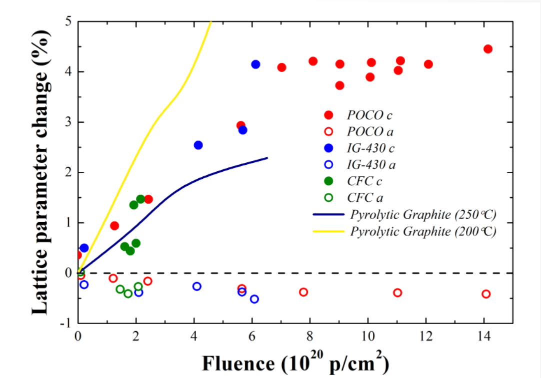
2) Couple techniques “use the right tool for the job”: pair multiple methods; leverage and compare to neutron irradiation data sets (where possible)

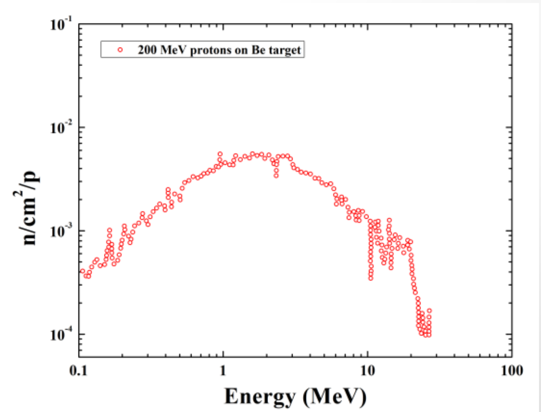
3) Science examples:

   a) High Energy Proton Targets
   b) Collimators for HL-LHC
   c) Fundamental Irradiation Materials Science

4) Summary and future







Multiscale characterization techniques are essential to assess irradiation effects in structural Materials

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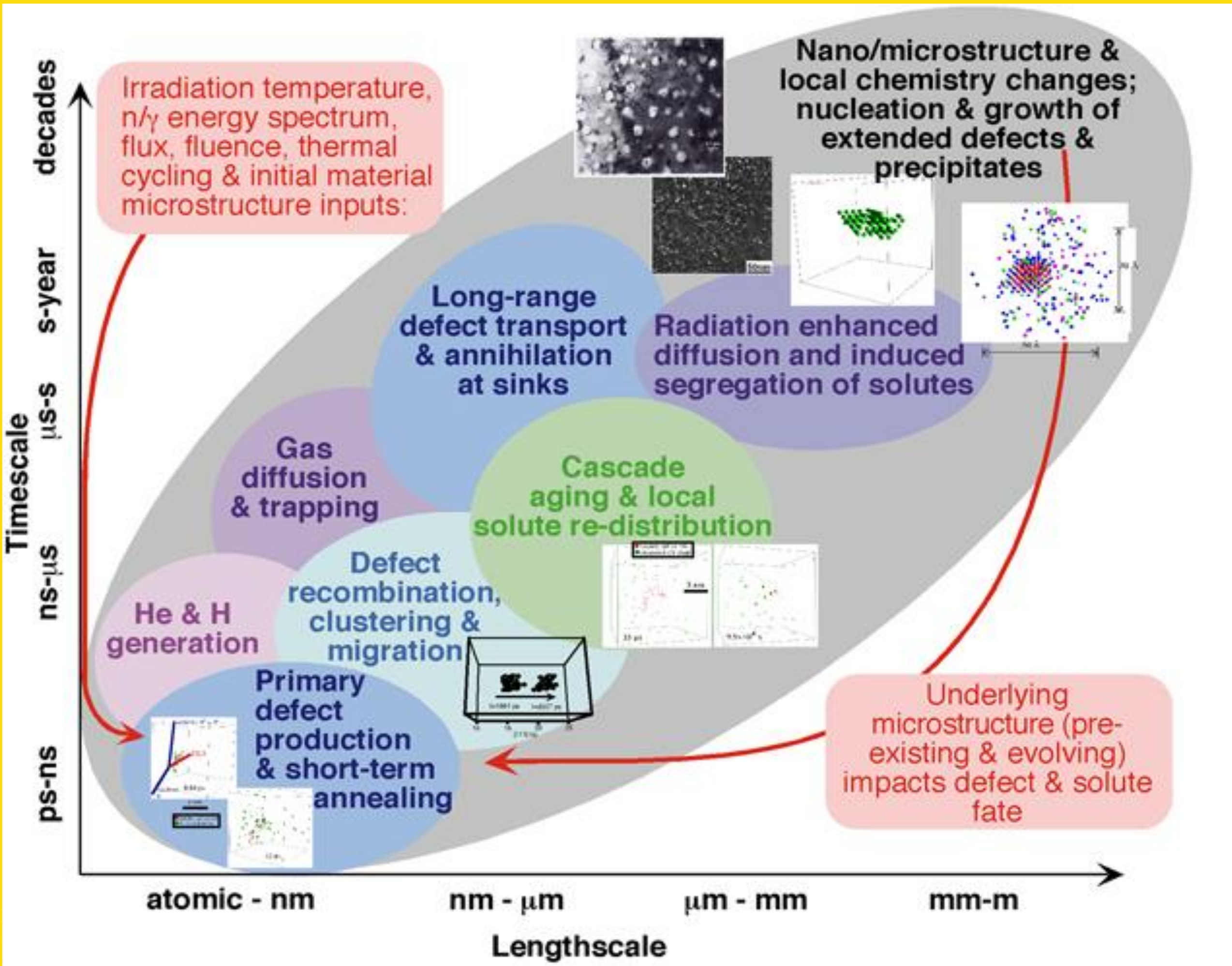
High Energy Accelerators, Fission & Fusion Reactors



JMR 30 (2015) 14

Challenge *– understanding of materials across multiple length and time scales*

A robust "toolbox" of characterization tools is necessary to tackle tough problems

Nick's lesson: Understand and quantify changes in bulk properties -mechanical, thermophysical, dimensional- and link to microstructure (and generate lots of experimental data)

Suite of PIE characterization capabilities

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Mechanical

- *Stress-strain*
- *3-point and 4-point bending*
- *Ultrasonic velocity measurements*

Physical properties

- *Thermal Expansion*
- *Thermal Conductivity*
- *Electrical resistivity*
- *Weight loss/gain*
- *Photon spectra*

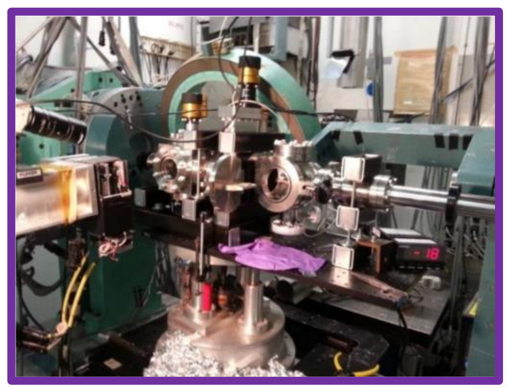
Atomic and Microstructural properties

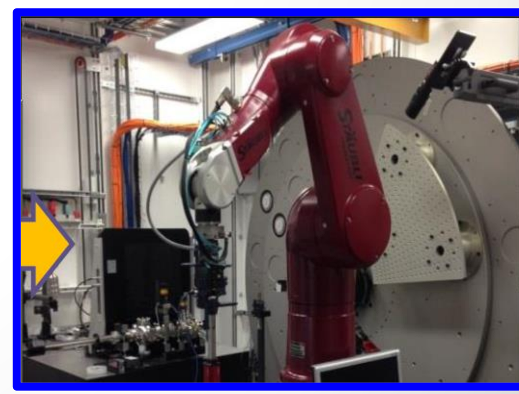
- NSLS (X17 - EDXRD)
- NSLS-II (XPD - XRD)

Simulation

FLUKA

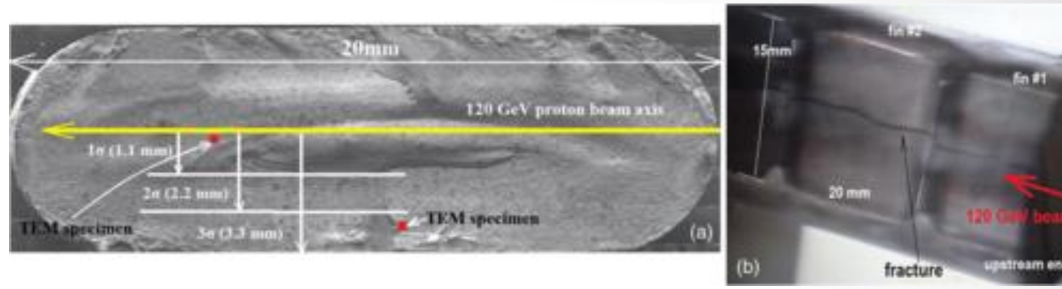
Productive space





High Energy Proton Targets - NT-02 neutrino target

- Multimegawatt power regime expected for future neutrino physics experiments
- Identifying life expectancy or fluence thresholds of target materials is of paramount importance
- Pinpointing the exact cause and target failure mode triggering the neutrino yield reduction is critical
- Segments of NT02 targets, post-irradiation, examined at the NSLS-II utilizing XRD, and complimentary TEM



NT-02 target (POCO graphite) irradiated with 120 GeV protons, 340 kW, peak fluence of 8.6 × 10^{21} protons/cm^2

XPD wiki

PHYSICAL REVIEW ACCELERATORS AND BEAMS 22, 041001 (2019)

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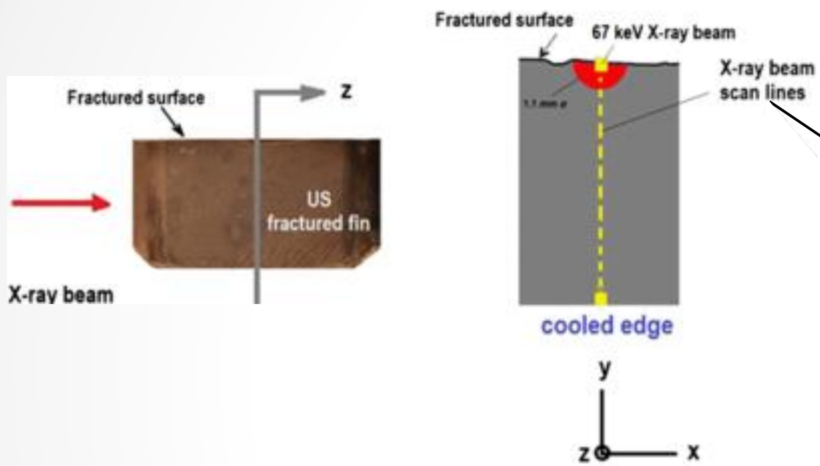
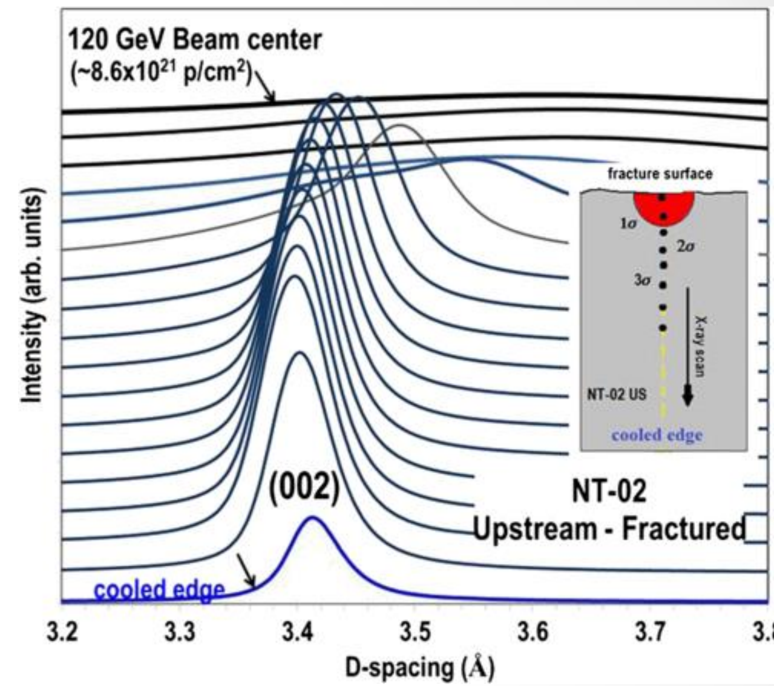
XRD mapping of irradiated targets fractured fin:



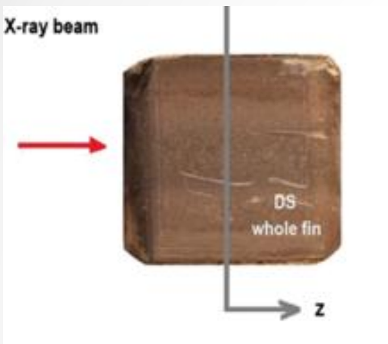


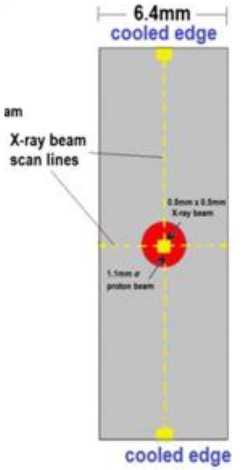
Microstructural Quantification from XRD include:

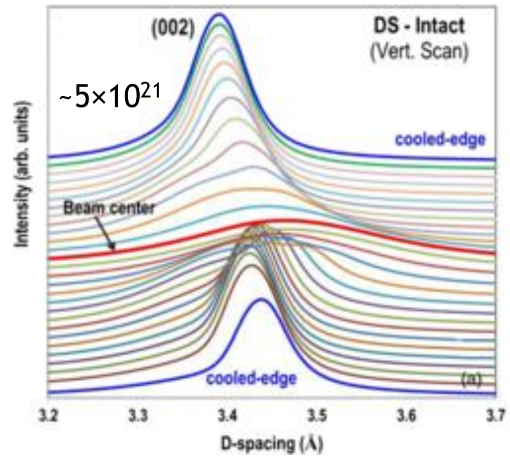
- *Increase in 002 d-spacing – extreme crystal swelling (10%*
- *Increase in width towards beam center*
- *Decrease in intensity of diffraction peaks*
- *Loss of peaks at ~1σ*

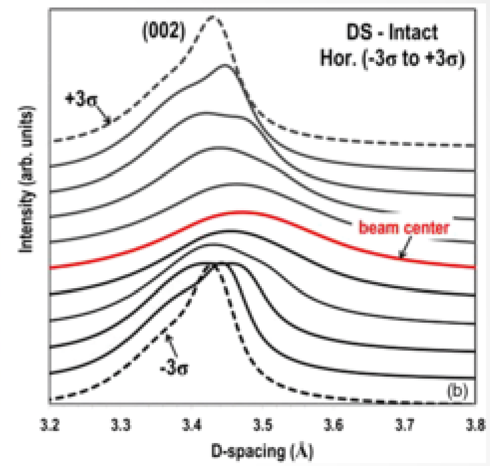
High Energy Proton Targets - NT-02 neutrino target

XRD mapping of irradiated targets intact fin:

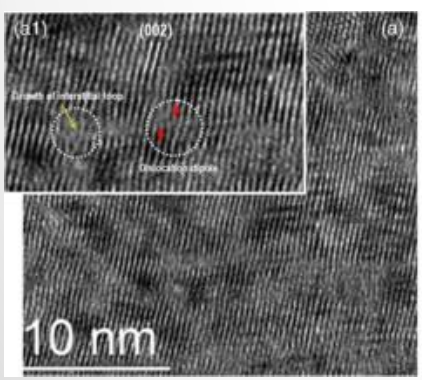




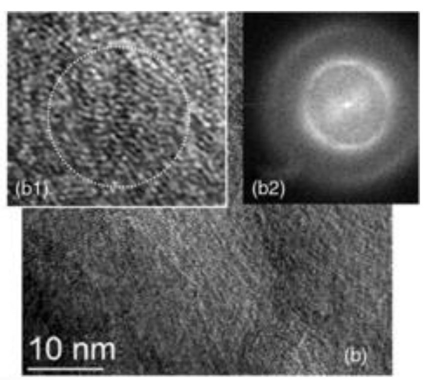


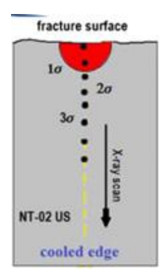


3σ



1σ





- *Complicated internal microstructure (ordered –nanocrystalline-disordered-amorphous*
- *TEM confirmation of amorphous material at 1σ*
- *Radiation damage caused failure in NuMi target*
- *Synergy in combined facilities – (NuMI + NSLS-II) matching science needs with experimental capabilities*

Collimators for the High Luminosity upgrade of the Large Hadron Collider (HL-LHC)

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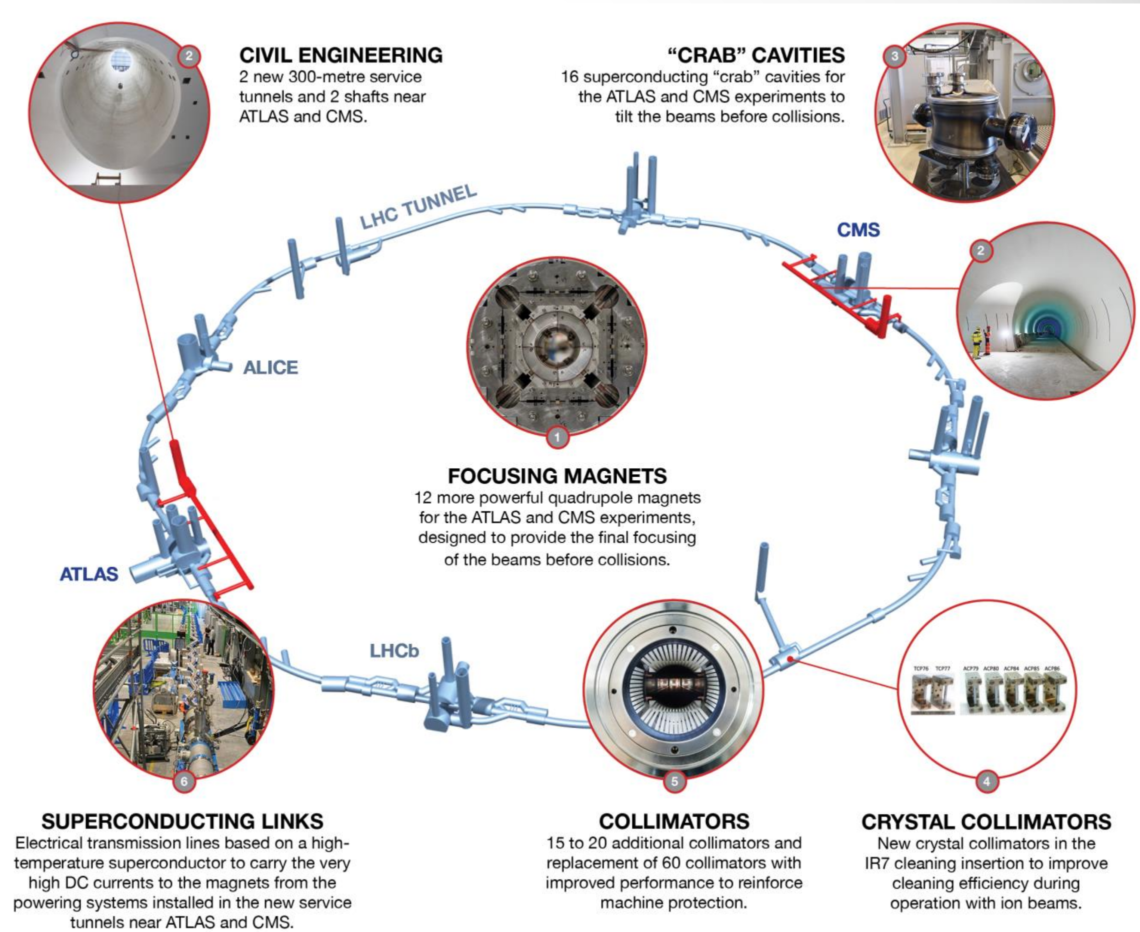
- Novel materials for beam-intercepting devices, such as collimators, are essential to protect machines from beam losses.
- The physical degradation of materials after low and moderate proton fluences is needed to determine their radiation stability and mechanical integrity
- Molybdenum-carbide-graphite compounds and CFCs irradiated at BLIP (various fluence-temperature combinations)
- PIE employing XRD, dilatometry to quantify the dimensional stability and crystallographic phase evolution

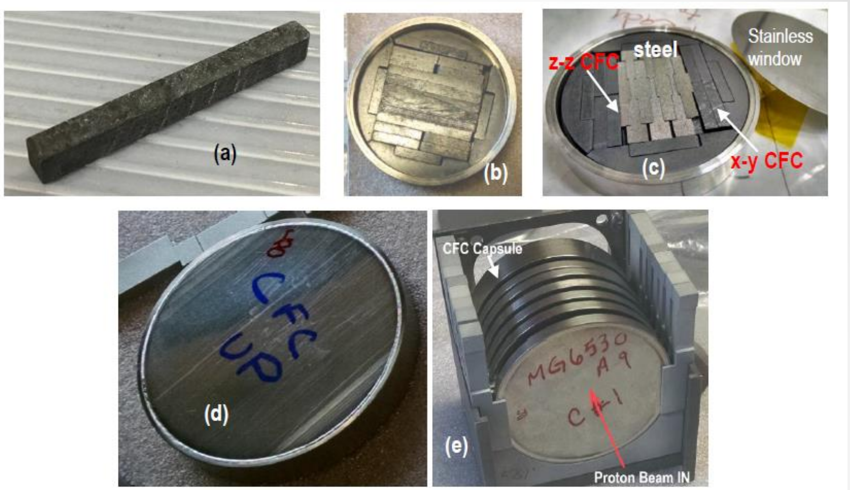


1.1 × 10^{15} p/cm^2 acute deposited energies of the order of ~1-10 MJ.
Long-term exposure ~5.0×10^{18} p/cm^2 over 12 years of ops operation

CERN

Irradiation and PIE campaigns for collimator materials

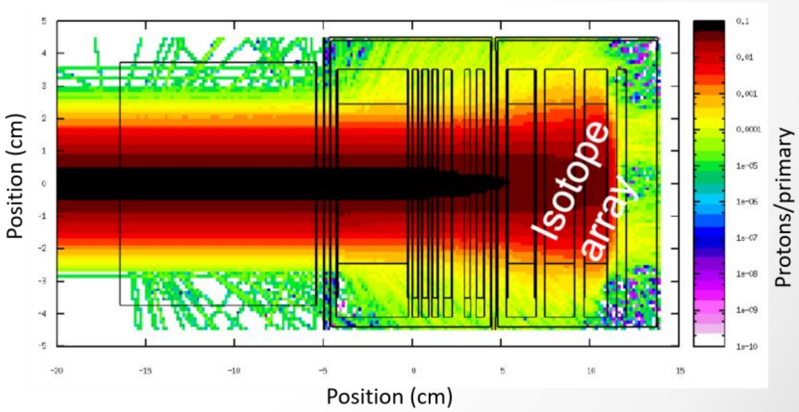
Stony Brook University



Specimens and experiments:

- Carbon fiber composites (two grades of AC-150K Tatsuno Co., Japan)
- Molybdenum graphite composites (four different grades from CERN jointly with the Brevetti Bizz Company)

- Temperatures ~200-500°C
- Doses – ~0.5-6 × 10^{20} p/cm^2
- PIE – Optical microscopy, Dilatometry, XRD, Ultrasonic velocity (elastic modulus)

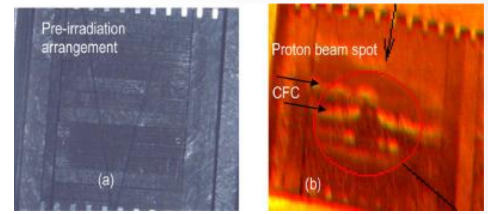


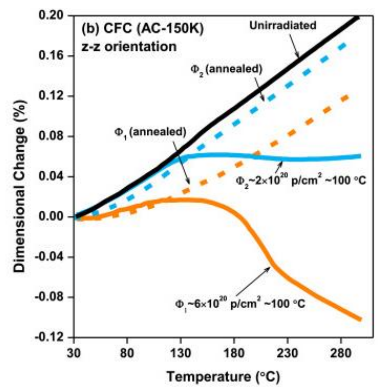
CFC - Carbon Trends 3 (2021) 100028
MoGr- Journal of Nuclear Materials 553 (2021) 153049

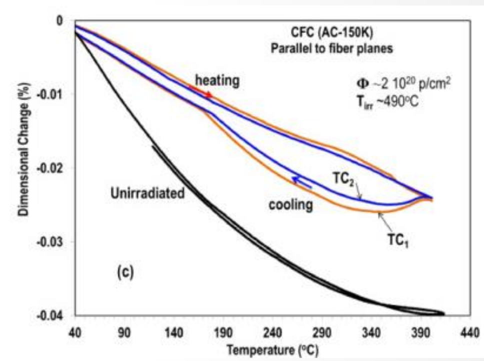
Irradiation and PIE campaigns for collimator materials

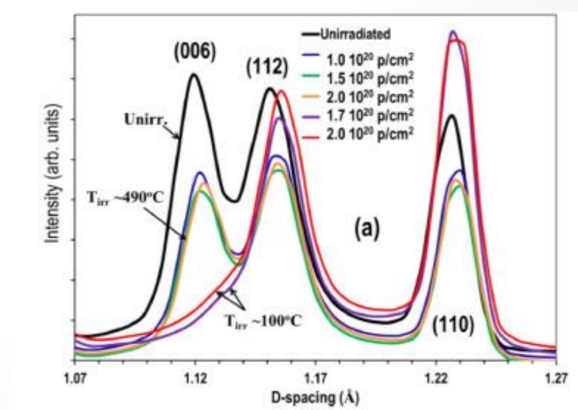
CFC results

- macroscopic degradation (fluence above 5×10^{20})
- Changes in the dimensional stability – dose, temperature, and orientation dependent (z-z, x-y)
- Some subtle differences in the Damage is non-repairable (for the low temps cycled)
- CFC experiences a high degree of damage (peak intensity reduction, broadening and increase in d-spacing) after low temperature irradiation.







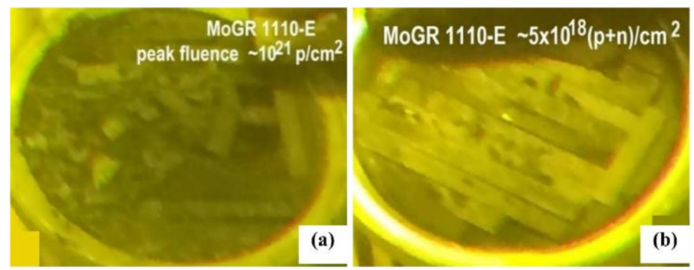


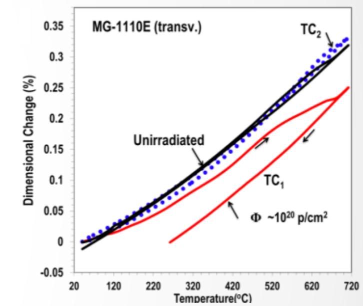
Irradiation and PIE campaigns for collimator materials

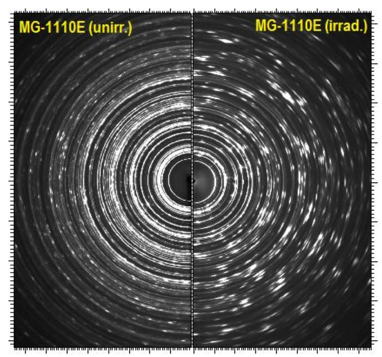
<u>MoGr results</u>

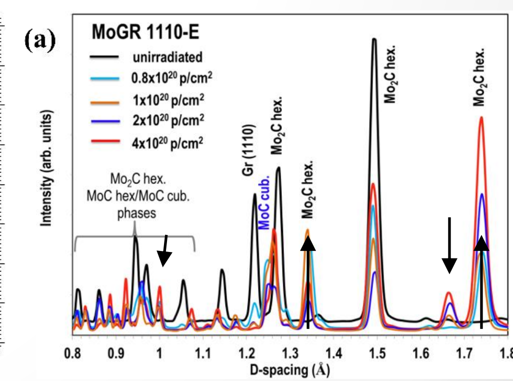
- Macroscopic degradation in CF containing MoGr after high and low fluence (difficult to find intact samples for dil)
- The large carbon fiber component results in an appreciably anisotropic microstructure;
- Material inhomogeneities (high fraction of hexagonal phases) and unreleased residual stress from fabrication (no post fabrication annealing cycle)
- Extensive texture in the microstructure, attributed to incomplete melting, carbon fiber bundles and significant anisotropy

Parameter	MG-1110E	MG-6530Aa	MG-6403Ga	MG-6403Fc
Density (g/cm^3)	3.7	2.5	2.5	2.5
% v Graphite	40.0	90.5	94.95	94.95
% v Mo	20.0	4.5	4.5	4.5
%v Ti	0.0	0.0	0.6	0.6
%v CFs[a]	40.0	5.0	0.0	0.0
Post-Sintering Temperature (°C)	No post sintering	2100	2100	2600
Coefficient of Thermal Expansion (CTE) (10^{-6} K^{-1})*	6.8	6.3	5.7	5.5
Thermal Conductivity (W m^{-1} K^{-1})	320	489.9	547	740









<u>Last light of NSLS</u>

Irradiation and PIE campaigns for collimator materials

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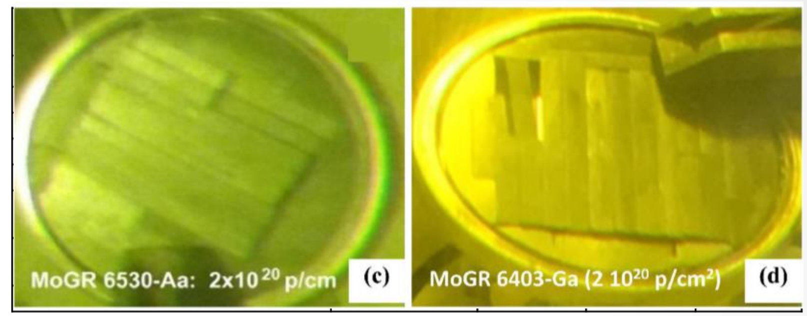
MoGr results

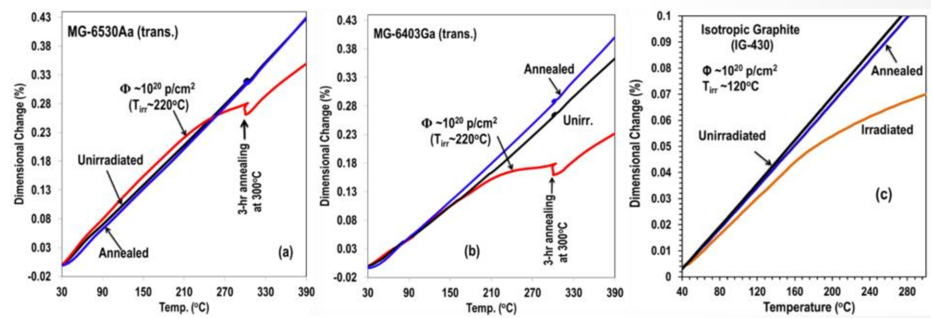
- Macroscopic degradation not observed in next gen grades

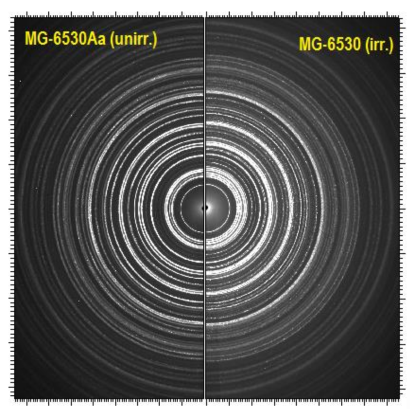
- MG-6530Aa, MG-6403Ga and MG-6403Fc aft 2×10^{20} p/cm^2 appear to be more radiation resistant – due to initial microstructure (pre-irradiation treatment important)

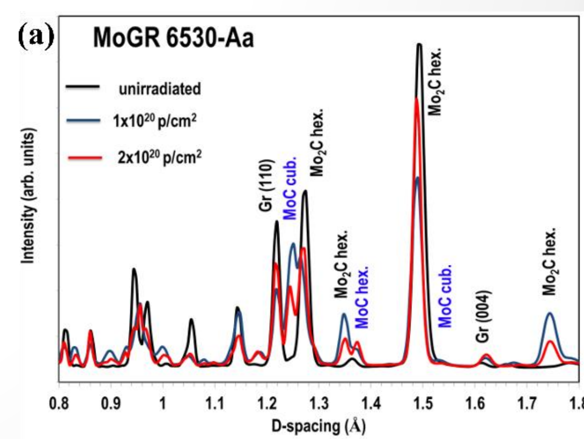
- Irradiation does induce new hexagonal Mo_2C phase in MG-6530Aa, but not significantly in MG-6403Ga and MG-6403Fc grades that both contain a small additions of Ti.

- These findings tie into and support the operational requirements of the HL-LHC. Next gen MoGr composites are excellent candidates for collimating structures of high-brightness machines









Last light of NSLS

Fundamental Irradiation Materials Science

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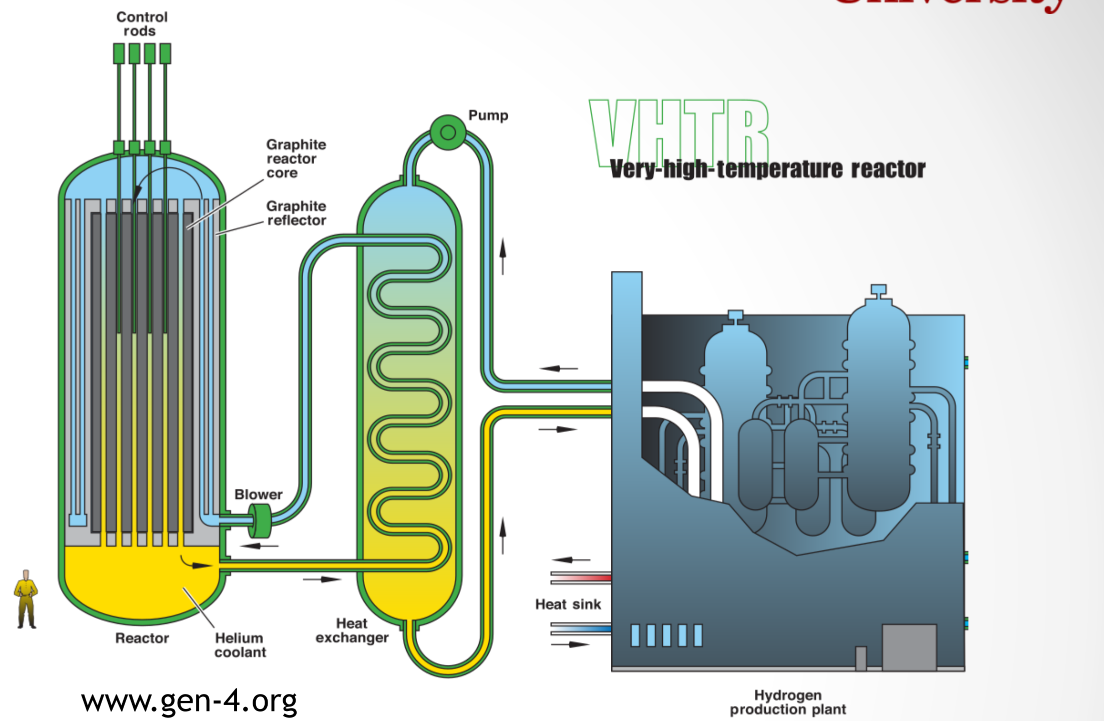
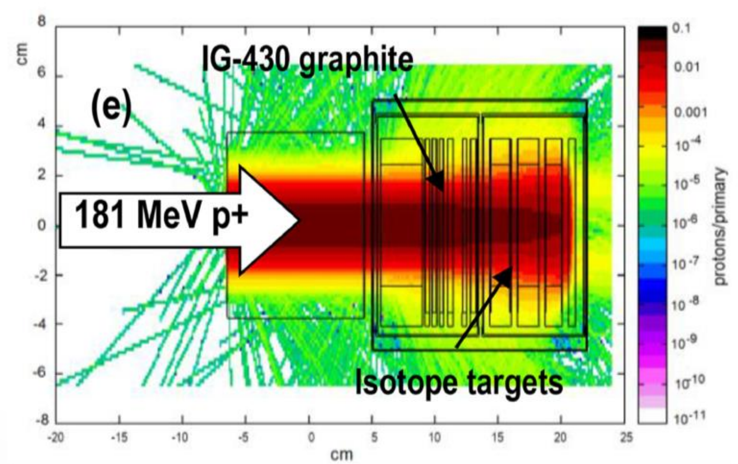
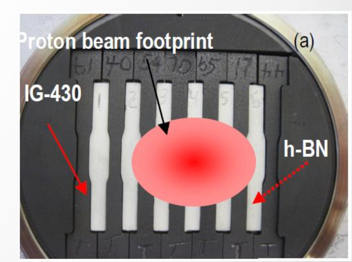
- IG-430 graphite interest to multi-MW power particle accelerators (LBNF, Neutrino Factory) advanced gen-iv power reactors (VHTR).
- Hexagonal BN with good thermal conductivity is a potential HE accelerator target material – high thermal conductivity, oxidation resistance, and can potentially improver thermal shock when composited
- Quantify the physical degradation of IG-430 and BN after low and moderate proton fluences is needed to determine their radiation stability and mechanical integrity



www.gen-4.org





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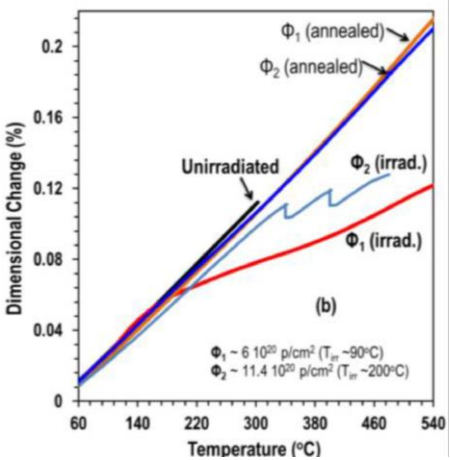
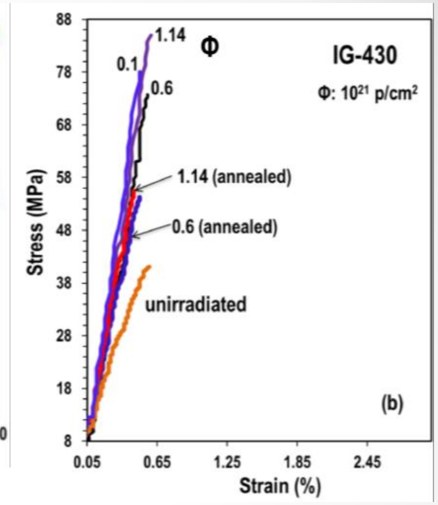
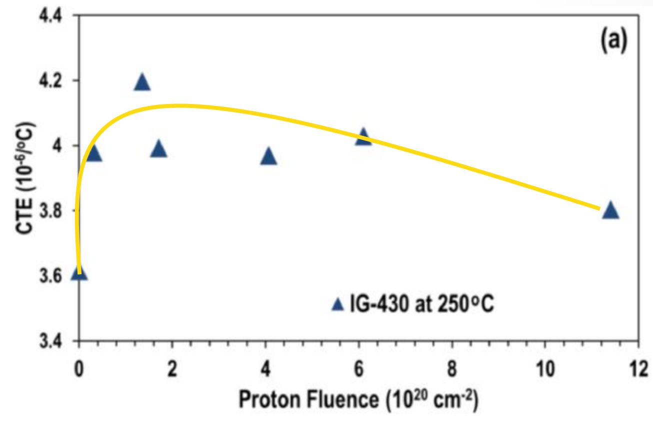
Thermophysical and mechanical results for low-moderate-high fluence p irradiation in IG430

- Changes/degradation in the mechanical and thermophysical properties of proton irradiated IG-430 are similar, but subtly different to those in neutron irradiated IG-430 (Young's modulus, strength, dimensional stability)
- Comparison of identical grades, proton and neutron irradiated, is planned for future study



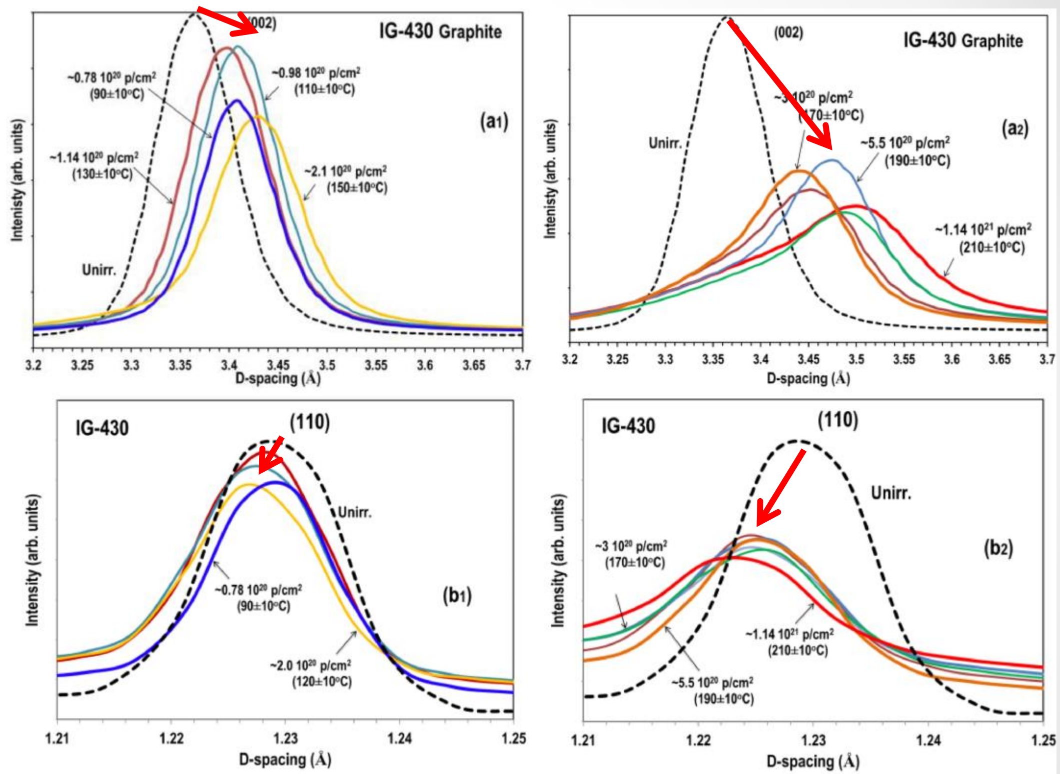




PHYS. REV. ACCEL. BEAMS 21, 053001 (2018)
J. NUCL. MATER. 542, 152438 (2020)

Fundamental Irradiation Materials Science

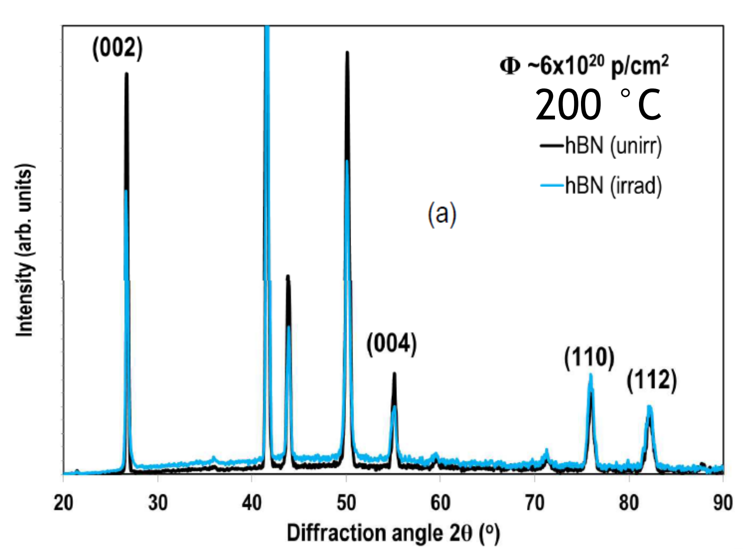
- Multiple changes/degradation in the microstructural properties are apparent. XRD is appreciably rich in atomic and microstructural information
- Expansion in the c-parameter and contraction in the a-parameter (consistent with previous studies in p- and n-irradiated graphite)
- Disorder is apparent in the change in peak heights and widths
- The amount of disorder and microstructural changes from quantitative XRD analysis (capturing the width, height of XRD peaks) is planned in the future.



Fundamental Irradiation Materials Science

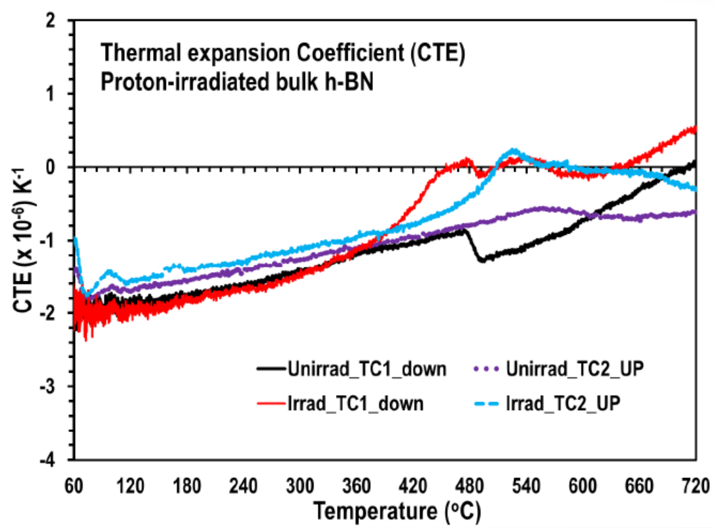
Results for low temp moderate fluence p irradiation in hBN

- HBN is very radiation stable!
- XRD peak shifting is almost extremely small
- Highest radiation dose achieved for BN…and it didn't misbehave
- Irradiation-induced defects perturb the thermal expansion behavior of hBN - amplitude of CTE after irradiation (origins?)



a_{un} = 2.5065
c_{un} = 6.6581

a_{irr} = 2.5053
c_{irr} = 6.6754



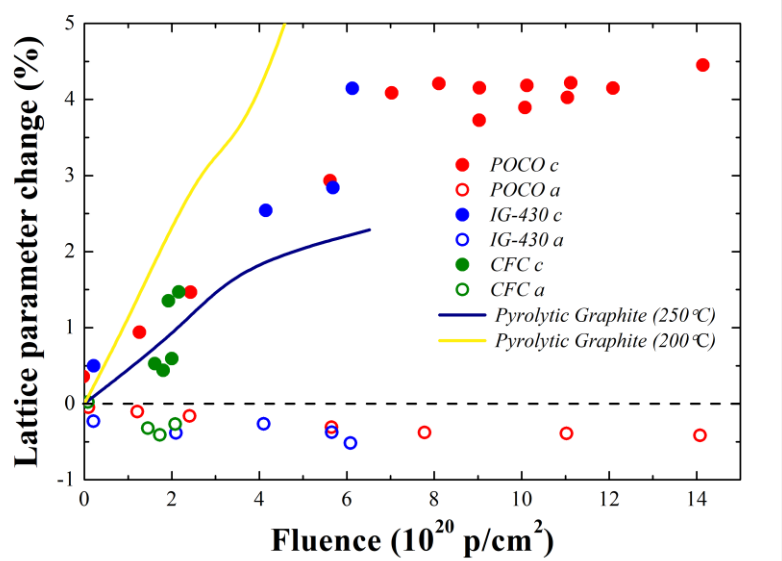
NIMB, 479, 110 (2020)

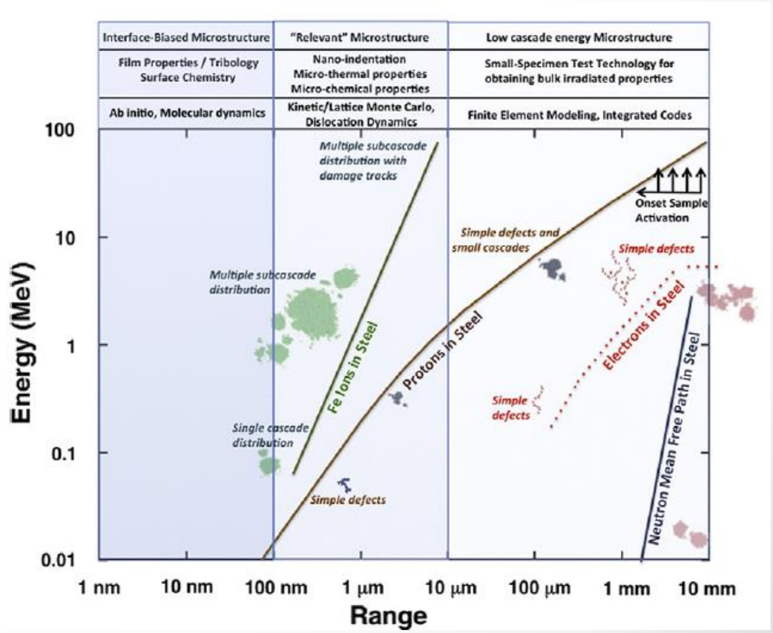
Summary

Nick Generated a tremendous amount of data for the academic community, and pioneered techniques to quantify the thermomechanical and microstructural effects of energetic irradiation:

I. *Targets (NuMi)*
II. *Collimators (HL-LHC)*
III. *Fundamental irradiation materials science*

Synchrotron-based characterization techniques (EDXRD and XRD) were particularly championed by Nick.





S.J. Zinkle, L.L. Snead / Scripta Materialia 143 (2018) 154–160

Future work:

- Lots – compare n an p irradiation results – XRD and physical properties (is there equivalence? Damage should be similar)
- Mixed field irradiations

Acknowledgements

NSLS and NSLS-II staff at X17 and XPD beamlines
NSLS-II Staff: H. Zhong, M Whitaker, Z. Zhong, S. Ghose, E. Dooryhee
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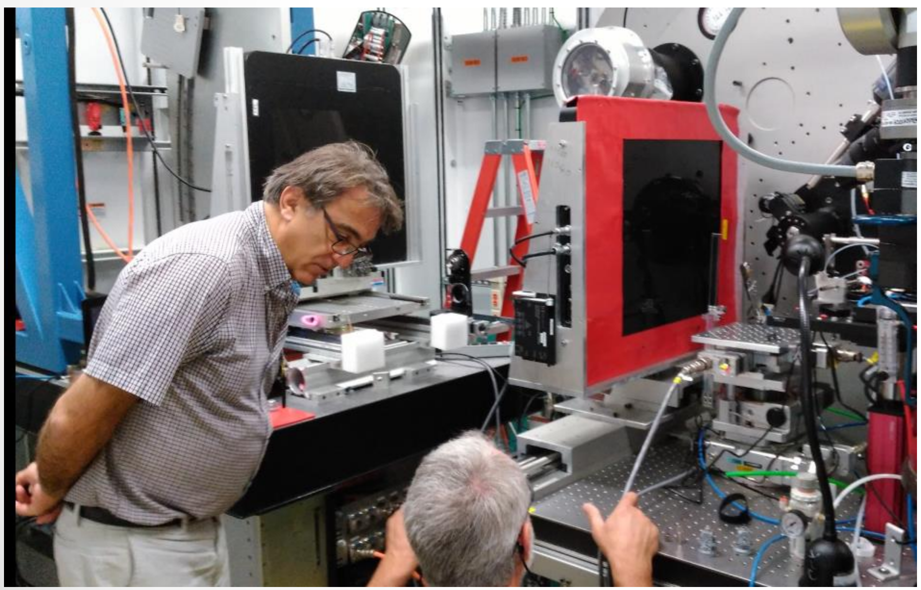
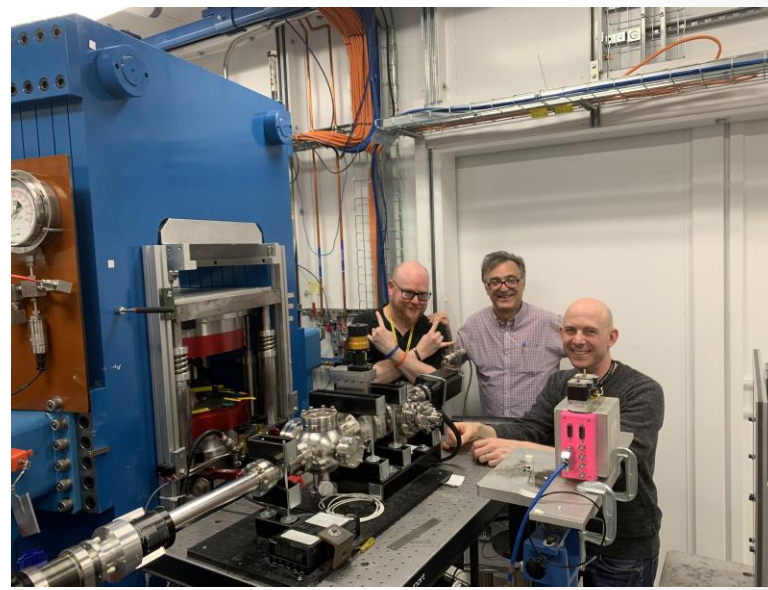
Fermilab: K Ammigan, P. Hurh,
National Center for Scientific Research Z. Kotsina
PNNL: A. Casella, D. J. Edwards, D. Senor
SBU: L Snead, M Botha-Snead





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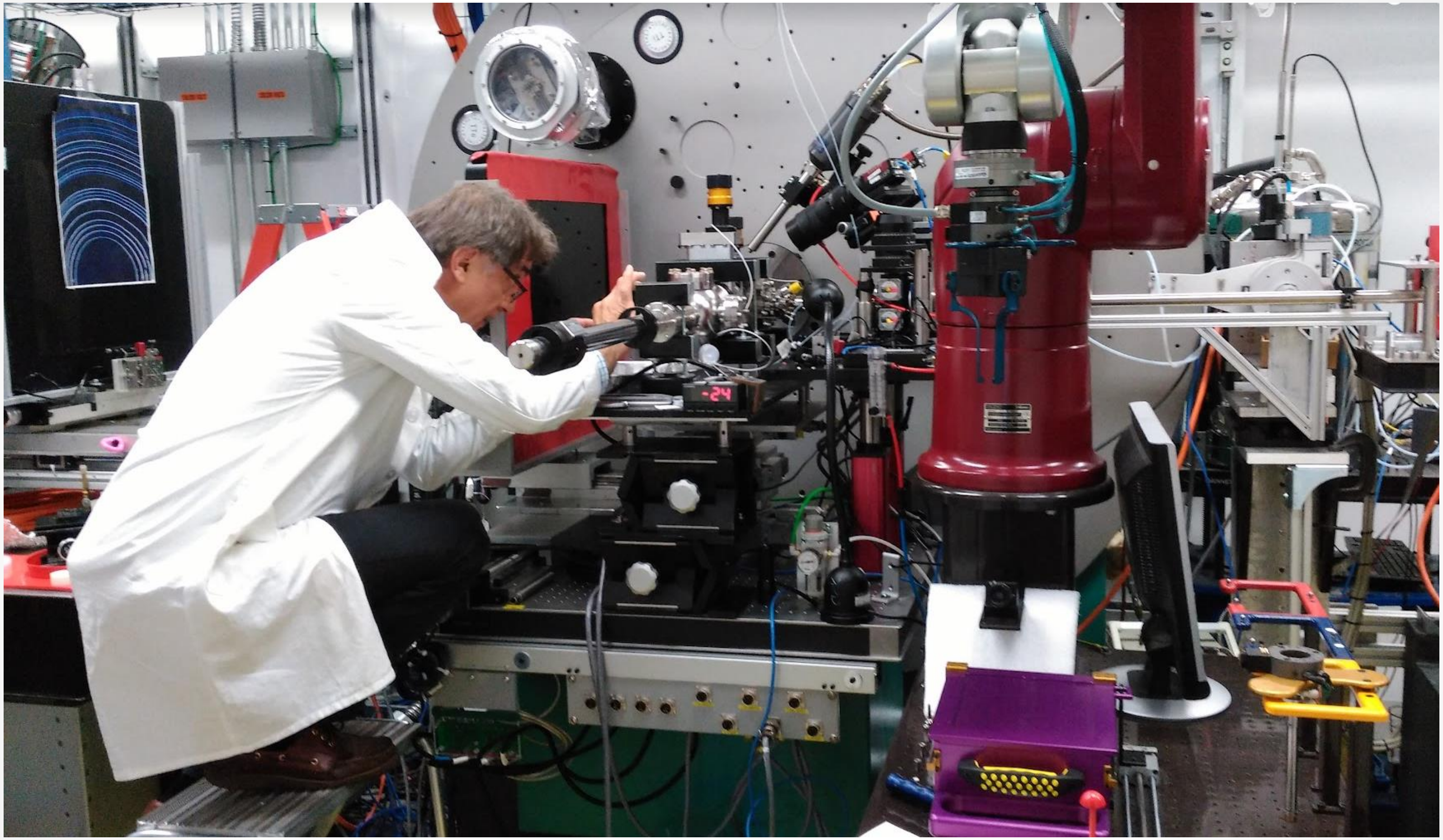
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We lost a mentor, friend and leader who worked actively to incite members, users and CERN staff

https://home.cern/news/news/experiments/flexible-and-accessible-hiradmat-facility-celebrates-its-tenth-anniversary

Nick has been one of my best friends since I met him two decades ago. He is the only friend who would show up more than once weekly in my office, unannounced, to chat for an hour or so about everything. He knew I was not doing "real work" in the office and that I enjoyed talking with him more than the unreal work.

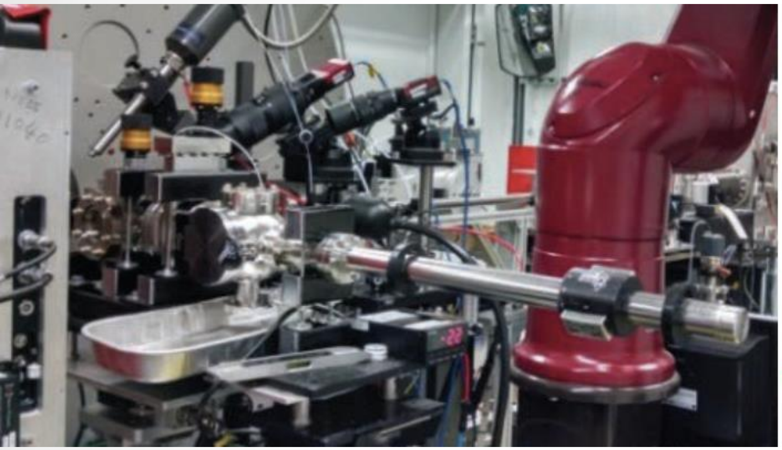
We talked about climate change, raising daughters of similar age, and their college applications. He introduced me to radiation damage to engineering materials, embrittlement, proper paperwork for experimenting on radioactive materials, the then-new book "Tesla", and the Tesla Science Center under construction in Shoreham.

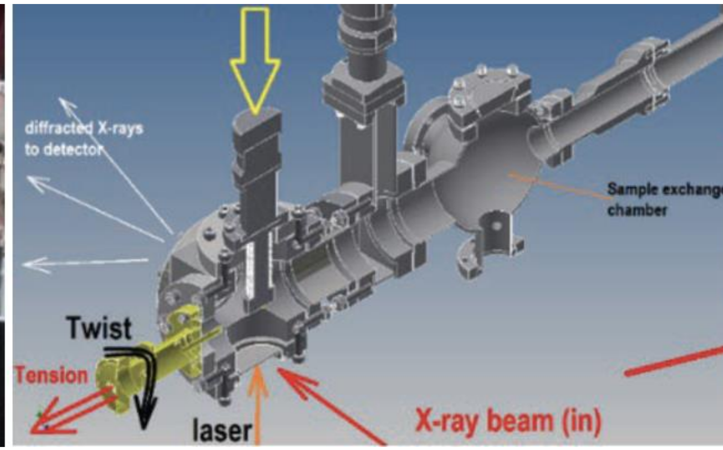
Nick is a Renaissance man – he knew so much about everything, whether the floor vibration or the latest trend in reactor design. He was so ready to spend his time sharing with me. He is humble, always asking me for advice on X-rays which I only know slightly better.

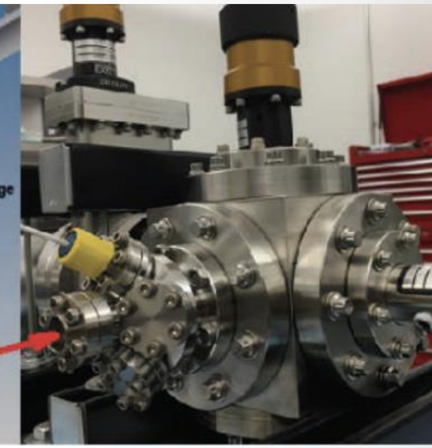
We talked about life and the timing of eventual retirement. He said on many of our chats, after the shutdown of the NSLS in 2014 and before the pandemic, that one thing that kept him going was to help the "young" people like me. I appreciate that, and I know he meant it.

Zhong

Nick Simos my friend: The Great Scientist, Engineer, Designer, Socialistic, and Very Enthusiastic Person.........Though we miss your physical presence, but we always can see, feel and nurture your footprints those marked around us.....







I had great opportunities to work with Nick starting from NSLS to NSLS II. The above picture shows XPD beamline and Nick's one of the versatile instrument for XRD measurements of samples under stress load. These works made me to be a co-author in most of his publications. He was a scientist with expertise in technology and with full of ideas.

We often discuss on many social & personal topics which made be know Nick more on his other good side. Nick was a great family man, mentor, teacher and friendly person, who always stay happy and make others happy too. Sanjit Ghose

Before we close the show, there's something I'd like to take a moment to say. Anybody that knows me, or has seen the UEC show before, knows that I'm not easily lost for words, nor am I one for writing things down, but in this case, I had to in order to make sure I could get through this properly. Last month, NSLS-II lost a cherished member of our community in Nik Simos. Nik was a constant fixture here at NSLS-II, and started an absolutely legendary career at Brookhaven Lab over thirty years ago. While his formal training was as a Mechanical Engineer, his work and his impact expanded far beyond this field and touched more areas than I have time to relay here. He was widely recognized as one of the premier scientists in the world working on the stability of sensitive accelerator facilities, and had a huge hand in the design and proper construction of the NSLS-II, as well as other facilities in the US such as the Spallation Neutron Source, and around the world from Europe to China and all points in between. His interests also included the study of irradiation damage in structural materials, which is how he grew such a close relationship with those of us fortunate enough to have worked with him at 28-ID here at NSLS-II. He not only conducted his work at the beamlines here, but he was always open to discussing new ideas and helping other users, particularly young scientists, and for that we as the User Community, and I personally, will be forever grateful.

While I can say some simple words about his contributions to science, I really knew Nik better as a friend. Though we met here at NSLS-II several years back, we quickly formed a fast friendship that went beyond our time at the Lab. I knew Nik as an amazing scientist, but also as a loving father and husband. I knew Nik as the guy who would crazily participate in the Polar Plunge to raise money for charity. I knew him as the guy that would come to my band's shows and rock along with us, always with a smile on his face. He always had a story that was uniquely his, and he always made sure to make you a part of it. I named my son after him, and I remember when I asked him if that would be alright, he told me he was flattered, and then regaled me with a story of the origins of the name. He was going to show me around Greece when my son was a little older, as it is the only place I've always wanted to visit, since I was 9 years old, but have never been. That trip looks like it's going to have to be a bit different than we had planned.

I asked around to a few people here at BNL who had the opportunity to work with Nik for a couple of brief things that I could say about him here today, and in every case I got back something that looked more like a novella than a bullet point. I think that simple fact says more than I possibly could. He was a giant in his field, and his expertise and contributions will be sorely missed. But more than that, he was a damn fine human being who will be missed more than any of my words can express. To commemorate Nik Simos and his incredible legacy, the NSLS-II UEC will be working with the team of 28-ID to put together a memorial tribute here at the Lab, and we will share more information on that as details become available. In the meantime, I would just say that we love you, Nik, and we miss you. Good Journey, my friend.

Thank you all for indulging me for a moment. That about brings us to the end of the show…

M. Whitaker