

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 ($\sim \$10/\text{pc}$ comparing with $> \$200/\text{pc}$)
- **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 PX² commissioning

Ambient **TDS** measurement: sample suitable for DAC, Si (15μm thick)

2015.1

design of the first version universal **membrane** cap

1st test on W-Al₂O₃ **heaters**

Website major revision: Tools

2015.2

Preparation for **TDS** beam time

received the 1st set of modified W-Al₂O₃ **heaters**

Website rearrange

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

Preparation for **TDS** beam time

Received the 1st version of universal **membrane** cap

Website revision: Techniques

2015.7

1st HP **TDS** on foresterite at **13IDC**

1st HP **TDS** on foresterite at **34ID**

Membrane tubing installed in 13BMC

2015.8

HP **TDS** measurement on **foresterite** up to **40GPa** at 13BMC

1st W-Al₂O₃ **heater** test in DAC

2015.9

Testing new inversion algorithm for **TDS** data analysis

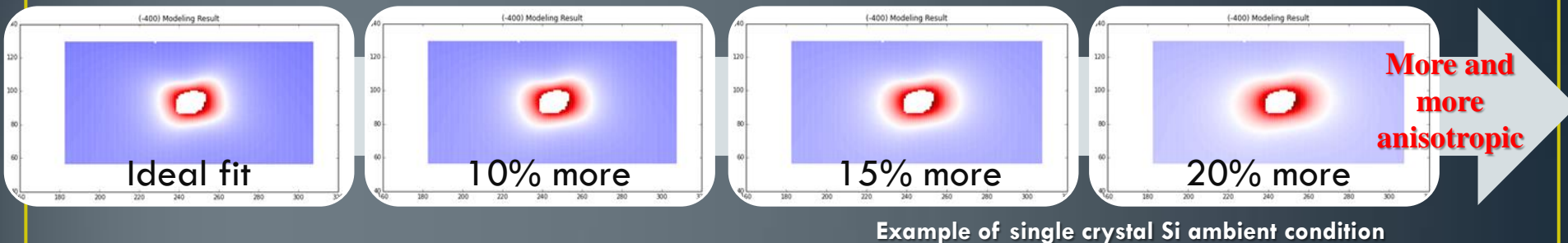
1st **heater** user: U of Arizona

Budget approved for adding optical system for **34ID**

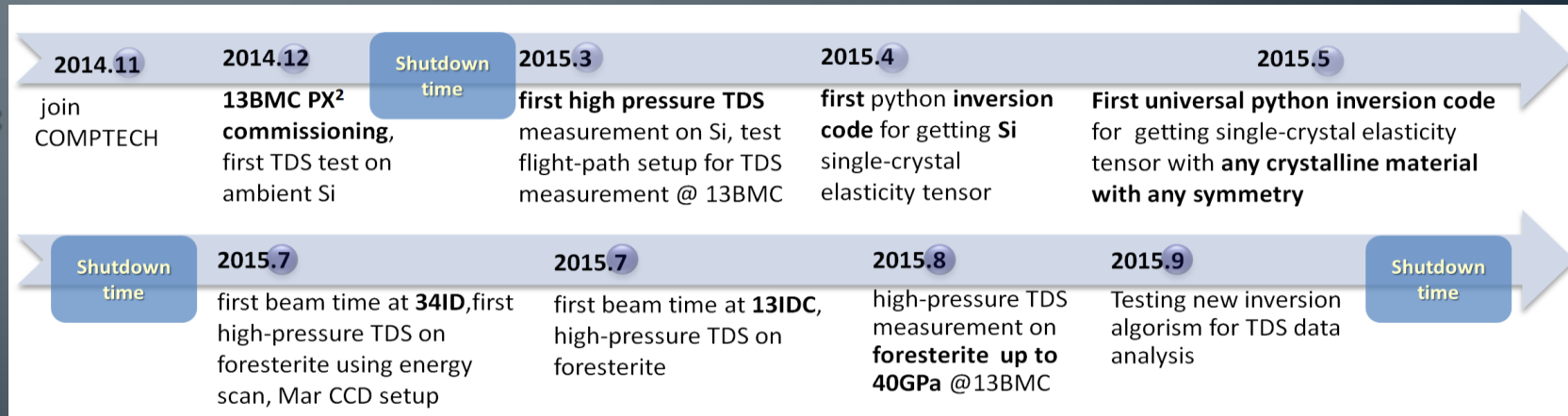
X-ray thermal diffuse scattering (TDS)

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:



Project timeline:



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)

- Current Status:

- Sample testing:

- Ambient condition: Si (100) (111) and **forsterite** (111)
 - High-pressure: Si (100) (1GPa); **forsterite** (111) (**~40GPa**)

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

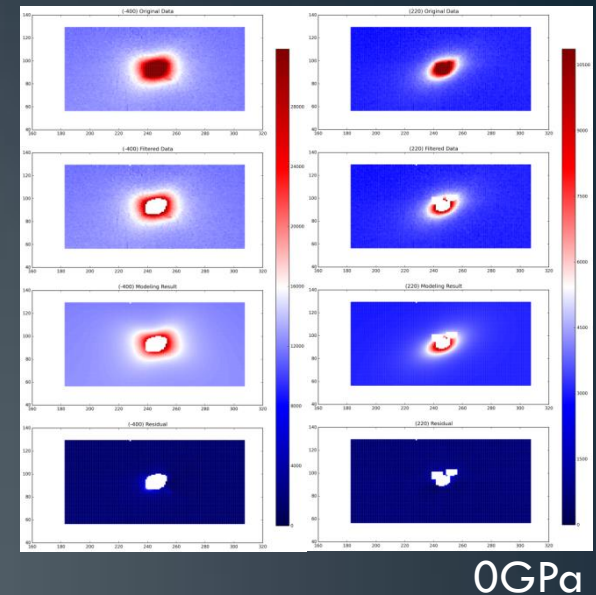
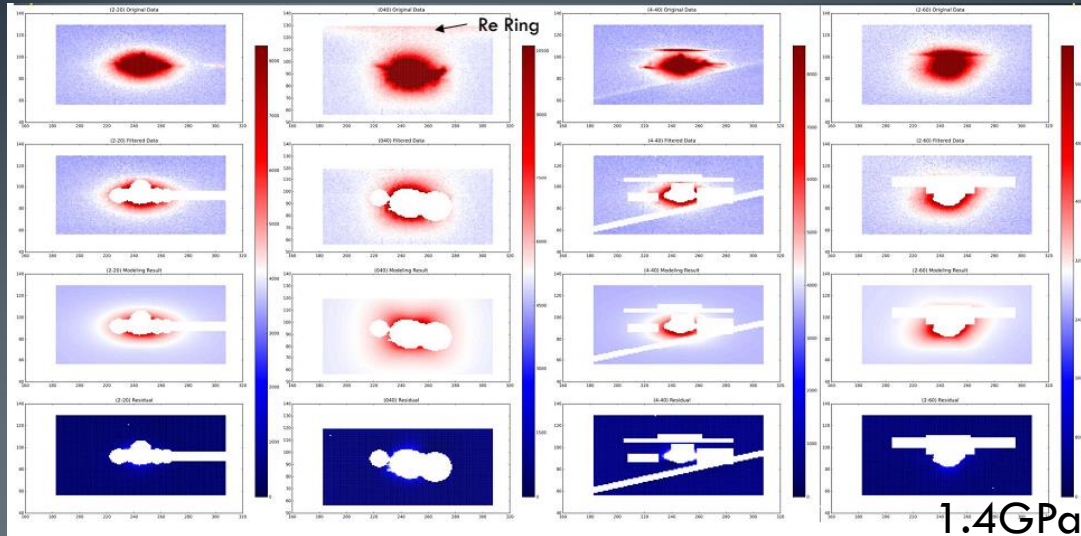
- Experimental Setup:

- Using flight path decrease background (originally designed for surface scattering experiment) **13BMC**
13IDC (beam size 30μm)
 - **Energy scan** to find Bragg 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)

- $C_{11}=156.6$; $C_{12}=61.2$; $C_{44}=74.6$ @0GPa
- $C_{11}=163.3$; $C_{12}=67.9$; $C_{44}=75.5$ @1.4GPa

PETRA III Germany

- Decremps et al. (PRB 2010) Using phonon imaging up to 7.9GPa

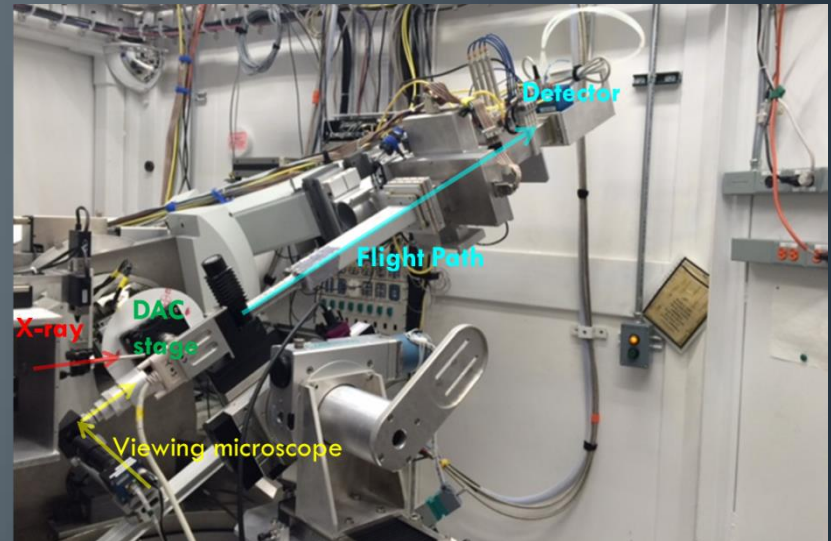
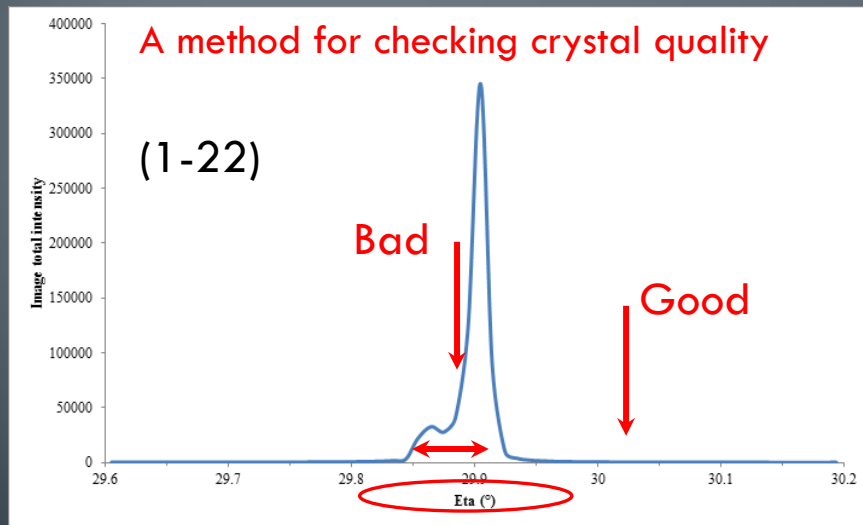
- $C_{11}=165.7$; $C_{12}=63.6$; $C_{44}=80.0$ @0GPa
- $C_{11}=172.1$; $C_{12}=71.5$; $C_{44}=80.0$ @1.4GPa

- This study: TDS

- $C_{11}=166.0(\text{fixed})$; $C_{12}=64.3(\pm 1.6)$; $C_{44}=79.1(\pm 0.8)$ @0GPa
- $C_{11}=172.0(\text{fixed})$; $C_{12}=66.3(\pm 3.1)$; $C_{44}=84.0(\pm 2.2)$ @1.4GPa

X-ray thermal diffuse scattering (TDS)

- Sample testing:
 - **forsterite (111) low symmetry (orthorhombic)** up to $\sim 40\text{GPa}$ @ 13BMC and IDC
 - **Geo-materials: silicate** **He pressure medium VS Ne pressure medium**



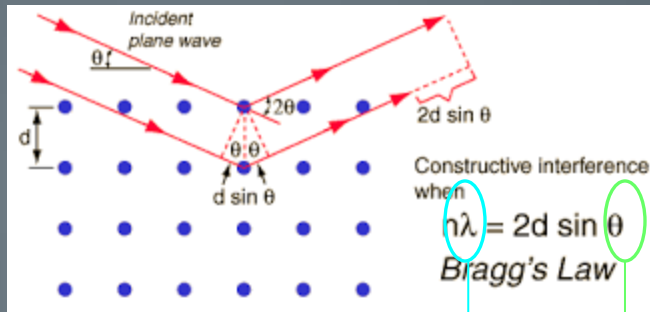
For olivine: ~ 0.1 up to 25GPa $< 0.3^\circ$ up to 32GPa $< 0.5^\circ$ up to 40GPa

- **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)

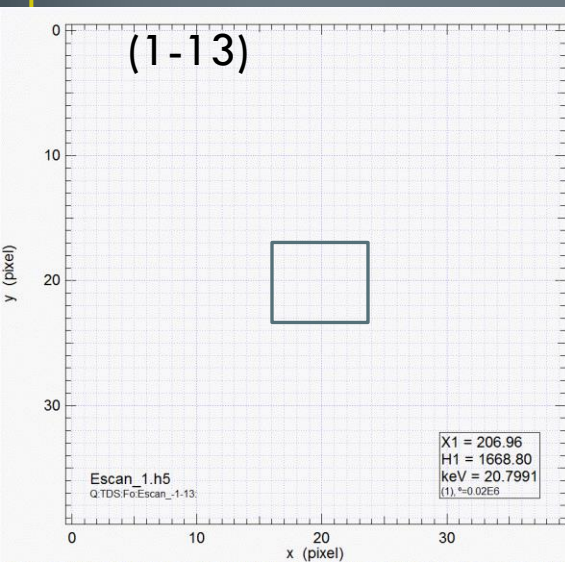
- Sample testing:
 - Energy scan approach at sector 34



Beam size < 500 nm

“Bad quality” crystal could be “good”

No rotation – “mechanical uncertainty” small



34ID

13IDC and BMC

Change X-ray energy:

Off diffraction condition

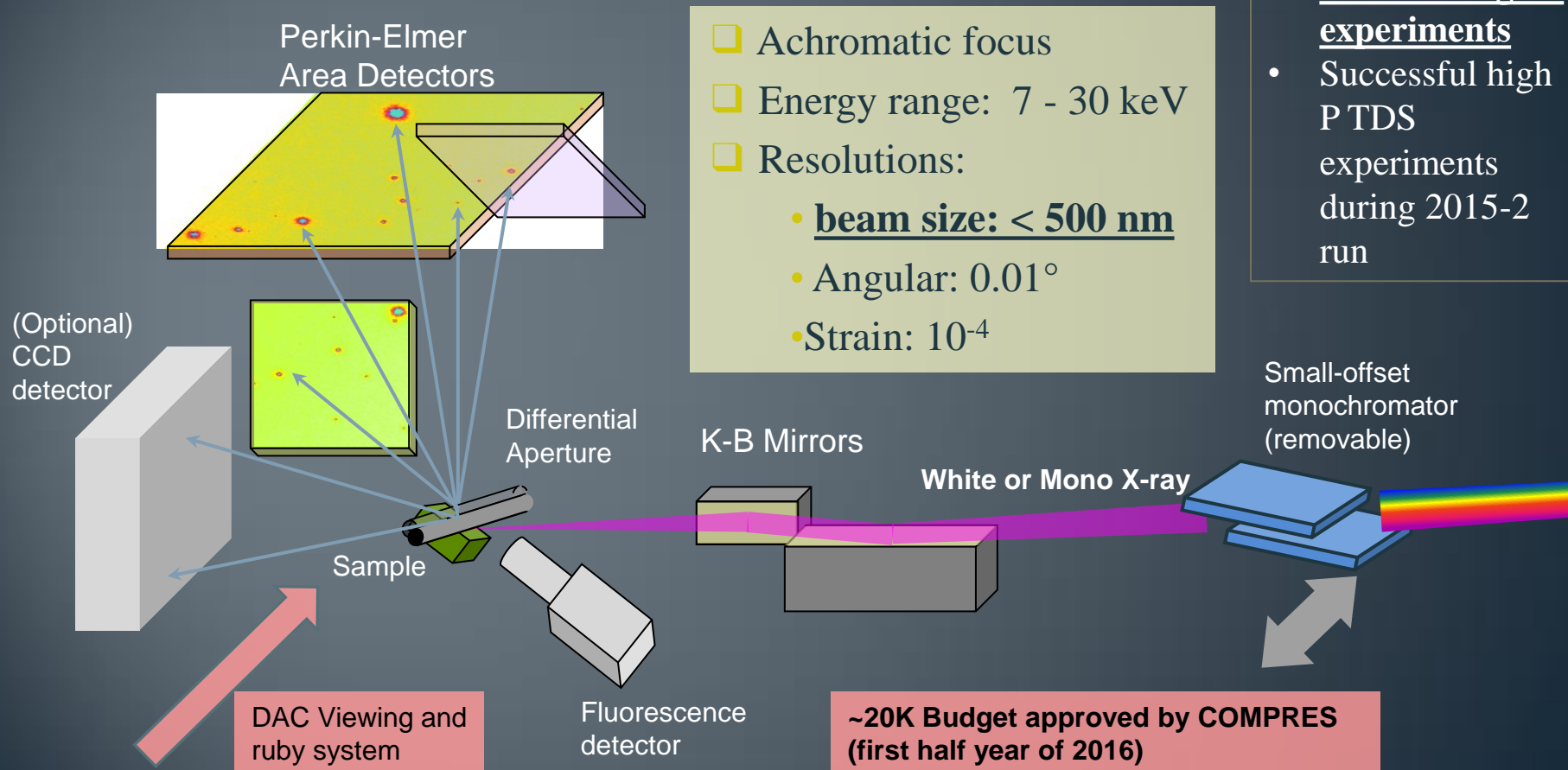
Diffraction condition

Off diffraction condition



Preferred access to existing beamlines

Sector 34 ID



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

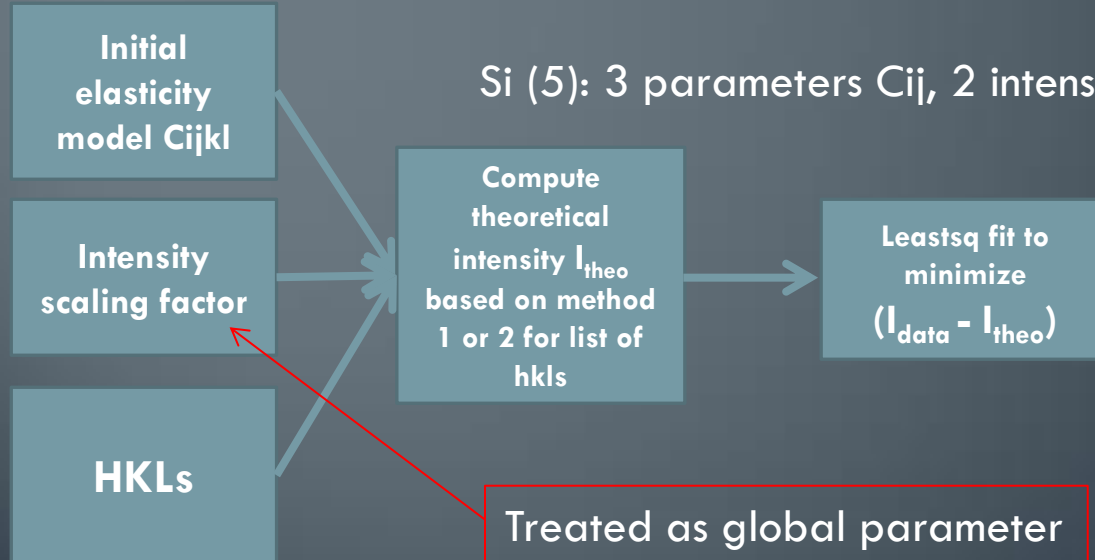
In 2015-2

Method 1: single crystal elasticity model

Method 2: Born-Von-Karman model

1 model for all, only based on C_{ij} s and hkl's.
Change C_{ij} s for different materials.

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



Si (5): 3 parameters C_{ij} , 2 intensity scaling factors for 2 images

Forsterite (28):

8 parameters for C_{ij}
18 intensity scaling factors for 18 TDS images



X-ray thermal diffuse scattering (TDS)

- Inversion code development

Only consider 1st order approximation – single-phonon scattering

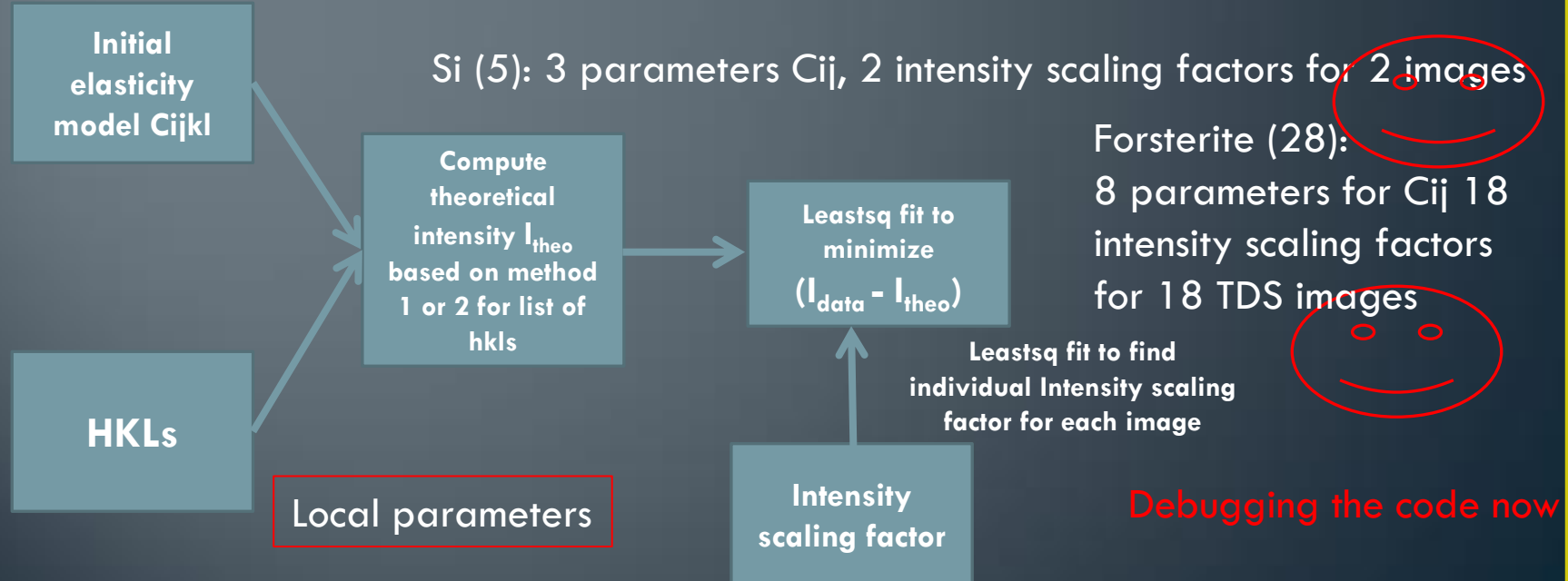
In 2015-2

Method 1: single crystal elasticity model

Method 2: Born-Von-Karman model

1 model for all, only based on C_{ij} s and hkl's.
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Micro force constant model for each pair of neighboring atoms – 1 material 1 model

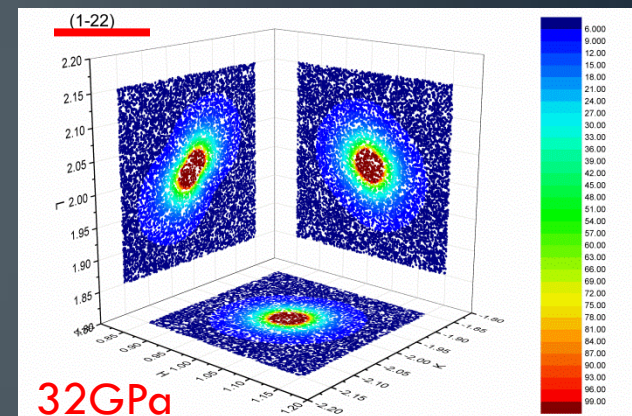
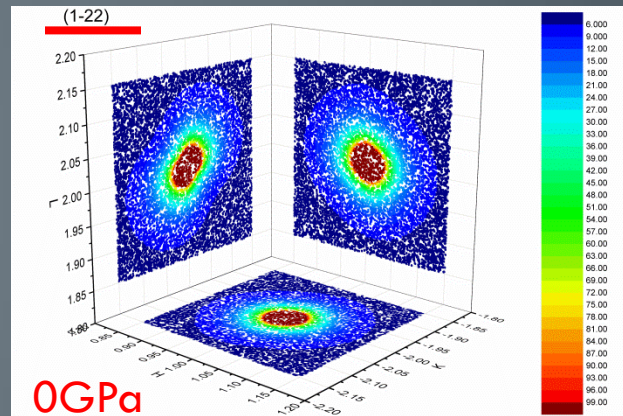


X-ray thermal diffuse scattering (TDS)

- **TDS simulation**

- Given elasticity model and HKL simulate the TDS signal

- Example of Olivine



Future work in last TAB meeting:

✓ **Reduce orientation Error:** flight path setup – only **two** bragg peaks are used for orientation determination

~✓ Standard measurement: **bench mark** for high pressure TDS measurement

Software interface: work with professional software engineers

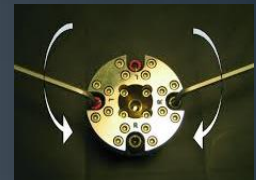
Working with theoreticians: under situation where trade-offs are large (e.g. low symmetry materials), tds only might not yield to a single solution, but will provide extra constrain to theoretical calculation.

High temperature effect

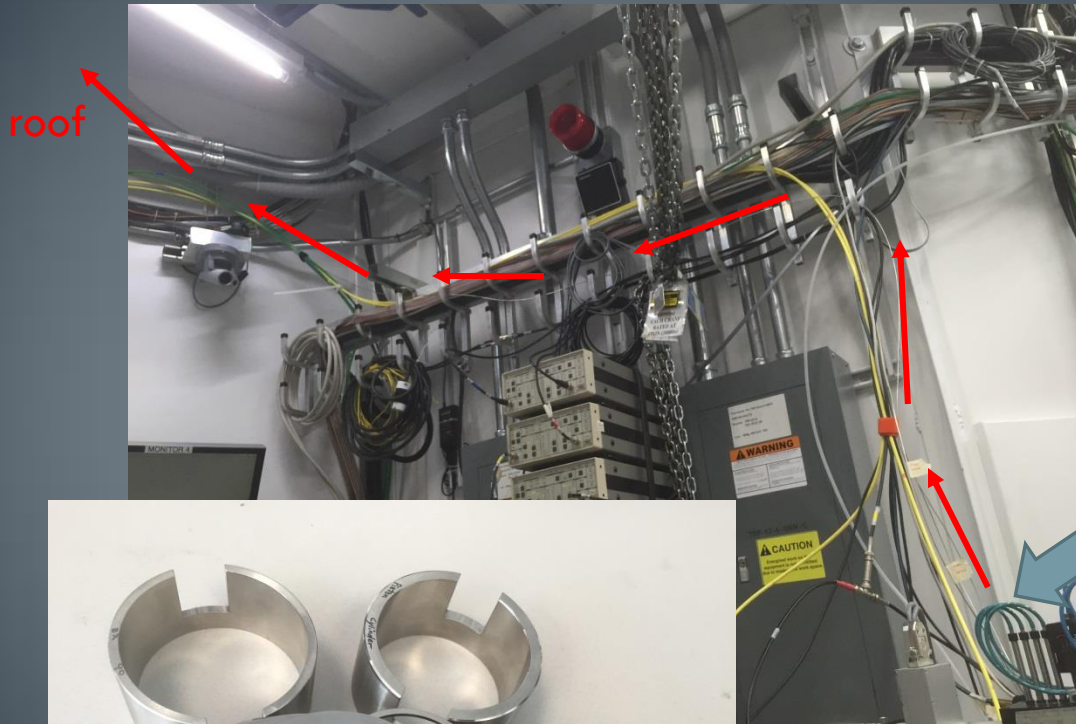
More exciting materials (seeking collaborations and invite users)

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 most DACs in different sizes: different spacers
 - **No lose in DAC opening**: ideal for single crystal studies
- **Project timeline:**
 - **Designed** 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
 - GE digital pressure **controller** ordered 08/2015
 - Gas **membranes** sent back to machine shop for **revision** 09/2015
- Expect:
 - Hopefully will get the membrane and pressure controller by 11/2015
 - Welcome COMPRES users to use!



Universal Membrane cap for most DACs



Membrane Holder

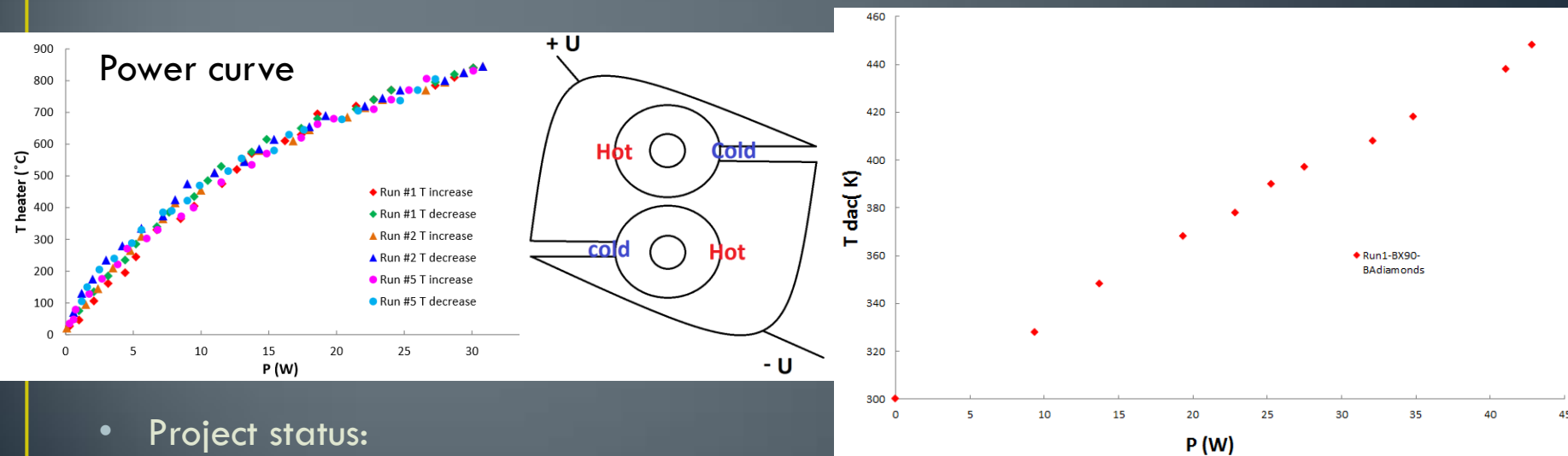
Membrane tubing in 13BMC
Installed
(with help of Mike Proskey)

Waiting:

1. Membrane controller
(ship out 09/23 today!)
2. Membranes

Standard “cheap” resistive heater for DAC

- Commercial W-Al₂O₃ 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-Al₂O₃ 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)

Website

<http://comptech.compres.us/>

COMPTECH Argonne, IL

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FACILITIES

ADVANCED PHOTON SOURCE, ARGONNE NATIONAL LABORATORY

SECTOR 3 ID-C: SINGLE-CRYSTAL X-RAY EXPERIMENTS FOR SOUND VELOCITY MEASUREMENTS


- Tutorial: single crystal XRD 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@anl.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


- Information on 13 BM-C
- BMC experimental hutches (pictures from Dr. Dongzhou Zhang)



- Contact: Dongzhou Zhang
Email: ddzhang@cars.uchicago.edu or dczhang@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)
- HP-CAT (APS Sector 16), High Pressure Collaborative Access Team
- APS Sector 3, Inelastic X-ray Nuclear Resonant Scattering Group

STANDARD DAC HEATER


OUR TARGET IS TO DESIGN A HEATER WITH FOLLOWING CHARACTERISTICS:

- Ready for use, minimal work to attach to a DAC.
- Well-calibrated, specifications have to be repeatable from piece to piece
- Reusable and not expensive
- Universal, fits as many types of DACs as possible
- Wide range of temperature if possible

- Commercial tungsten heaters sealed within evacuated ceramic capsules can reach 1000 K within 30 s and are very stable. However their sizes are not suitable for DAC experiments. We plan to redesign the commercial W heaters to fit in the BX-90 cell, modified 3-pin and 4-pin Merrill-Bassett cells, which are the most widely used resistive heated DACs in the mineral physics community.

PROJECT STATUS:

- Test of original W heater (lower curves), which heating from single heater turned by using two heaters together



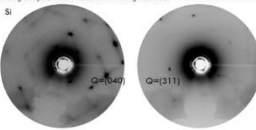
- Instructions for using the originally designed heater available: [Download](#)

Address: 9700 S. CASS AVE., BUILDING 434A, ARGONNE, IL 60439 TEL: (630) 252-0441 COPYRIGHT © 2015 COMPTECH

TDS EXPERIMENT INVOLVE 3 STEPS:

- Single crystal diffraction to get orientation matrix and unit cell parameters.
- Picking up Bragg peaks with proper h,k,l ns.
- Measuring TDS signal focused on the selected Bragg peaks at slightly off-Bragg condition.

- Si single-crystal TDS measurement using mar CCD:



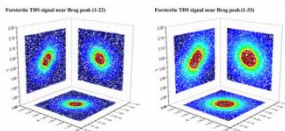
TDS DATA ANALYSIS INVOLVE 4 STEPS:

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

PROJECT STATUS:

- Ambient condition measurement: single-crystal Si and MgO.
- High-pressure measurement: single-crystal Si at room temperature and one high pressure in 2015-1 cycle.
- Software: preliminary python-Fortran code developed based least sq fitting
- 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. HP-CAT, 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:
powered by Jin Zhang
Notes by Jin Zhang and Przemek Dera
- 2015 2nd meeting:
powered 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: jnz@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?