X-ray Diffraction studies of irradiated Materials at BNL Experimental Facilities - N. Simos (Oct. 9, 2014)

MATERIALS:

Graphite polymorphs, h-BN, Be, AlBeMet, Tungsten, Molybdenum, Glidcop, Mo-Gr, Cu-CD, carbon fiber composites, superalloys (Ti6Al4V, s-INVAR and gum metal) and metal-metal interfaces

Irradiations:

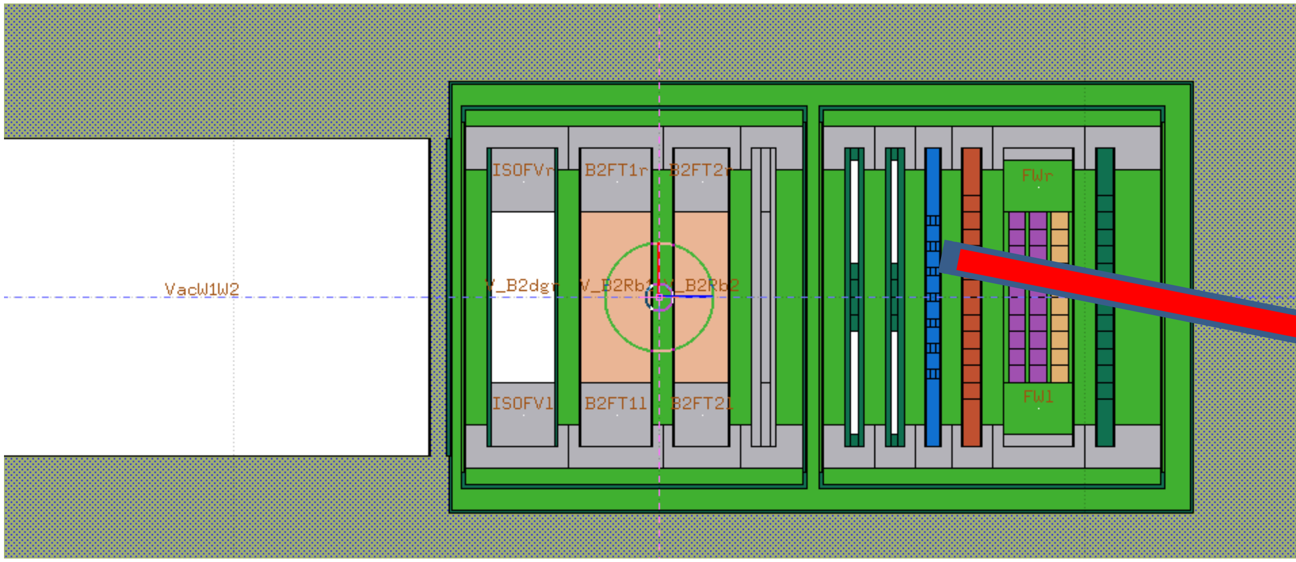
118-200 MeV Protons at BNL BLIP
Fast Neutrons at BNL BLIP
28 MeV Protons at Tandem
Neutrons at Tandem (low temperature)

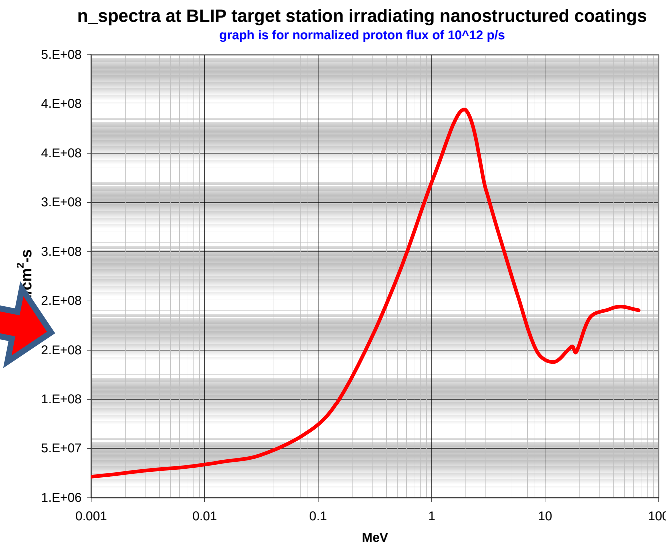
X-ray Studies (completed)

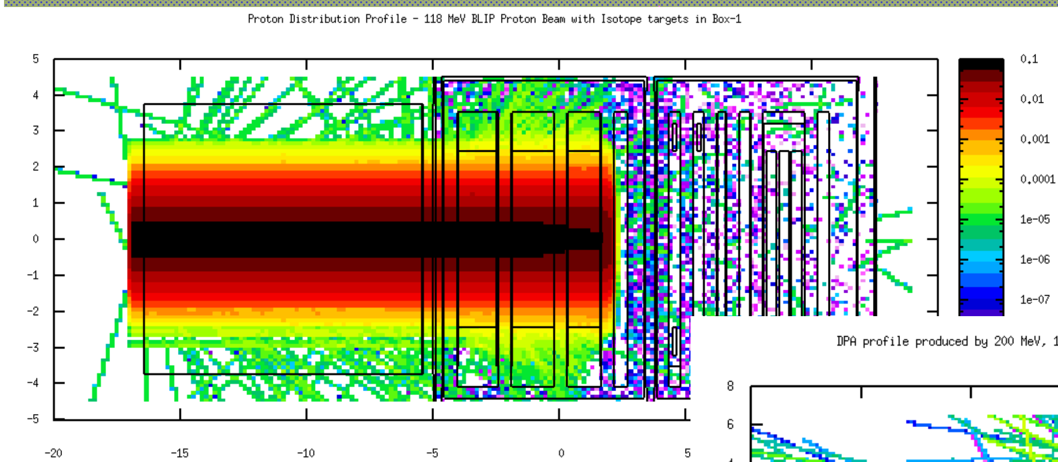
(a) using monochromatic high energy X-rays
(B) high energy x-rays EDXRD (Phase I & Phase II)

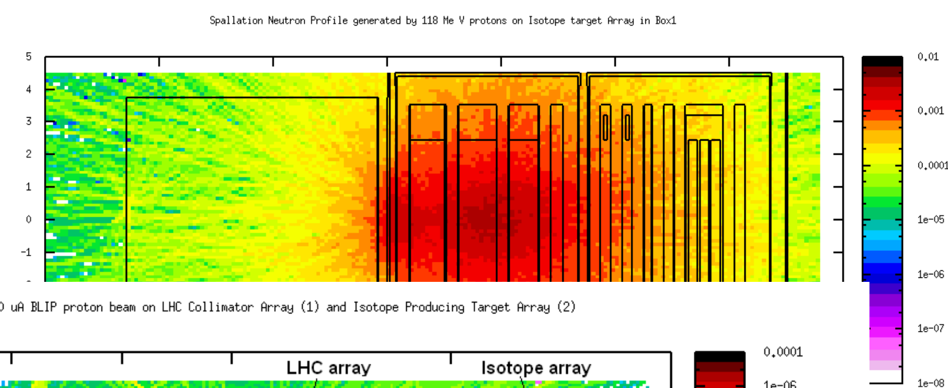
MICROSCOPY (at CFN): SEM/EDS, annealing, DSC and TG/DTA

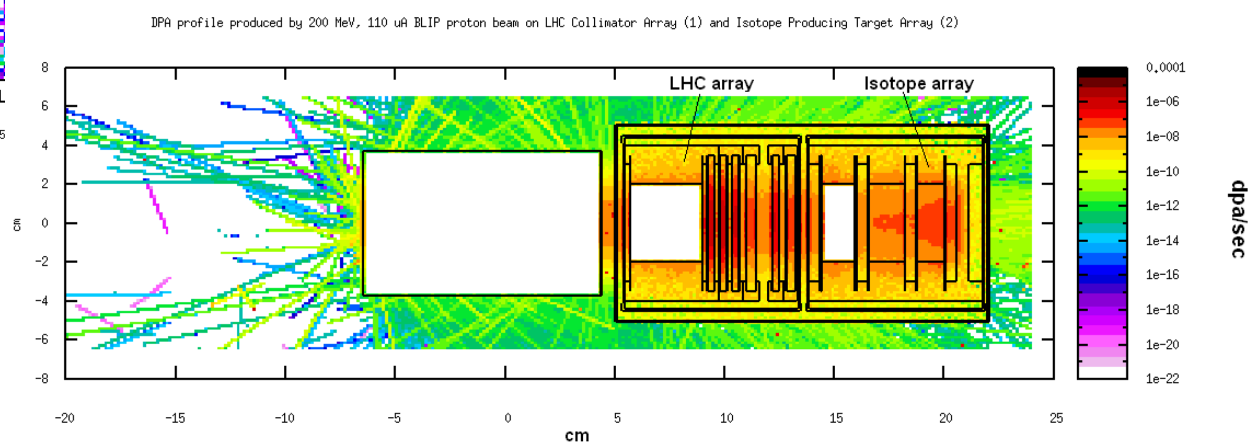
Spallation Neutron Irradiation at BLIP





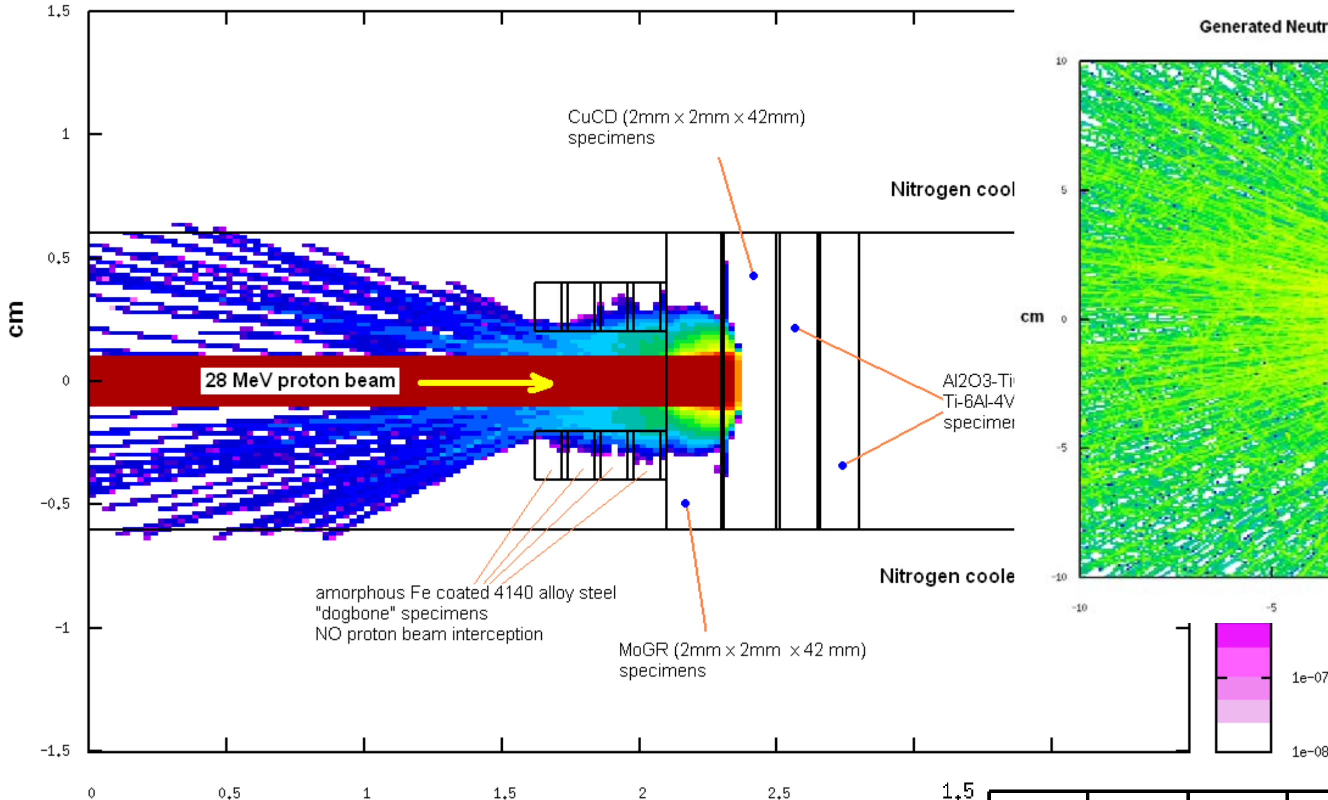


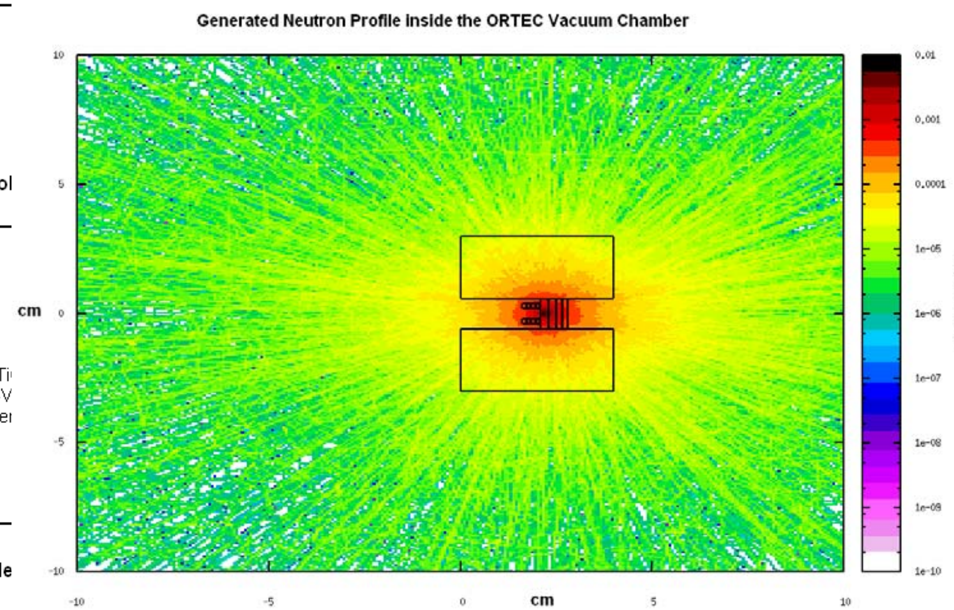


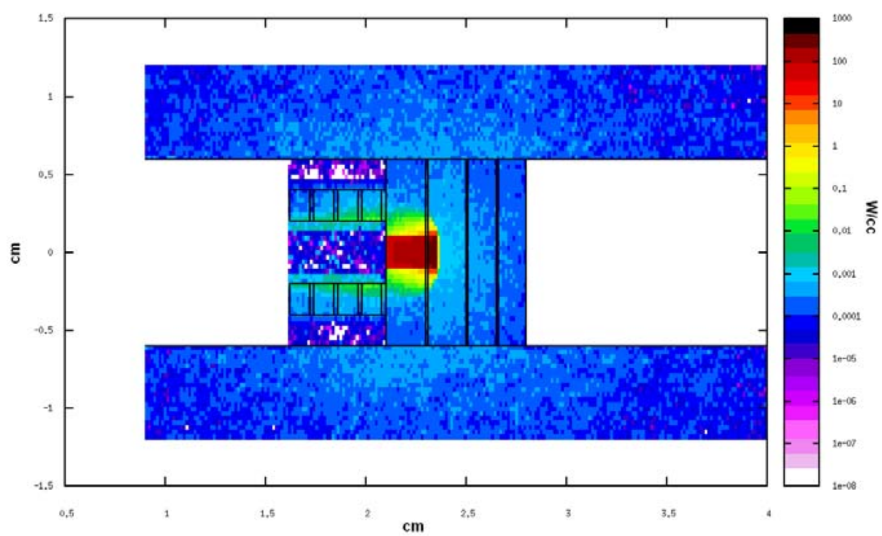


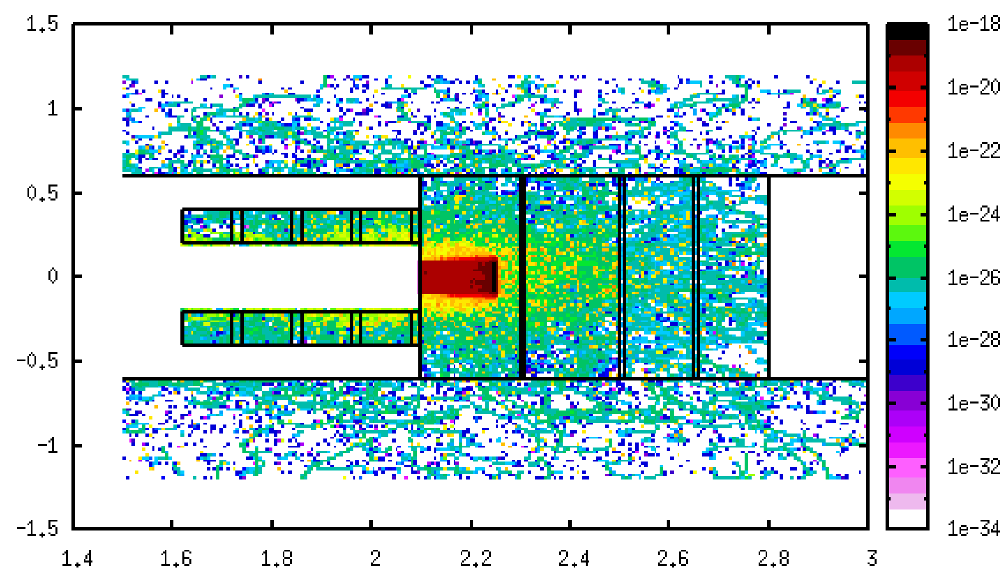
28 MeV Proton Irradiation at Tandem

Localized Damage
Followed by EDXRD Studies









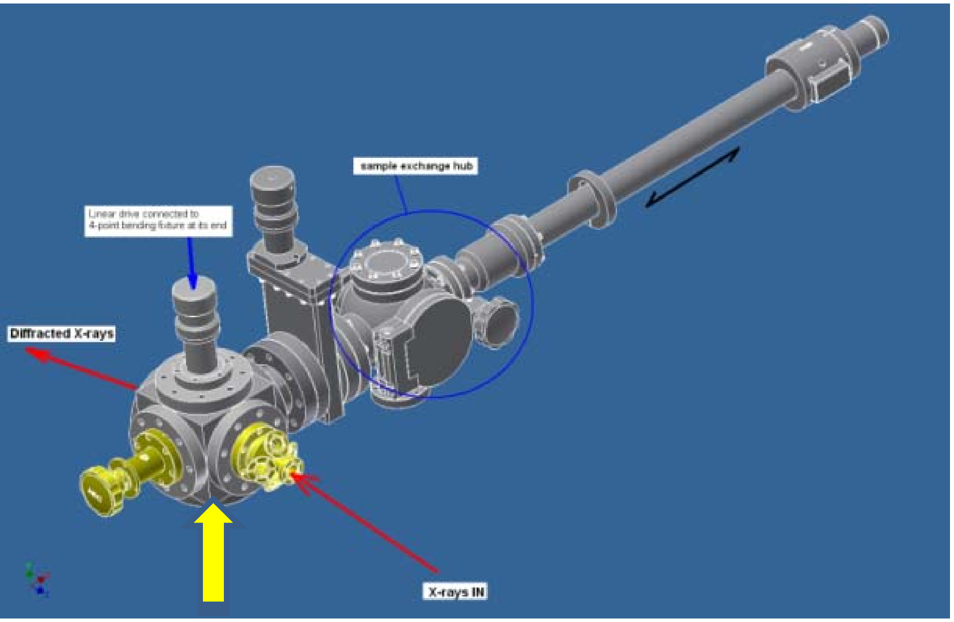
Multi-functional stage capable of handling

Real size irradiated specimens, under vacuum and four point bending state of stress

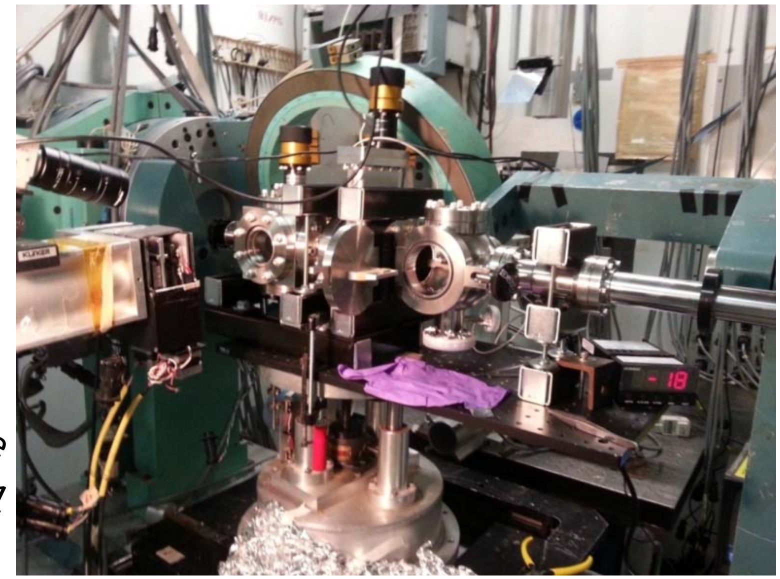
and eventually

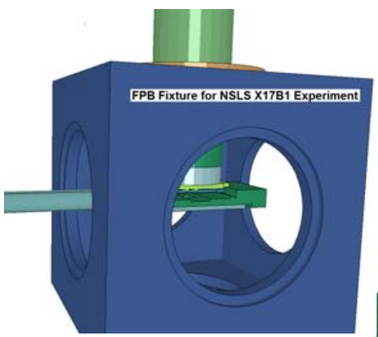
Heating/annealing via a portable, collimated laser beam

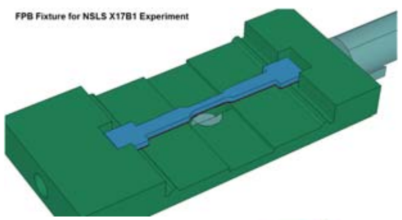
Tensile stress-strain test

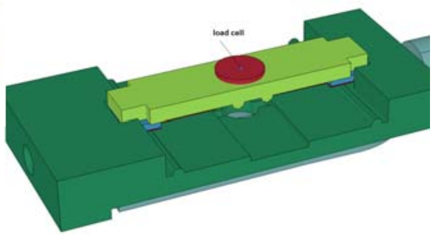


From concept to a versatile experimental stage at X17B1 beamline at NSLS

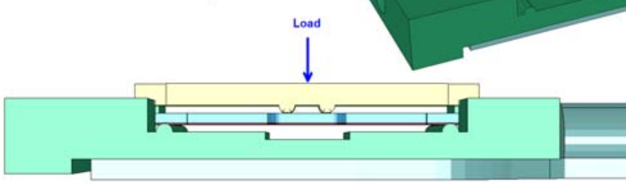


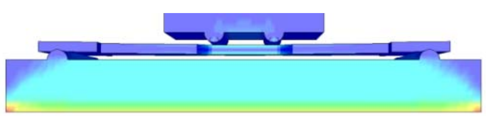




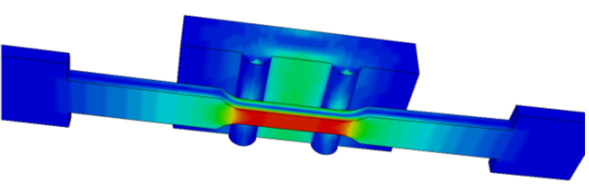


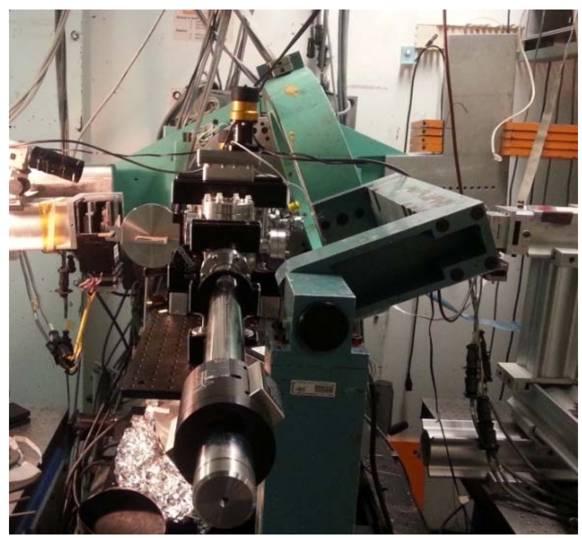
FPB Fixture for NSLS X17B1 Experiment

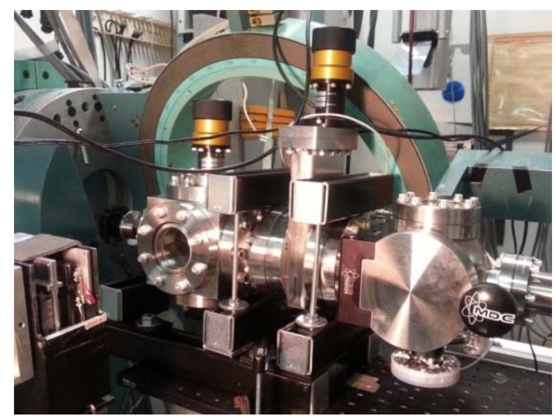


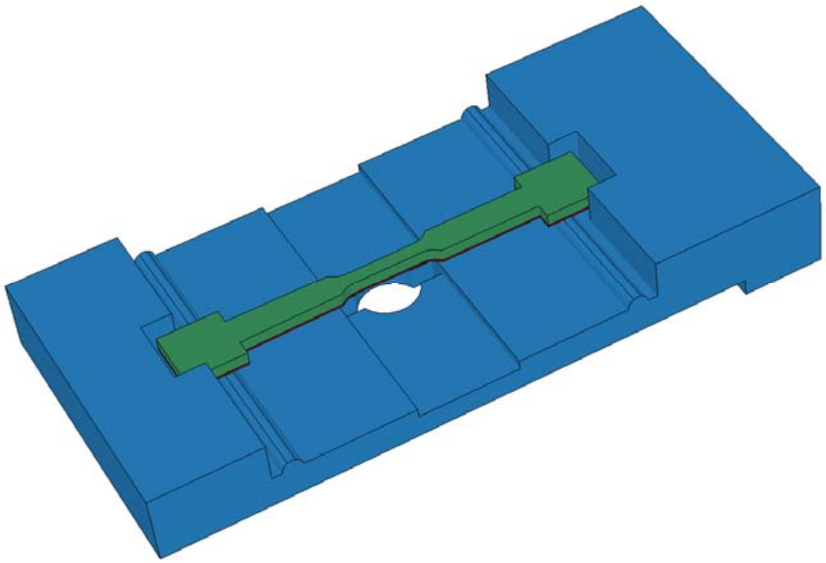


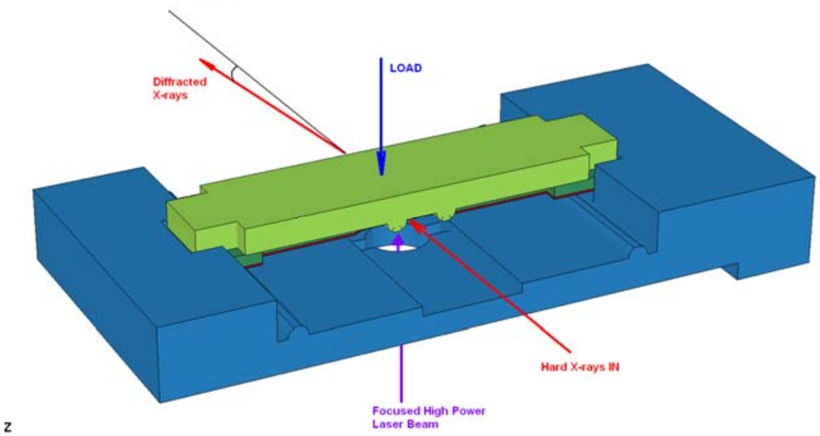
FPB of 1mm-thick Poco Graphite Tensile Test Specimen

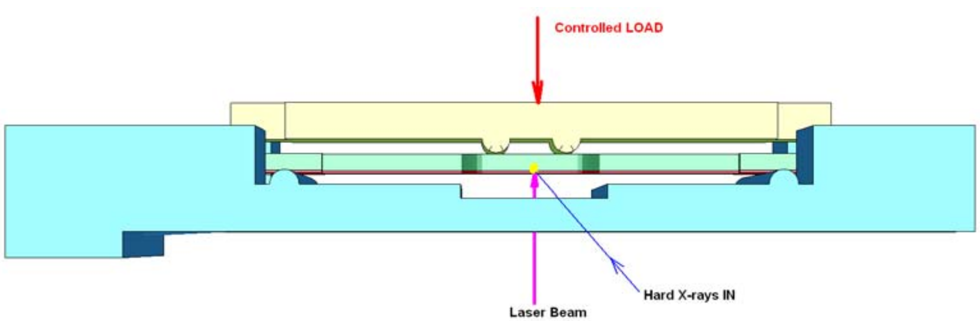


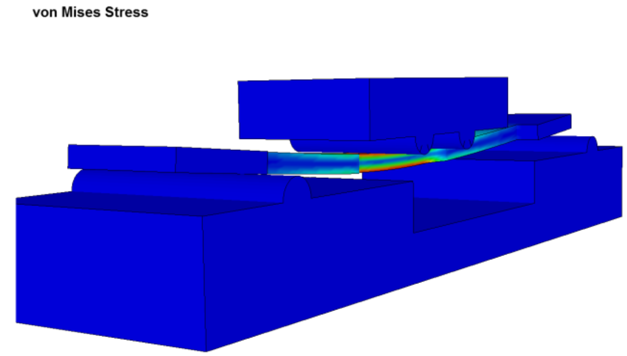




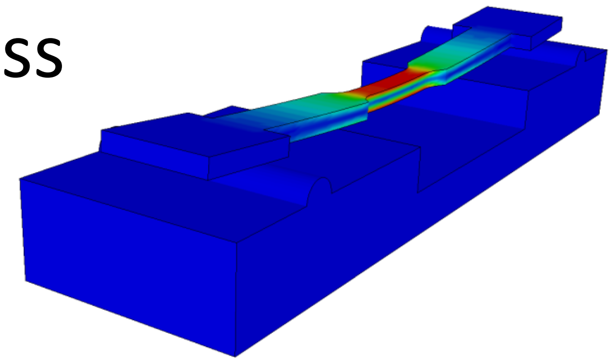


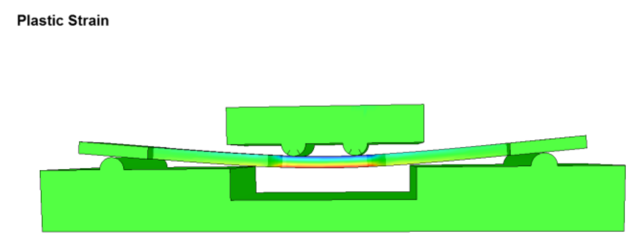




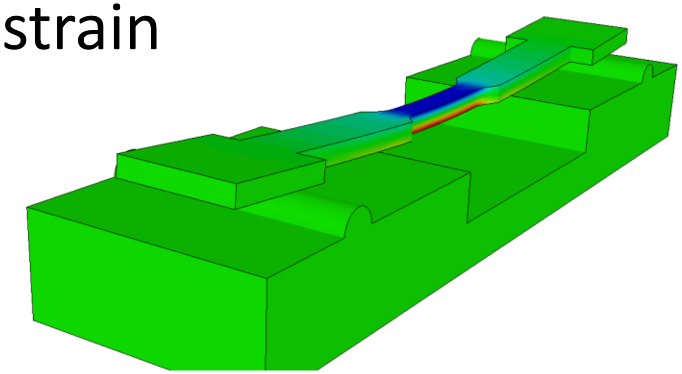


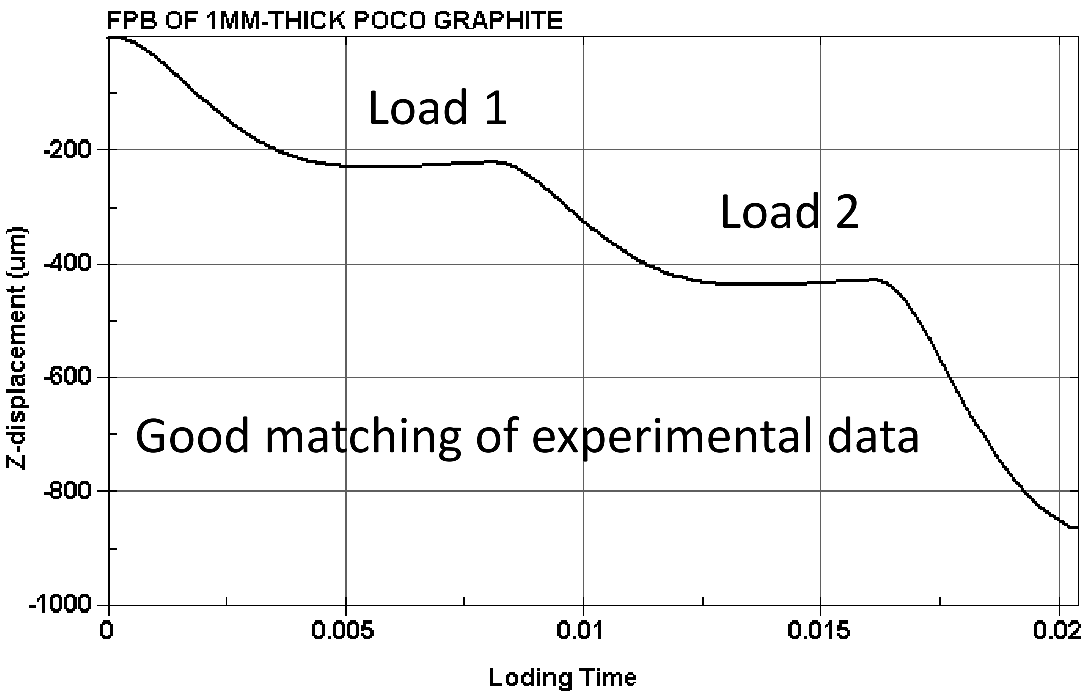
stress

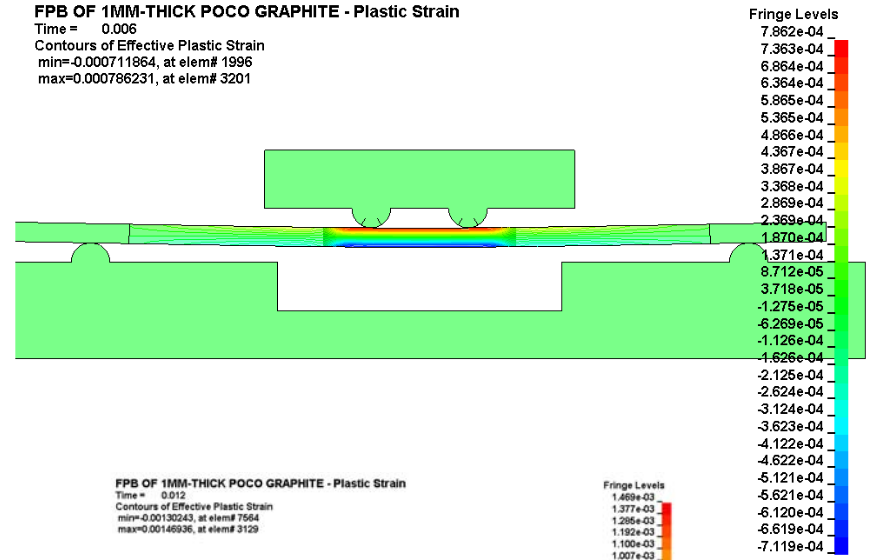


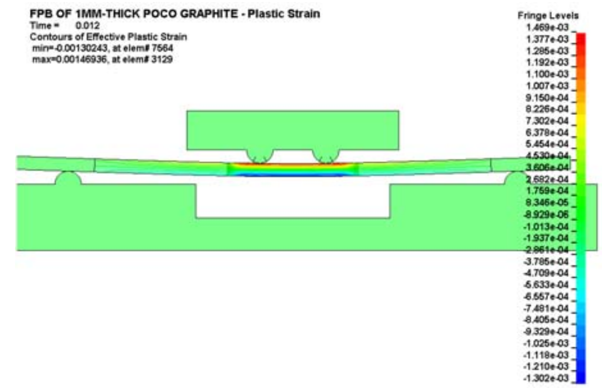


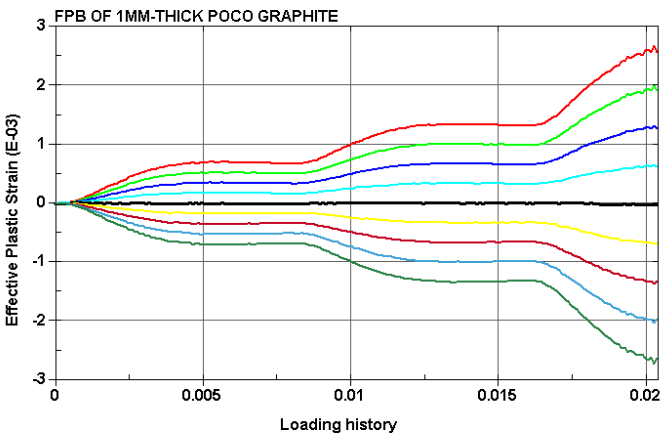
strain

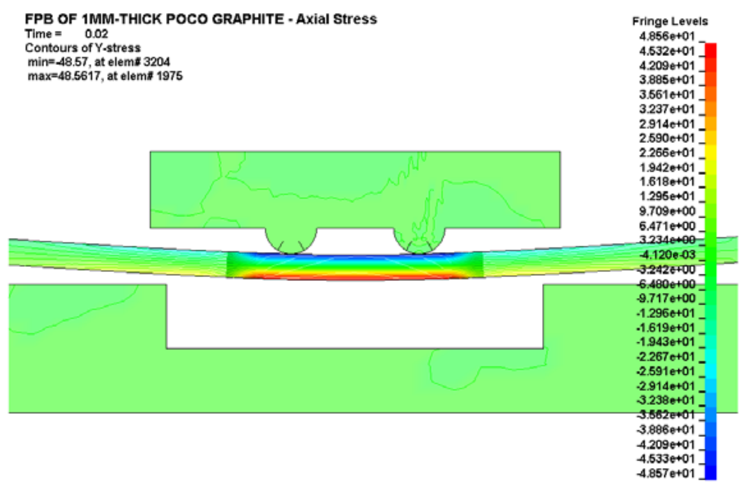



FPB OF 1MM-THICK POCO GRAPHITE
Load 1
Load 2
Good matching of experimental data
Z-displacement (um)
-200
-400
-600
-800
-1000
0
0.005
0.01
0.015
0.02
Loding Time


FPB OF 1MM-THICK POCO GRAPHITE - Plastic Strain
Time = 0.006
Contours of Effective Plastic Strain
min=-0.000711864, at elem# 1996
max=0.000786231, at elem# 3201
Fringe Levels
7.862e-04
7.363e-04
6.864e-04
6.364e-04
5.865e-04
5.365e-04
4.866e-04
4.367e-04
3.867e-04
3.368e-04
2.869e-04
2.369e-04
1.870e-04
1.371e-04
8.712e-05
3.718e-05
-1.275e-05
-6.269e-05
-1.126e-04
-1.626e-04
-2.125e-04
-2.624e-04
-3.124e-04
-3.623e-04
-4.122e-04
-4.622e-04
-5.121e-04
-5.621e-04
-6.120e-04
-6.619e-04
-7.119e-04


FPB OF 1MM-THICK POCO GRAPHITE - Plastic Strain
Time = 0.012
Contours of Effective Plastic Strain
Fringe Levels


FPB OF 1MM-THICK POCO GRAPHITE
Effective Plastic Strain (E-03)
3
2
1
0
-1
-2
-3
0
0.005
0.01
0.015
0.02
Loading history


FPB OF 1MM-THICK POCO GRAPHITE - Axial Stress
Time = 0.02
Contours of Y-stress
min=-48.57, at elem# 3204
max=48.5617, at elem# 1975
Fringe Levels
4.856e+01
4.532e+01
4.209e+01
3.885e+01
3.561e+01
3.237e+01
2.914e+01
2.590e+01
2.266e+01
1.942e+01
1.618e+01
1.295e+01
9.709e+00
6.471e+00
3.234e+00
-4.120e-03
-3.242e+00
-6.480e+00
-9.717e+00
-1.296e+01
-1.619e+01
-1.943e+01
-2.267e+01
-2.591e+01
-2.914e+01
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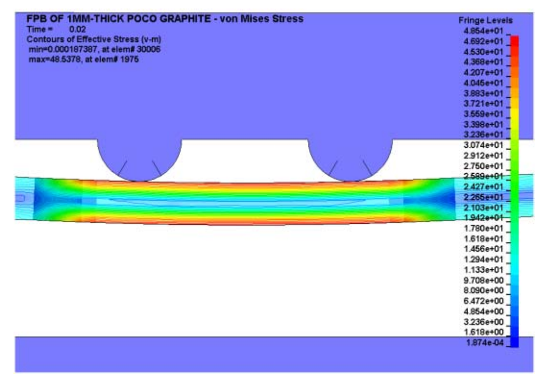
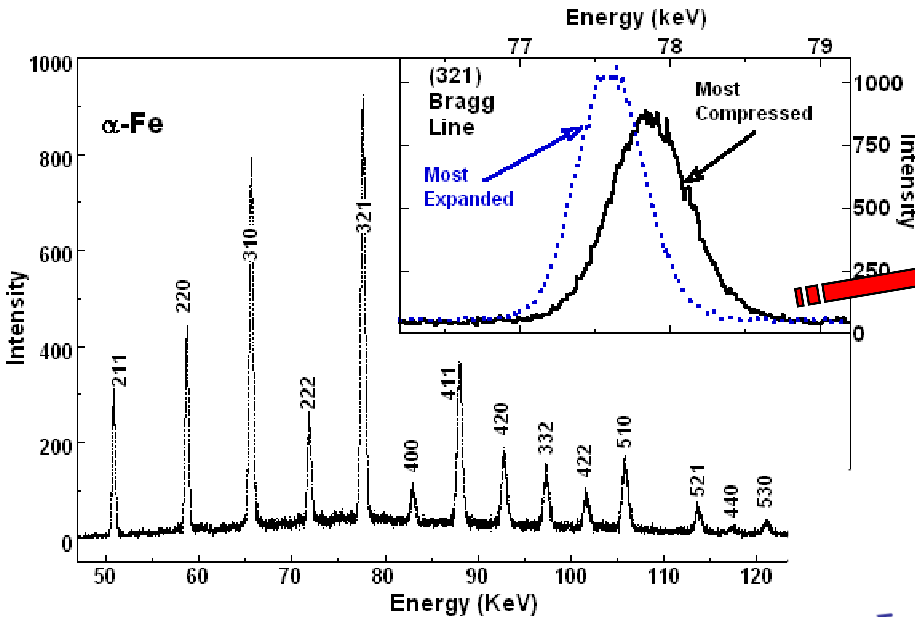
-4.857e+01

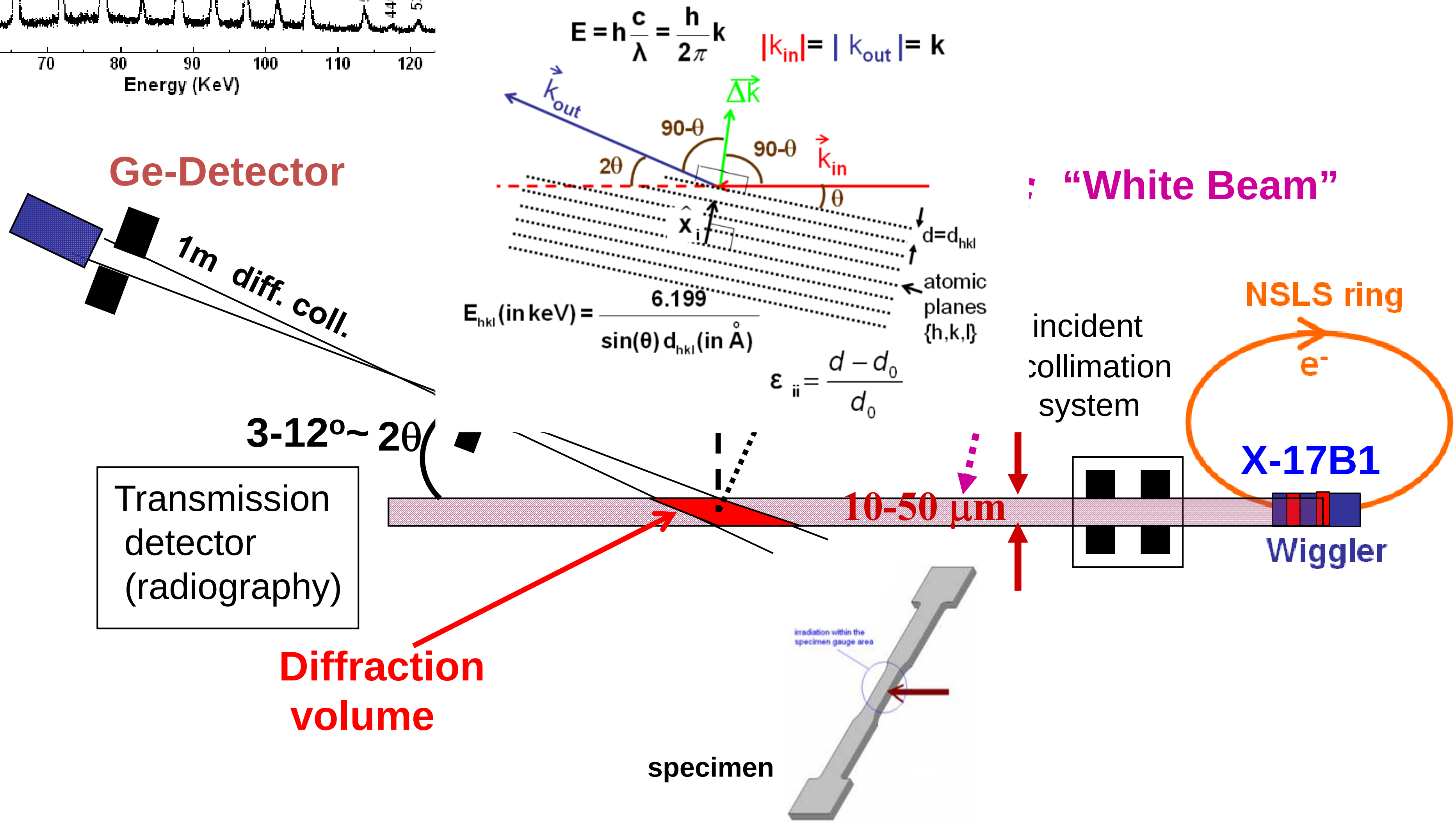

FPB OF 1MM-THICK POCO GRAPHITE - von Mises Stress
Fringe Levels

STRAIN MAPPING
Energy Dispersive Diffraction Mode



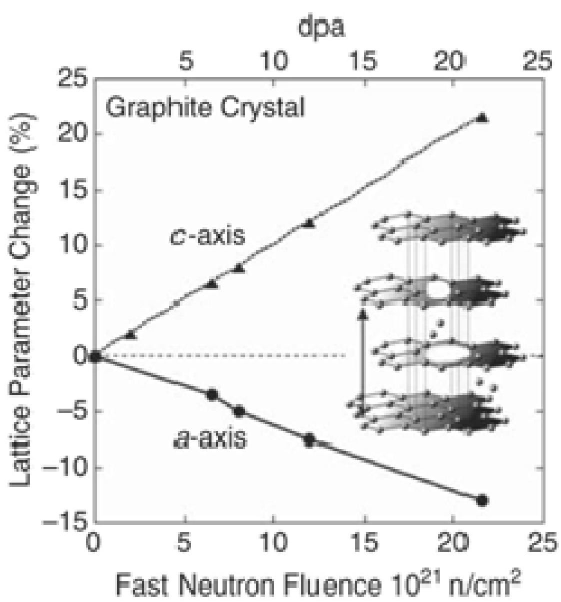
$$E_{hkl}\,[\text{in keV}] = \frac{6.199}{d_{hkl}\sin\theta} \;\rightarrow\; \varepsilon = \frac{\Delta d}{d_0}$$

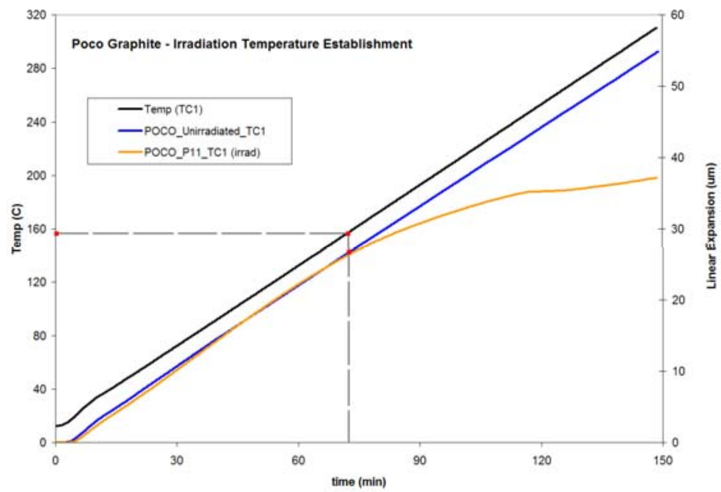
Like having imbedded inter-atomic strain gauges !!!!

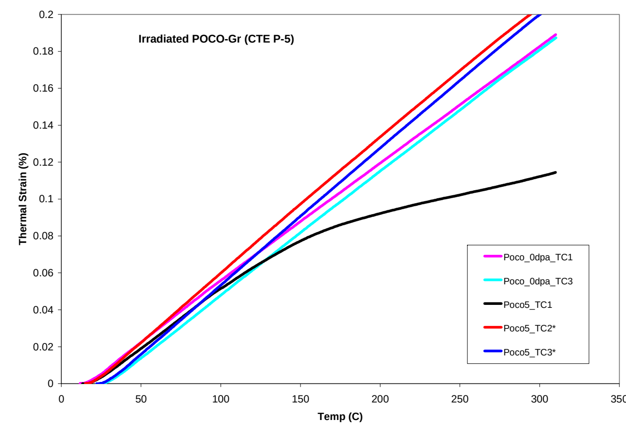


Graphite

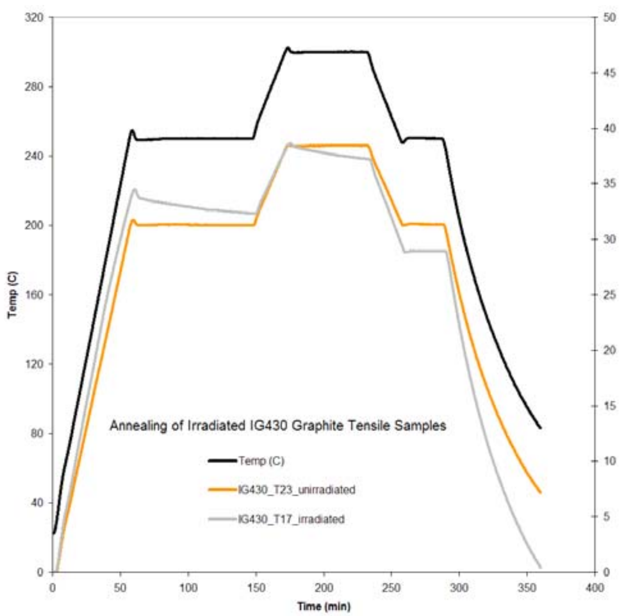
Important to know what occurs during irradiation and post-irradiation annealing (mobilization of interstitials/vacancies)

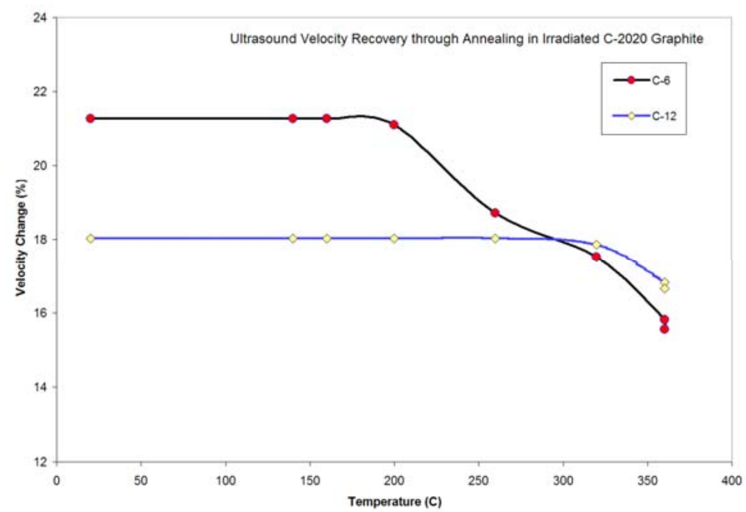


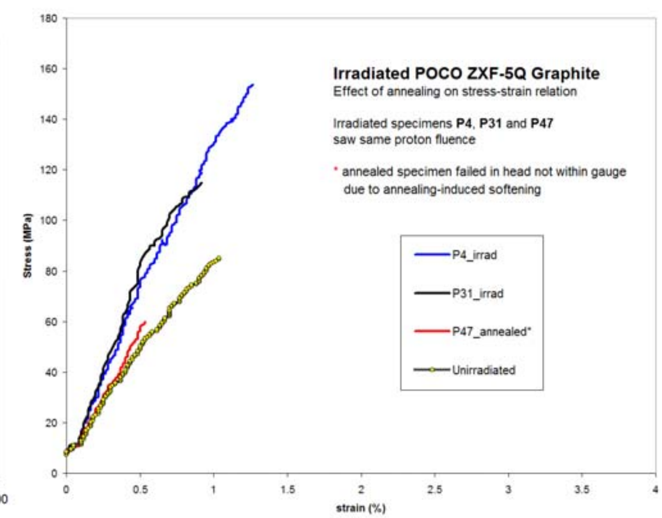


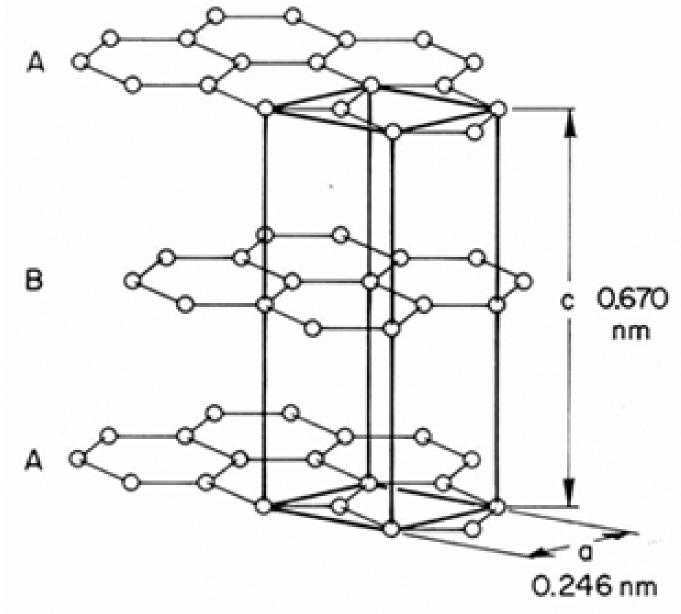


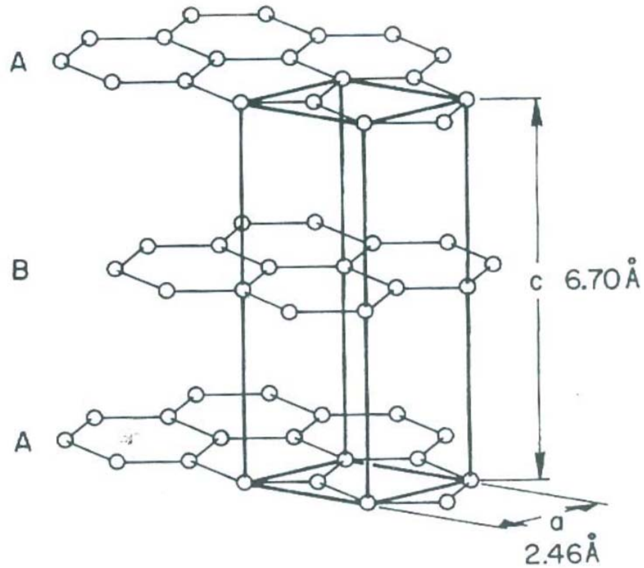
This is what we observe in BULK
What happens at the crystal level?
How is E is affected or is strain in crystal related to bulk?

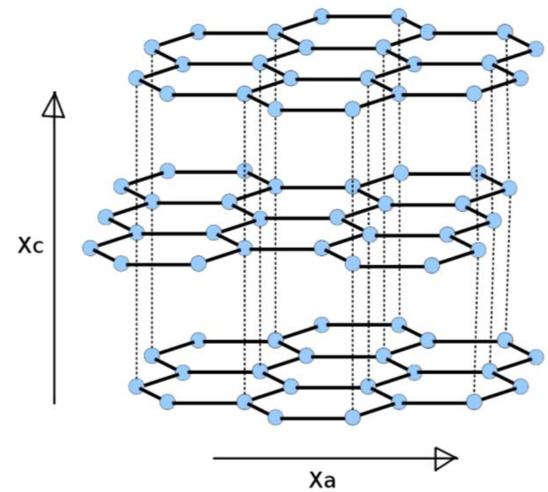


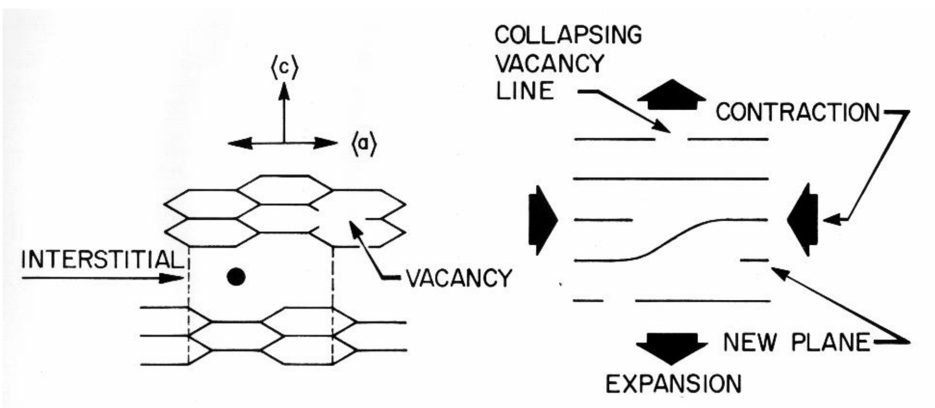










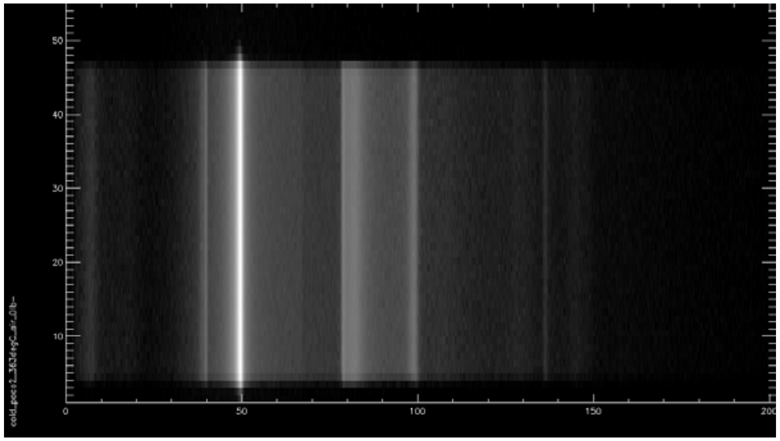


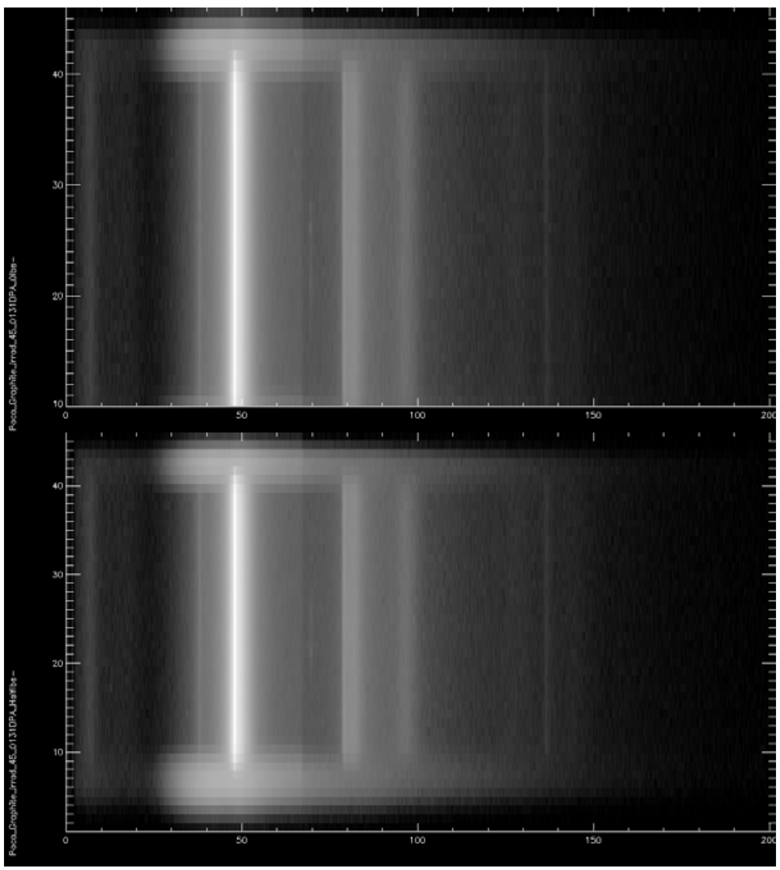
Interstitial defects will cause crystallite **growth** perpendicular to the layer planes (c-axis direction)

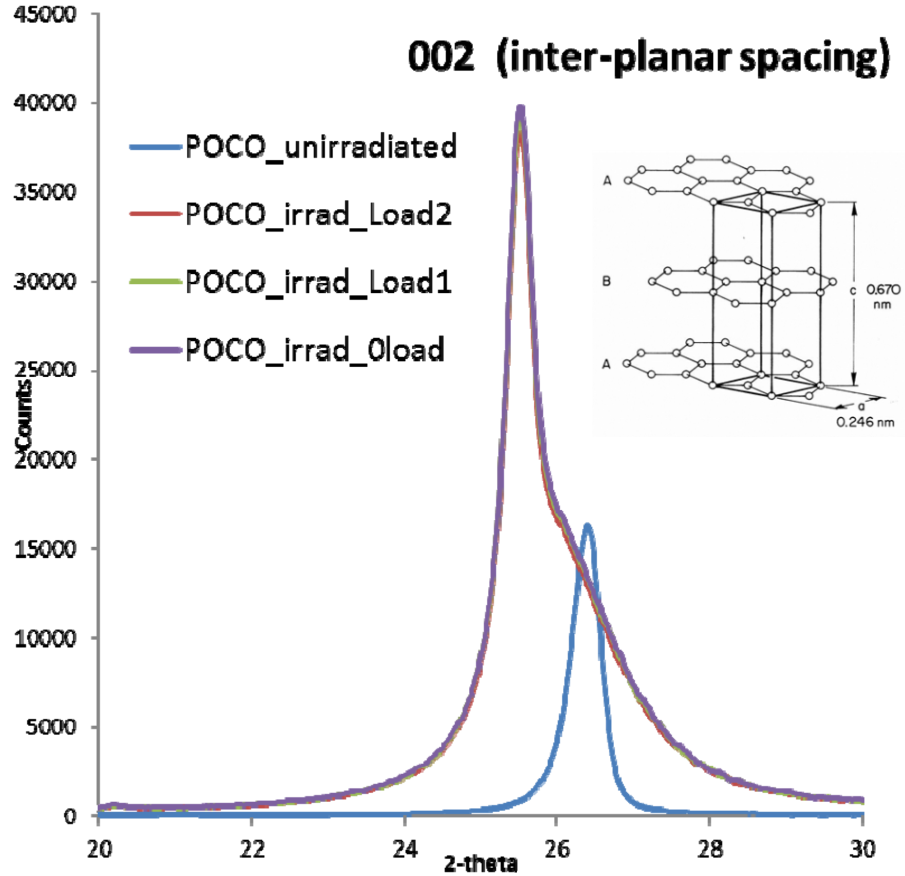
Coalescence of vacancies will cause a **shrinkage** parallel to the layer planes (a-axis direction)

Graphite

Various grades, including Carbon fiber composites under different irradiations

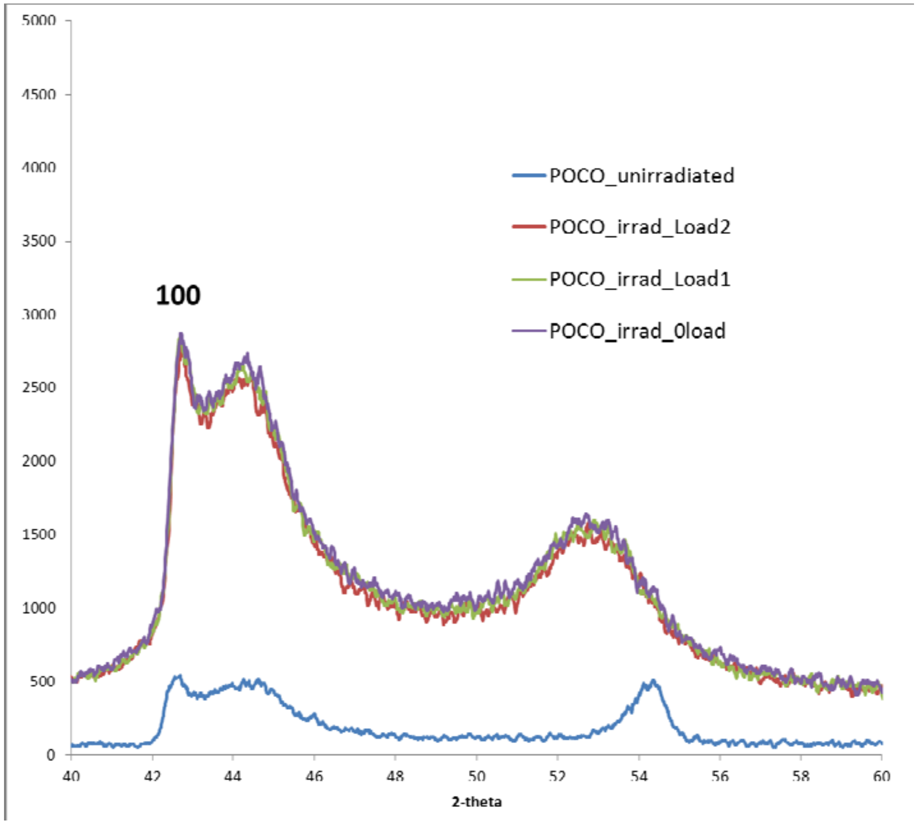


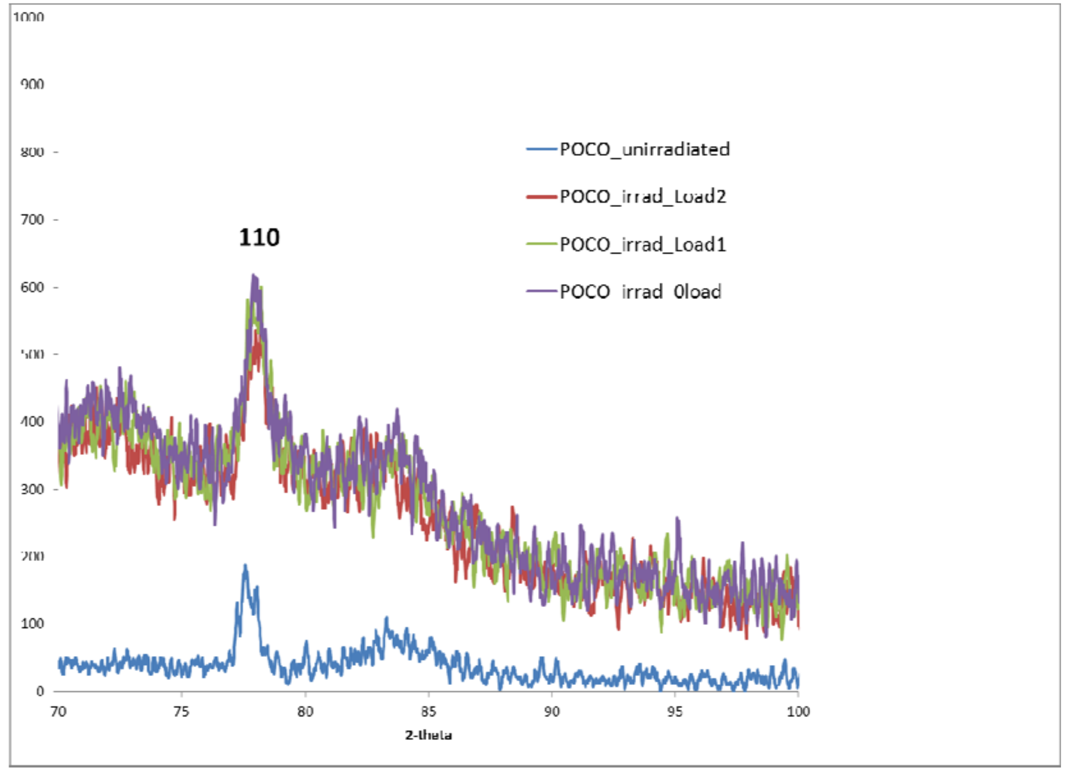


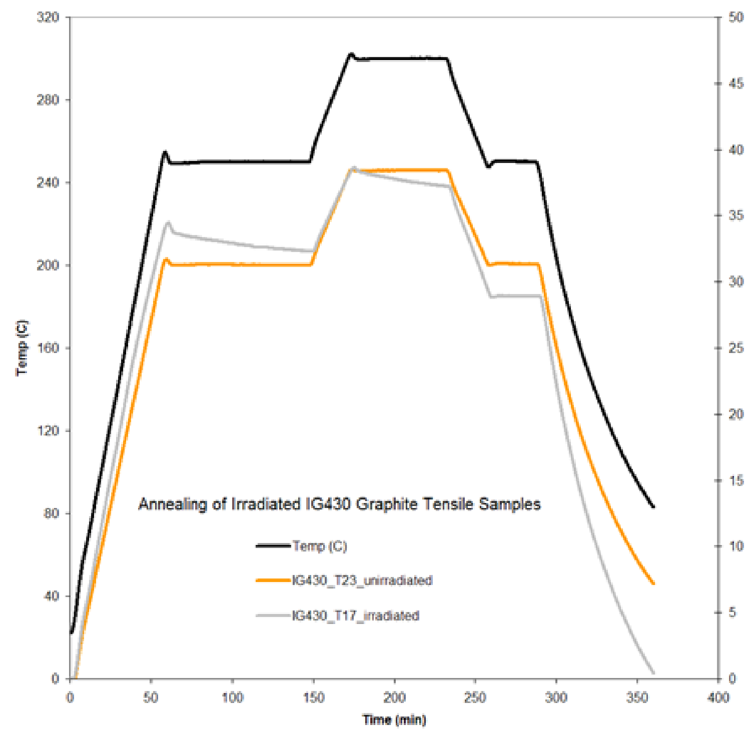


This 002 peak also broadens asymmetrically, with a bias towards smaller angles indicating an increase in average interlayer distance.

The (002) diffraction spot also broadens in single crystal images, suggesting a range of values for the interlayer distance



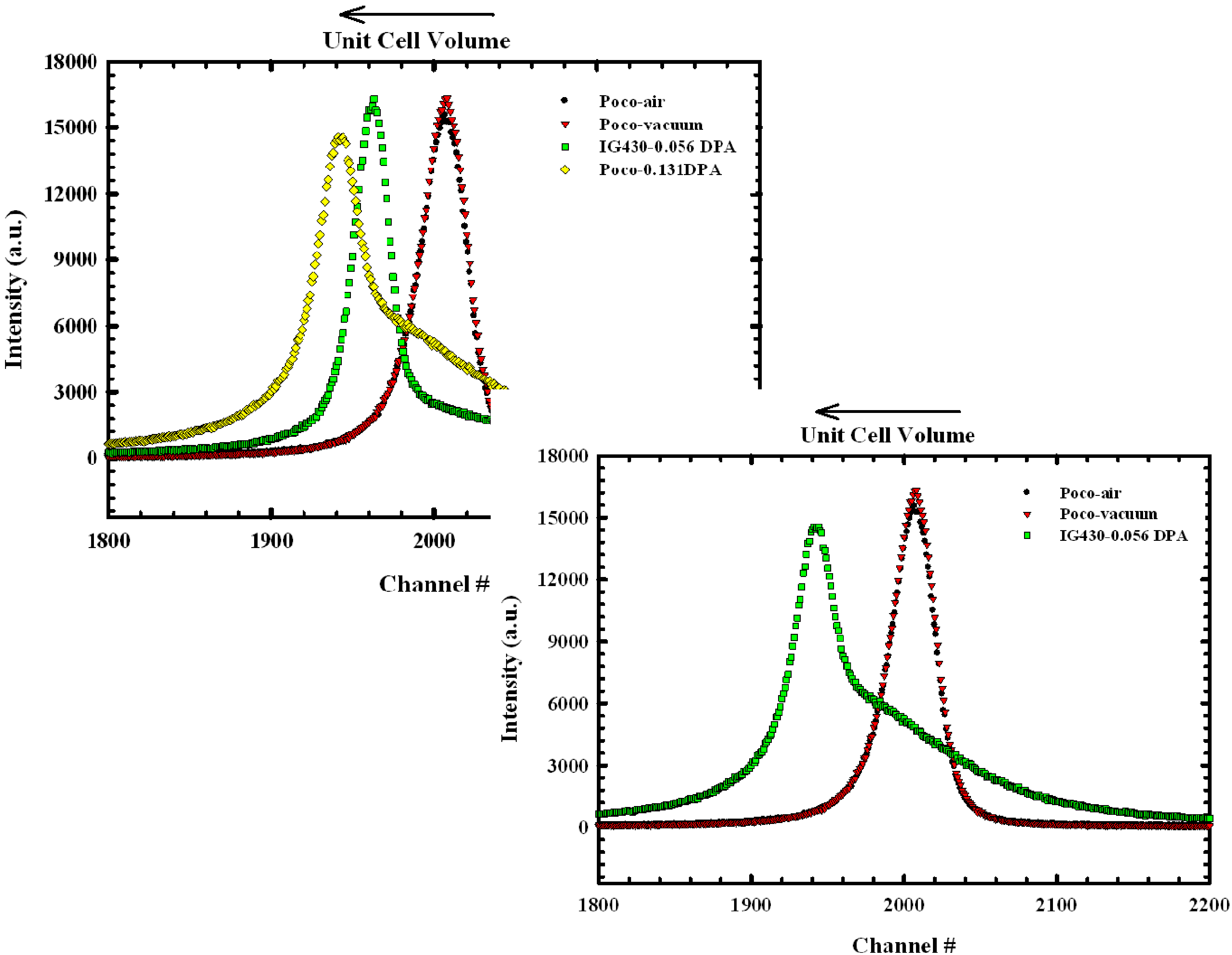




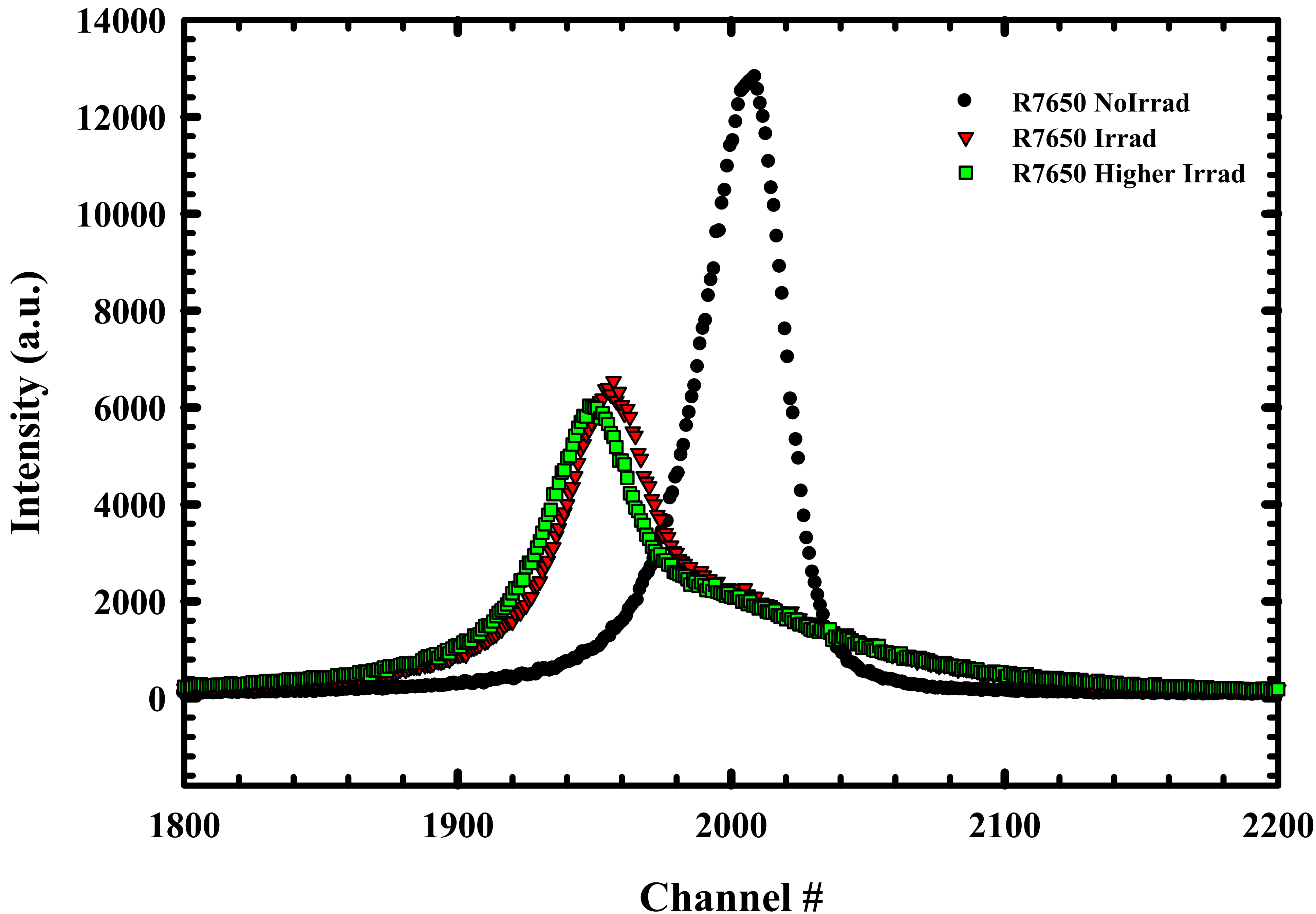
Goal is to correlate post-irradiation annealing observed macroscopically with shifts observed in XRD

Global volumetric changes vs. crystal-level changes

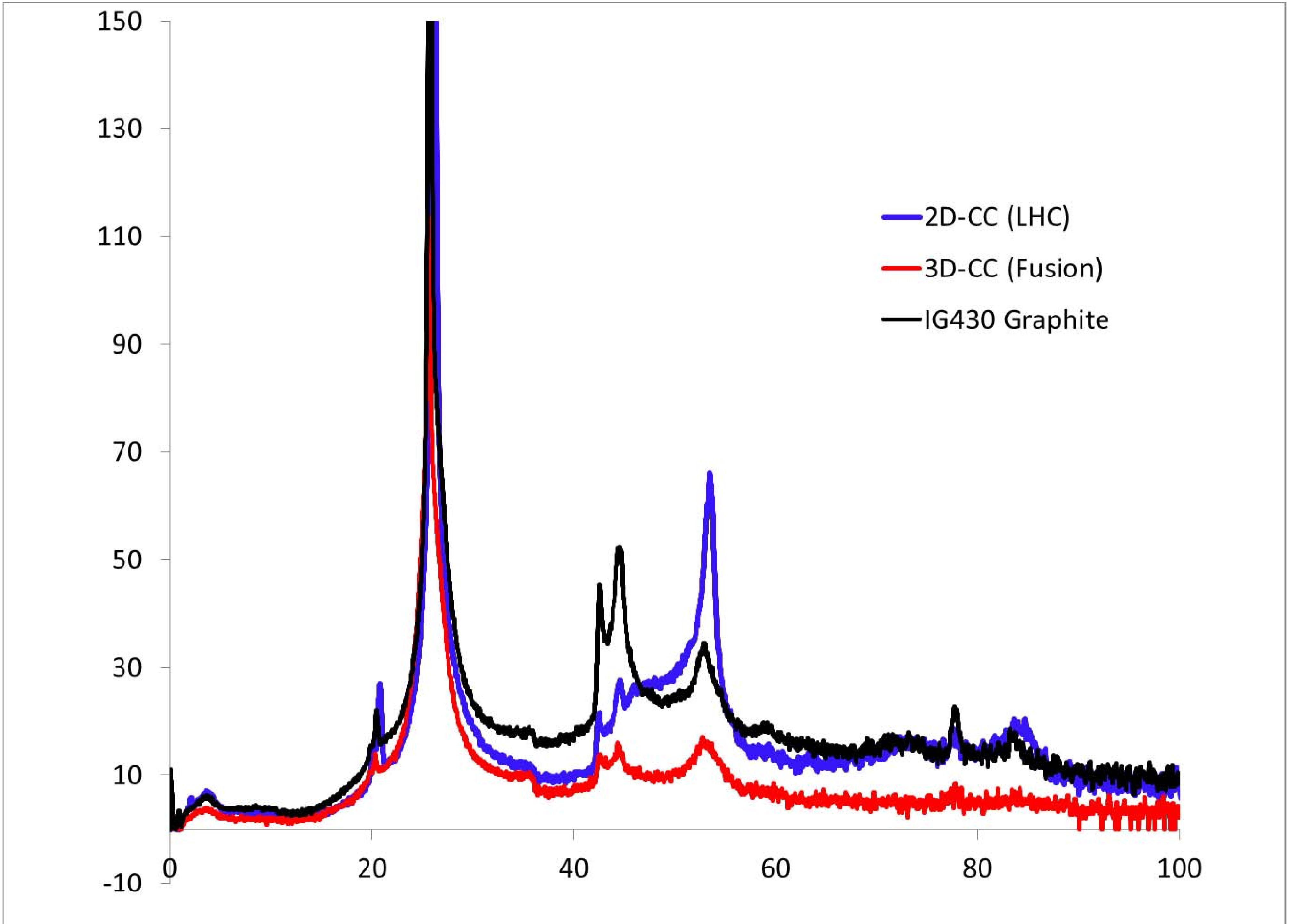
Activation Energy

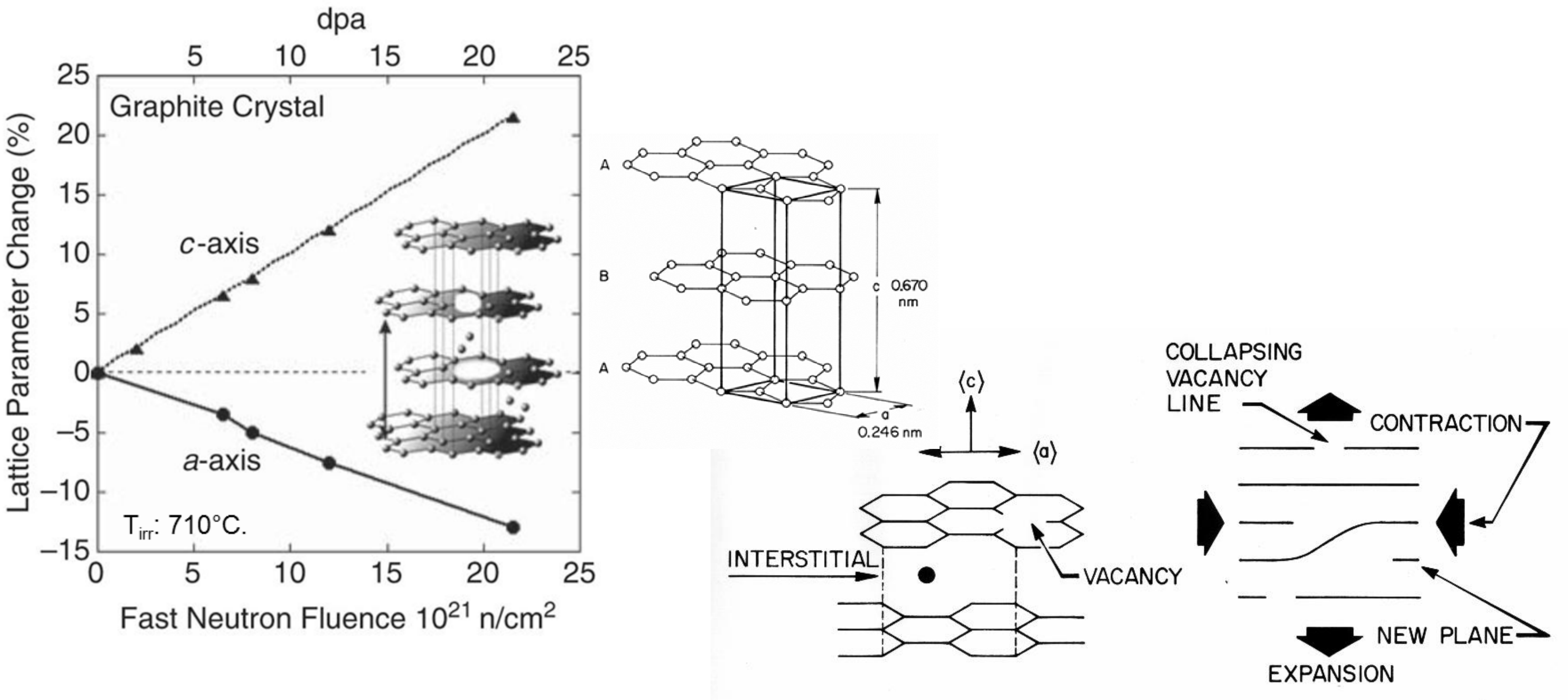


Unit Cell Volume
18000
15000
12000
9000
6000
3000
0
Intensity (a.u.)
Poco-air
Poco-vacuum
IG430-0.056 DPA
Poco-0.131DPA
1800
1900
2000
Channel #
Unit Cell Volume
18000
15000
12000
9000
6000
3000
0
Intensity (a.u.)
Poco-air
Poco-vacuum
IG430-0.056 DPA
1800
1900
2000
2100
2200
Channel #



R7650 NoIrrad
R7650 Irrad
R7650 Higher Irrad
Intensity (a.u.)
Channel #
14000
12000
10000
8000
6000
4000
2000
0
1800
1900
2000
2100
2200


150
130
110
90
70
50
30
10
-10
0
20
40
60
80
100
2D-CC (LHC)
3D-CC (Fusion)
IG430 Graphite



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