COMPTECH TAB meeting 2016-1

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01/22/2016 Argonne, IL

Brief introduction

- X-ray Thermal Diffuse Scattering (TDS)
  - **X-ray thermal diffuse scattering (TDS)** can be used to measure: **single-crystal** elastic properties of any crystalline materials (include **opaque materials**) using regular diffraction setups. Incorporation of **DAC** in TDS experiment is possible.
  - New **anisotropic elasticity measurement method** for high-pressure mineral physics field.
- Universal Membrane cap for most DACs
  - convert screw-driven DAC into membrane-driven DAC – **remote pressure control**
  - Universal cap that could **fit different DACs without losing DAC opening**
- Standard "cheap" resistive heater for DAC
  - **Cheap, reusable**, standard heaters (~$10/pc comparing with >$200/pc)
- Website Modification
  - **TAB** meeting ppts, Notes
  - **Tools**: including instructions for downloads
  - **Techniques and Facilities**: into to all COMPRES facilities at APS; Updates to technical development
- Facilities
  - PX^2 commissioning, membrane tubing system in 13BMC
  - Sector 34, add-in viewing and optical system for high-pressure experiments
- Data base
  - IEDA-kickoff workshop

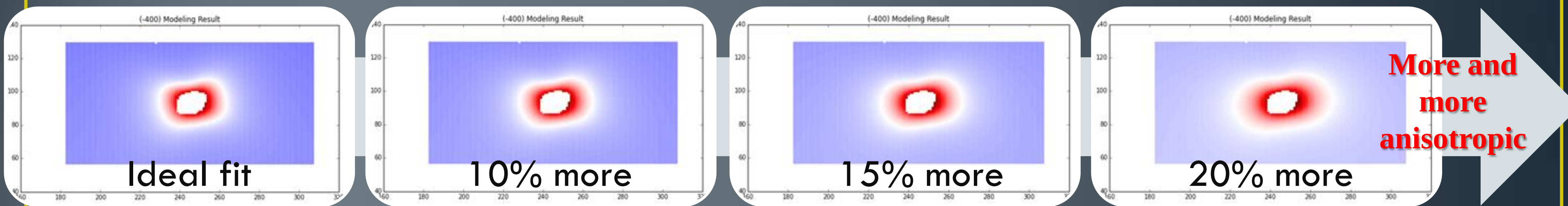
Time Line:

2014.11	join COMPTECH		
2014.12	13BMC PX2 commissioning	Ambient TDS measurement: sample suitable for DAC, Si (15um thick)	
2015.1	design of the first version universal membrane cap	1st test on W-Al2O3 heaters	Website major revision: Tools
2015.2	received the 1st set of modified W-Al2O3 heaters	Website rearrange	Preparation for TDS beam time
2015.3	1st TDS measurement with flight-path setup at 13BMC	1st high pressure (HP) TDS measurement on Si at 13BMC	
2015.4	1st python code for getting Si single-crystal elasticity tensor from TDS: based on BVK model	TDS sensitivity test	
2015.5	1st python code for getting single-crystal elasticity tensor from TDS: crystals (any symmetry)	Website revision: Facilities	
2015.6	Received the 1st version of membrane cap	Website revision: Techniques	COMPRES annual meeting present TDS results
2015.7	1st HP TDS on foresterite at 34ID	1st HP TDS on foresterite at 13IDC	Membrane tubing installed in 13BMC
2015.8	HP TDS measurement on foresterite up to 40GPa at13BMC	1st W-Al2O3 heater test in DAC	
2015.9	Testing new inversion algorism for TDS data analysis	1st heater user: U of Arizona	
2015.10	MS&T meeting in OH (1week); advertise PX2	TDS at 34 beam time	Data Base: IEDA Alliance Kickoff workshop
2015.11	TDS at BMC beam time	UNM visit (3days)	1st use of membrane control system at 13BMC during beam time
2015.12	AGU meeting present TDS results	Membrane cap finished and tested with DAC for the 1st time	TDS at IDC beam time
2016.1	Portable optical and sample viewing System Design	Testing new TDS data extraction code for both 34IDE data and IDC BMC	

X-ray thermal diffuse scattering (TDS)

X-ray thermal diffuse scattering (**TDS**) is caused by **lattice thermal vibrations** (phonons).

Shape of the TDS signal tells how anisotropic the sound velocities are:



Example of single crystal Si ambient condition

Project timeline:

2014.11 join COMPTECH

2014.12 **13BMC PX²** **commissioning,** first TDS test on ambient Si

Shutdown time

2015.3 **first high pressure TDS** measurement on Si, test flight-path setup for TDS measurement @ 13BMC

2015.4 **first** python **inversion code** for getting **Si** single-crystal elasticity tensor

2015.5 **First universal python inversion code** for getting single-crystal elasticity tensor with **any crystalline material with any symmetry**

Shutdown time

2015.7 first beam time at **34ID**, first high-pressure TDS on foresterite using energy scan, Mar CCD setup

2015.7 first beam time at **13IDC**, high-pressure TDS on foresterite

2015.8 high-pressure TDS measurement on **foresterite up to 40GPa** @13BMC

2015.9 Testing new inversion algorism for TDS data analysis

Shutdown time

<u>Major effect during 2015.10-2016.1:</u>

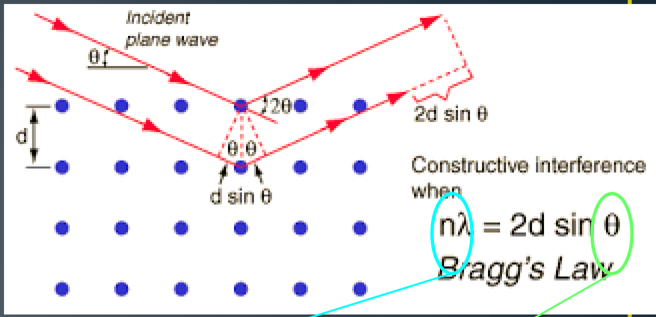
<u>New Data extraction code:</u> 34IDE (python code) and 13IDC, BMC (IDL program within ATREX)

<u>New inversion algorism:</u> intensity scaling factor fitting within each individual image

X-ray thermal diffuse scattering (TDS)

- Current Status:
  - **Sample testing:**
    - **Ambient condition: Si (100) (111) and forsterite (111)**
    - **High-pressure: <u>Si (100) (~ 6GPa)</u>; forsterite (111) (~40GPa)**
  - **Experimental Setup:**
    - **Angular scan approach + Pilatus100K:13BMC 13IDC (beam size 30μm)**
    - **Energy scan to find Brag peaks, then monochromatic beam for TDS measurement (MarCCD) 34ID**
  - **Software development:**
    - **TDS calculation : micro BV model for Si and Macro-single crystal elasticity model for all crystals**
    - **<u>Data inversion</u> : new optimized inversion code with better efficiency for lower symmetry materials**
    - **<u>Data extraction:</u> graphical output from ATREX for Angular scan data (with Przemek)**

      data output from IgorLaueGo + python for energy scan data (with Ruqing)

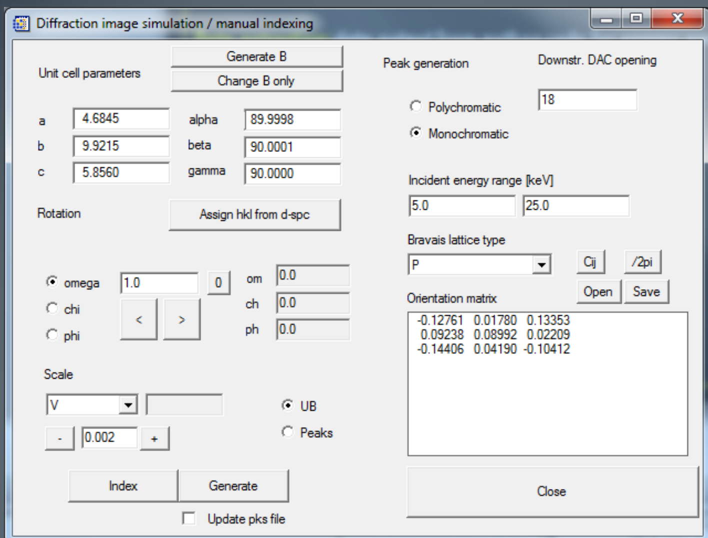


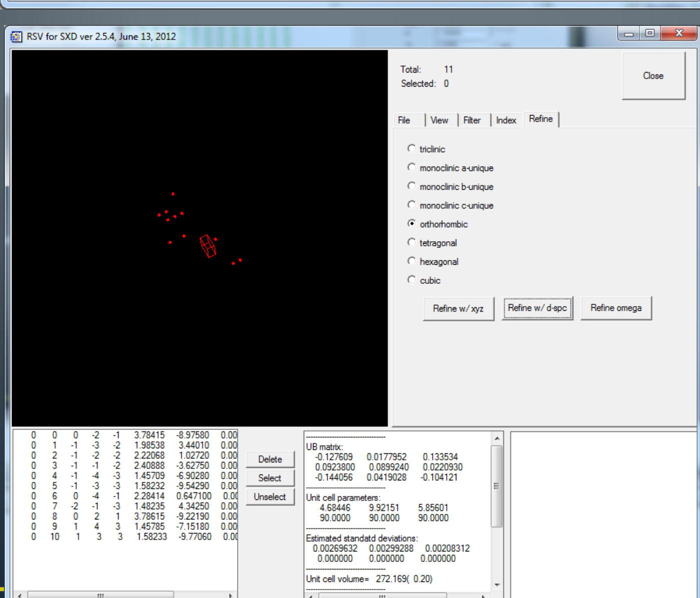
34ID

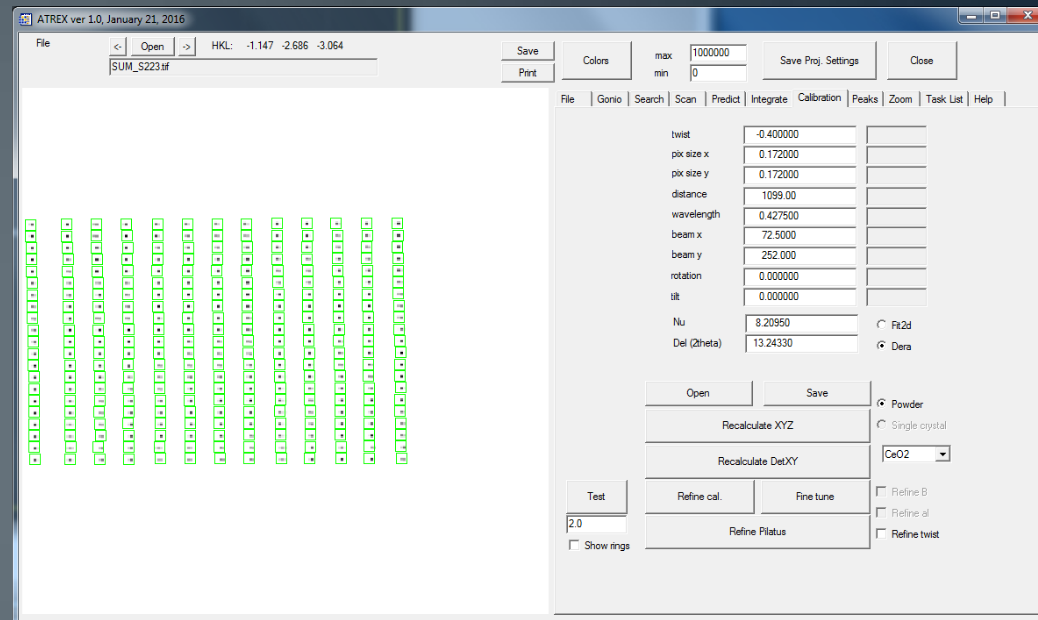
13IDC and BMC

Data extraction: graphical output from ATREX for Angular scan data (with Przemek)

1. Crystal orientation: 2 peaks (partially solved by using >8 peaks) – but SPEC has limitation: no symmetry constrain
2. Detector X-Y to HKL conversion: originally assuming polynormial fit H(x,y) K(x,y) , L(x,y)





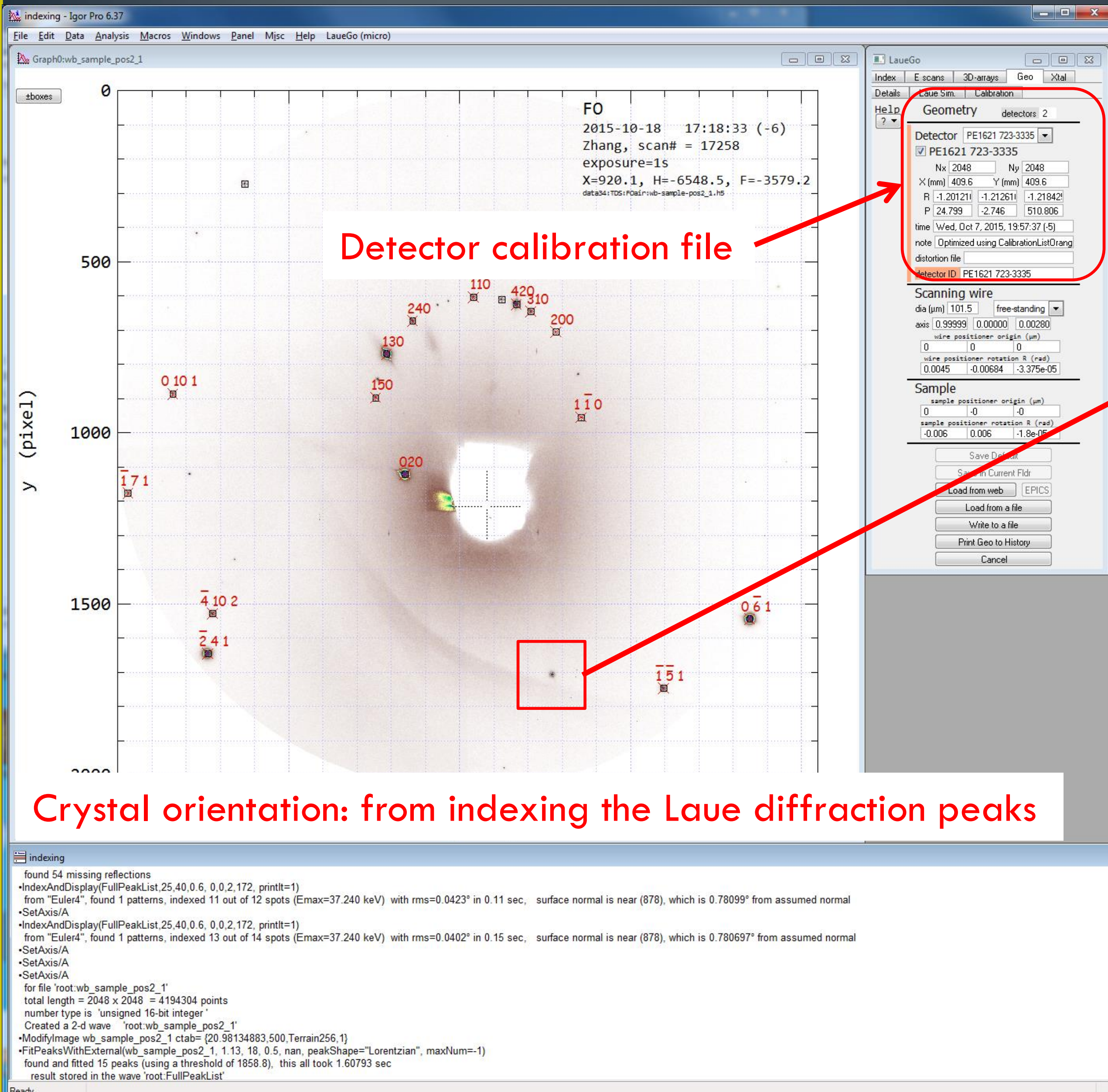


1. Utilizing the obtained 11 peaks and angles to get Lattice parameters with constrained symmetry (SPEC does not do that – it treats everything as triclinic)

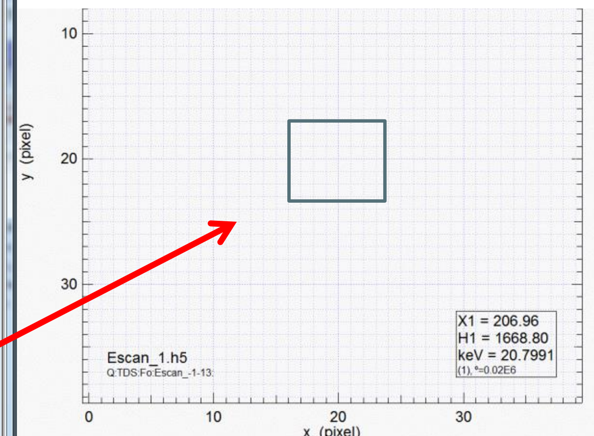
2. UB matrix can be read into the ATREX software for hkl calculation of each pixel.

3. Detector calibration can be made according to the mesh scan result.

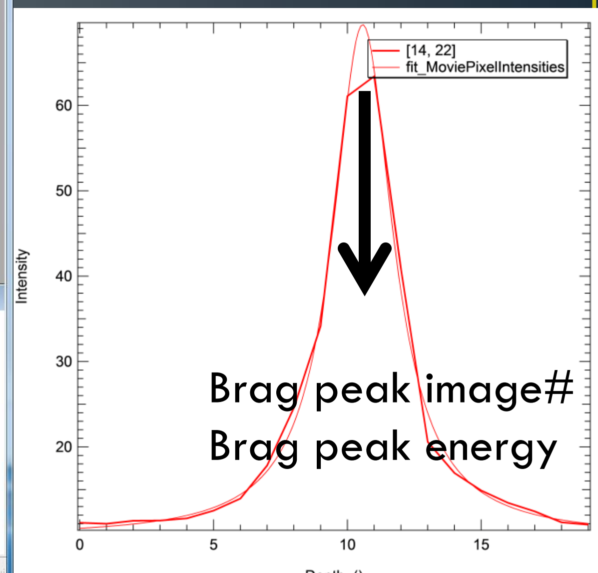
Data extraction: data output from IgorLaueGo + python code for energy scan data



Make movie: Energy scan for individual peak in ROI



Plot intensity VS image number:



Data extraction: data output from IgorLaueGo + python code for energy scan data

```
In [1]: import numpy as np
        import sys
        import os
        # Input working directory: change folder accordingly
        workdir=r'C:\Users\jzhang\Dropbox (UH Mineral Physics)\HIGP_Mineral_Physics_Team\TDS\Fo009GPa34\1DataOriginal'
        os.chdir(workdir)
        # make sure xmlutils.py is located in current working directory
        import xmdutils as xu
        print os.getcwd()

        C:\Users\jzhang\Dropbox (UH Mineral Physics)\HIGP_Mineral_Physics_Team\TDS\Fo009GPa34\1DataOriginal
```

```
In [2]: ## Define detector info and geometry, for one run, the numbers does not change. ##
        ## Use Igor to read the saved geometry file.##
        mar165 = xu.Detector(2048,2048,162.361/2048,162.361/2048)
        mar165_geo = xu.DetectorGeometry([-1.238,-15.689,153.471],
                                         [0.00752321,-0.01143958,3.14097174])
```

Detector calibration file

```
In [3]: ## Define crystal orientation (a*,b*,c*) from Igor indexing result ##
        astar = np.array((-8.787488, +0.469981, -9.857230))* 1.0e9
        bstar = np.array((+2.723327, +5.083838, -2.185390)) * 1.0e9
        cstar = np.array((+6.338666, -5.946492, -5.934293)) * 1.0e9
        aas = np.linalg.norm(astar)
        bbs = np.linalg.norm(bstar)
        ccs = np.linalg.norm(cstar)
        samp_rot_mat = np.vstack((astar/aas,bstar/bbs,cstar/ccs)).T
```

Crystal orientation

```
In [5]: ## get q vec of the defined HKL peak ##
        filefolder = r'C:\Users\jzhang\Dropbox (UH Mineral Physics)\HIGP_Mineral_Physics_Team\TDS\TDSdata\2015-2\34IDE\BX
        filename = r'Escan_54.h5' # use highest image intensity to identify which image the Brag peak is exactly located
        h5img = xu.XmdH5Data(os.path.join(filefolder,filename))

        ## Calculate the Brag peak absolute pixel X and Y
        # Input fitted Brag peak positions from Igor, Igor only read relative position
        peakX = h5img.roi_startX + 19.4544
        peakY = h5img.roi_startY + 20.0588
        print "Brag peak position:", "(",peakX, peakY,")"

        hklname = '(-131)' #Input ideak HKL name
        HKLxeng = 23.5985 # Input X-ray energy (keV)
        pk_hkl = np.array([-1.0,3.0,1.0])    #Input integer ideal HKL
        pk_cen_xyz = xu.pixels_to_xyzs([peakX],[peakY],mar165,mar165_geo)
        pk_cen_q = xu.pxyzs_to_qs(pk_cen_xyz,HKLxeng)
        pk_qc = np.linalg.norm(pk_cen_q)
        print 'current hkl:',pk_hkl

        # test computing hkl of the Bragg peak, obtained numbers should be similar to theoretical HKL
        testhkl = xu.qs_to_ortho_hkls(pk_cen_q[0],np.array((aas,bbs,ccs)),samp_rot_mat)
        print 'Real hkl is', testhkl

        Brag peak position: ( 662.4544 1060.0588 )
        current hkl: [-1.  3.  1.]
        Real hkl is [-1.00149768  3.00925398  0.9993432 ]
```

Calculate hkl based on
X-ray energy
And pixel position

Double check:
ideal hkl
calculated hkl

Data inversion: optimized inversion with better efficiency for lower symmetry materials

In 2015-2 TAB meeting

Only consider 1st order approximation – single-phonon scattering

Method 1: single crystal elasticity model = **Method 2: Born-Von-Karman model**

1 model for all, only based on Cijs and hkls. Change Cijs for different materials.

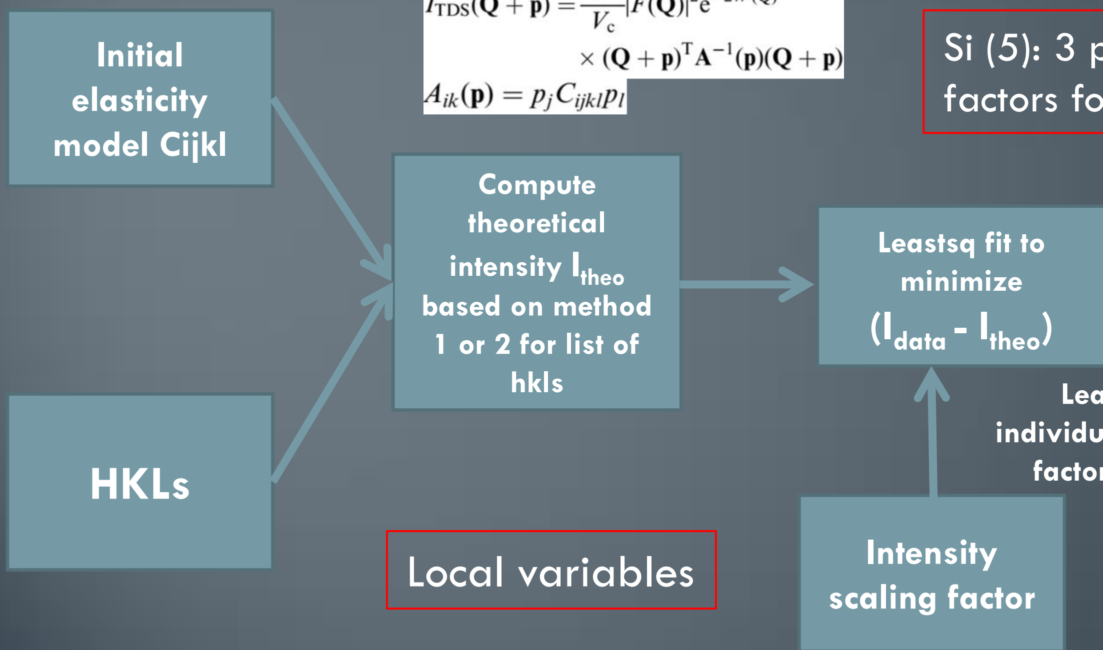
Micro force constant model for each pair of neighboring atoms – **1 material 1 model**

$$I_{TDS}(\mathbf{Q}+\mathbf{p}) = \frac{k_B T}{V_c}|F(\mathbf{Q})|^2 e^{-2W(\mathbf{Q})} \times (\mathbf{Q}+\mathbf{p})^T \mathbf{A}^{-1}(\mathbf{p})(\mathbf{Q}+\mathbf{p})$$

$$A_{ik}(\mathbf{p}) = p_j C_{ijkl} p_l$$

Si (5): 3 parameters Cij, 2 intensity scaling factors for 2 images

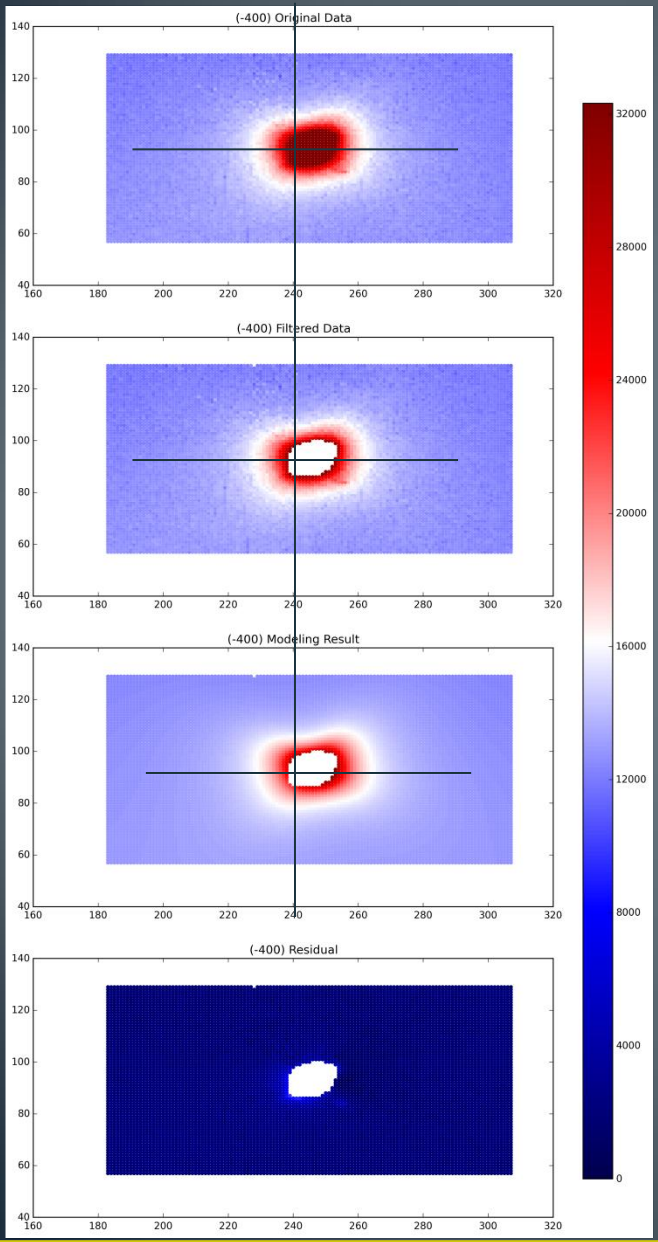
In 2015-3 TAB meeting



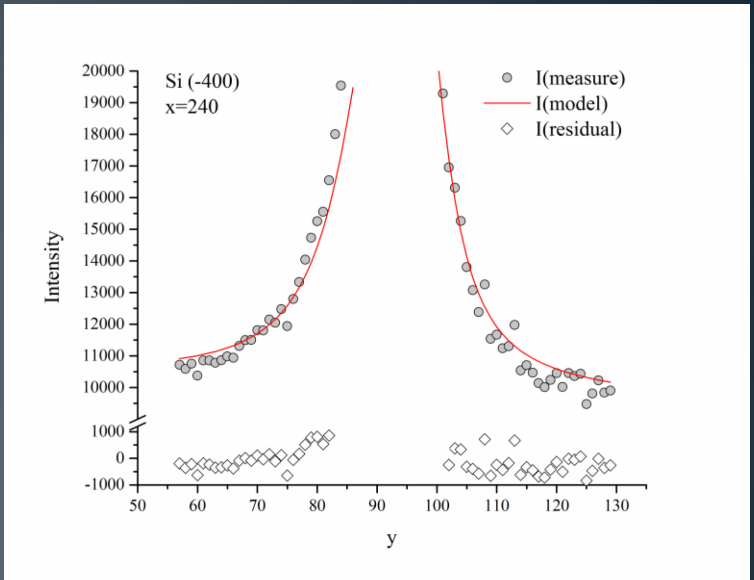
Forsterite (28):
8 parameters for Cij +18 intensity scaling factors for 18 TDS images

New code gives exactly the same answer to Si when treating the intensity scaling factors as local variables VS global variables.

Si result: line profiles

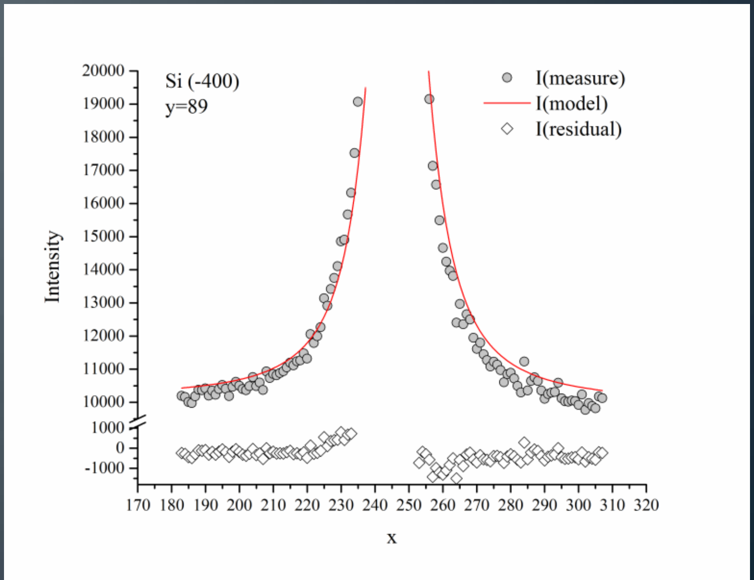


Vertical cut



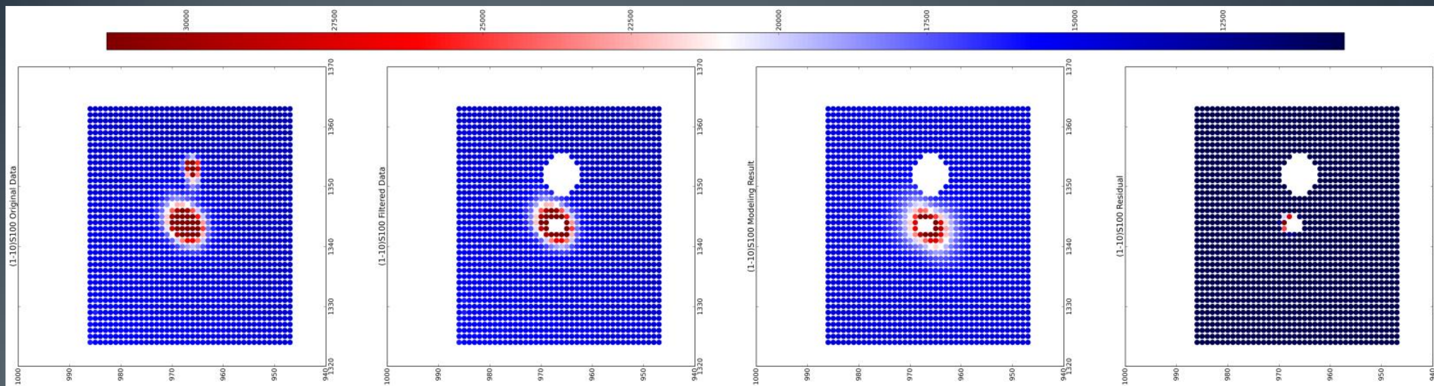
I residual~10^2
I measure ~10^4
Fitting uncertainty ~a few percent

horizontal cut

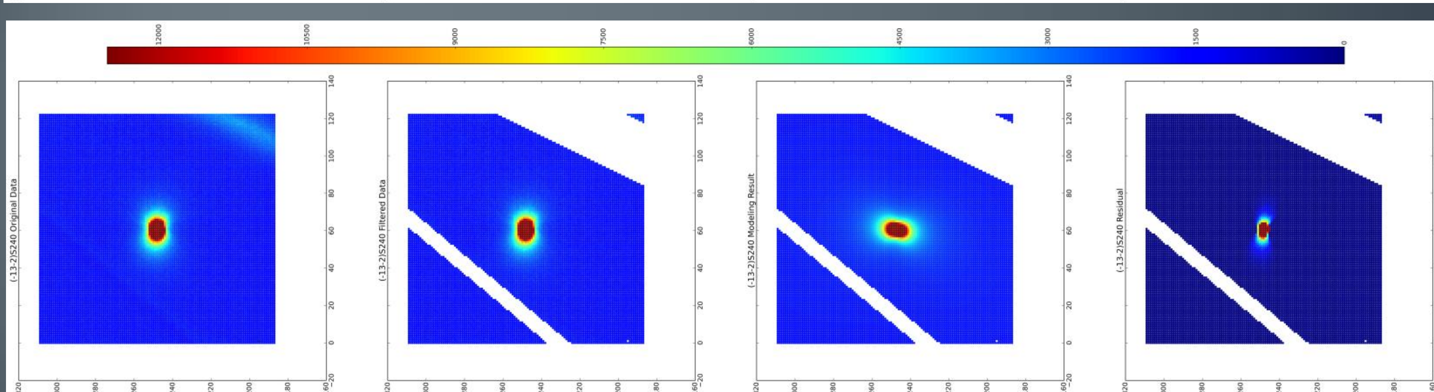


Forsterite result

beam time in 2016-1:34IDE



34IDE
0GPa



13IDC
9GPa

Future work:

1. Figure out what was the problem with forsterite data:

Collaborate with Dr. B. Winkler (Germany):

First principle calculation – predict TDS + Cijs

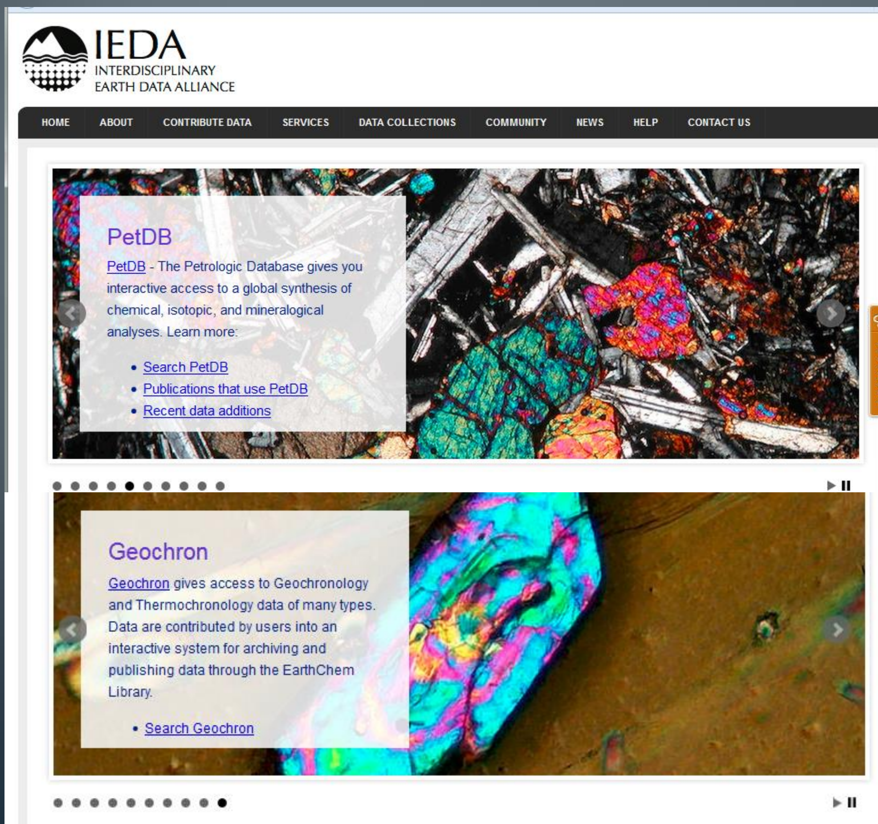
Treat his theoretically generated TDS as real data and invert the Cijs.

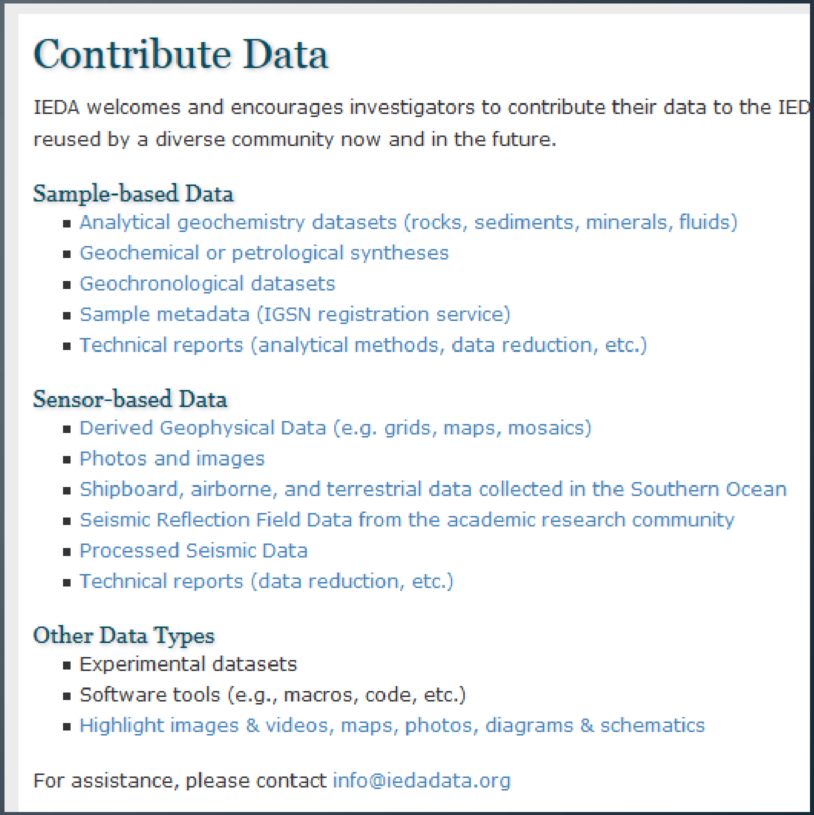
See if this inverted Cijs = Cijs from first principle calculation.

2. Start writing up the first paper describing the method and the our results on single-crystal Si up to ~6 GPa.

Data base – IEDA kickoff workshop

- Importance of having a mineral physics data base:
  - EOS, elasticity, rheology
  - Phase diagram, element partitioning





Contribute Data

IEDA welcomes and encourages investigators to contribute their data to the IED
reused by a diverse community now and in the future.

Sample-based Data

- Analytical geochemistry datasets (rocks, sediments, minerals, fluids)
- Geochemical or petrological syntheses
- Geochronological datasets
- Sample metadata (IGSN registration service)
- Technical reports (analytical methods, data reduction, etc.)

Sensor-based Data

- Derived Geophysical Data (e.g. grids, maps, mosaics)
- Photos and images
- Shipboard, airborne, and terrestrial data collected in the Southern Ocean
- Seismic Reflection Field Data from the academic research community
- Processed Seismic Data
- Technical reports (data reduction, etc.)

Other Data Types

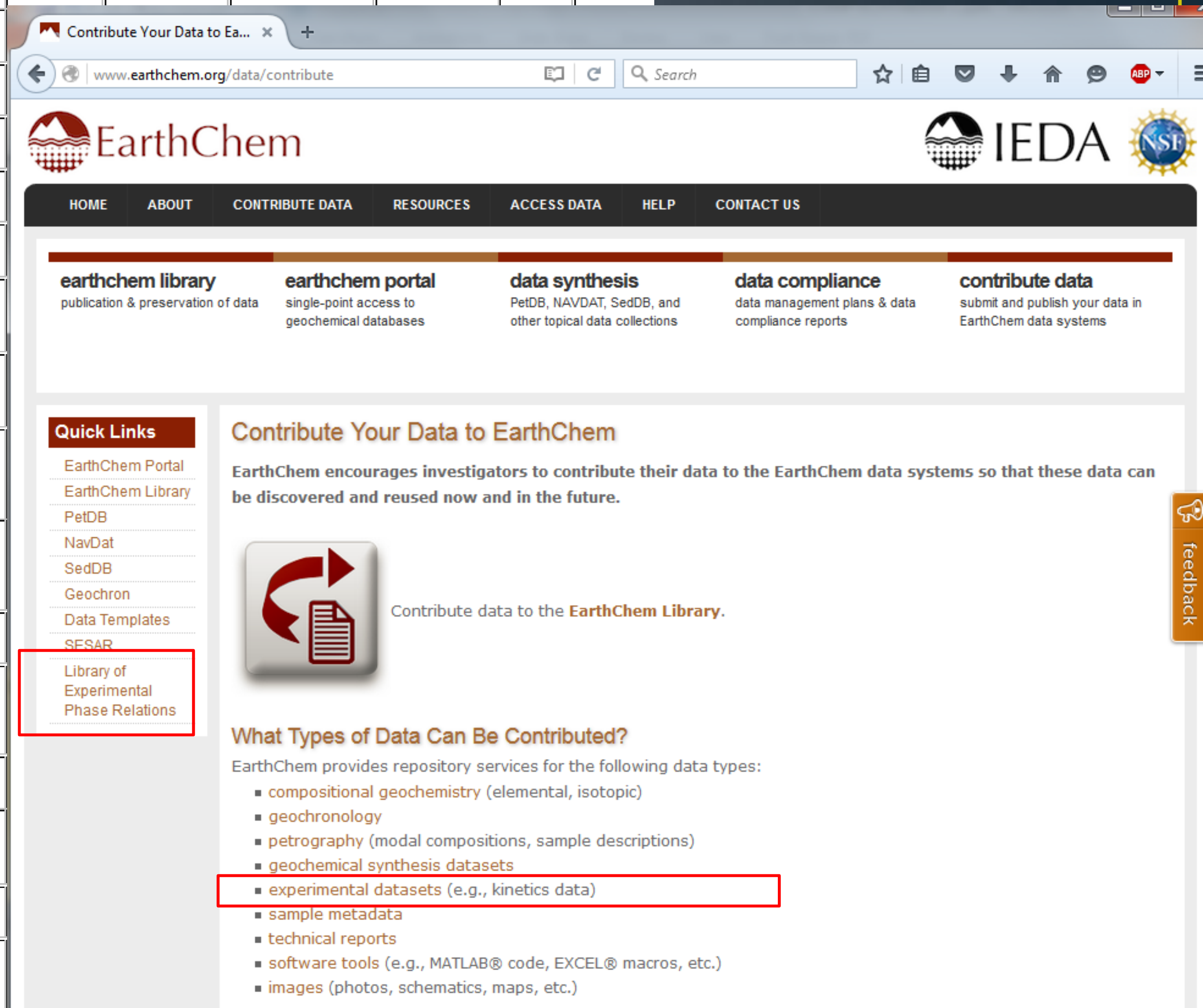
- Experimental datasets
- Software tools (e.g., macros, code, etc.)
- Highlight images & videos, maps, photos, diagrams & schematics

For assistance, please contact info@iedadata.org

What do they have?

Where do we fit to?

Data System	Description	Find Data	Geophysical Data	Oceanographic Data	Geochemical Data	Images	Sample Info
Antarctic and Southern Ocean Data Portal (ASODS)	Data collected in the Southern Ocean	Search	X	X	Links to	X	X
Academic Seismic Portal (ASP) at LDEO	Seismic Reflection Field Data from the academic research community	Search	X				
Academic Seismic Portal (ASP) at UTIG	Processed Seismic Data						
Deep Lithosphere Dataset	Petrology of mantle xenoliths						
EarthChem Portal	Access to geochemical data in federated databases						
EarthChem Library	Geochemical datasets and data syntheses						
Geochron	Geochronological and thermochronological data						
GeoPRISMS Data Portal	Data from interdisciplinary research along continental margins						
Global Multi-Resolution Topography (GMRT)	Global compilation of seafloor bathymetry and land topography						
MARGINS Data Portal	Data from interdisciplinary research along continental margins						
MediaBank	High-quality images and videos for education and outreach						
PetDB	Petrological database of the ocean floor						
Ridge 2000 Data Portal	Data from interdisciplinary research at mid-ocean ridges, from mantle to microbe						
SedDB	Geochemical data of marine sediments						
System for Earth Sample Registration (SESAR)	Sample Catalog & Registry for the International Geo Sample Number						
VentDB	Hydrothermal spring geochemistry data system						
USAP-DCC	Metadata and data from NSF-funded Antarctic research programs						



Universal Membrane cap for most DACs

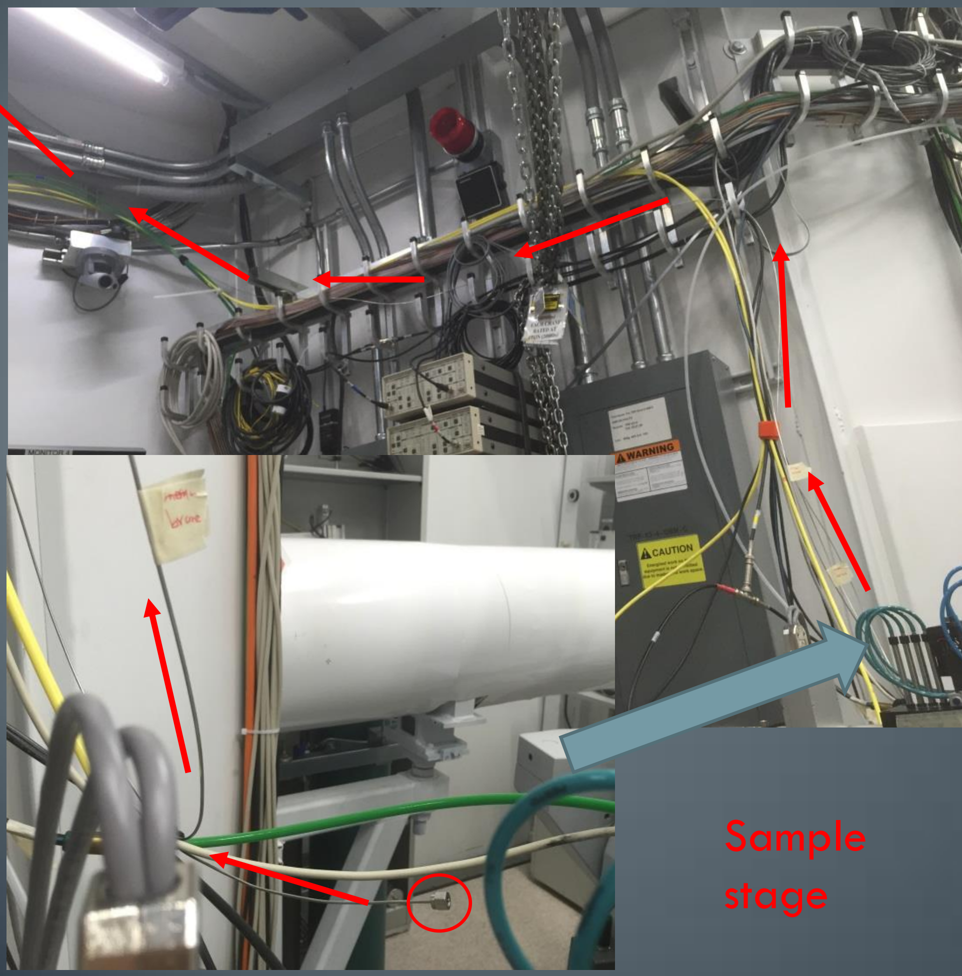
- Advantage of Membrane cap – convert screw-driven DAC into membrane-driven DAC
- Remote precise pressure control
- Currently available membrane caps
  - Too many DACs in different sizes – one cap for each type
  - Limitations to the DAC opening – IXS, Brillouin, single x-stal XRD

- Our target:
  - **One cap** for most DACs in different sizes: different spacers
  - **No lose in DAC opening**: ideal for single crystal studies

- **Project timeline:**
  - **Design**ed the first edition of membrane cap 01/2015
  - Received all **body parts** of membrane cap 06/2015
  - Stainless steel **tubing** ordered and installed in 13BMC 07-08/2015
  - Gas **membranes** sent back to machine shop for **revision** 09/2015
  - GE digital pressure **controller** ordered 08/2015 received 10/2015
  - GE pressure controller and tubing installed and **user used the system** in 11/2015
  - Received the modified membrane in 11/2015, **tested the membrane caps offline** in 12/2015

Welcome COMPRES users to use the membrane cap + control system from 2016-1!

Membrane system setup at PX^2



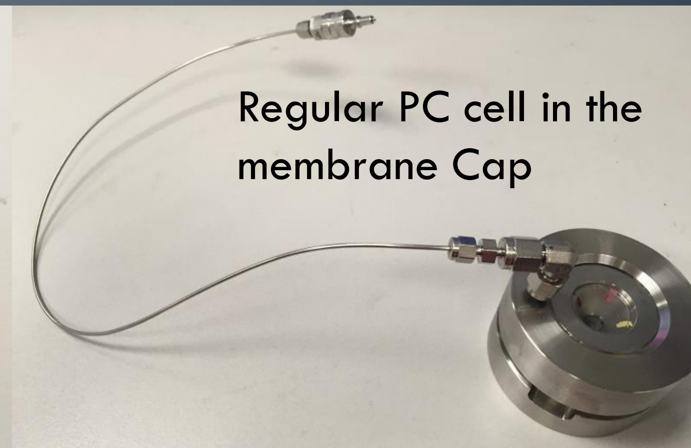
Membrane tubing (ss, swagelok)
Controller (GE pace5000)
Regulator (3000psi)

Installed in 13BMC
(with help of Mike Proskey)

Users used membrane control and tubing in BMC in 2015-3 run with their own Membrane Caps

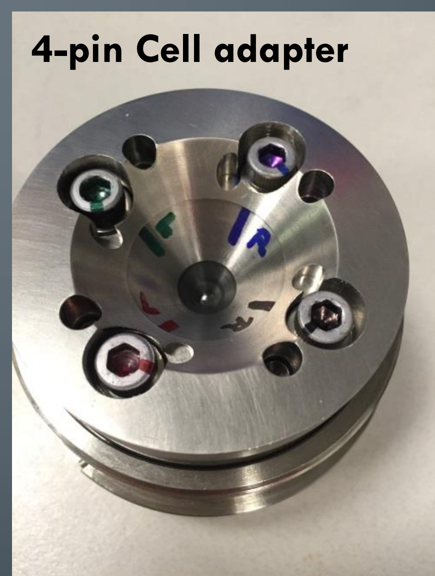
Universal Membrane cap for most DACs


Membrane Cap

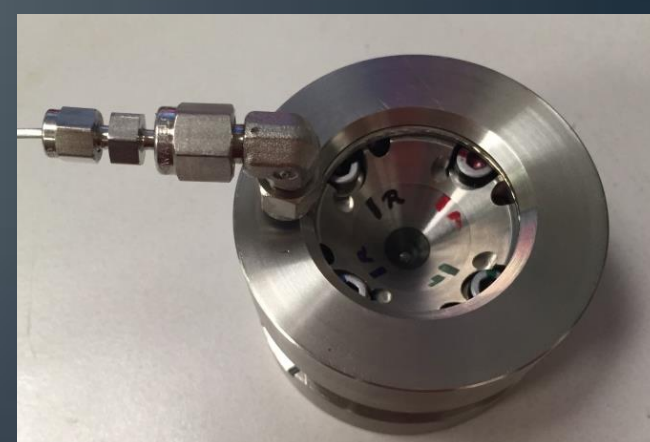

Regular PC cell in the membrane Cap


4-pin Cell 100° opening


Membrane


4-pin Cell adapter

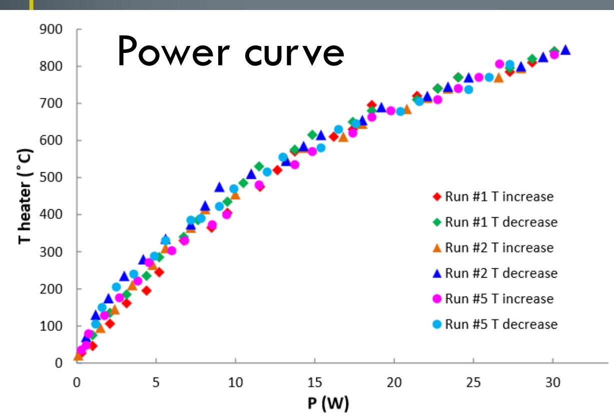
4-pin Cell within Membrane Cap

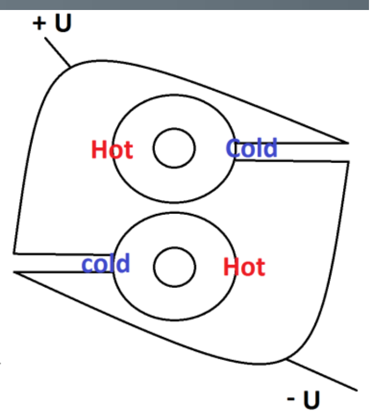


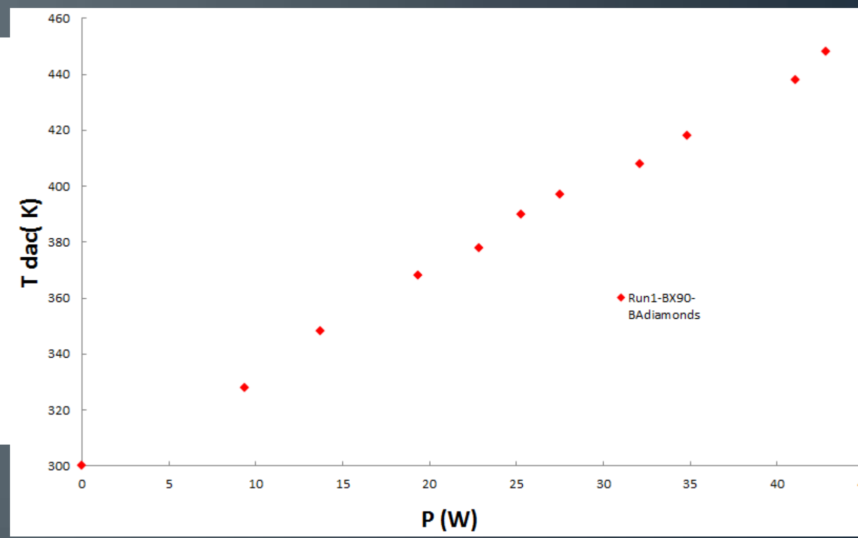
Tested offline in Dec. 2015 successfully

Standard "cheap" resistive heater for DAC

- Commercial W-Al_2O_3 metal ceramics heater can reach 1000 K within 30 s, very stable and cheap (<$10/pc).
- Ready for use, reproducible specifications, reusable, and not expensive
- Calibration and modification of the W-Al_2O_3 heater: (procedures online updated)







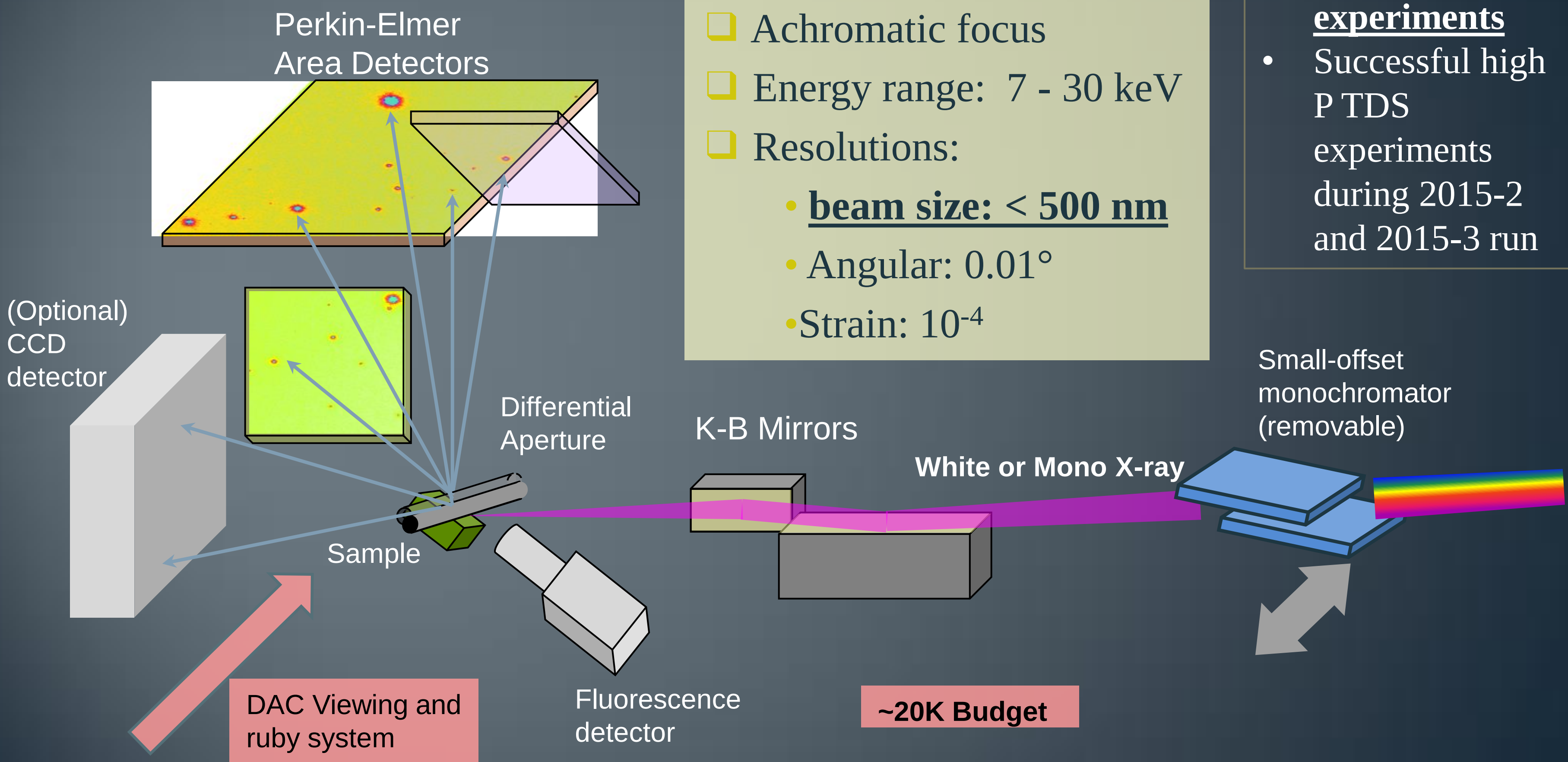
- Project status:
  - 1st test with DAC: with **single** heater – sample chamber reached highest T 450K
  - 1st supply to COMPRES users: (Dan Shim and Kurt Leinenweber)
  - 2nd supply to COMPRES users: (Zhenxian Liu)
  - Other testers: (Bin Chen, Xiaojing Lai, Yi Hu, Jay Bass)

Preferred access to existing beamlines

Sector 34 ID

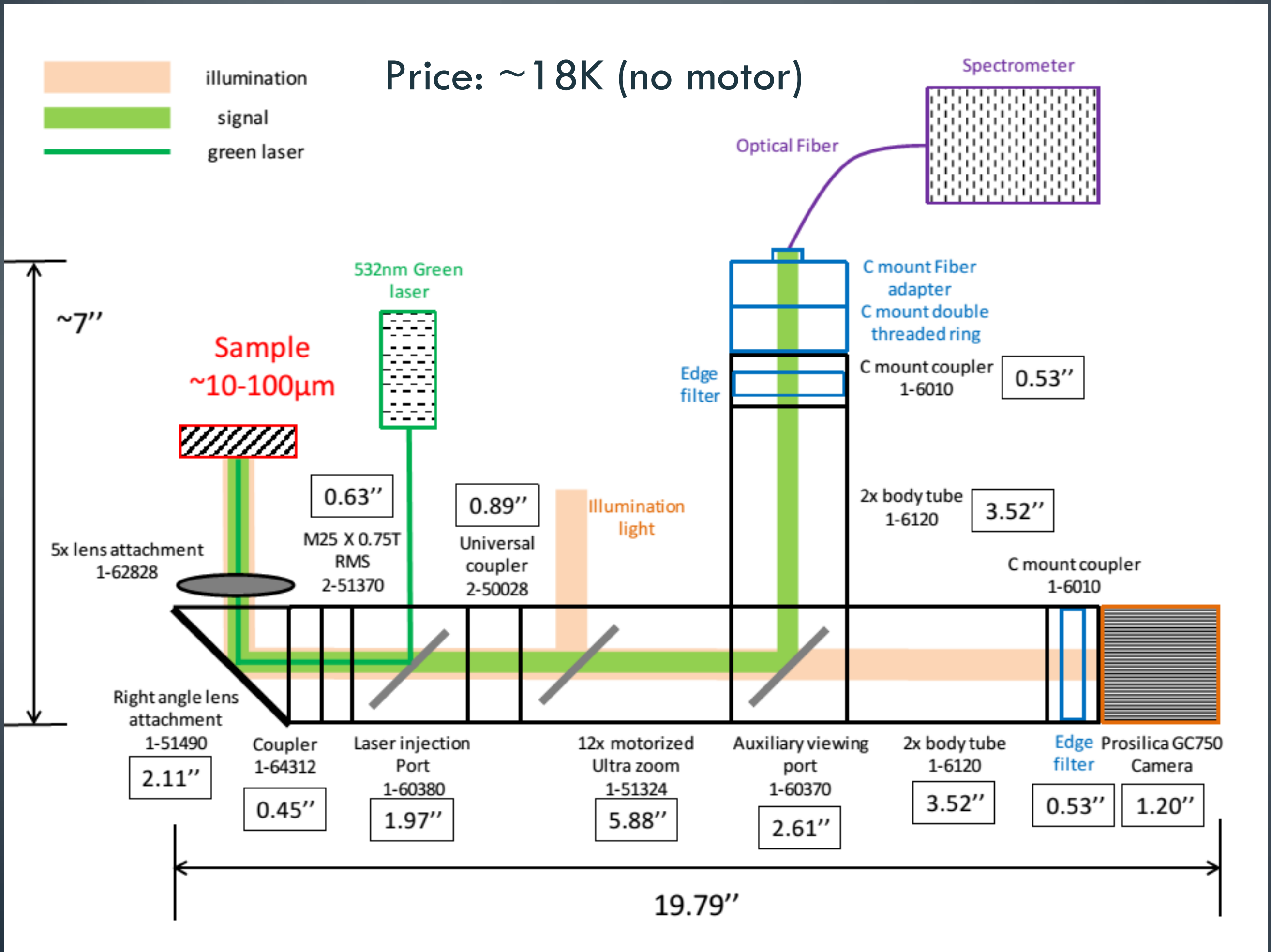
- **Ideal for high P experiments**
- Successful high P TDS experiments during 2015-2 and 2015-3 run

- Achromatic focus
- Energy range: 7 - 30 keV
- Resolutions:
  - **beam size: < 500 nm**
  - Angular: 0.01°
  - Strain: 10^{-4}



Beamline Contact: Wenjun Liu <wjliu@anl.gov>; Ruqing Xu <ruqingxu@anl.gov>; Jon Tischler <tischler@anl.gov>

Portable viewing and ruby system



All light path Integrated into the tube

- Difficult to "misalign"
- Compact
- for sector 34IDE

And other non-high pressure beamlines

http://comptech.compres.us/

Thanks! Questions?