5h Joint HiLumi-LARP Meeting & 24th LARP Collaboration Meeting
Fermilab 11-13 May 2015

Post irradiation analysis of LHC collimator material samples at BNL facilities

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Acknowledgments:

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CERN, Geneva, Switzerland

Outline

* Motivation
* Status of the work
* Thermo-physical characterization at CFC
* X-rays diffraction studies at NSLS I
* Conclusions

Motivation

LHC collimators are exposed to high radiation dose level during the normal operation of the machine. It may lead to dramatic changes in the material properties.

Main goal of the study is:

Identify all other parameters that, together with radiation, contribute to material damage

Irradiation exposes materials to extreme operation conditions and very high temperatures are reached.

High temperature can induce changes in the material, in terms of:

1. Crystal structure
2. Phase transformations
3. Oxidation of the surface

What are we doing to understand these aspects?

Status of the work

BLIP

- 200 MeV proton irradiation (8 weeks): COMPLETED
- Neutron irradiation from 112 MeV protons (several months not continously): COMPLETED
- Cooling of highly radioactive samples in Hot Cell Lab 66: ON-GOING

TANDEM VAN DER GRAAF

- 28 MeV proton irradiation + sample cooling: COMPLETED

NSLS (National Synchrotron Light Source)

- X-Ray diffraction studies for phase and strain mapping of some cold and irradiated samples: COMPLETED
- Data analysis: ON-GOING

CFN (BNL Center of Functional Nanomaterials)

- Annealing, SEM and thermo-physical analysis: ON-GOING

IEF (BNL Isotope Extraction Facility)

- Activity and dose measurements per sample: COMPLETED
- γ-spectra for selected samples: PARTIALLY COMPLETED

Thermo-physical characterization at CFN

CFN Lab

At the Center of Functional Nanomaterials (CFN), we performed:

Annealing

CVD Furnace (vacuum atmosphere)

Mo: 460°C, 660°C, 1000°C
MoGR: 400°C, 660°C, 720°C
CuCD: 400°C, 660°C

SEM/EDS Analysis

SEM JEOL 7600F

Mo: RT + annealed at 460°C, 1000°C
MoGR: RT + annealed at 400°C, 660°C
CuCD: RT + annealed at 400°C, 660°C

Thermal Analysis

PerkinElmer DSC

Mo: 580°C (ramp: 6°C/min)

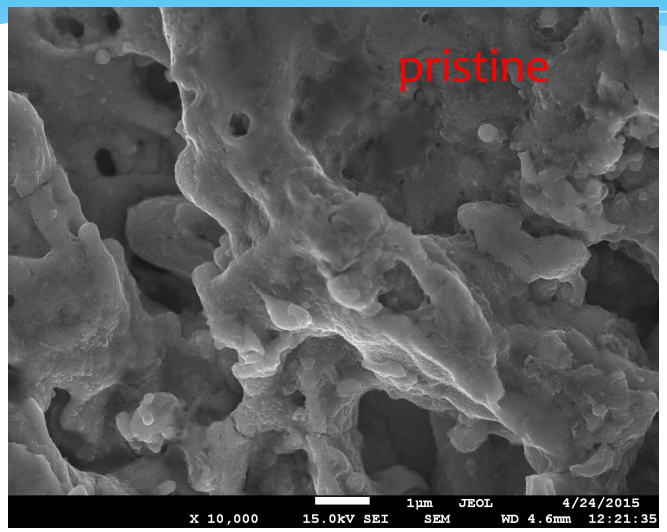
PerkinElmer TG/DTA

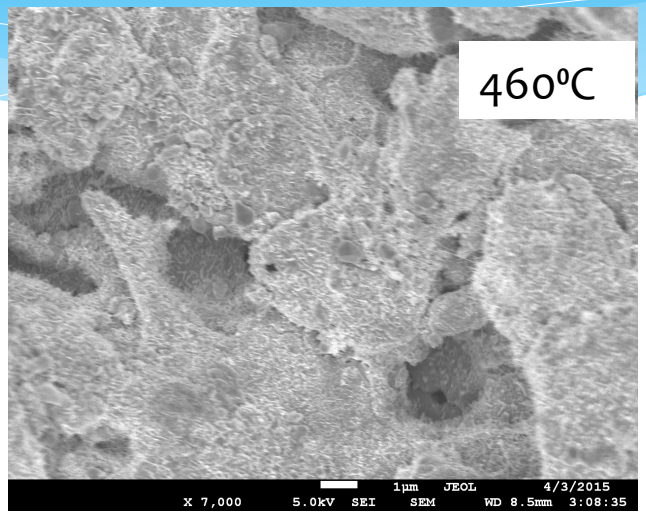
Mo: 980°C (Oxygen atmosphere, ramp: 5°C/min)

Data analysis on-going
Results not presented today!

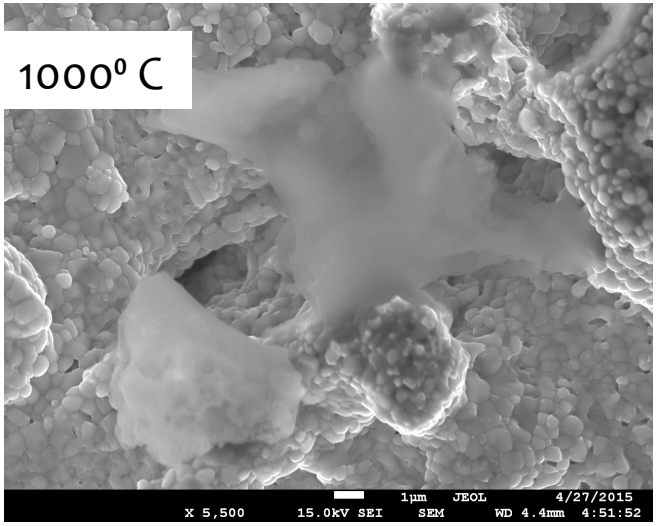
So far, the studies concerned ONLY UNIRRADIATED samples.

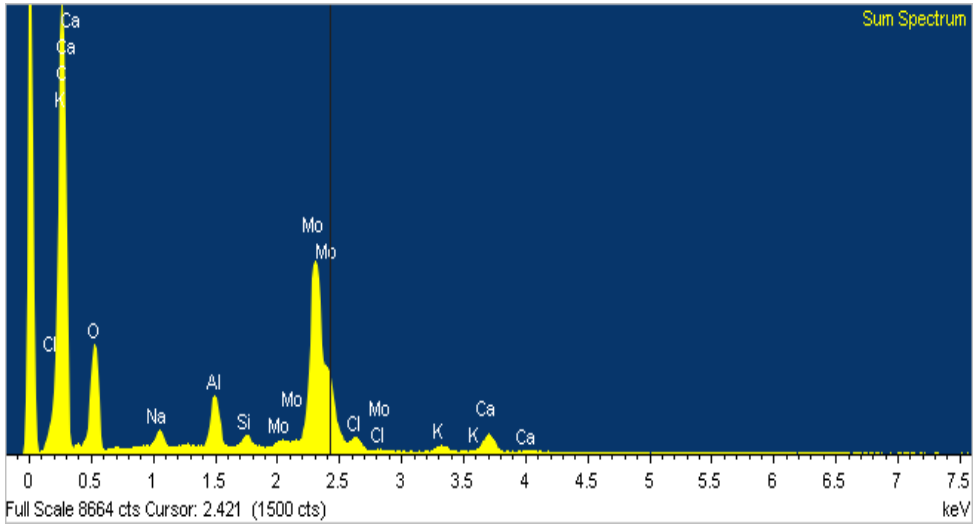
SEM/EDS: Mo



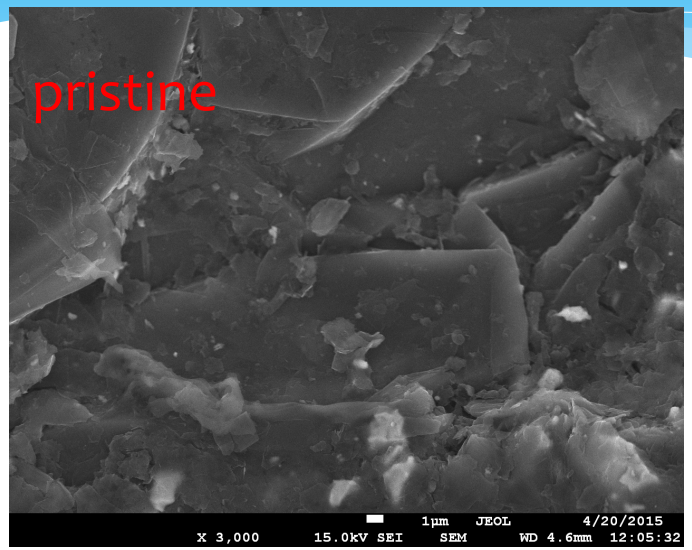


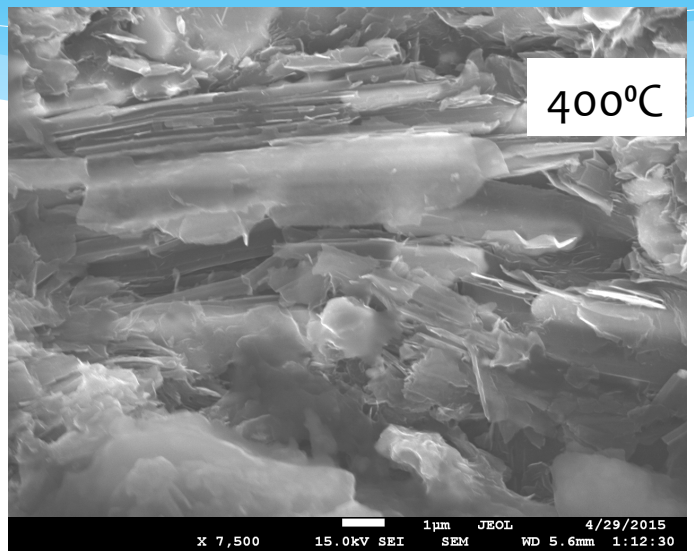
Changes in structure at high tempereture: crystallize



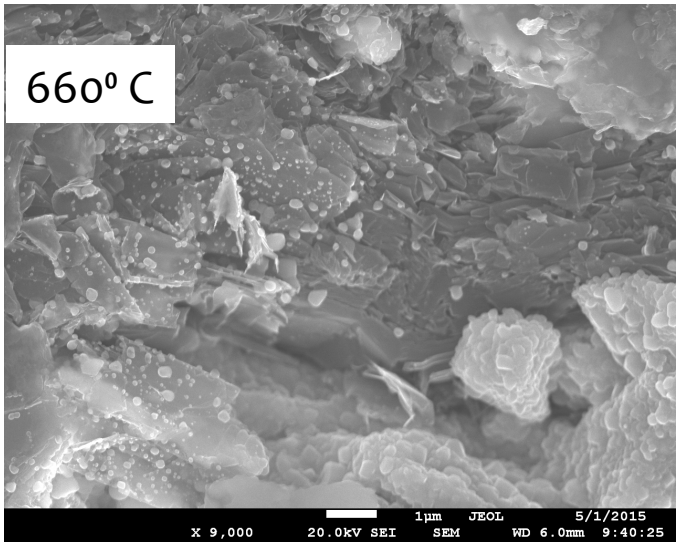


SEM/EDS: MoGr

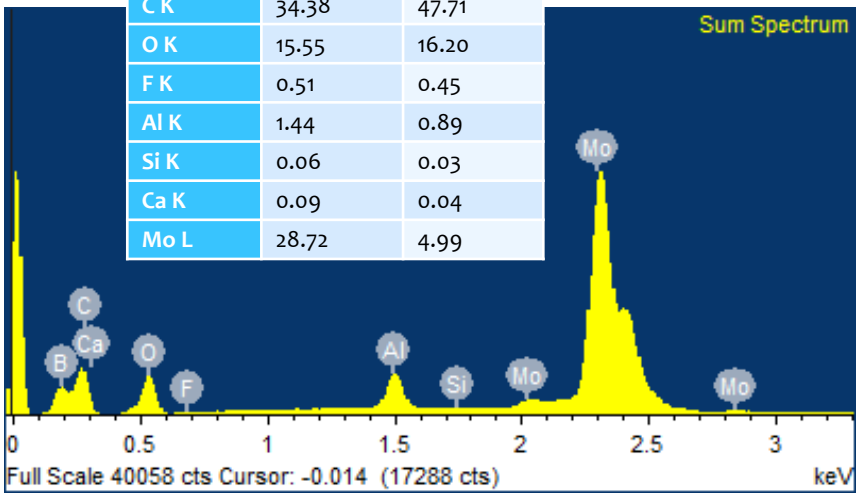




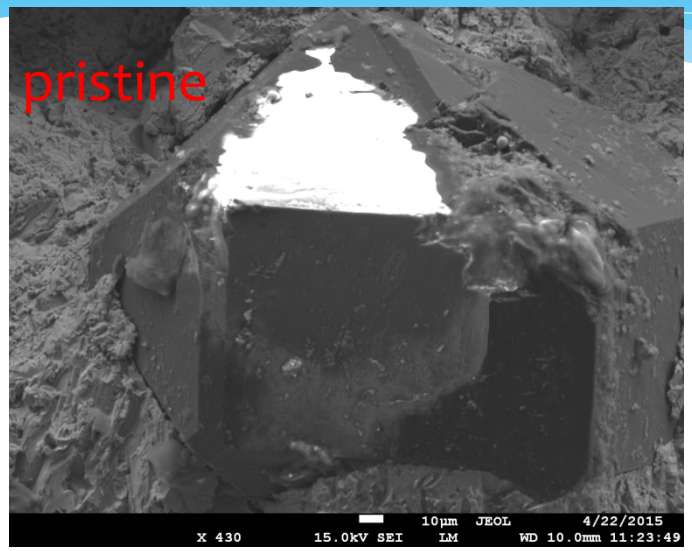
Changes in structure at high tempereture: crystallize

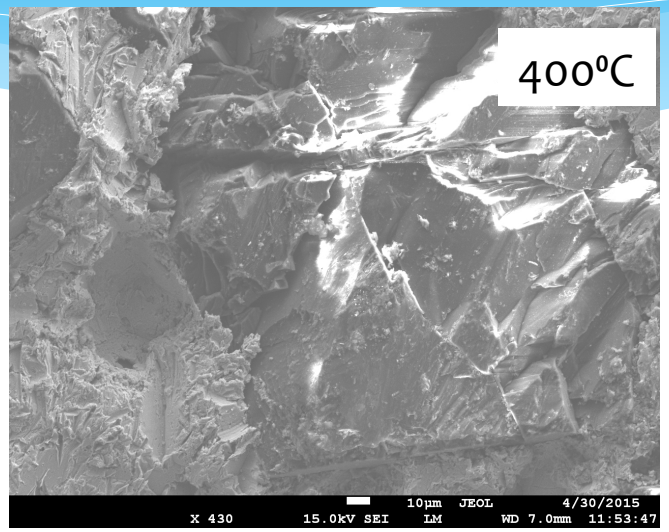


B K	19.25	29.68
C K	34.38	47.71
O K	15.55	16.20
F K	0.51	0.45
Al K	1.44	0.89
Si K	0.06	0.03
Ca K	0.09	0.04
Mo L	28.72	4.99

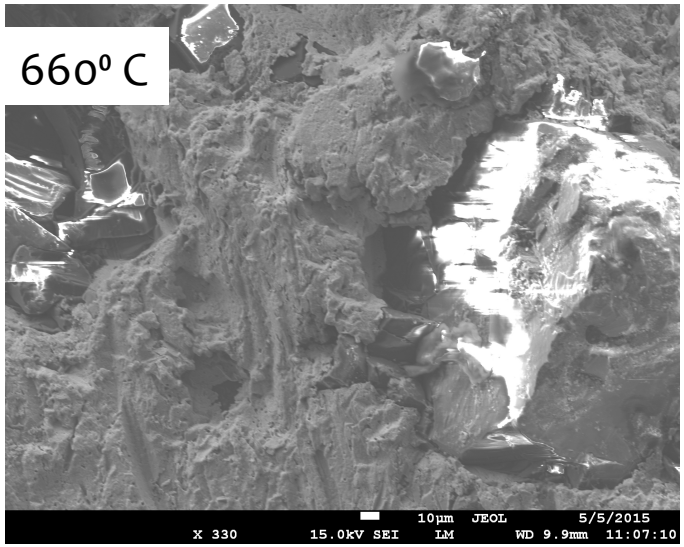


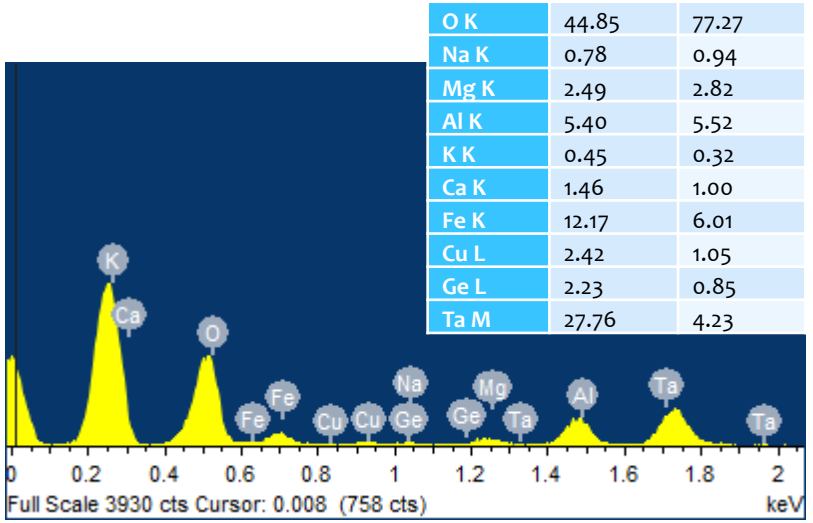
SEM/EDS: CuCD





Preliminary analysis: no relevant changes are visible. Further investigation needed.





O K	44.85	77.27
Na K	0.78	0.94
Mg K	2.49	2.82
Al K	5.40	5.52
K K	0.45	0.32
Ca K	1.46	1.00
Fe K	12.17	6.01
Cu L	2.42	1.05
Ge L	2.23	0.85
Ta M	27.76	4.23

Ongoing and future work at CFN

Annealing

CVD Furnace (vacuum atmosphere)

Mo: 460°C, 660°C, 1000°C
MoGR: 400°C, 660°C, 720°C
CuCD: 400°C, 660°C, 720°C

SEM/EDS Analysis

SEM JEOL 7600F

Mo: RT + annealed at 460°C, 1000°C
MoGR: RT + annealed at 400°C, 660°C, 720°C
CuCD: RT + annealed at 400°C, 660°C, 720°C

Thermal Analysis

PerkinElmer DSC

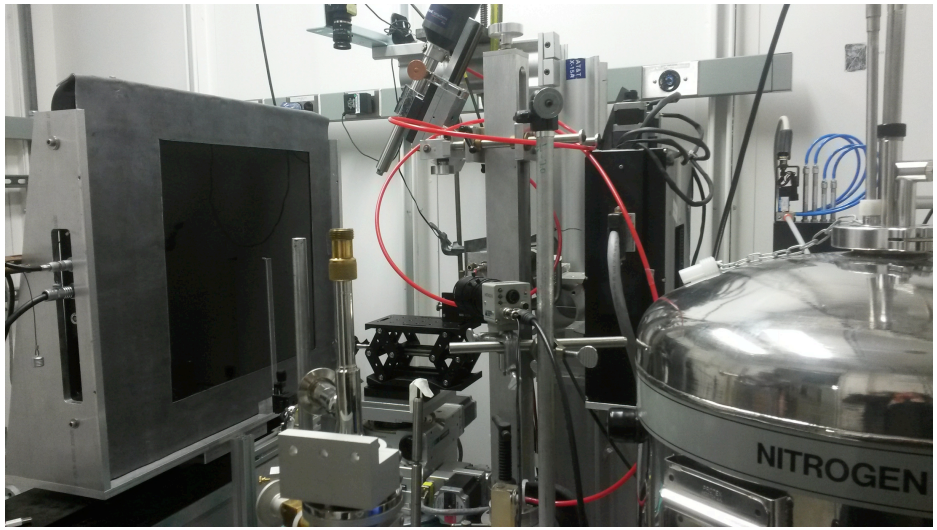
Mo: 580°C
MoGR: 580°C

PerkinElmer TG/DTA

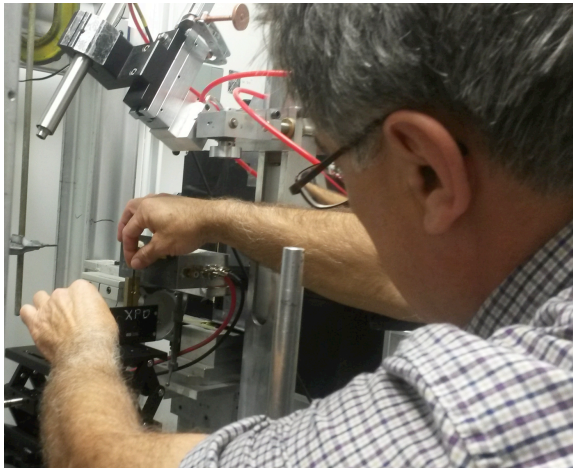
Mo: 980°C (Oxygen atmosphere)
Mo: 980°C (Inert atmosphere)
MoGR: 980°C (Oxygen atmosphere)
MoGR: 980°C (Inert atmosphere)
CuCD: difficult to cut

X-rays diffraction analysis at NSLS

2D XRD at X17A beamline at NSLS



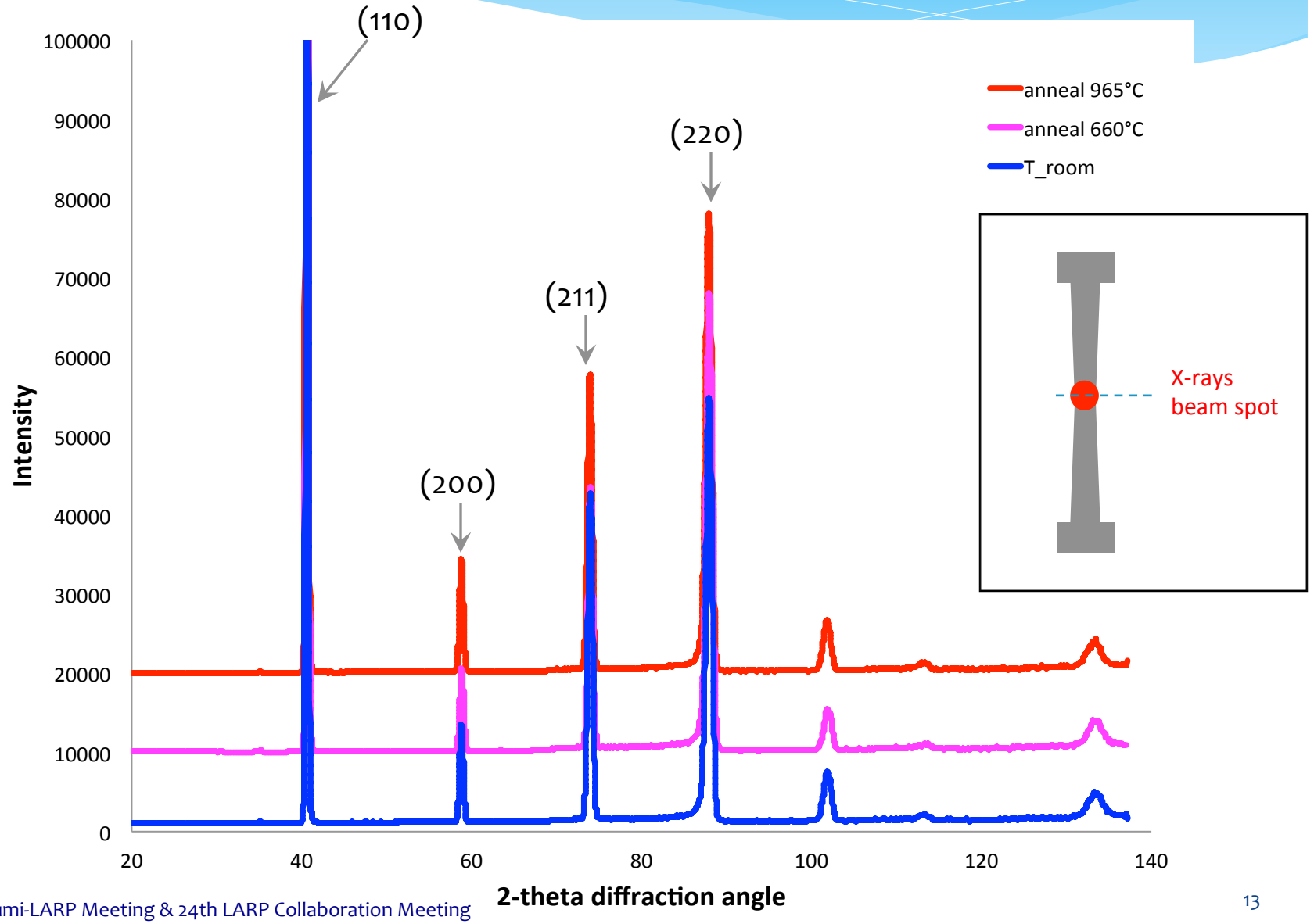
- **Monochromatic** X-Ray beam
- Energy = **60-70 keV**
- Beam spot size: 0.5 x 0.5 mm

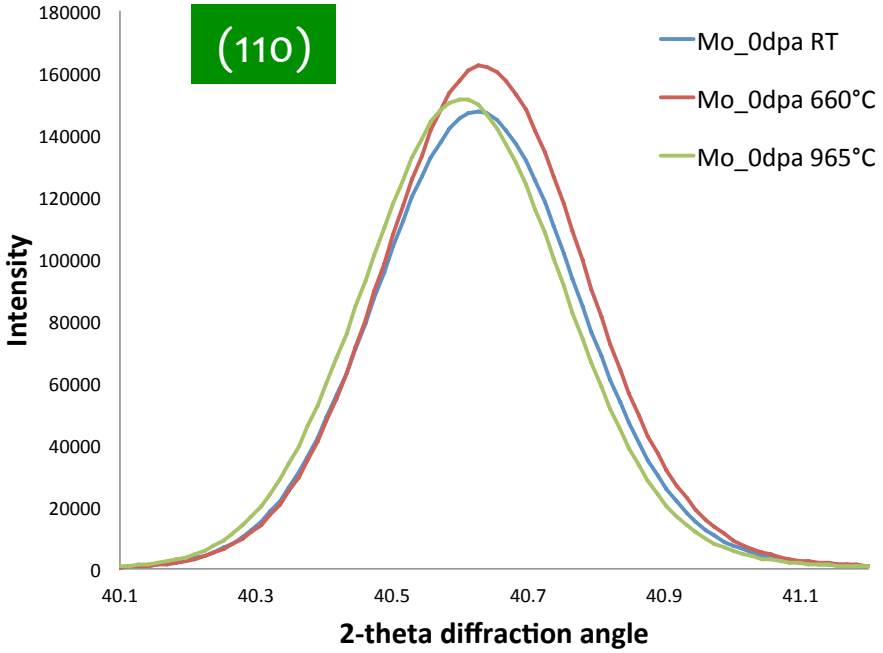


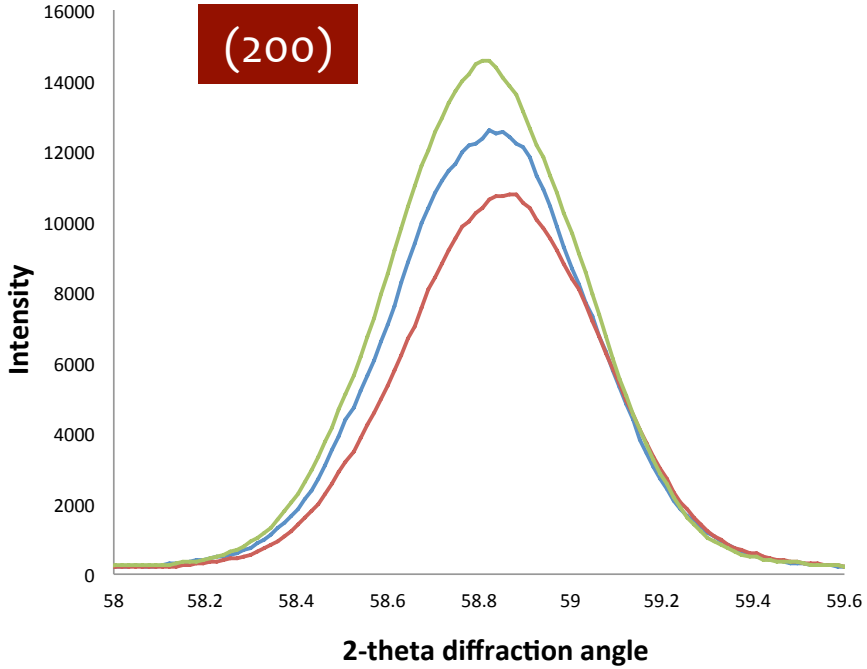
Results are presented for **Molybdenum:**

- unirradiated + annealing (660°C, 965°C)
- Irradiated at Tandem (28 MeV protons)

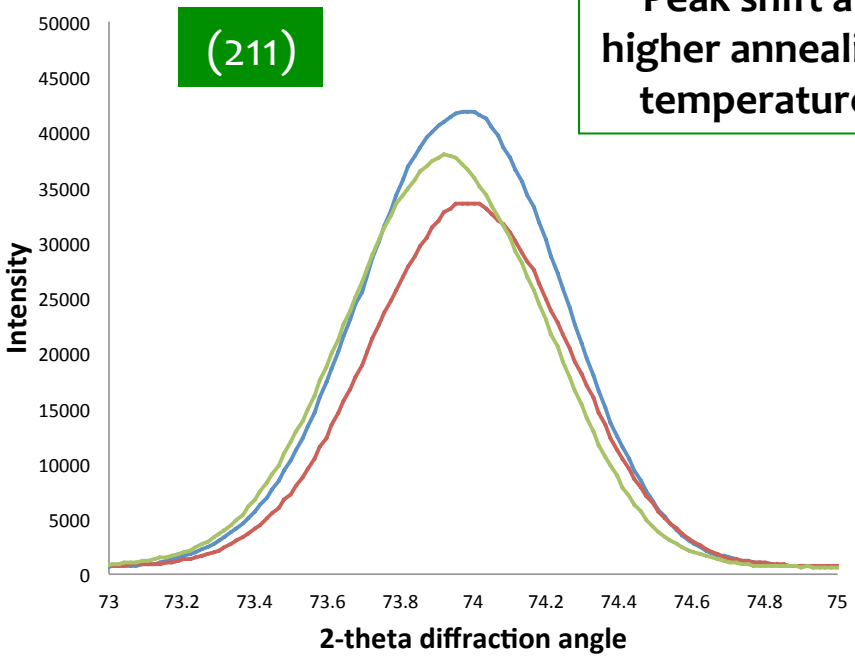
Mo – 0dpa – annealed (April'14)

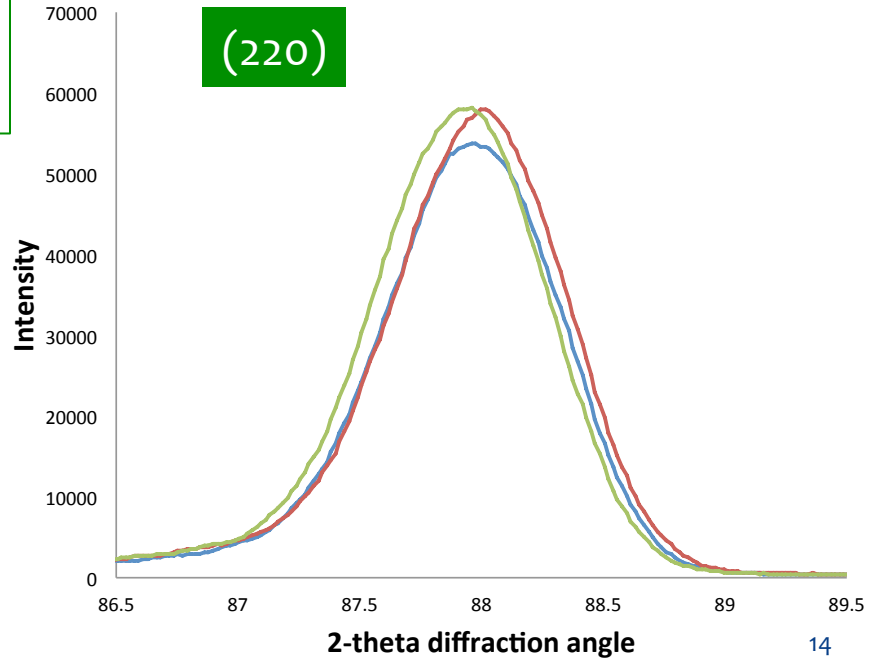




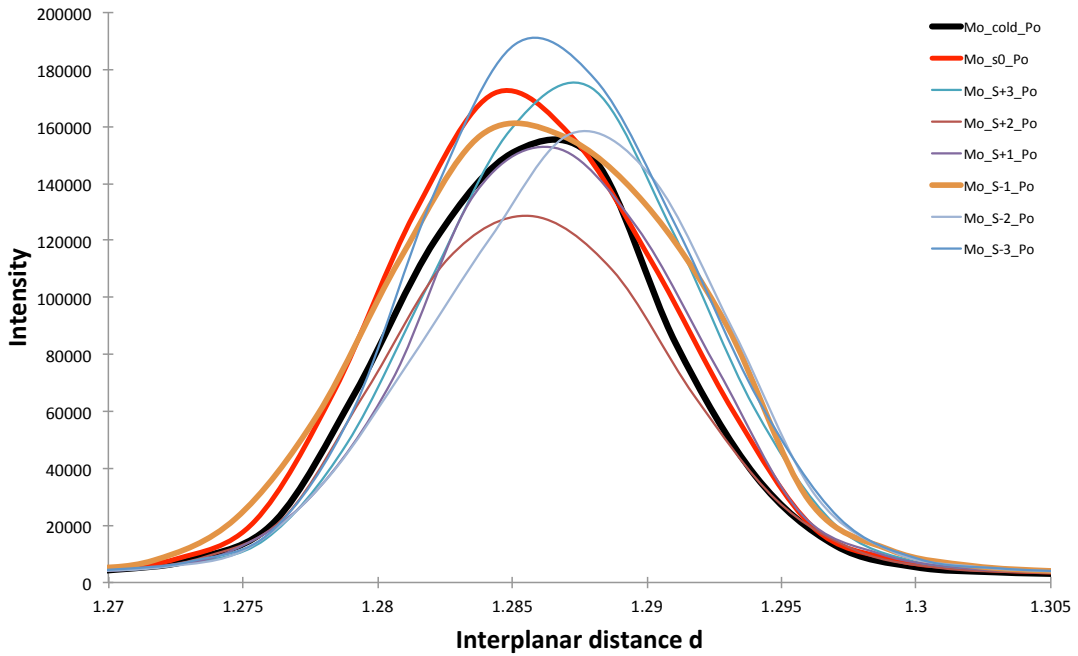


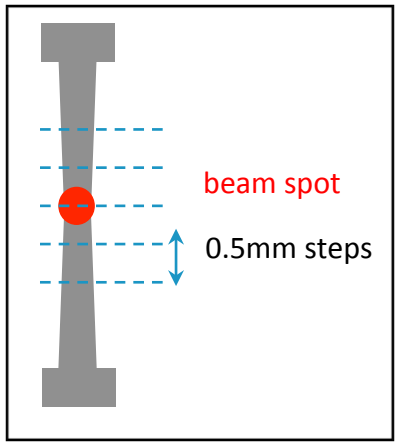
Peak shift at higher annealing temperature

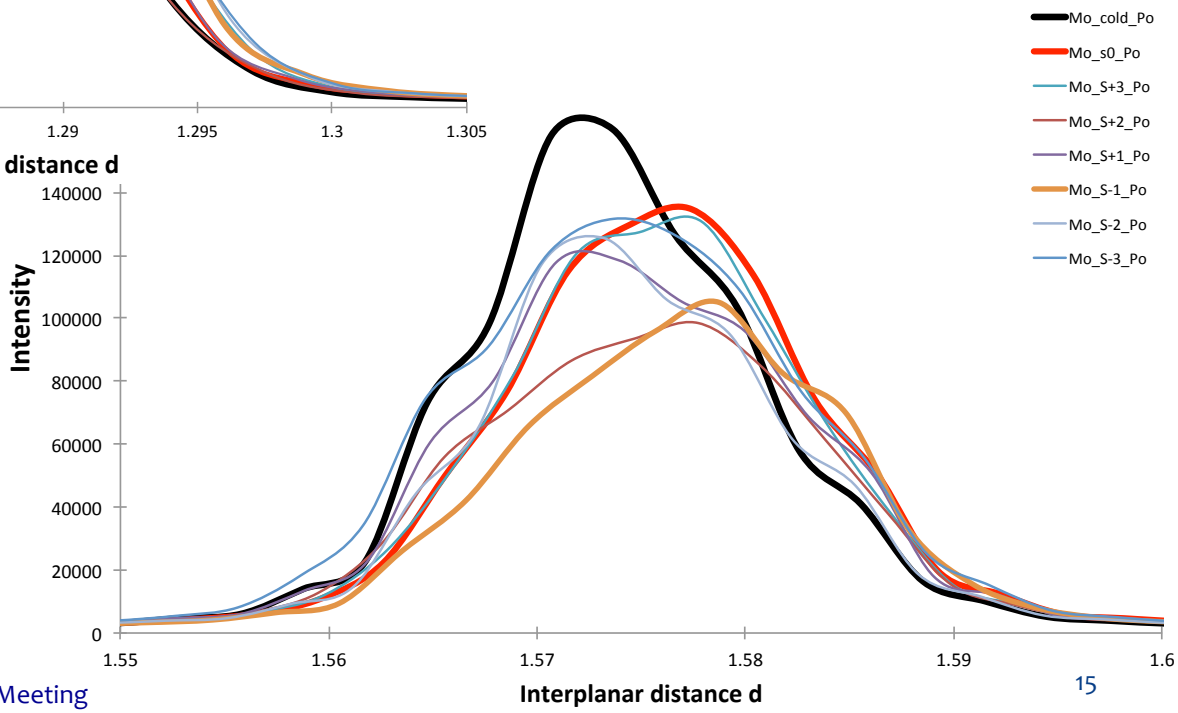




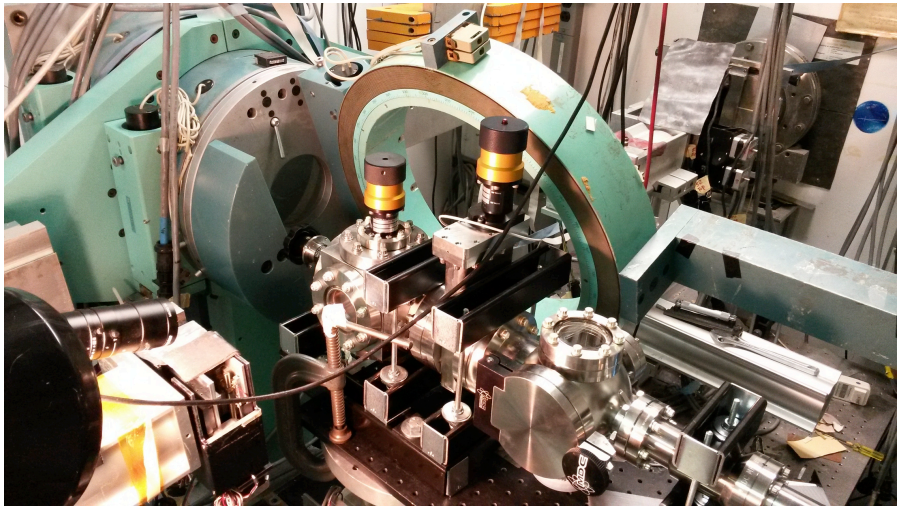
Mo – TANDEM (Sept.'14)







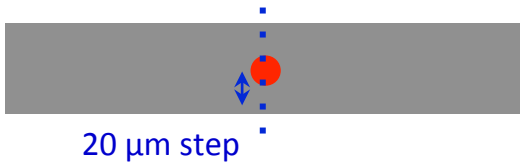
EDXRD at X17B1 beamline at NSLS



- Continuous **white** radiation
- Energy **up to 200 keV** (bulk analysis)
- **Fixed angle** 2θ (good for in-situ measurements)
- **Energy distribution** of scattered photons analyzed by a semiconductor detector

Discretized scan along sample thickness

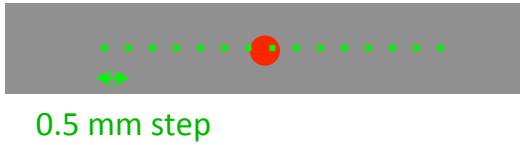
beam spot = 20 µm x 1 mm



Mo - TANDEM

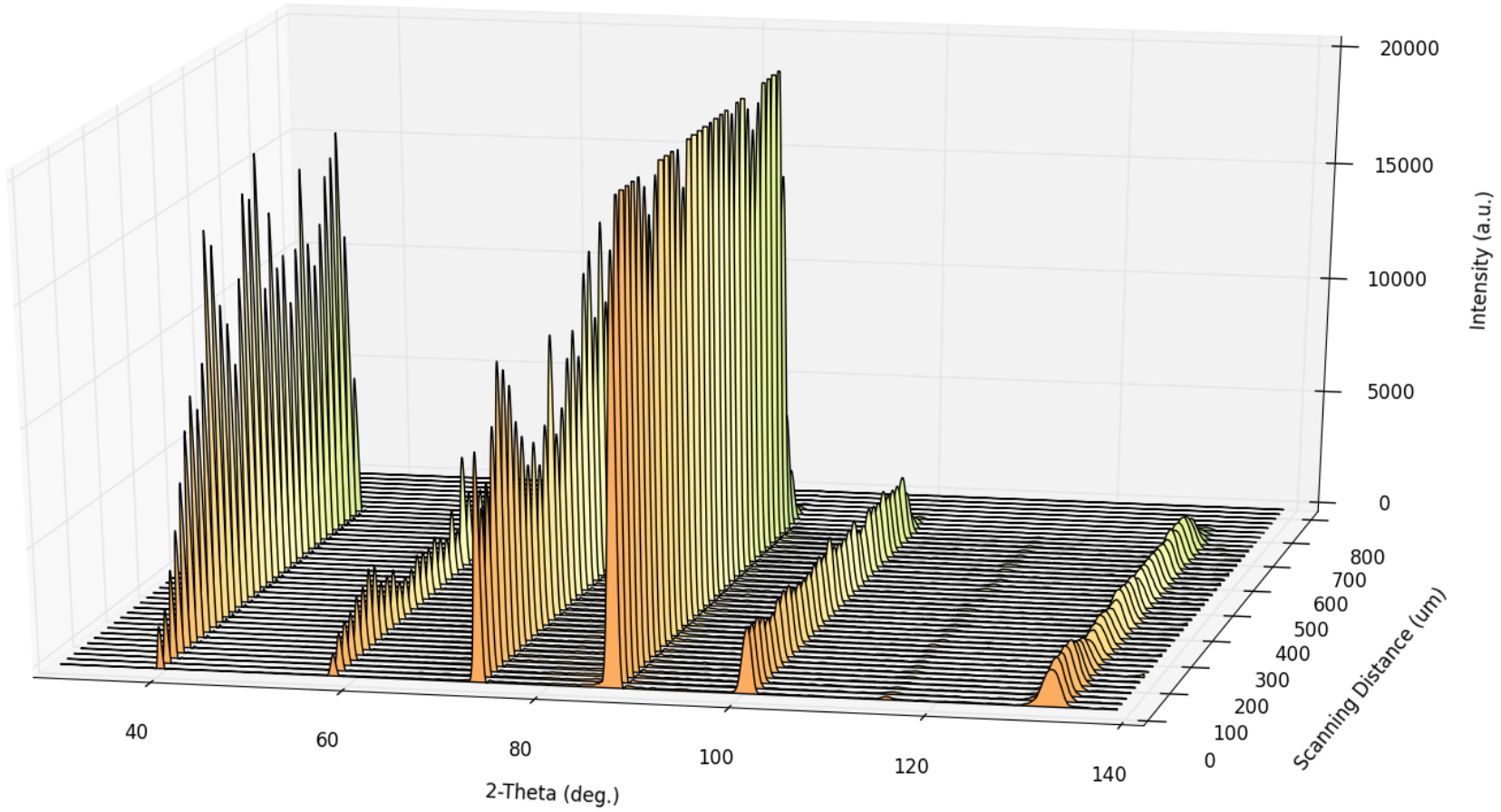
Discretized scan along sample length

beam spot = 20 µm x 0.5 mm

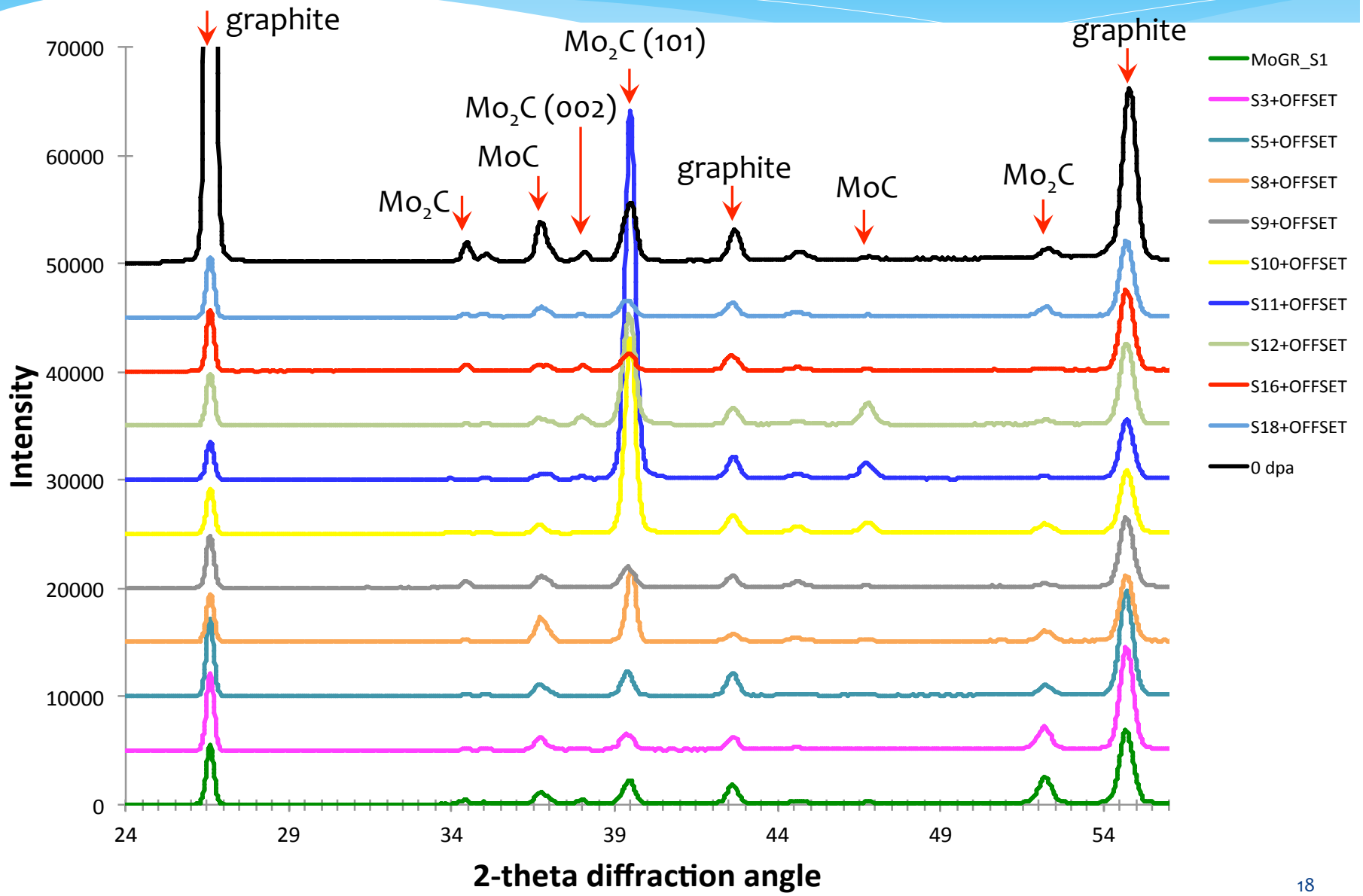


MoGR – BLIP: "last NSLS light"

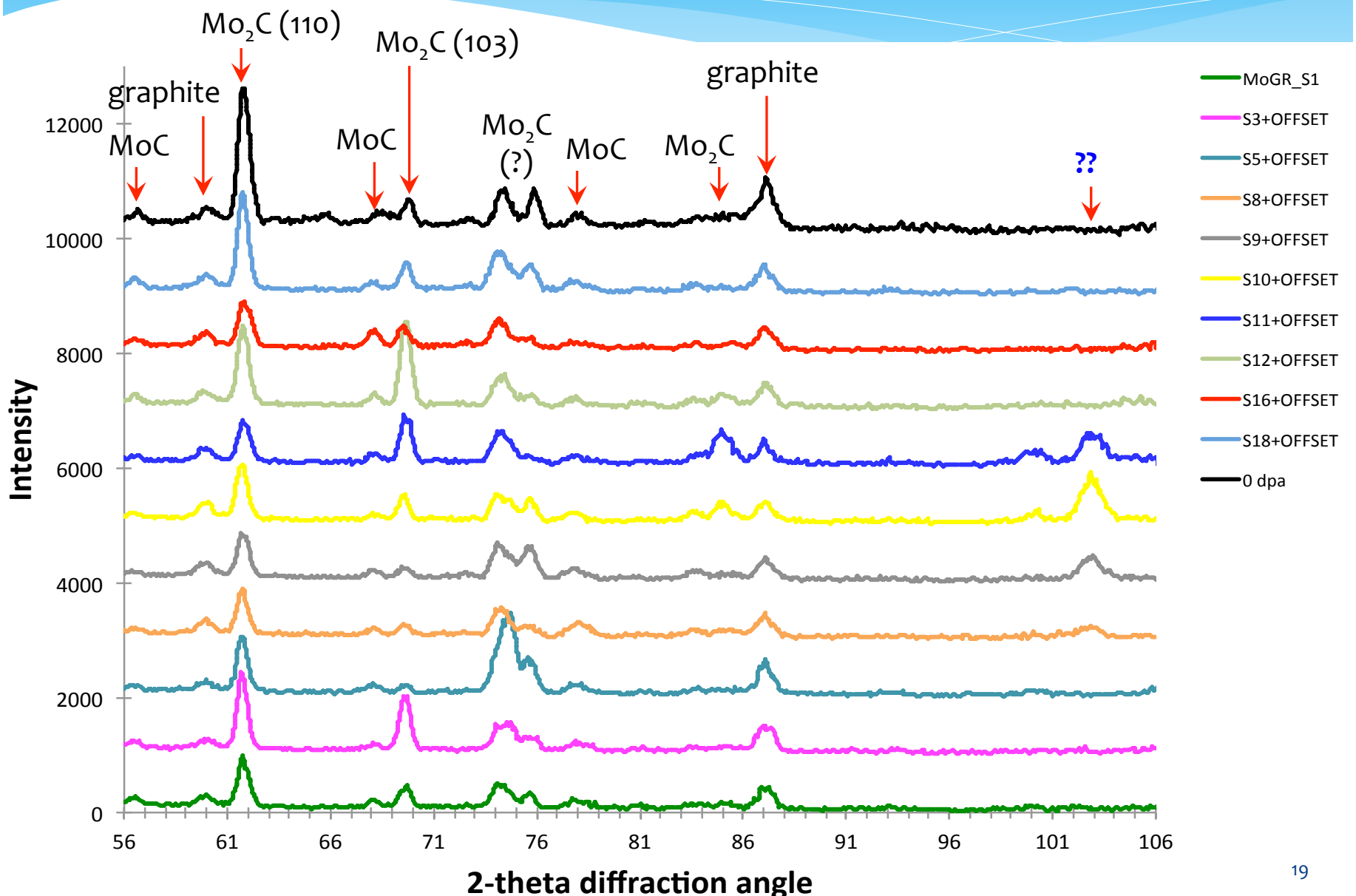
Mo – TANDEM


20000
15000
10000
5000
0
Intensity (a.u.)
800
700
600
500
400
300
200
100
0
Scanning Distance (um)
40
60
80
100
120
140
2-Theta (deg.)

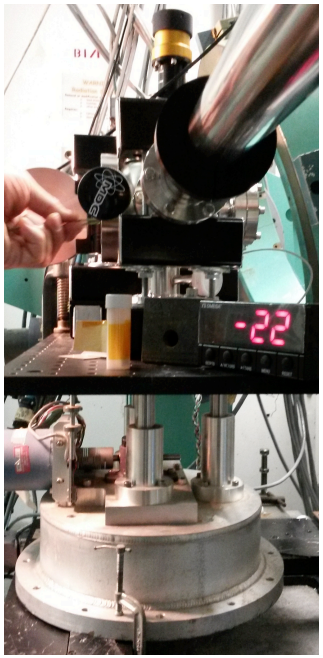
MoGR – BLIP: "last NSLS light" (I)

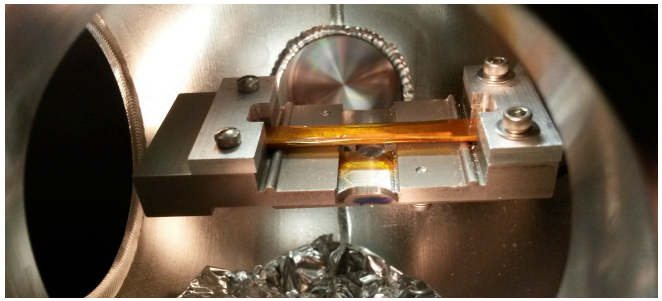


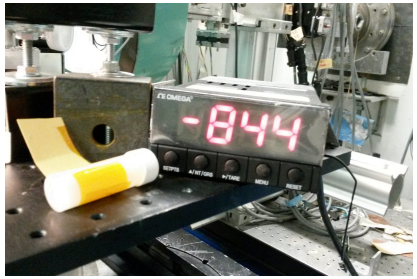
MoGR – BLIP: "last NSLS light" (II)



EDXRD at X17B1: phase and stress mapping

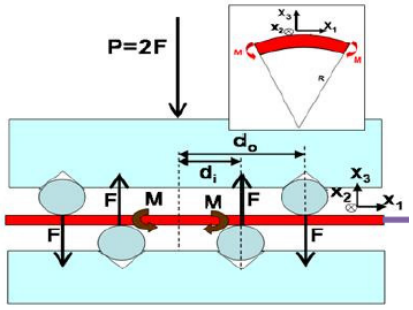






Load on sample placed in 4-points bending fixture

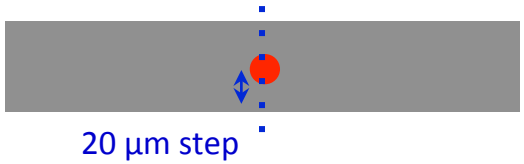
Allow calculation of elastic constants of the material.

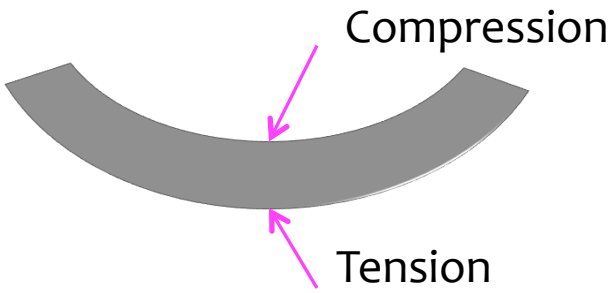


4-Point Bending

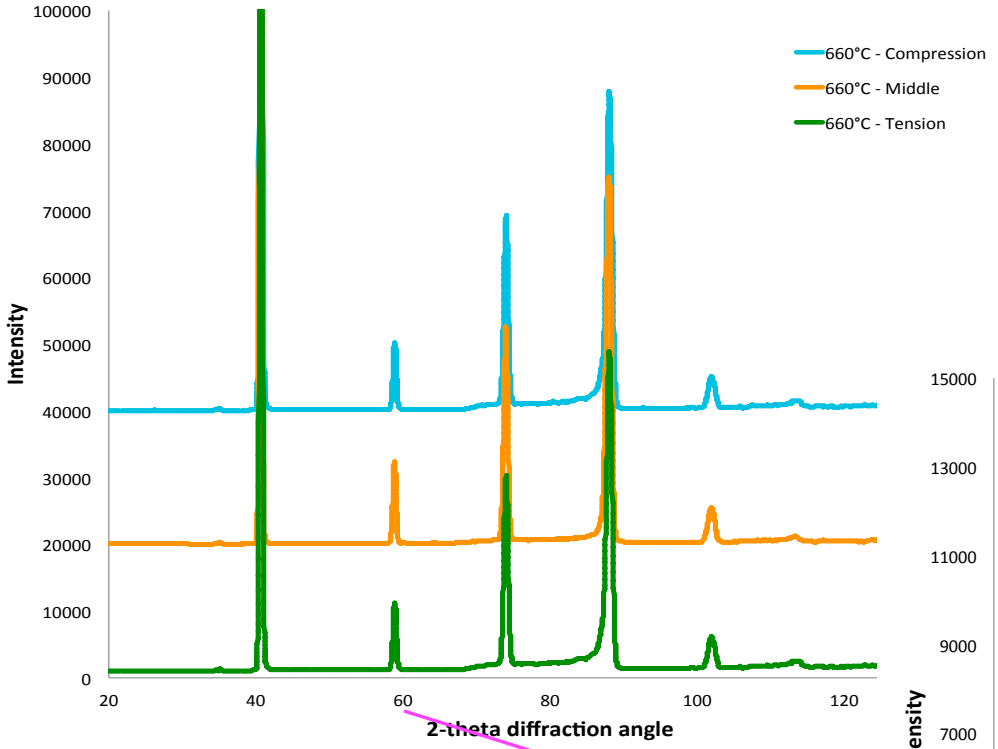
Discretized scan along sample thickness

beam spot = 20 µm x 1 mm

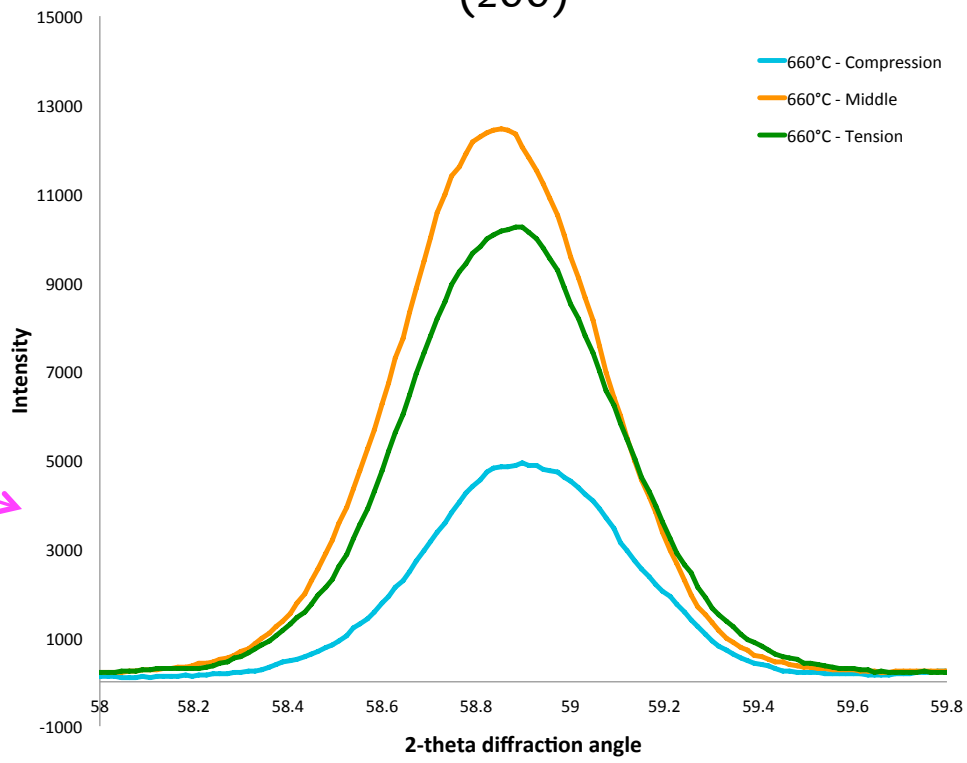




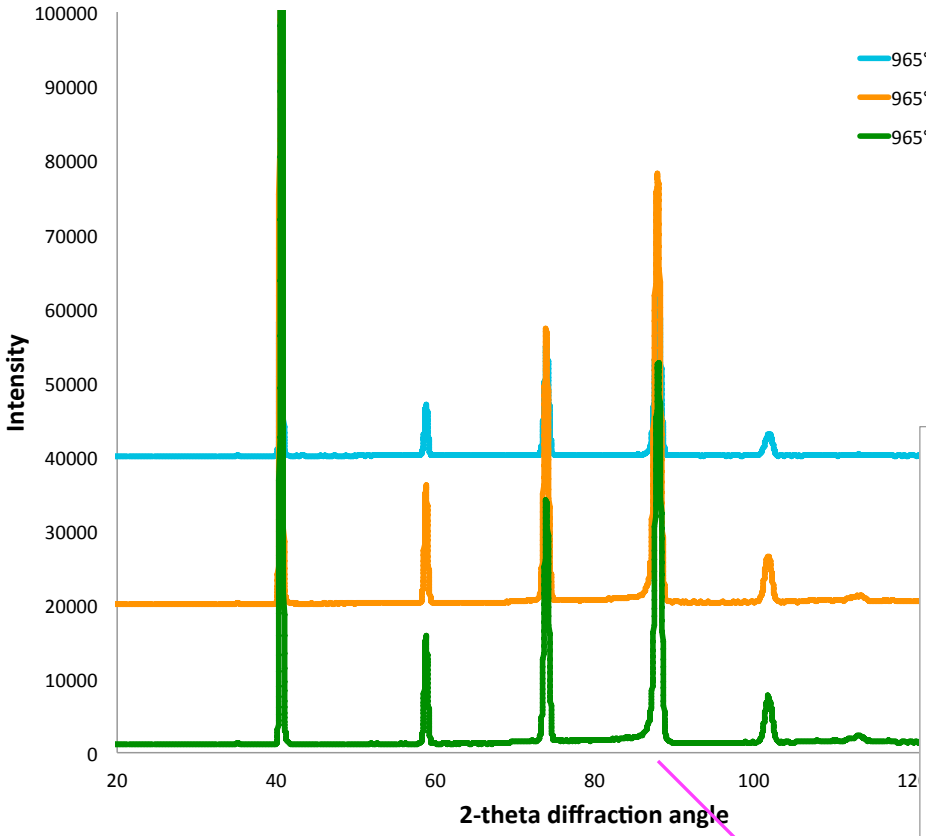
Mo – odpa – 660°C – 50lbs



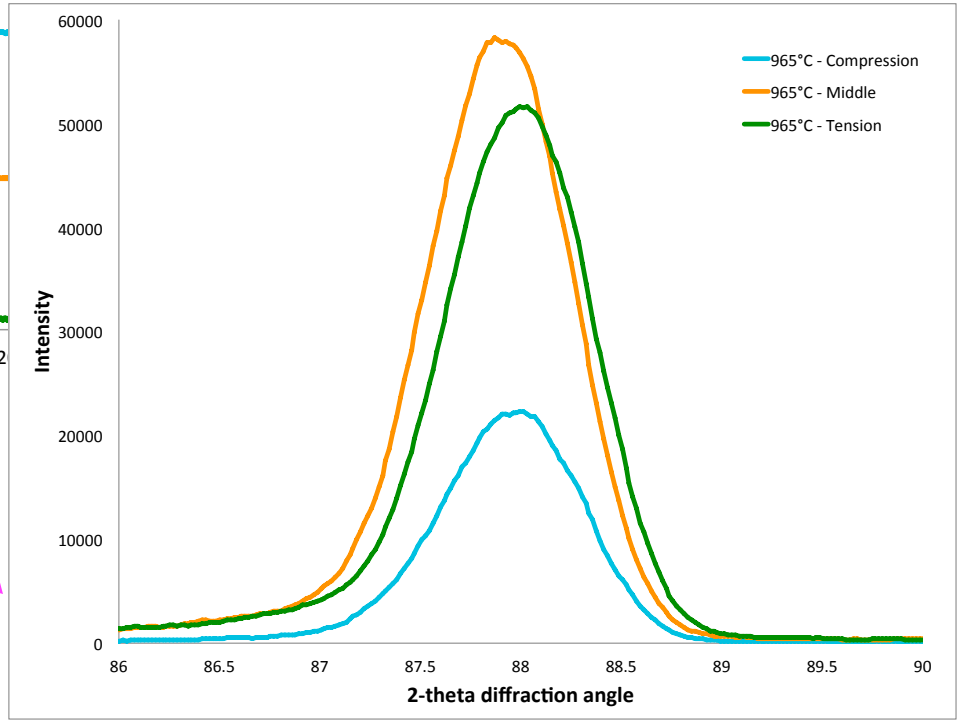
(200)



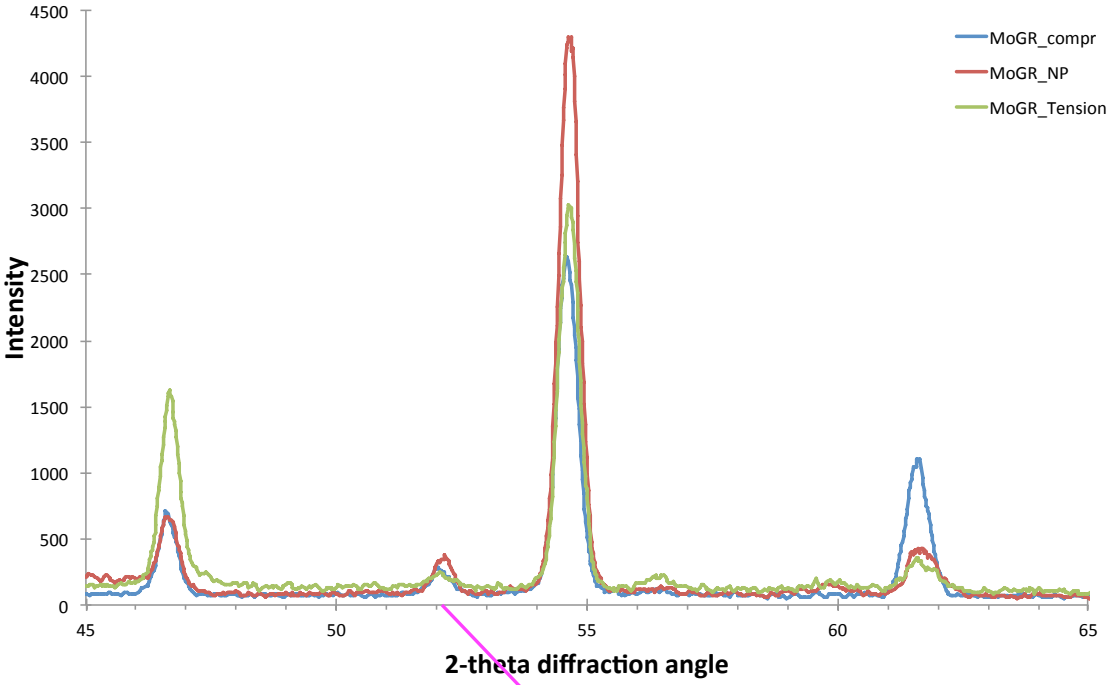
Mo – odpa– 965°C– 50lbs



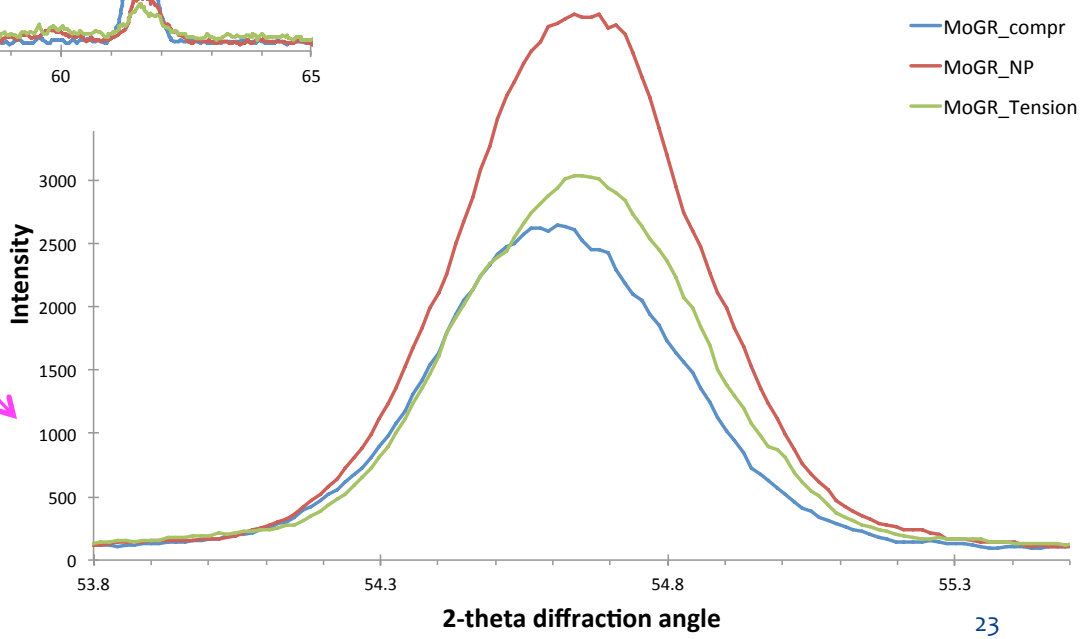
(220)



MoGr – TANDEM – 50lbs



Peak of Mo_2C



Wrap up

* Extensive work of LHC collimator materials characterization performed at BNL
* Focus on role of temperature affecting parameter and structural changes in materials
* Characterization of pristine samples at CFN:
  - Annealing up to high T
  - Microscopical observations
  - Thermal analysis (oxidation and phase transformation)
* X-rays data analysis from NSLS experiments on:
  - Unirradiated annealed Mo (loaded and unloaded)
  - Mo irradiated at TANDEM (unloaded)
  - MoGR irradiated at TANDEM (loaded)
  - "last light" MoGR irradiated at BLIP for few hours (unloaded)

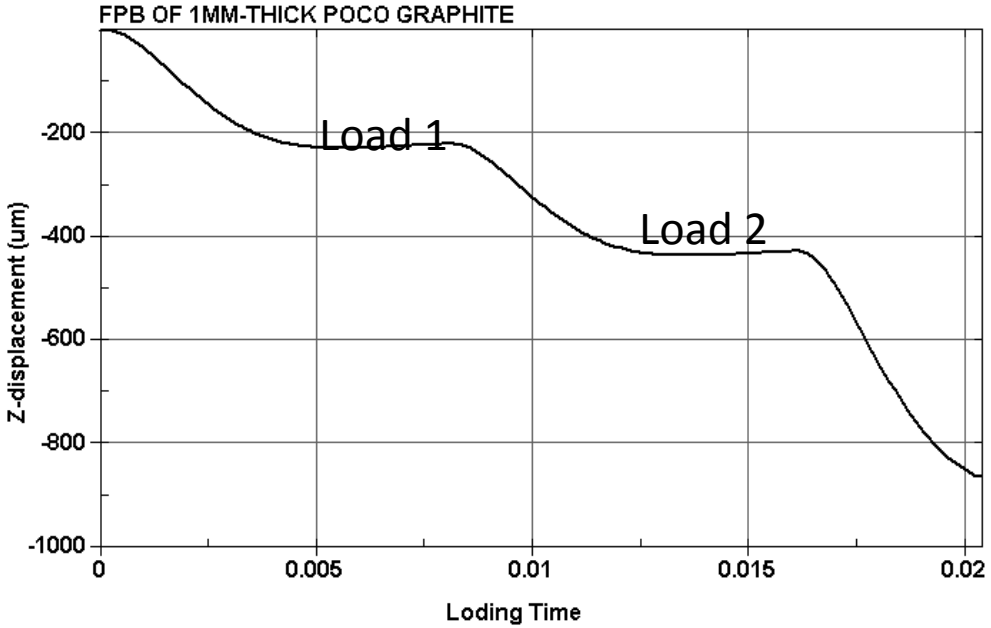
Plans for the future

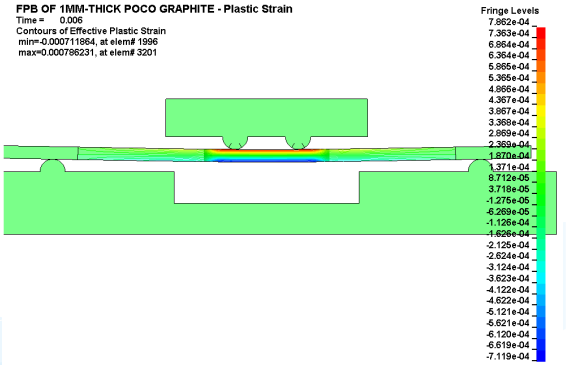
* Finalize thermal characterization at CFN on Mo and MoGr (possibly also CuCD)
* Follow up XRD analysis on all available data from last run in September 2014
* Start post-irradiation analysis on hottest samples (BLIP irradiated), still waiting to cool down to be handled
* Proposal for further XRD studies at new NSLS II beamline
* Plan additional irradiation tests on latest developed grades of collimator materials

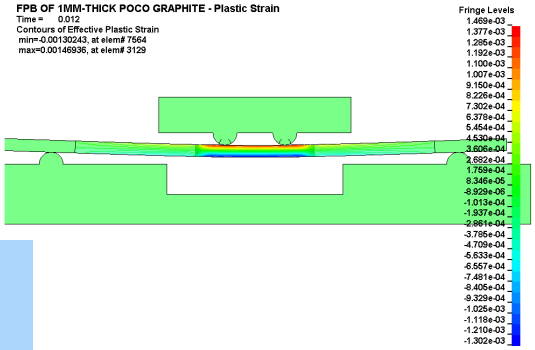
Thank you for your attention

Let's now take some time to discuss…

BACKUP slides

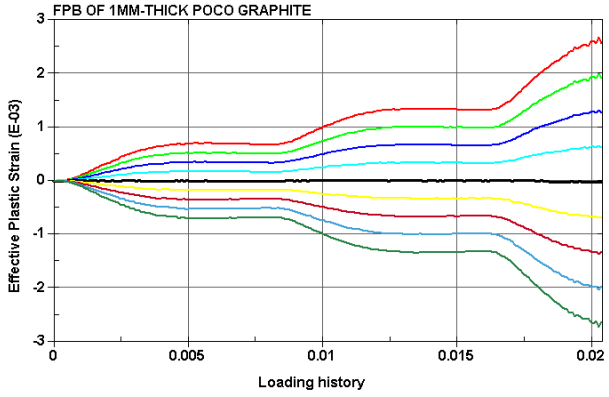


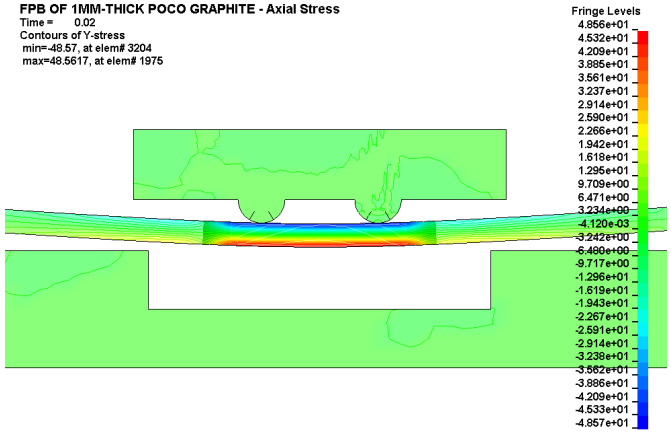


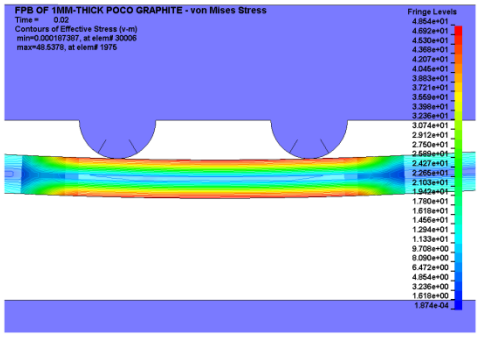


EXCELLENT matching with the experimental data at X17B1 using x-rays and loading to assess the displacement.

GOAL: LATTICE strain and global strain are very different in magnitude







MoGR – protons at BLIP (III)