COMPTECH TAB meeting 2015-3

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COMPTECH Officer @ APS, ANL

09/23/2015 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
  - Sector 34, add-in viewing and optical system for high-pressure experiments

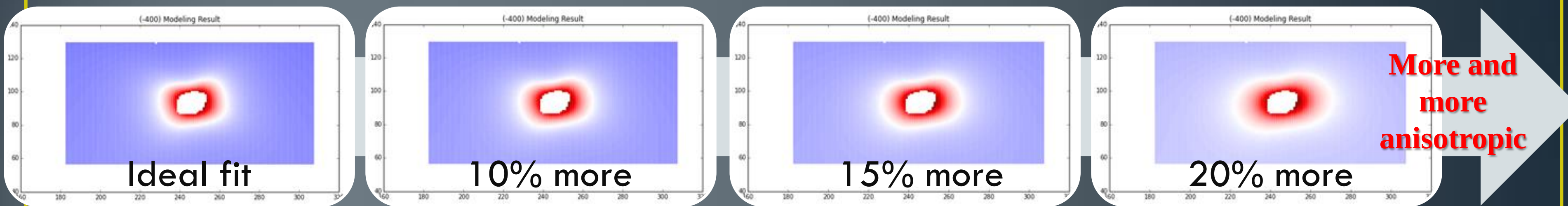
Time line of COMPTECH

- **2014.11** — join COMPTECH
- **2014.12** — 13BMC PX^2 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 universal membrane cap | Website revision: Techniques | Preparation for TDS beam time
- **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-Al_2O_3 heater test in DAC
- **2015.9** — Testing new inversion algorism for TDS data analysis | 1st heater user: U of Arizona
- Budget approved for adding optical system for 34ID

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

Publication: 2015Fall-2016Spring;

Engaging users: 2016Summer-2016Fall (workshop TDS, collaboration: sample)

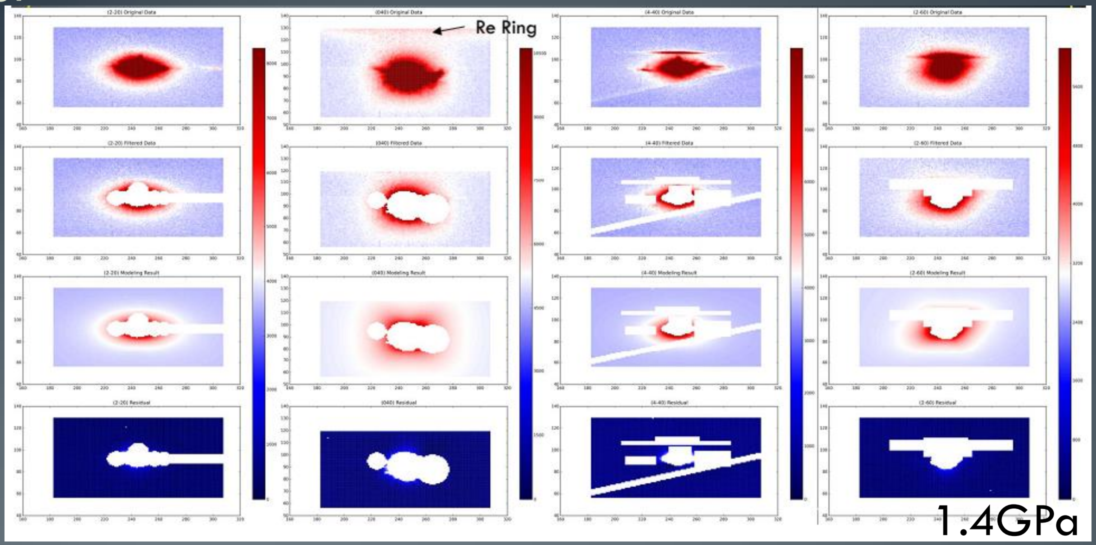
New technique: starting 2017Spring (survey in the workshop: XANES XAFS Raman?)

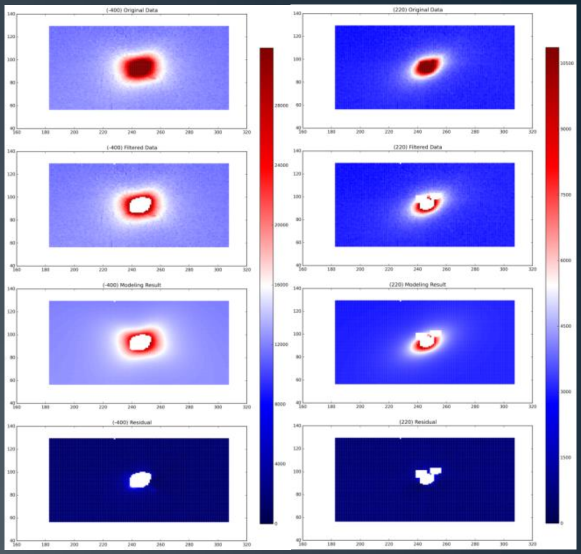
X-ray thermal diffuse scattering (TDS)

1 beam time in 2015-1:
13BMC
3 beam times in 2015-2:
13IDC
13BMC
34ID

- Current Status:
  - **Sample testing:**
    - **Ambient condition: Si (100) (111) and forsterite (111)**
    - **High-pressure: Si (100) (1GPa); forsterite (111) (~40GPa)**
  - **Experimental Setup:**
    - **Using flight path decrease background (originally designed for surface scattering experiment) 13BMC 13IDC (beam size 30μm)**
    - **Energy scan to find Brag peaks, then monochromatic beam for TDS measurement (MarCCD) 34ID**
  - **Software development:**
    - **Python: micro-force-constant model for Si**
    - **Python: Macro-single crystal elasticity model for crystals with different symmetry**
    - **Python: new optimized inversion code with better efficiency for lower symmetry materials**

- Si

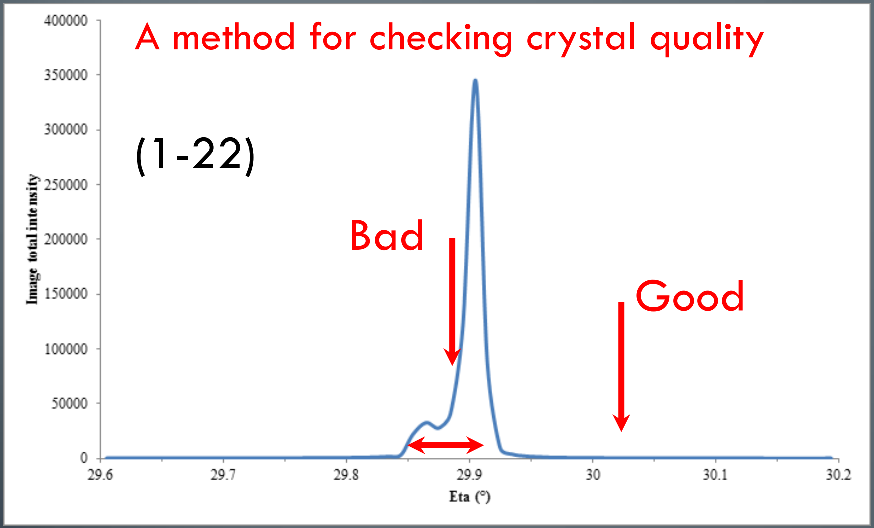


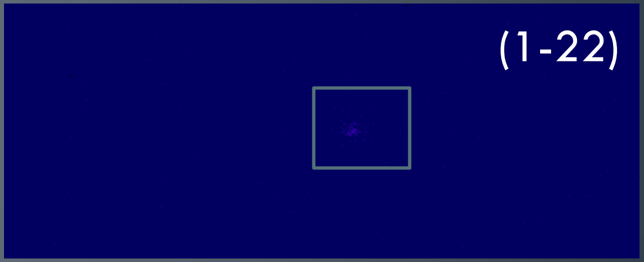


- B. Winkler (U of Frankfurt, 1st principle calculation) (meet during COMPRES 2015 annual meeting)
  - C11=156.6; C12=61.2; C44=74.6 @0GPa
  - C11=163.3; C12=67.9; C44=75.5 @1.4GPa
- Decremps et al. (PRB 2010) Using phonon imaging up to 7.9GPa
  - C11=165.7; C12=63.6; C44=80.0 @0GPa
  - C11=172.1; C12=71.5; C44=80.0 @1.4GPa
- TDS calculation, this study:
  - C11=166.0(fixed); C12=64.3(±1.6); C44=79.1(±0.8) @0GPa
  - C11=172.0(fixed); C12=71.6 (±3.1); C44=78.2(±2.7) @1.4GPa

X-ray thermal diffuse scattering (TDS)

- Sample testing:
  - **forsterite (111) low symmetry (orthorhombic) up to ~40GPa**
    - **Geo-materials: silicate**   **He pressure medium VS Ne pressure medium**





Change the **Eta angle**:
Off diffraction condition
to diffraction condition
to off diffraction condition

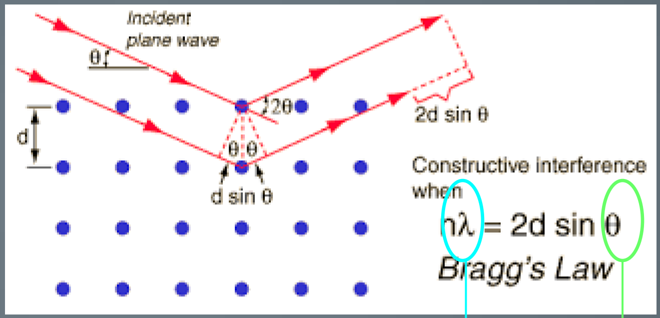
For olivine: ~0.1 up to 25GPa <0.3 ° up to 32GPa <0.5 ° up to 40GPa

- **Accurate determination of orientation matrix (12-20 peaks used instead of just 2)**

Less fitting parameters (No parameter is needed to account for orientation uncertainty)

X-ray thermal diffuse scattering (TDS)

- Sample testing:
  - **Energy scan approach at sector 34**

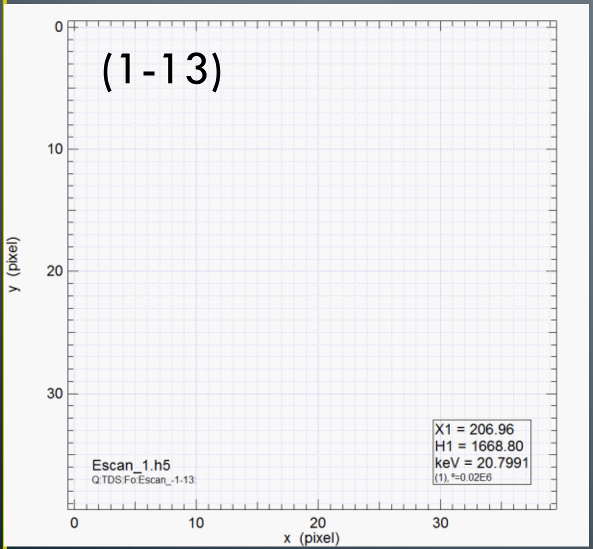


Beam size <500 nm

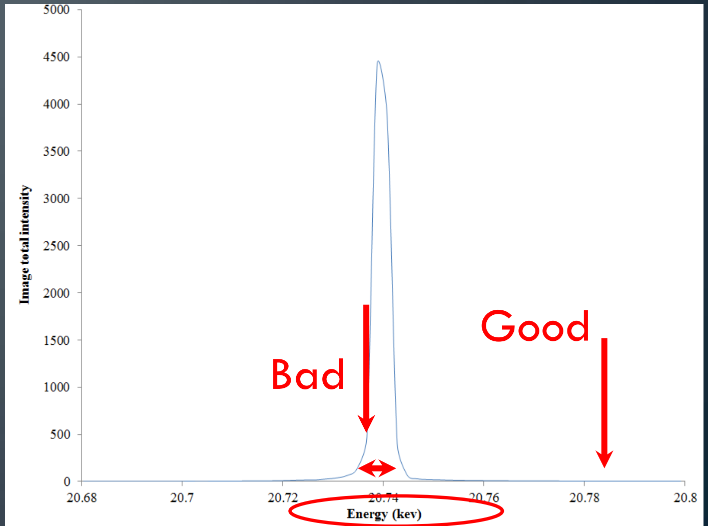
"Bad quality" crystal is not "bad" anymore

34ID

13IDC and BMC

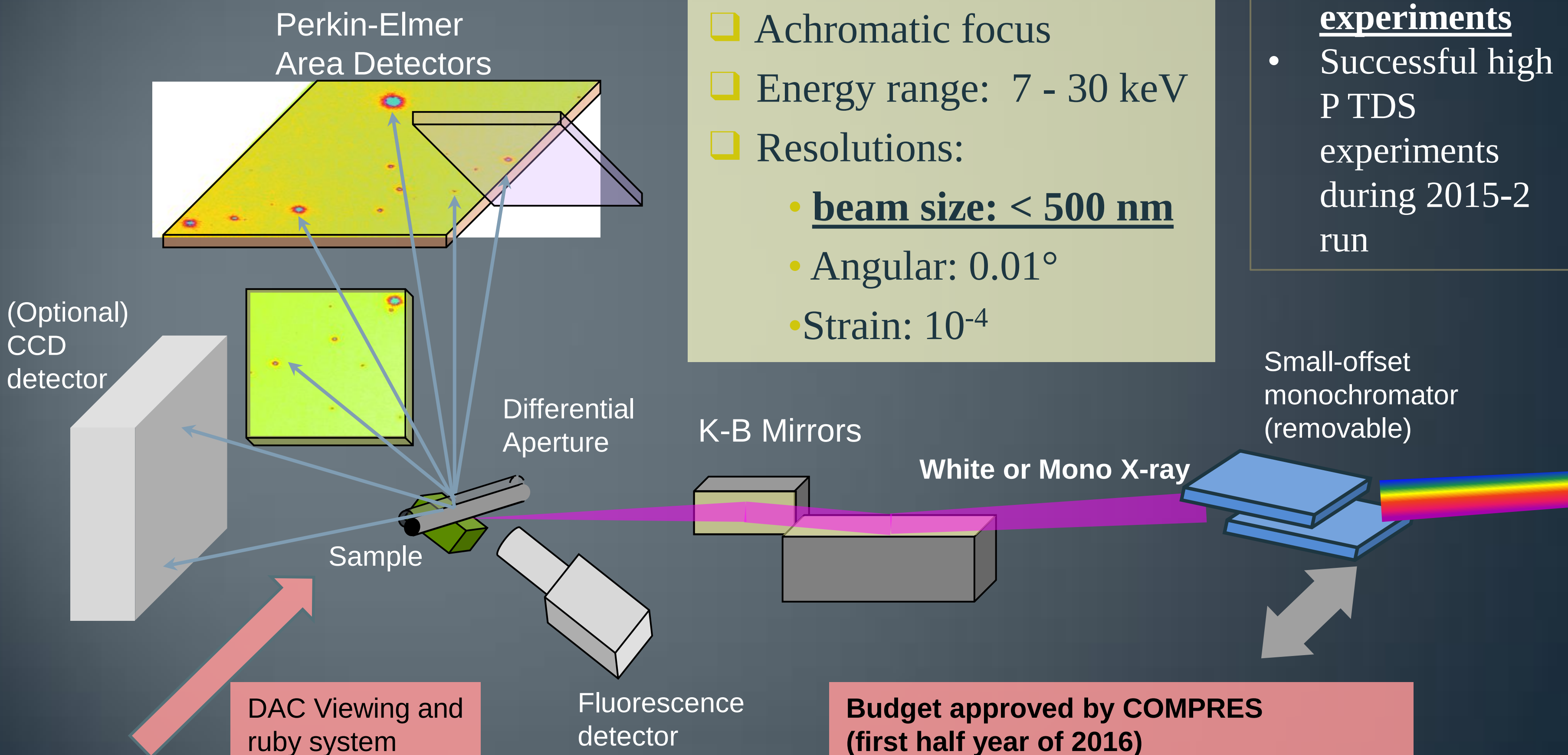


Change the **energy**:
Off diffraction condition
to diffraction condition
to off diffraction condition



Preferred access to existing beamlines

Sector 34 ID



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

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

Budget approved by COMPRES (first half year of 2016)

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

X-ray thermal diffuse scattering (TDS)

- **Inversion code development**

Only consider 1st order approximation – single-phonon scattering

In 2015-2

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

Initial elasticity model Cijkl

Intensity scaling factor

HKLs

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

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

Treated as global parameter

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

Fo (27):
9 parameters for Cij
18 intensity scaling factors for 18 TDS images

X-ray thermal diffuse scattering (TDS)

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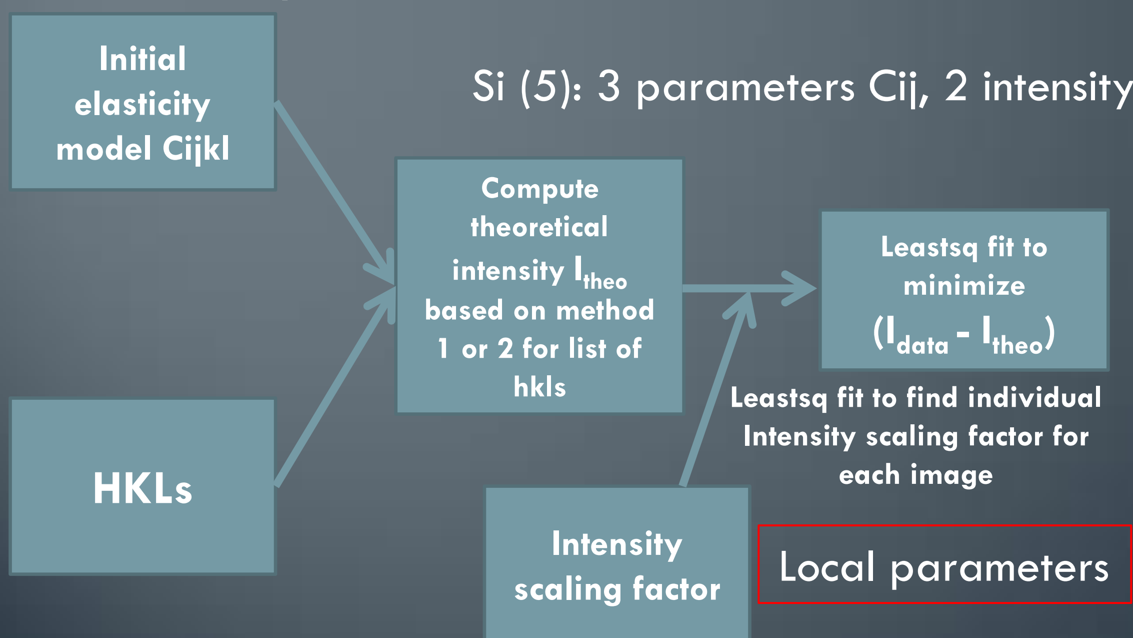


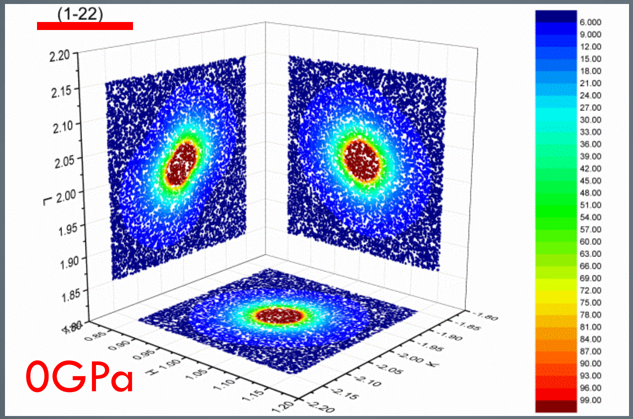
Si (5): 3 parameters Cij, 2 intensity scaling factors for 2 images

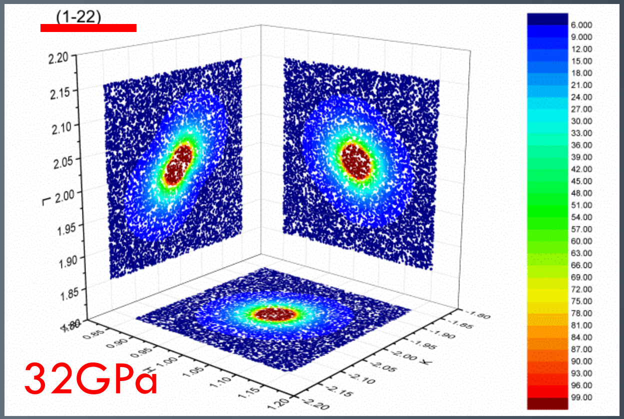
Fo (27):
9 parameters for Cij
18 intensity scaling factors for 18 TDS images

Debugging the code now

X-ray thermal diffuse scattering (TDS)

- **TDS simulation**
  - **Given elasticity model and HKL simulate the TDS signal**





Future work in last TAB meeting:

√ **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

<u>Software interface</u>: work with professional software engineers

<u>Working with theoreticians</u>: 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.

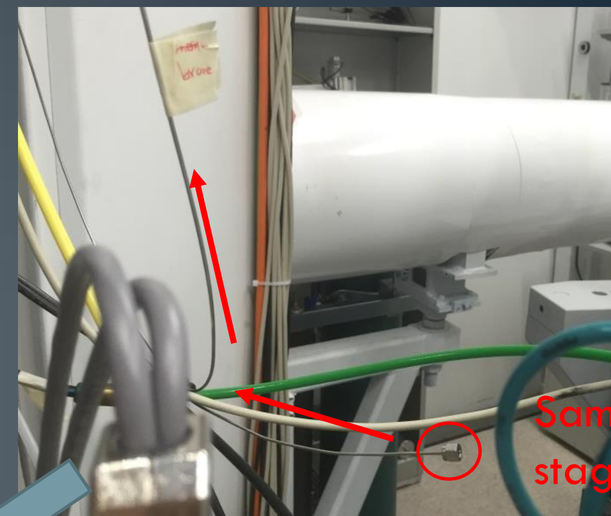
High temperature effect **More exciting materials (seeking collaborations and invite users)**

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 all** DACs in different sizes: different spacers
  - **No lose in DAC opening**: ideal for single crystal studies
- Project status:
  - Designed the first edition of membrane cap 01/2015
  - Received all body parts of membrane cap 06/2015
  - Stainless steel tubing installed in 13BMC already 07/2015
  - GE digital pressure controller ordered 08/2015
  - Gas membranes sent back to machine shop for revision 09/2015
- Project status:
  - Hopefully will get the membrane and pressure controller by 11/2015
  - Welcome COMPRES users to use!

Universal Membrane cap for most DACs



Membrane tubing in 13BMC installed

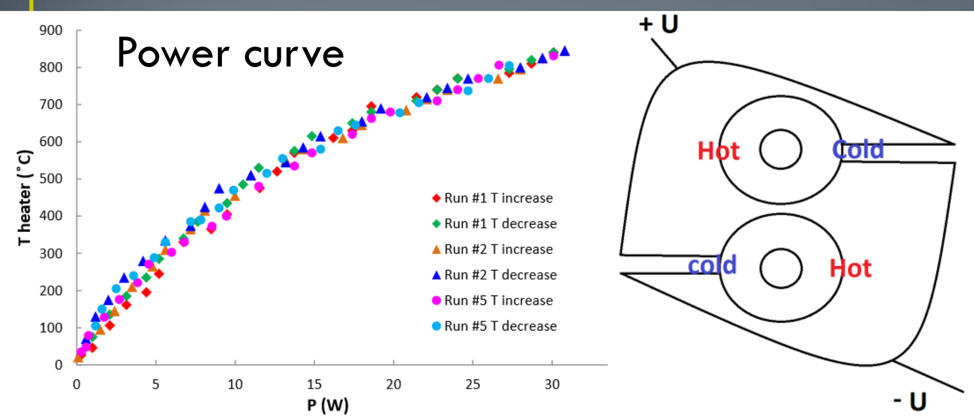
Membrane Holder

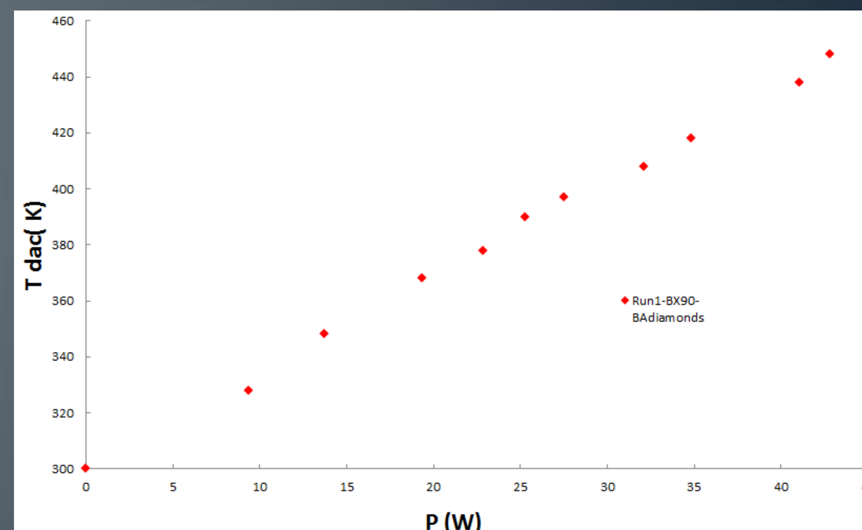
Waiting:

1. Membrane controller
2. Membranes

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])

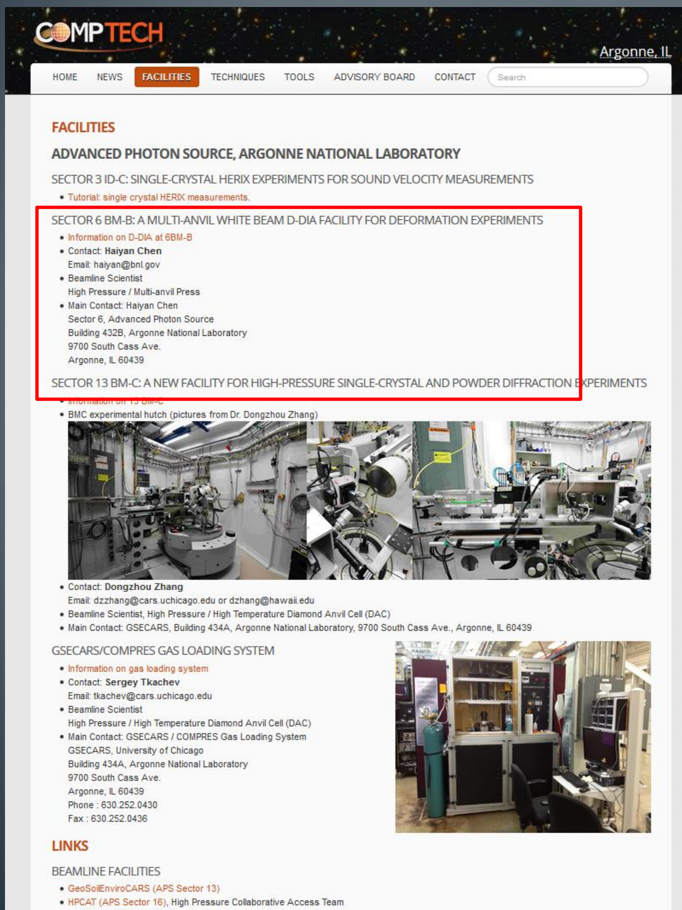




- Project status:
  - 1st test with DAC: with single heater – sample chamber reached highest T 450K
  - 1st supply to COMPRES users: University of Arizona (Dan Shim and Kurt Leinenweber)

Website

http://comptech.compres.us/



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FACILITIES

ADVANCED PHOTON SOURCE, ARGONNE NATIONAL LABORATORY

SECTOR 3 ID-C: SINGLE-CRYSTAL HERIX EXPERIMENTS FOR SOUND VELOCITY MEASUREMENTS
- Tutorial: single crystal HERIX measurements.

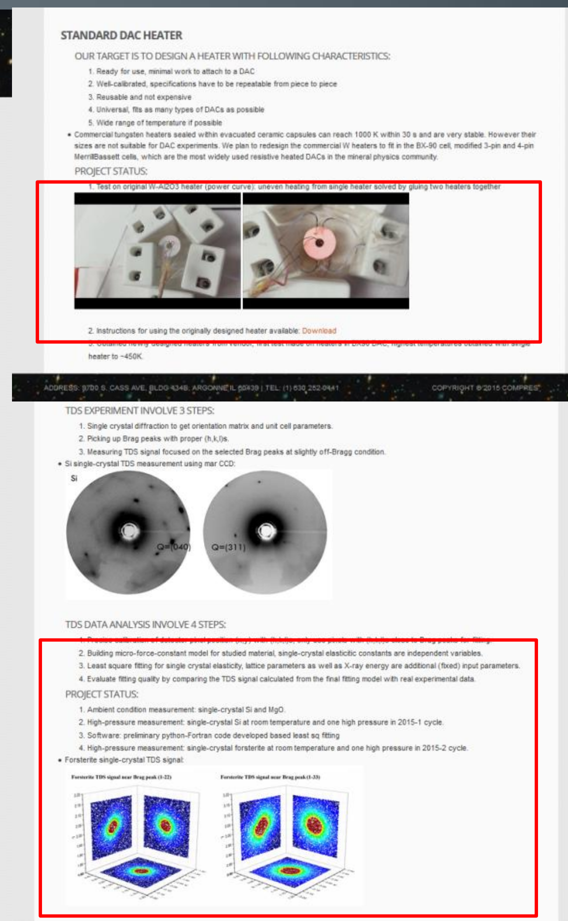
SECTOR 6 BM-B: A MULTI-ANVIL WHITE BEAM D-DIA FACILITY FOR DEFORMATION EXPERIMENTS
- Information on D-DIA at 6BM-B
- Contact: Haiyan Chen
  Email: haiyan@bnl.gov
- Beamline Scientist
  High Pressure / Multi-anvil Press
- Main Contact: Haiyan Chen
  Sector 6, Advanced Photon Source
  Building 432B, Argonne National Laboratory
  9700 South Cass Ave.
  Argonne, IL 60439

SECTOR 13 BM-C: A NEW FACILITY FOR HIGH-PRESSURE SINGLE-CRYSTAL AND POWDER DIFFRACTION EXPERIMENTS
- BMC experimental hutch (pictures from Dr. Dongzhou Zhang)
- Contact: Dongzhou Zhang
  Email: dzzhang@cars.uchicago.edu or dzhang@hawaii.edu
- Beamline Scientist, High Pressure / High Temperature Diamond Anvil Cell (DAC)
- Main Contact: GSECARS, Building 434A, Argonne National Laboratory, 9700 South Cass Ave., Argonne, IL 60439

GSECARS/COMPRES GAS LOADING SYSTEM
- Information on gas loading system
- Contact: Sergey Tkachev
  Email: tkachev@cars.uchicago.edu
- Beamline Scientist
  High Pressure / High Temperature Diamond Anvil Cell (DAC)
- Main Contact: GSECARS / COMPRES Gas Loading System
  GSECARS, University of Chicago
  Building 434A, Argonne National Laboratory
  9700 South Cass Ave.
  Argonne, IL 60439
  Phone : 630.252.0430
  Fax : 630.252.0436

LINKS

BEAMLINE FACILITIES
- GeoSoilEnviroCARS (APS Sector 13)
- HPCAT (APS Sector 16), High Pressure Collaborative Access Team



STANDARD DAC HEATER

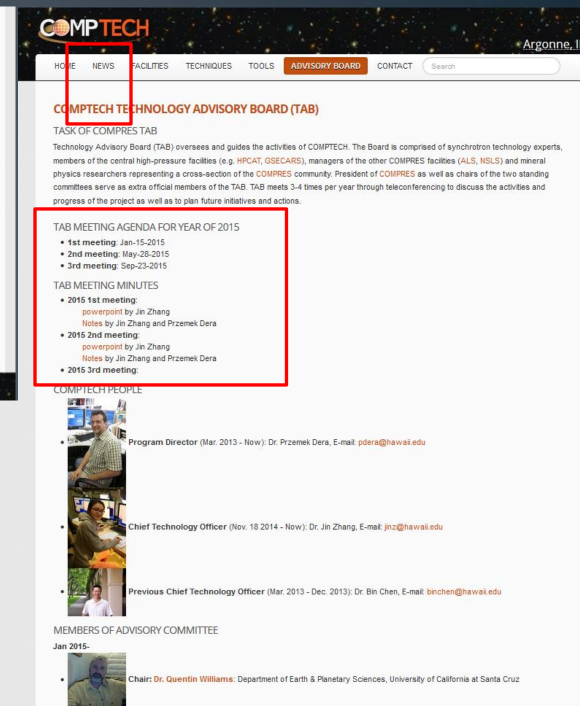
OUR TARGET IS TO DESIGN A HEATER WITH FOLLOWING CHARACTERISTICS:
1. Ready for use, minimal work to attach to a DAC
2. Well-calibrated, specifications have to be repeatable from piece to piece
3. Reusable and not expensive
4. Universal, fits as many types of DACs as possible
5. Wide range of temperature if possible

PROJECT STATUS:
2. Instructions for using the originally designed heater available: Download

TDS EXPERIMENT INVOLVE 3 STEPS:
1. Single crystal diffraction to get orientation matrix and unit cell parameters.
2. Picking up Brag peaks with proper (h,k,l)s.
3. Measuring TDS signal focused on the selected Brag peaks at slightly off-Bragg condition.
- Si single-crystal TDS measurement using mar CCD:

TDS DATA ANALYSIS INVOLVE 4 STEPS:
2. Building micro-force-constant model for studied material, single-crystal elasticitc constants are independent variables.
3. Least square fitting for single crystal elasticity, lattice parameters as well as X-ray energy are additional (fixed) input parameters.
4. Evaluate fitting quality by comparing the TDS signal calculated from the final fitting model with real experimental data.

PROJECT STATUS:
1. Ambient condition measurement: single-crystal Si and MgO.
2. High-pressure measurement: single-crystal Si at room temperature and one high pressure in 2015-1 cycle.
3. Software: preliminary python-Fortran code developed based least sq fitting
4. High-pressure measurement: single-crystal forsterite at room temperature and one high pressure in 2015-2 cycle.
- Forsterite single-crystal TDS signal:



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COMPTECH TECHNOLOGY ADVISORY BOARD (TAB)

TASK OF COMPRES TAB

Technology Advisory Board (TAB) oversees and guides the activities of COMPTECH. The Board is comprised of synchrotron technology experts, members of the central high-pressure facilities (e.g. HPCAT, GSECARS), managers of the other COMPRES facilities (ALS, NSLS) and mineral physics researchers representing a cross-section of the COMPRES community. President of COMPRES as well as chairs of the two standing committees serve as extra official members of the TAB. TAB meets 3-4 times per year through teleconferencing to discuss the activities and progress of the project as well as to plan future initiatives and actions.

TAB MEETING AGENDA FOR YEAR OF 2015
- 1st meeting: Jan-15-2015
- 2nd meeting: May-28-2015
- 3rd meeting: Sep-23-2015

TAB MEETING MINUTES
- 2015 1st meeting:
  powerpoint by Jin Zhang
  Notes by Jin Zhang and Przemek Dera
- 2015 2nd meeting:
  powerpoint by Jin Zhang
  Notes by Jin Zhang and Przemek Dera
- 2015 3rd meeting:

COMPTECH PEOPLE
- Program Director (Mar. 2013 - Now): Dr. Przemek Dera, E-mail: pdera@hawaii.edu
- Chief Technology Officer (Nov. 18 2014 - Now): Dr. Jin Zhang, E-mail: jinz@hawaii.edu
- Previous Chief Technology Officer (Mar. 2013 - Dec. 2013): Dr. Bin Chen, E-mail: binchen@hawaii.edu

MEMBERS OF ADVISORY COMMITTEE

Jan 2015-
- Chair: Dr. Quentin Williams: Department of Earth & Planetary Sciences, University of California at Santa Cruz

Thanks! Questions?