COMPTECH TAB meeting 2015-2

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05/28/2015 Argonne, IL

Update on Projects

- X-ray Thermal Diffuse Scattering
- Multi-grain X-ray diffraction
- Universal Membrane cap for most DACs
- Standard “cheap” resistive heater for DAC
- Website Modification

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.
- Project Status:
  - **Sample testing:**
    - **Ambient condition: Si (100) (111) and MgO (100)**
    - **High-pressure: Si (100)**
  - **Experimental Setup:**
    - **Using flight path decrease background (originally designed for surface scattering experiment)**
  - **Software development:**
    - **Python: micro-force-constant model & macro-single crystal elasticity model**

X-ray thermal diffuse scattering (TDS)

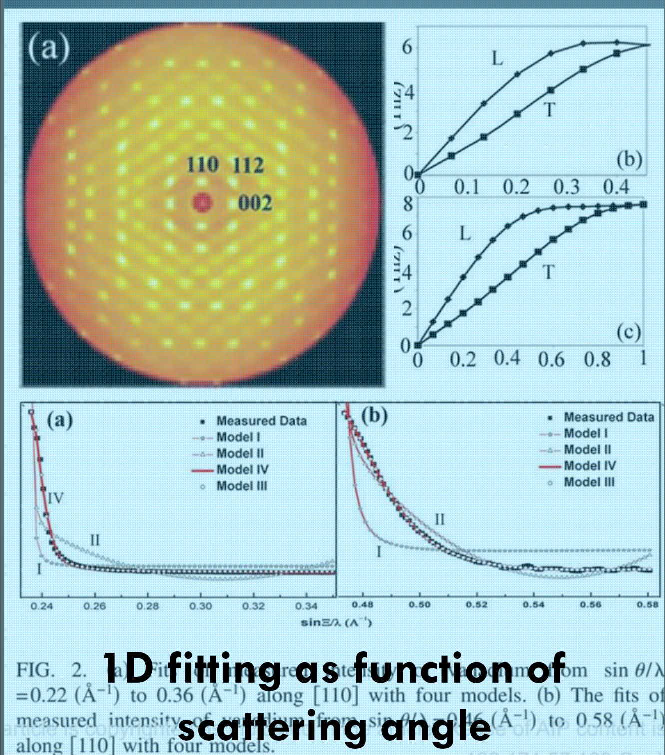
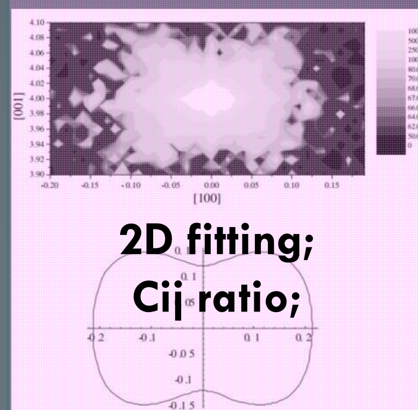


FIG. 2. (a) Fit... along [110] with four models. (b) The fits of measured intensity of vanadium ... along [110] with four models.

1D fitting as function of scattering angle

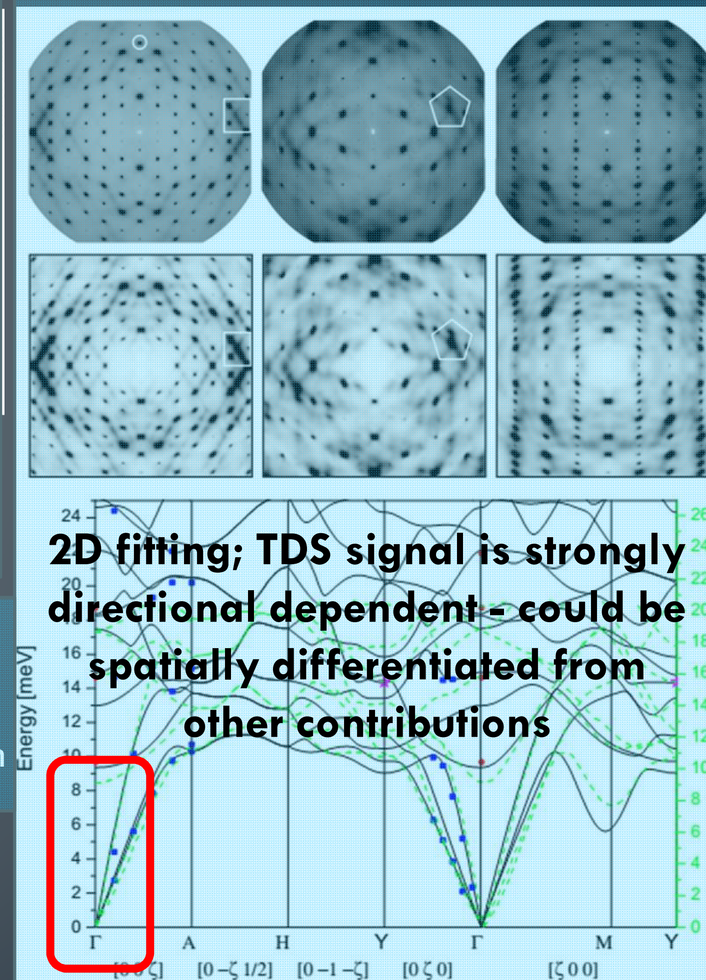
Ding et al. 2006 TDS on **vanadium**
Ambient condition measurement



2D fitting; Cij ratio;

Ohtsu et al. 2008 CdTe
Phase transition at 3.79 GPa

Wehinger et al. 2013 Coesite
Phonon dispersion



2D fitting; TDS signal is strongly directional dependent – could be spatially differentiated from other contributions

Experiment is relatively simple data explanation is difficult – micro force-constant model between the neighbor atoms are needed (1st and higher orders).

X-ray thermal diffuse scattering (TDS)

Only consider 1st order approximation – single-phonon scattering

Method 1: - Cij ratios

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

=?=

Method 2: simplified traditional approach

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

$$I_{\mathrm{TDS}}(\mathbf{Q}+\mathbf{p}) = \frac{k_{\mathrm{B}}T}{V_{\mathrm{c}}}|F(\mathbf{Q})|^2 \mathrm{e}^{-2W(\mathbf{Q})} \times (\mathbf{Q}+\mathbf{p})^{\mathrm{T}}\mathbf{A}^{-1}(\mathbf{p})(\mathbf{Q}+\mathbf{p})$$

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

Vc is the volume of an unit cell, F(Q) is the structure factor of the unit cell and $e^{-2W(Q)}$ is the Debye–Waller factor.

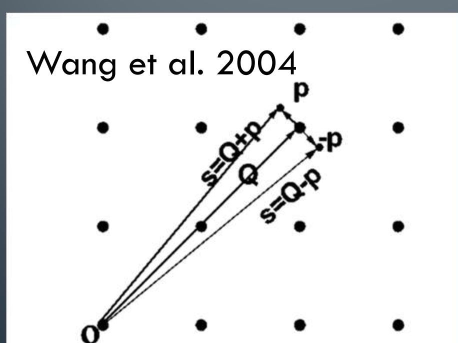
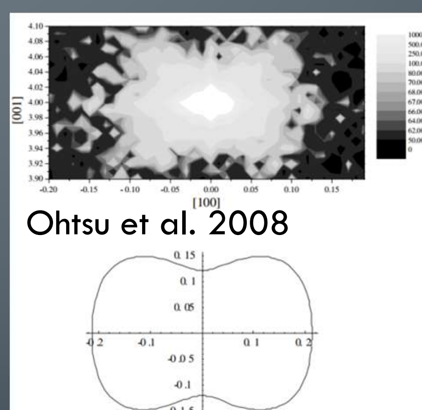
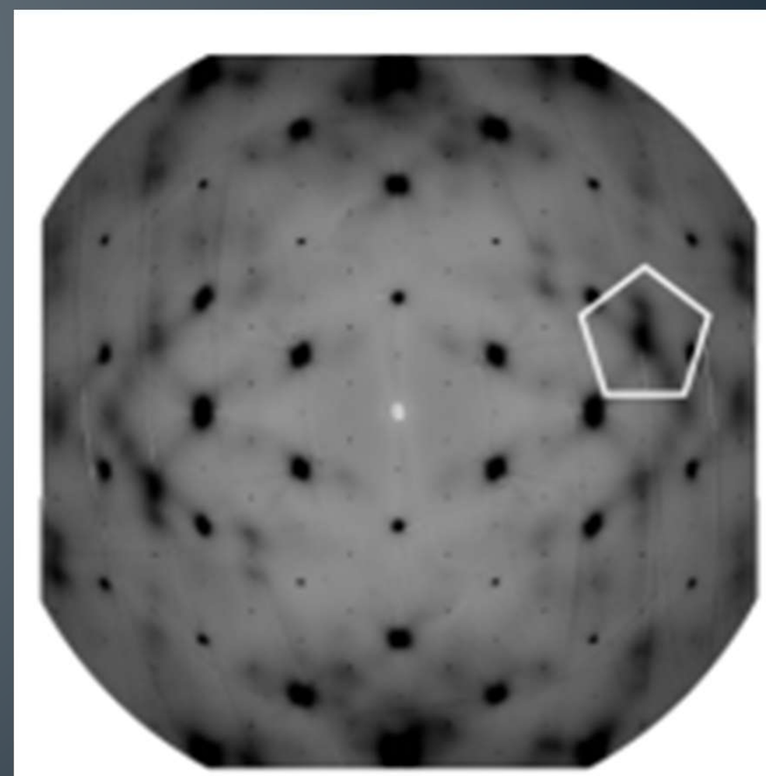


Fig. 1. Schematic diagram showing the relation between diffraction vector **s**, the reciprocal lattice vector **Q**, and lattice wave vector **p**: **s** = **Q** ± **p**.





X-ray thermal diffuse scattering (TDS)

Method 1: Cij ratios =?= **Method 2: Micro force constant model**

- **Test on single crystal Si under ambient conditions:**

Know cij, hkl, calculate tds intensity

- **Method 1: Python module written by Jin**
- **Method 2: Fortran module from Dr. Ruqing Xu, which is callable from python**

Obtained images (known hkls, hkl0 and intensity I_{data})

Starting Cij model, Intensity scaling factor

hkl off set (we never measure exactly at brag peak)

→ **Compute theoretical intensity I_{theo} based on method 1 or 2 for list of hkls**

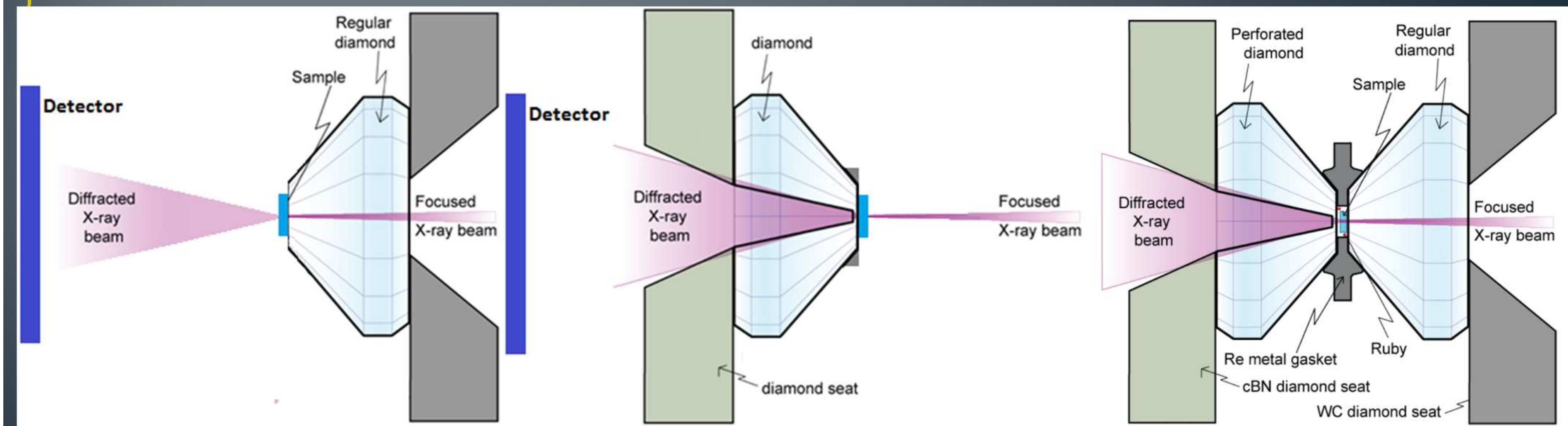
→ **Leastsq fit to minimize (I_{data} - I_{theo})**

→ **Final Cij model Intensity scaling factor etc.**

Inversion code for Cijs written by Jin using python

X-ray thermal diffuse scattering (TDS)

Ambient condition TDS → DAC no pressure transmitting medium → High-pressure TDS with Ne as pressure transmitting medium



Well-defined samples: Si, MgO etc. → Any materials

Si: ambient condition and 1.4 GPa

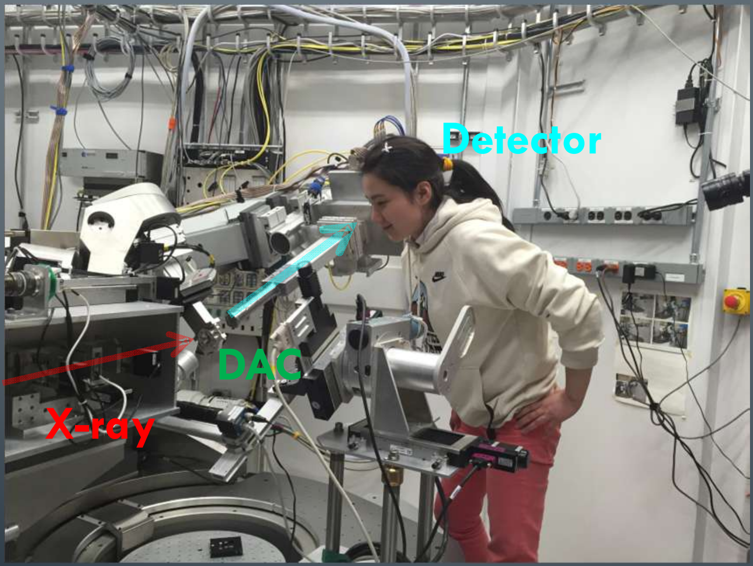
MgO: ambient condition

X-ray thermal diffuse scattering (TDS)

- **Collaboration:**
  - **Sector 34 beamline scientist Dr. Ruqing Xu (TDS analysis)**
  - **GSECARS beamline scientist Dr. Peter Eng**

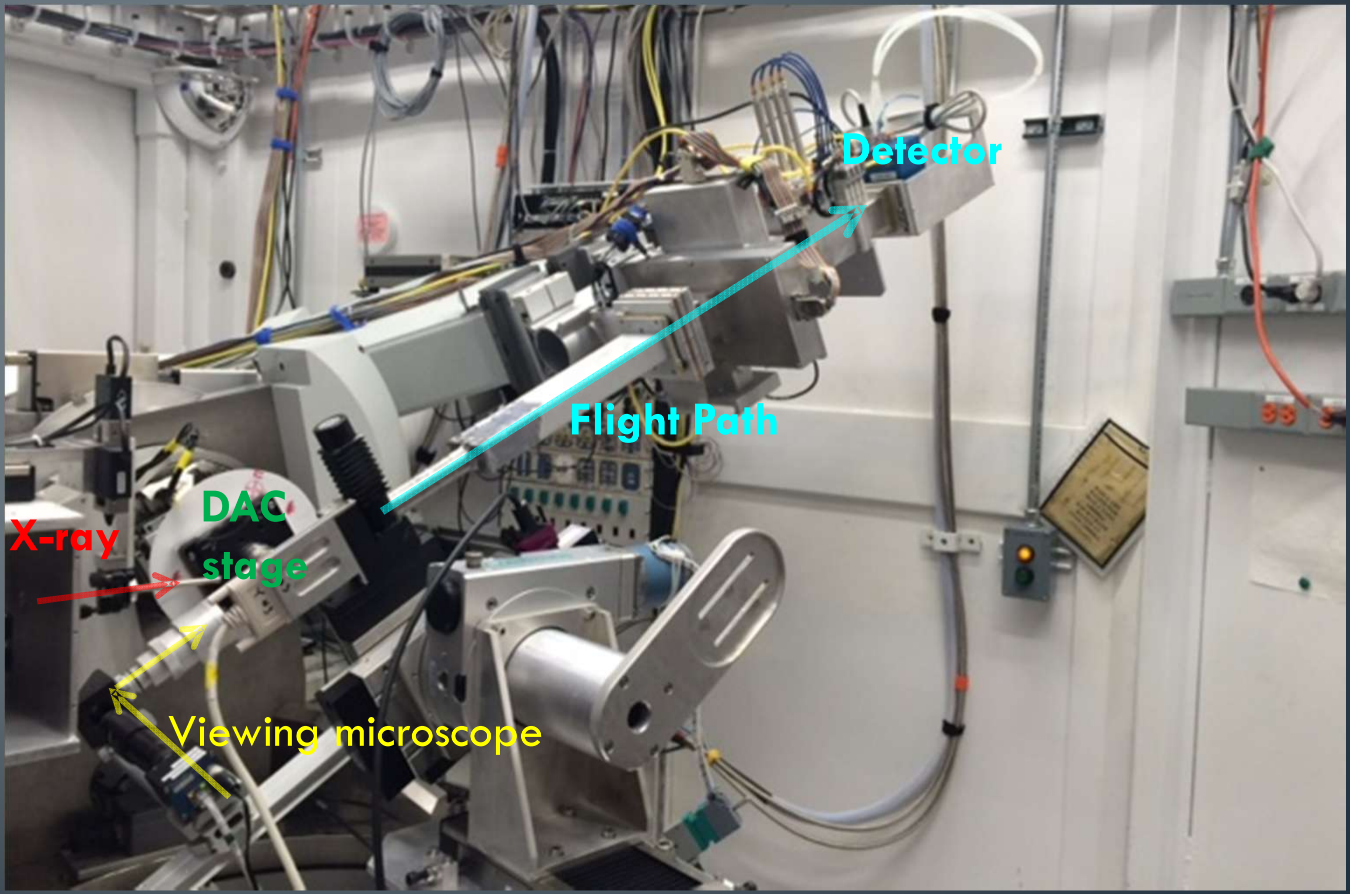
    and Dr. Joanne Stubbs (Surface scattering)

Special setup - flight path: filter out unwanted signal



X-ray thermal diffuse scattering (TDS)

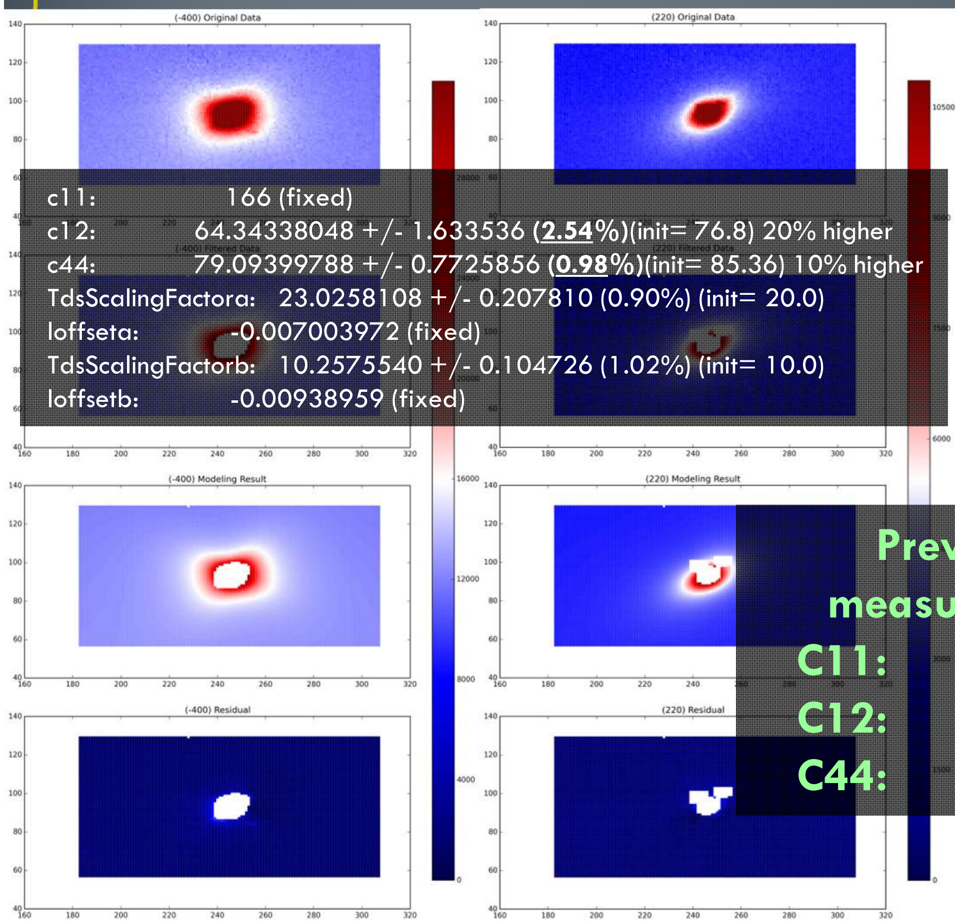
- **flight path: filter out unwanted signal**



X-ray thermal diffuse scattering (TDS)

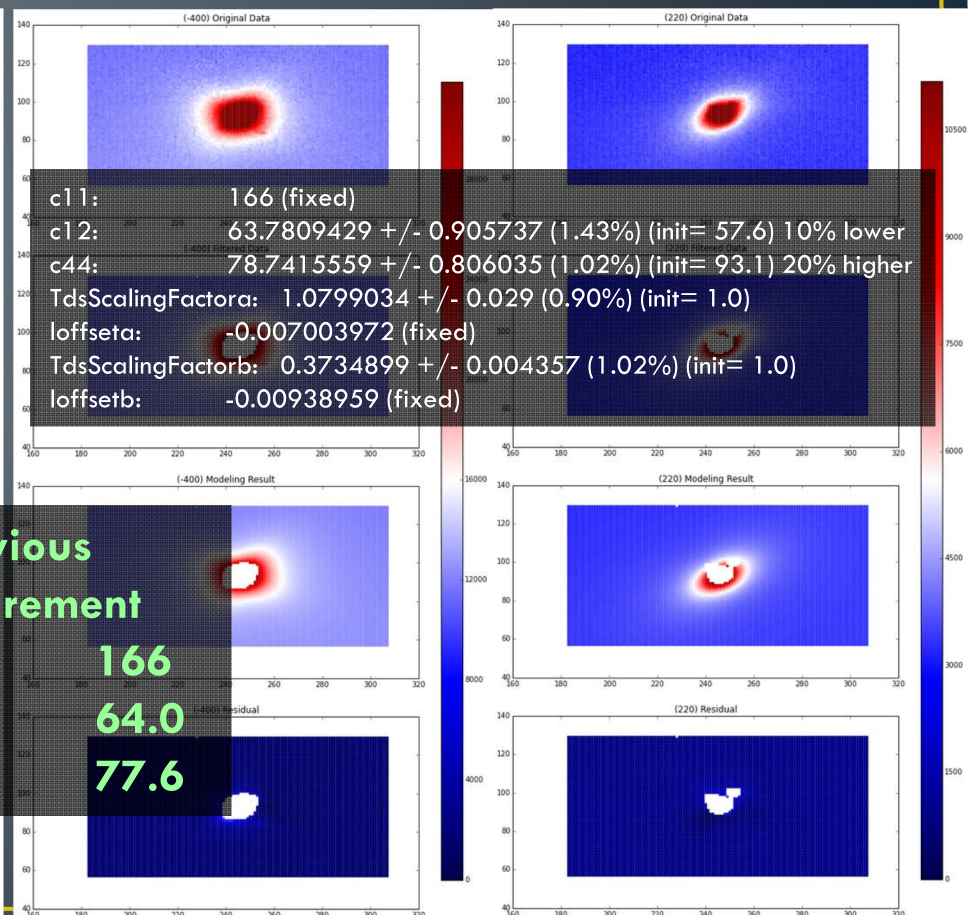
- **Test on single crystal Si: surface quality ultra high**

Method 1: Python module written by Jin



c11: 166 (fixed)
c12: 64.34338048 +/- 1.633536 (**2.54%**)(init= 76.8) 20% higher
c44: 79.09399788 +/- 0.7725856 (**0.98%**)(init= 85.36) 10% higher
TdsScalingFactora: 23.0258108 +/- 0.207810 (0.90%) (init= 20.0)
Ioffseta: -0.007003972 (fixed)
TdsScalingFactorb: 10.2575540 +/- 0.104726 (1.02%) (init= 10.0)
Ioffsetb: -0.00938959 (fixed)

Method 2: Fortran module from Dr. Ruqing Xu



c11: 166 (fixed)
c12: 63.7809429 +/- 0.905737 (1.43%) (init= 57.6) 10% lower
c44: 78.7415559 +/- 0.806035 (1.02%) (init= 93.1) 20% higher
TdsScalingFactora: 1.0799034 +/- 0.029 (0.90%) (init= 1.0)
Ioffseta: -0.007003972 (fixed)
TdsScalingFactorb: 0.3734899 +/- 0.004357 (1.02%) (init= 1.0)
Ioffsetb: -0.00938959 (fixed)

Previous measurement

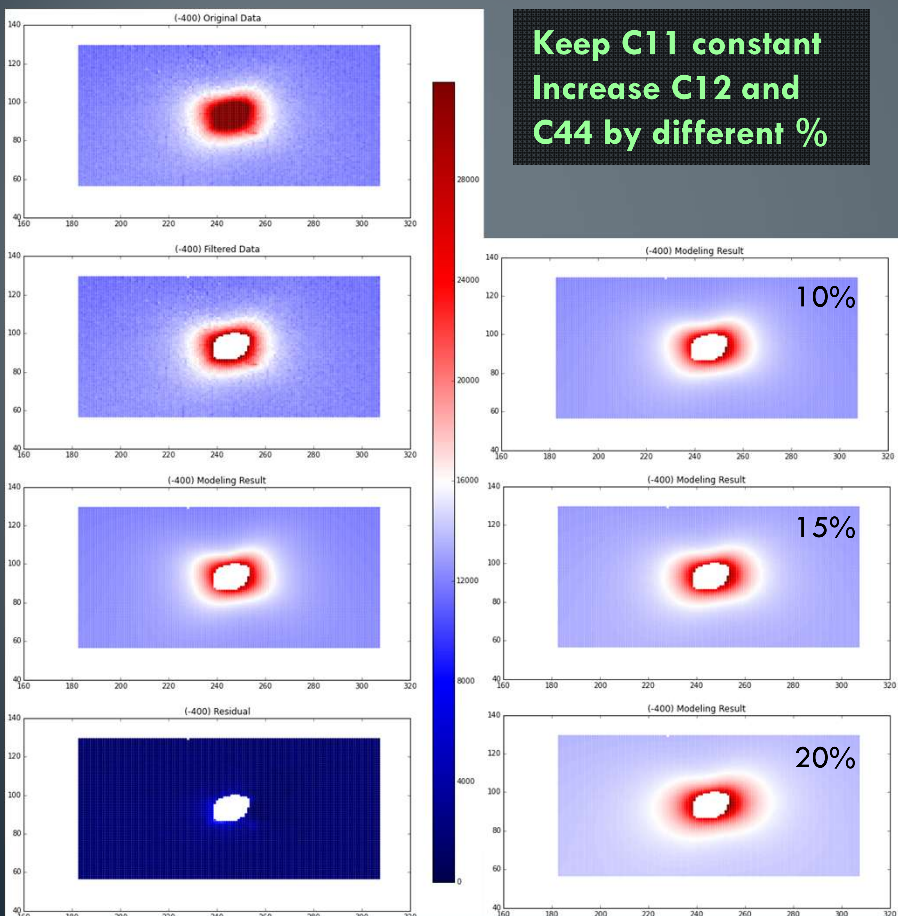
C11:	166
C12:	64.0
C44:	77.6

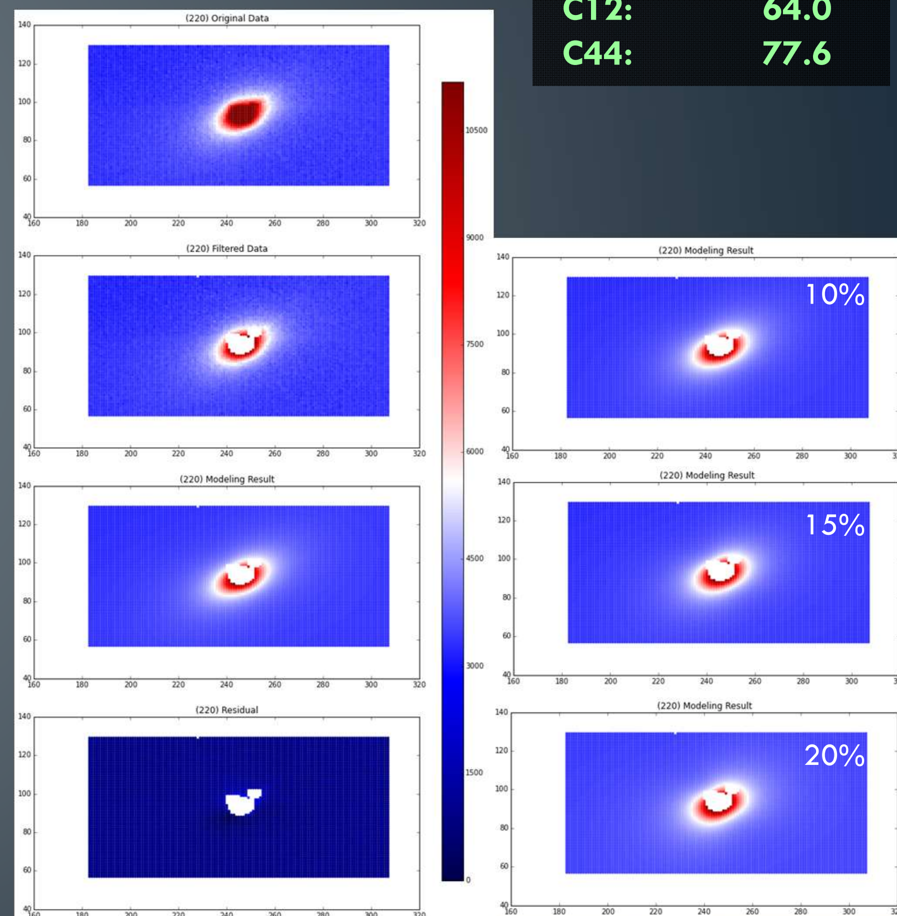
X-ray thermal diffuse scattering (TDS)

- Test on single crystal Si: accuracy of the measurement

ideal values	
C11:	166
C12:	64.0
C44:	77.6

Keep C11 constant
Increase C12 and
C44 by different %

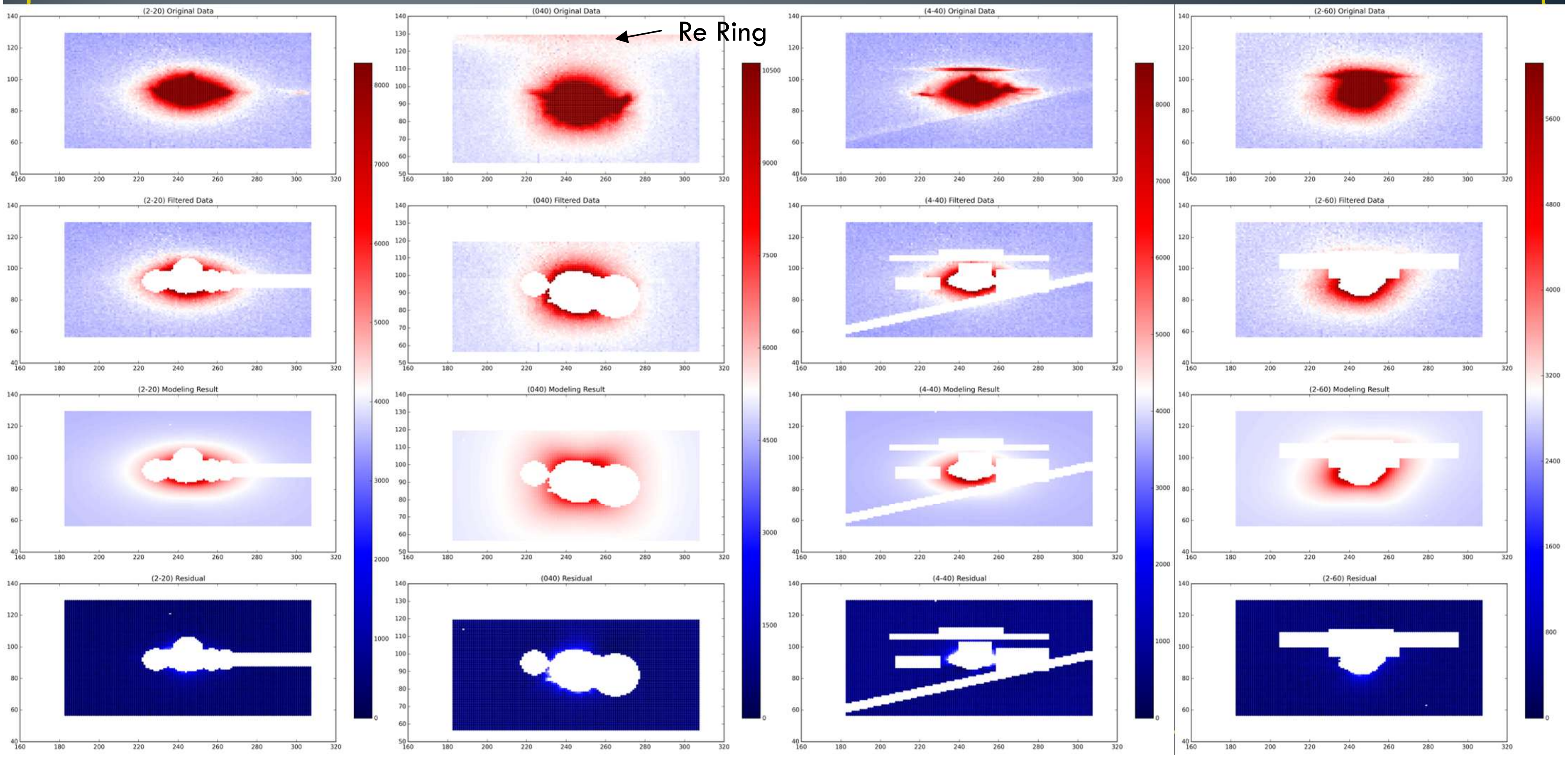




X-ray thermal diffuse scattering (TDS)

- Test on single crystal Si: 1.4 GPa
  - Si crystal is very brittle; Peaks broadened; A lot more features show up

No Cij values reported for Single crystal Si under pressure



X-ray thermal diffuse scattering (TDS)

- Short summary
  - Optimized experimental setup:
    - Flight path: minimize background contribution
    - Filter out signals from diamond, air, pressure medium
  - Development of tds fitting code
    - Simple fitting using single-phonon assumption worked
    - Not necessary to use micro-force-constant models
  - Uncertainty of less than 5% expected for TDS measurement
- Future work
  - Reduce Error: sample orientation for the flight path setup – only two brag peaks are used for orientation determination
  - **Standard measurement: bench mark for high pressure TDS measurement**
  - Software interface: work with professional software engineers
  - Working with theoreticians: under situation where trade-offs are large (e.g. low symmetry materials), tds only might not yield to a single solution, but will provide extra constrain to theoretical calculation.

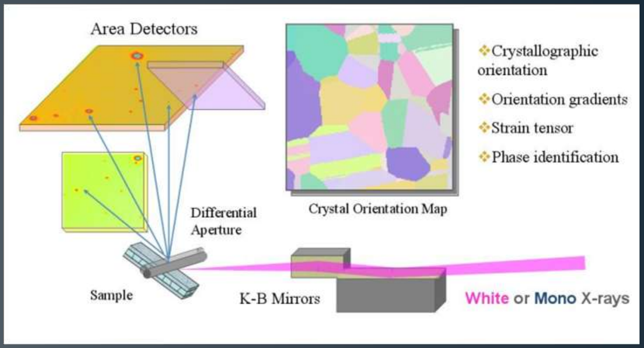
X-ray thermal diffuse scattering (TDS)

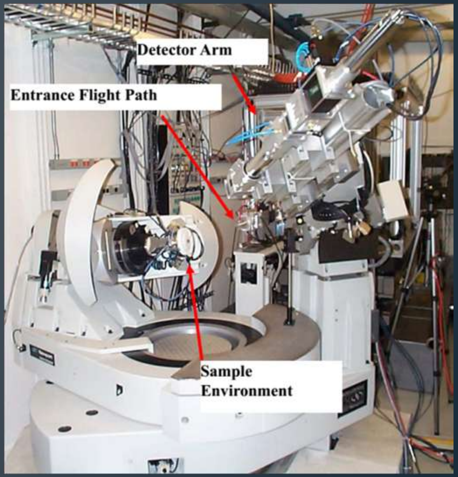
- Test on single crystal MgO: ambient condition
  - Data not good
  - Investigation: Crystal quality not good – crystal mosaicity determined from rocking curve is high (~1.2-1.4°)
- Found new MgO samples
  - Checked in UIUC X-ray lab in Chemistry, one small crystal might be useable
- Fosterite? Low symmetry
  - Well determined at high P, measure by Zha et al. through Brillouin
- Garnet? Not that interesting but high symmetry
  - Pyrope/almandine, almost acoustically isotropic
- Bridgemenite? Low symmetry but interesting
  - Single crystal elasticity at HP not well-determined…

Suggestions: material choice

X-ray thermal diffuse scattering (TDS)

- **Future beam time in 2015-2**
  - **34IDE** Laue/energy scan approach + CCD
    - **Advantages:**
      - **Hkls more precisely determined:** the sample does not have to be rotated
      - **Nano-beam:** measure super small crystals/seek better spot on a big sample
    - **Disadvantages:**
      - **CCD detector**
      - **Loss of flux due to super tight focus**
  - **13IDC** Single crystal, angular scan approach + flight path/CCD
    - **Advantages:** **High flux: ID beam line; flight path: filtered unwanted signal**
    - **Disadvantages:** **focus ~30-50um, hkl determination could be improved**
  - **13BMC** Single crystal, angular scan approach + flight path/CCD
    - **Advantages:** **focus ~15um; flight path: filtered unwanted signal**
    - **Disadvantages:** **focus ~15um; low flux: BM beam line, hkl determination could be improved**



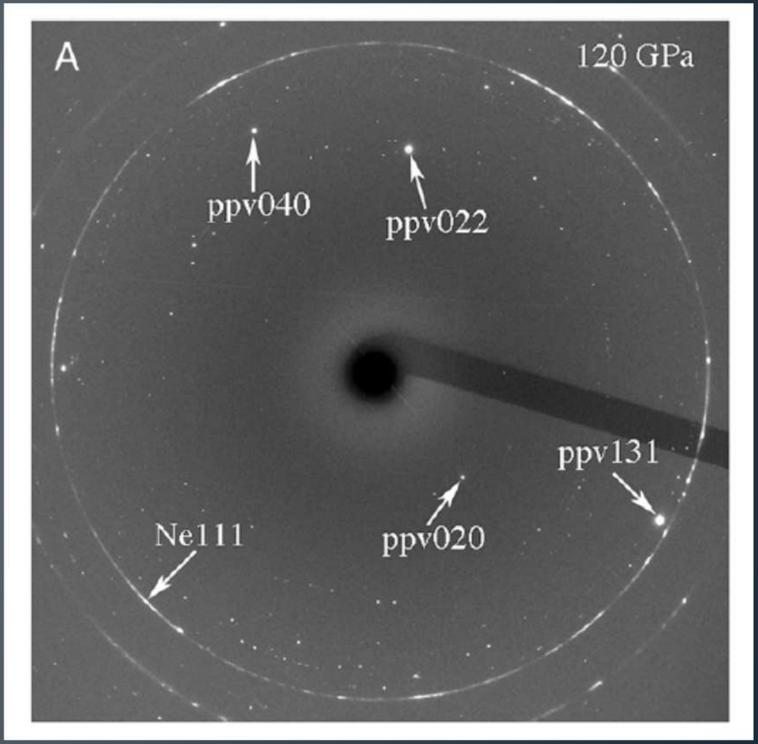


Multi-grain X-ray diffraction

- Reliable analysis of data from high-pressure experiments that involve samples in-between the single crystal and powder state has been very high on the wish list of mineral physics researchers for several years.

- There has been a number of recent very exciting high-pressure discoveries that resulted from laser heating experiments and produced coarse powder samples of new unquenchable phases, e.g. Fe_4O_5 (Lavina et al. 2009, PNAS), ppv (Zhang et al. 2013, PNAS), H-phase (Zhang et al. 2014, Science 2014), carbonates (Merlini et al. 2012, EPSL), with analysis performed using the multigrain approach.

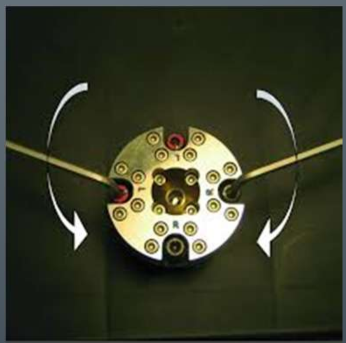
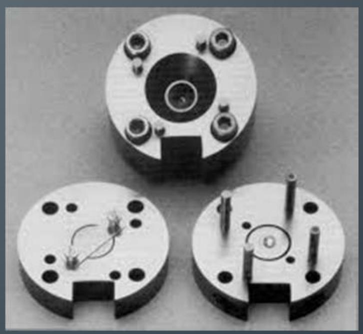
- The same approach has also been applied to study lattice preferred orientation development in rheological experiments is DACs (NISR et al. 2014, HPR).



Zhang et al. 2013, PNAS. Data from 16IDB

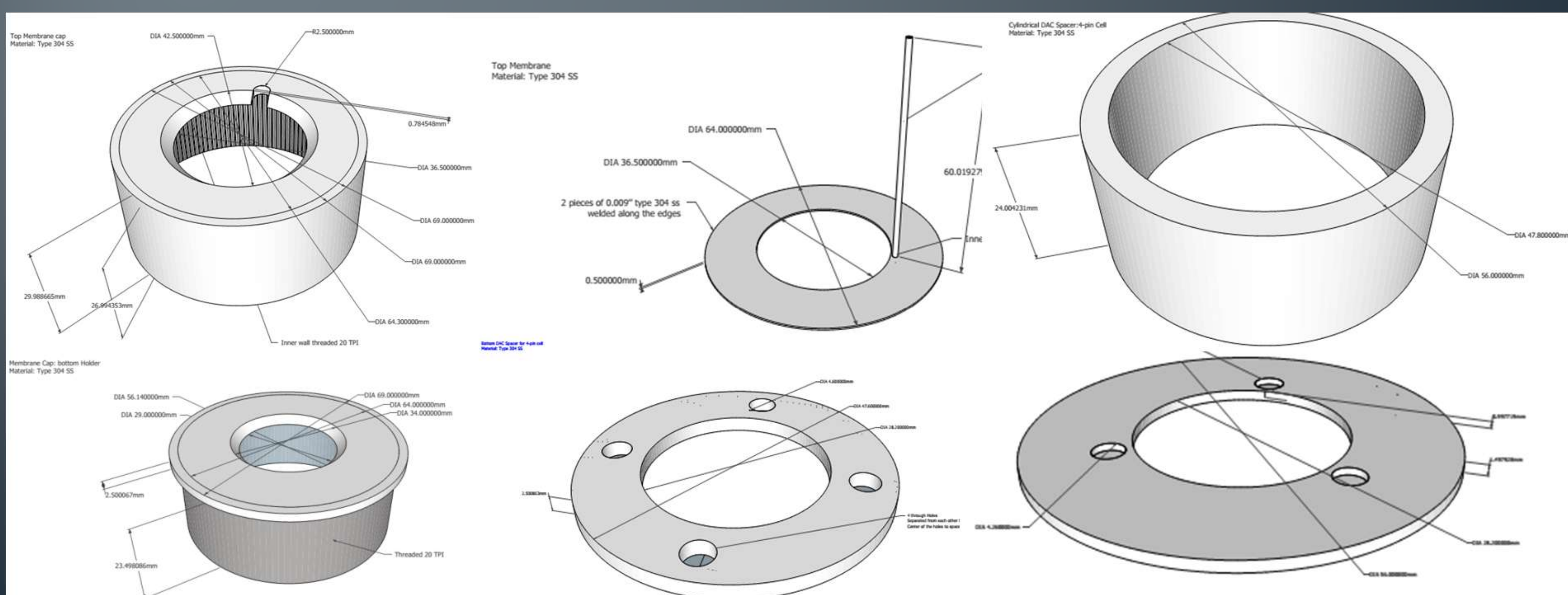
Universal Membrane cap for most DACs

- 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
  - Most are good only for compression (**Many thanks to Stas Sinogeikin**: solved by **double membrane cap design**)
- Our target:
  - Modify the membrane cap after previous designs (e.g. Yale, UIUC, especially Stas' design).
  - **One cap for all** DACs in different sizes: different spacers
  - **No lose in DAC opening**: ideal for single crystal studies

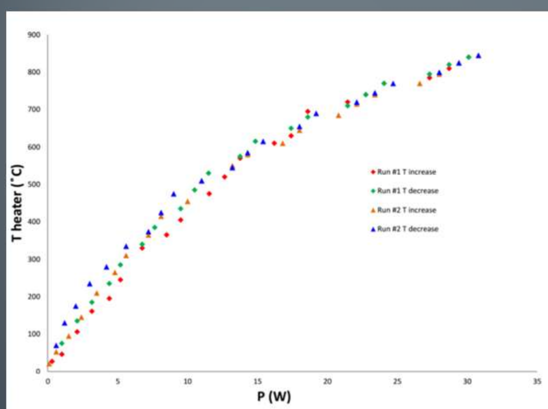

Universal Membrane cap for most DACs

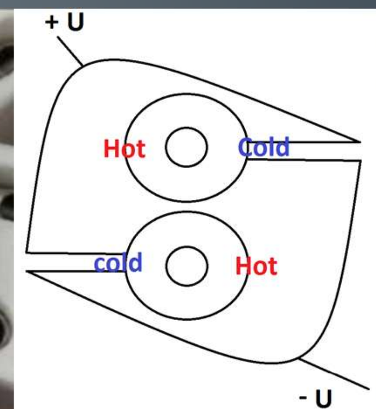
- Strategy
  - Single membrane cap – waiting for machining in Hawaii
  - Double membrane cap
  - Online resource: extensive test with perspective users
  - Order available for all COMPRES users through UH machine shop
  - **Drucker pressure controller will be ordered through COMPRES**



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 published)

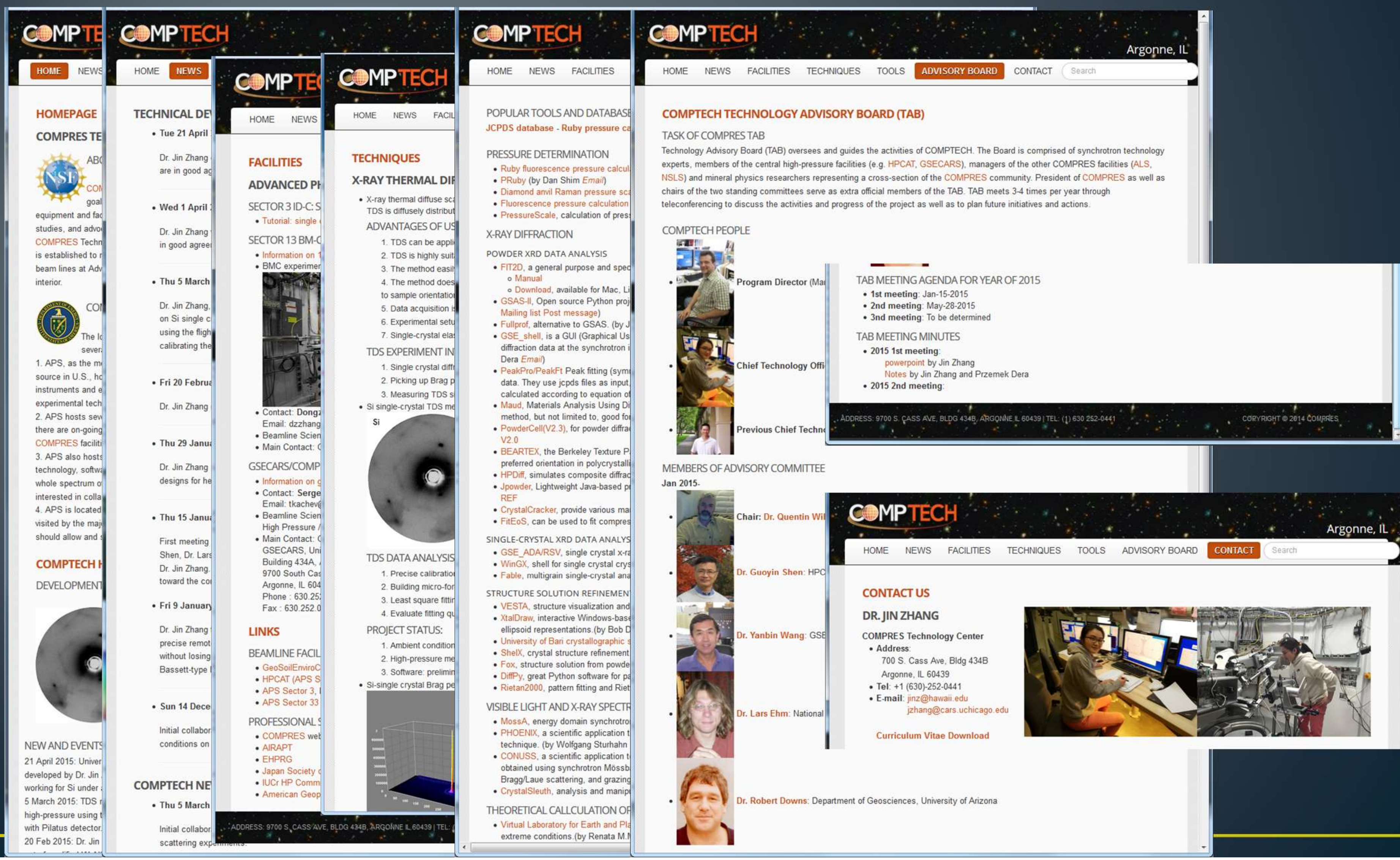




- Modification of W-Al_2O_3 heater:
  - The above tested heater: ID 4mm; 6-10 Ohm model
  - Modified heater: ID 6.5mm; 2 Ohm model
  - Received the modified heaters
  - Waiting for further testing

Website

http://comptech.compres.us/



Multigrain analysis - introduction

Goals

- Provide reliable, easy to use, sufficiently automated, and optimized for high-pressure applications software solution for analysis of multigrain data.
- Develop a reliable data collection methodology.

Funding

- This activity does not use COMPTECH resources, though Jin's involvement in pilot experiments at HPCAT in April is gratefully acknowledged.
- Supported by NSF Geoinformatics project Advanced Tools for Extreme Xtallography (ATREX).
- Now also part of new NSF software Infrastructure for Sustained Innovation project (PI Matt Newville).
- Now also part of new NASA SERA project (PIs David Blake and Bob Downs).

Personnel

- Harold Garbeil - software engineer, UH
- Linda Martel – web services and outreach, UH
- Yi Hu – PhD Student, UH
- Hannah Shelton – PhD Student, UH

Collaborators

- Dongzhou Zhang – PX^2 collaborator, UH @ APS
- Jin Zhang – COMPTECH collaborator, UH @ APS
- Jesse Smith - HPCAT
- Yue Meng - HPCAT

Multi-grain X-ray diffraction

- Development of reliable and optimized heating protocol which will reproducibly yield optimal samples. (GUP proposals13IDD and 16IDB)
- Development of optimized data collection strategy which will guarantee best data quality, minimize effects of sample moving with respect to the beam during data collection, maximize data coverage, etc. (GUP proposals 13IDD and 16IDB)
- Development of software that will allow carrying out the data analysis in a manner simple enough for at least partial on-the-fly data interpretation. (ATREX software development project)

Synthesis and Microdiffraction at Extreme Pressures and Temperatures

Barbara Lavina[1], Przemyslaw Dera[2], Yue Meng[3]

[1]High Pressure Science and Engineering Center, Department of Physics and Astronomy, **University of Nevada, Las Vegas**, [2]GeoSoilEnviroCARS, **University of Chicago**, [3]High Pressure Collaborative Access Team, **Carnegie Institution of Washington**

The laser heated diamond anvil cell combined with synchrotron micro-diffraction techniques allows researchers to explore the nature and properties of new phases of matter at extreme pressure and temperature (PT) conditions. Heterogeneous samples can be characterized *in situ* under high pressure by 2D mapping and combined powder, single-crystal and multigrain diffraction approaches.

Published **October 7, 2013**. Keywords: Physics, x-ray diffraction, geochemistry, geophysics, solid-state physics, high-pressure, high-temperature, Diamond anvil cell, micro-diffraction, novel materials, iron oxides, mantle mineralogy

Project activities in 2015-1

Software

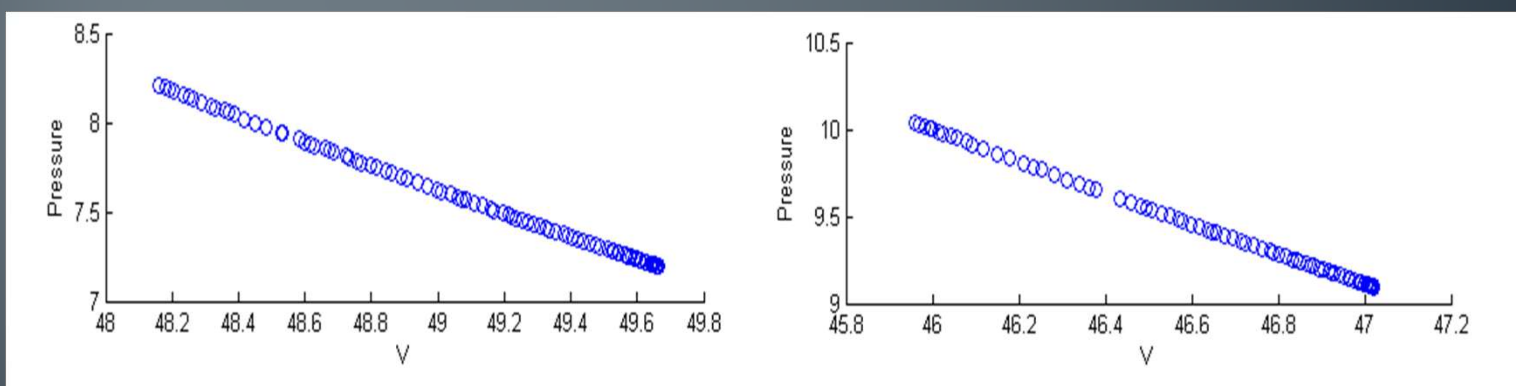
- Conversion of IDL GSE_ADA (now ATREX) code to Python and migration to open source distribution through GitHub (on going, project repositories for IDL and Python code already available on GitHub)
- Enhancement of functionality of GSE_ADA code towards increased automation and handling of multicrystal, as well as time resolved single-crystal data (on going, major improvements in handling spatial overlaps and overall automation in the IDL code)
- Support for automated massive serial data analysis (1000s of pressure points, TBytes of image data) from Pilatus 1M
- Data collection software for single crystals/multigrain using Pilatus 1M and membrane-driven DAC in continuous compression mode.

Experiments & methodology

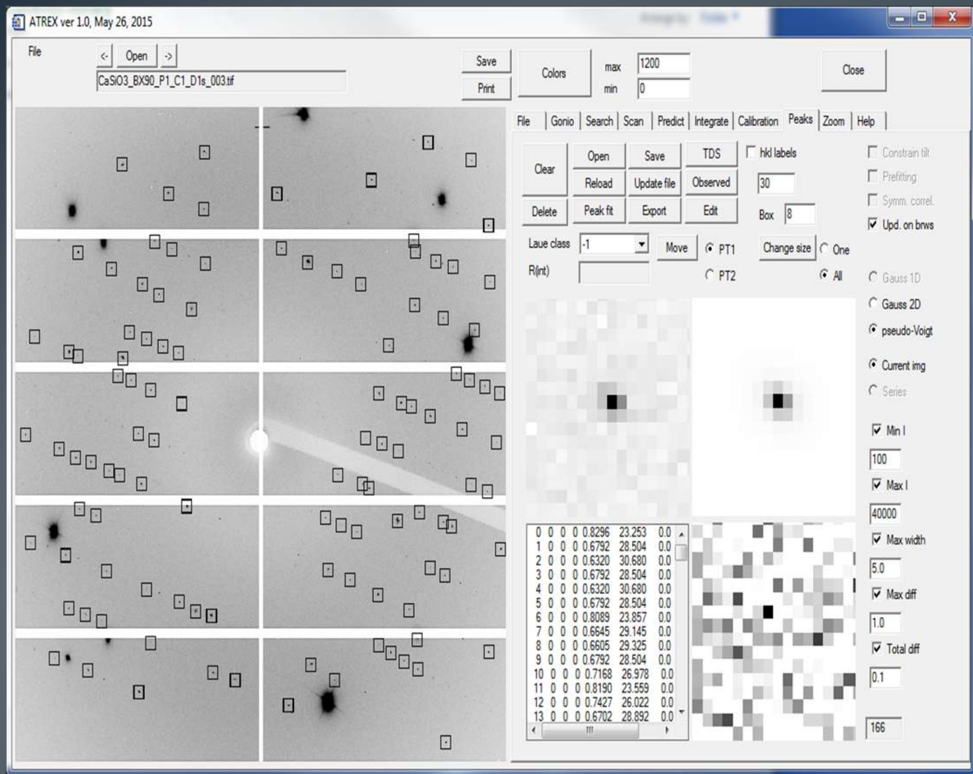
- Conducted 2 GUP experiments (February and April) at HPCAT 16IDB involving elements of multigrain/time resolved techniques:
  - oFs100 single crystal (ambient temperature + resistive heating)
  - oFs100 single crystal + multigrain
  - cFs100 single crystal
  - CaSiO3 wollastonite single crystal + multigrain (ambient temperature + laser heating)
- Worked on automated rastering technique in data collection

Pilatus 1M single crystal and multigrain experiments:

- Constrain hysteresis of reversible phase transition
- Obtain reliable compressibility information over a narrow pressure range (1-2 GPa) from hundreds of measurements at very small, continues pressure increments
- Monitor progress of phase transition though phase fraction analysis
- Be able to work with multiple crystallographically complex (low symmetry, large unit cell) phases and carry out automated analysis

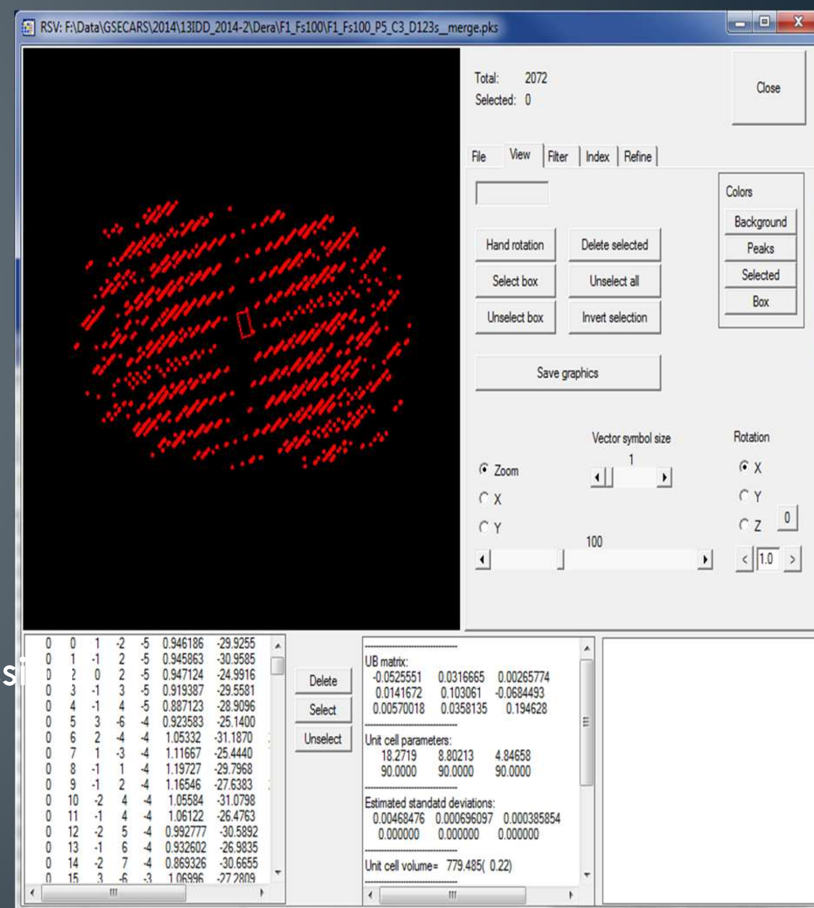


Unit cell volume of Ne vs. pressure in continuous compression time resolved experiments with oFs100



CaSiO3 at 10 GPa – serial processing of time-resolved SXD data from Pilatus 1M

oFs at 30 GPa – fully automated data processi



Plans for 2015-2

Software

- Finalize enhancements of functionality of ATREX IDL code.
- Continue code conversion to Python
- Development of online documentation and training materials
- Further improvements in support for automated massive serial data analysis from Pilatus 1M

Experiments & methodology

- Another 2 GUP experiments (July and August) at HPCAT 16IDB involving elements of multigrain/time resolved techniques:
    - Cristobalite and $Be(OH)_2$
    - oFs100 and cFs100