COMPTECH TAB meeting 2015-3

Jin Zhang
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

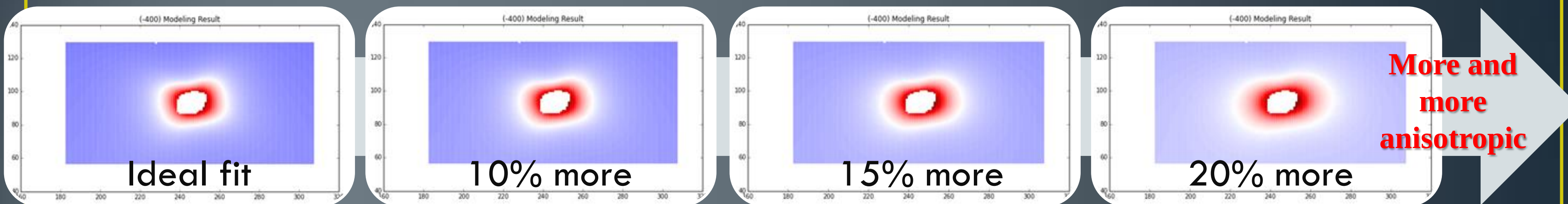
Time line of COMPTECH

- **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** — 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** 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)

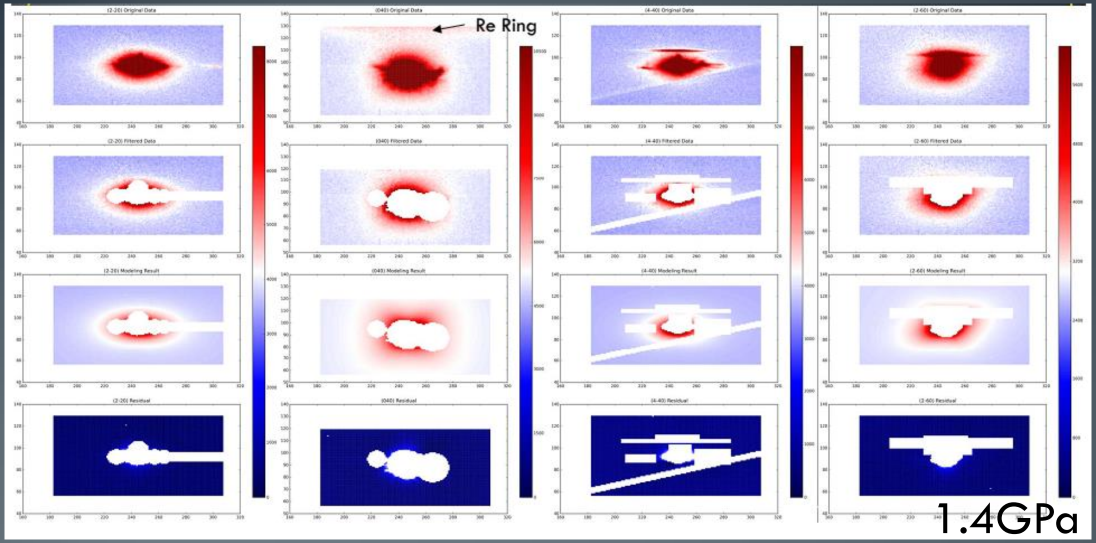
New technique: starting 2017Spring (survey in the workshop: XANES XAFS Raman?)

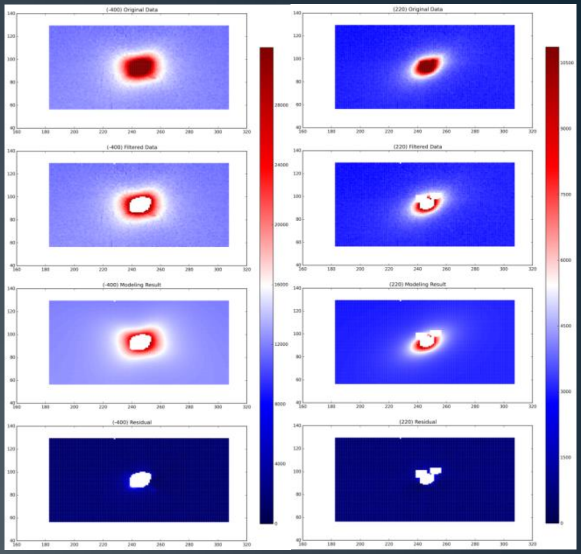
X-ray thermal diffuse scattering (TDS)

- 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**

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

- Si





0GPa

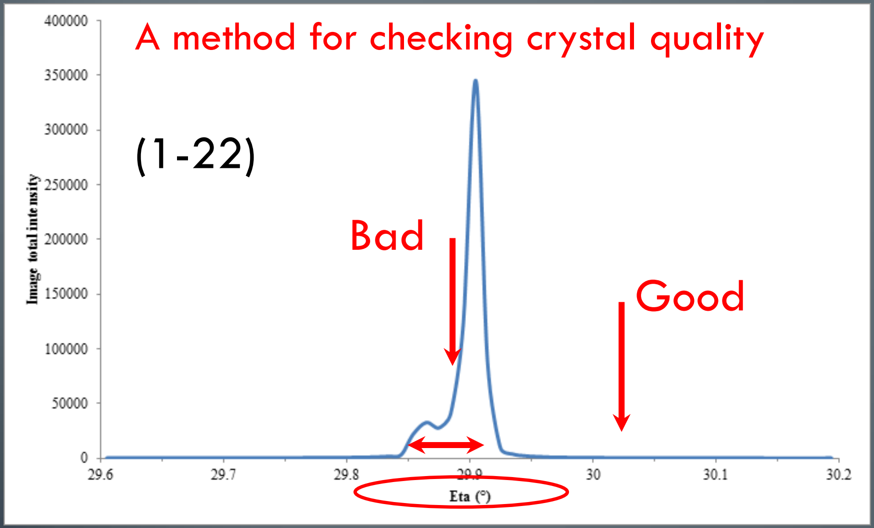
- **B. Winkler (U of Frankfurt, 1st principle calculation) (meet during COMPRES 2015 annual meeting)**
  - **C11=156.6; C12=61.2; C44=74.6 @0GPa**
  - **C11=163.3; C12=67.9; C44=75.5 @1.4GPa**

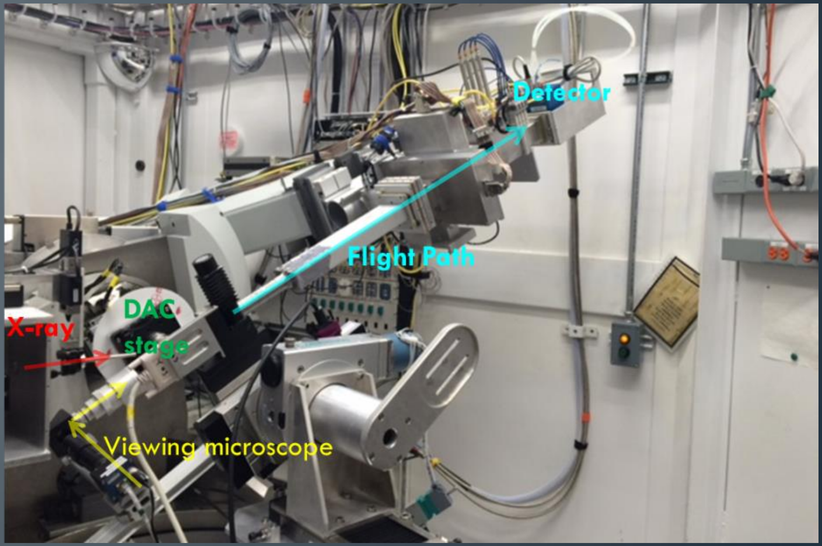
PETRA III Germany

- **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**
- **This study: TDS**
  - **C11=166.0(fixed); C12=64.3(±1.6); C44=79.1(±0.8) @0GPa**
  - **C11=172.0(fixed); C12=66.3 (±3.1); C44=84.0(±2.2) @1.4GPa**

X-ray thermal diffuse scattering (TDS)

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





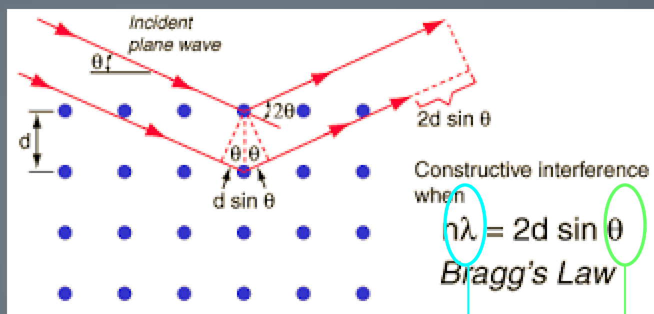
For olivine: ~0.1 up to 25GPa <0.3 ° up to 32GPa <0.5 ° up to 40GPa

- **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)

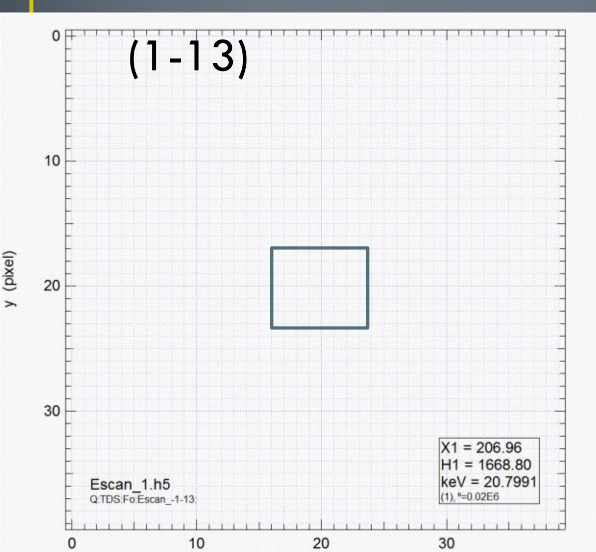
- Sample testing:
    - **Energy scan approach at sector 34**



Beam size <500 nm
"Bad quality" crystal could be "good"

No rotation – "mechanical uncertainty" small

34ID | 13IDC and BMC



Change **X-ray energy**:

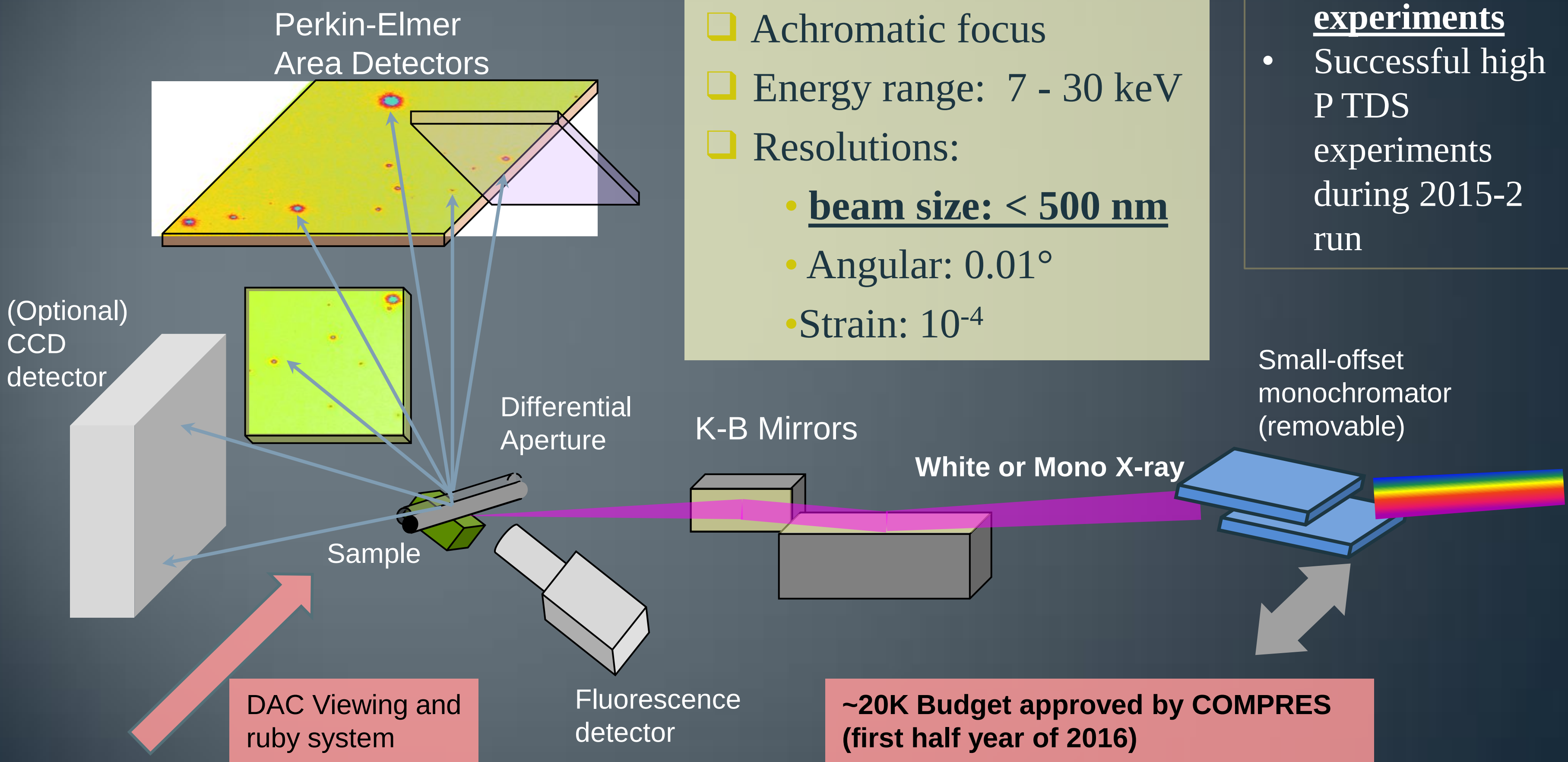
Off diffraction condition

Diffraction condition

Off diffraction condition

Preferred access to existing beamlines

Sector 34 ID



- 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

~20K 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

Forsterite (28):
8 parameters for Cij
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 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

HKLs

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

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

Leastsq fit to find individual Intensity scaling factor for each image

Intensity scaling factor

Local parameters

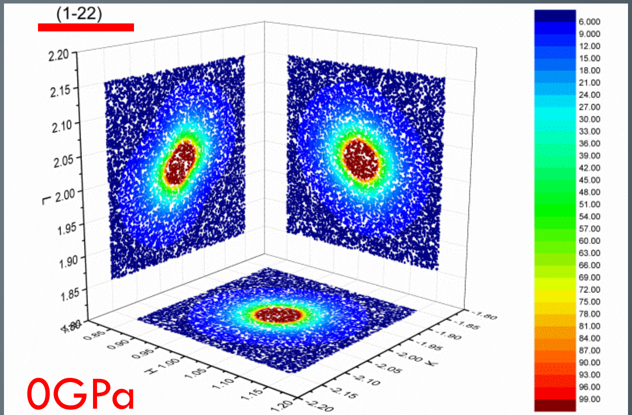
Si (5): 3 parameters Cij, 2 intensity scaling factors for 2 images

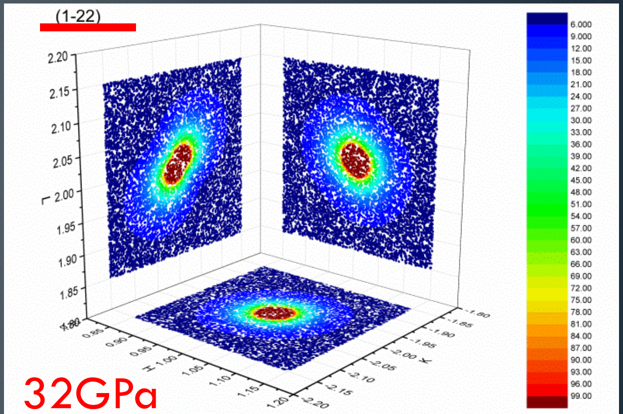
Forsterite (28):
8 parameters for Cij 18 intensity scaling factors for 18 TDS images

Debugging the code now

X-ray thermal diffuse scattering (TDS)

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





Future work in last TAB meeting:

√ **Reduce orientation Error**: 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.

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:**
  - **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
  - 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 tubing in 13BMC
Installed
(with help of Mike Proskey)

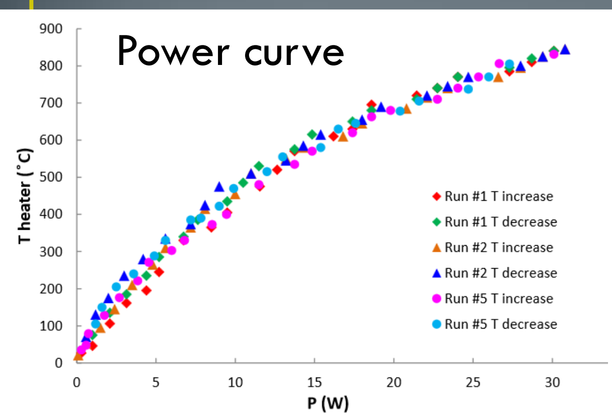
Membrane Holder

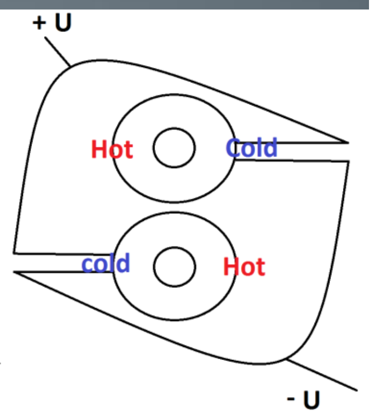
Waiting:

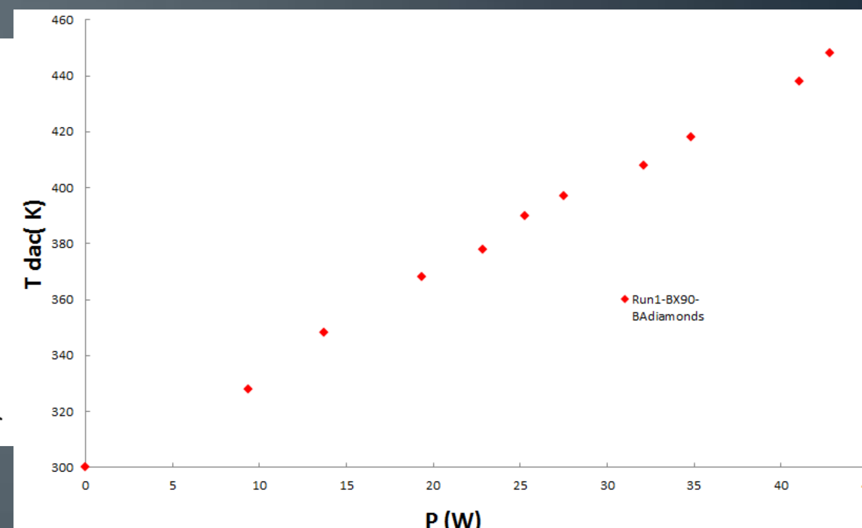
1. Membrane controller
   (ship out 09/23 today!)
2. Membranes

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)



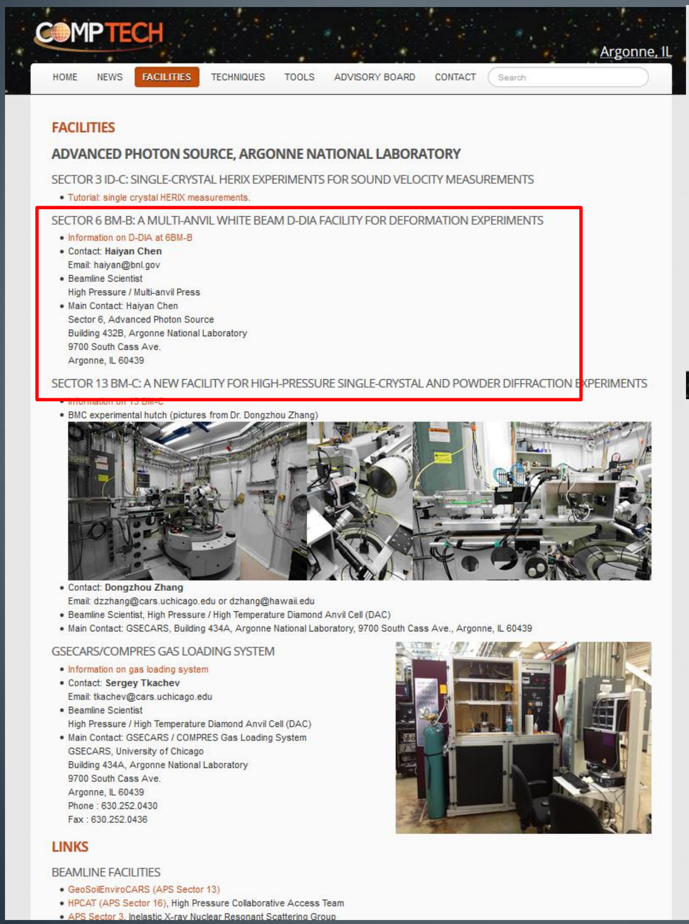


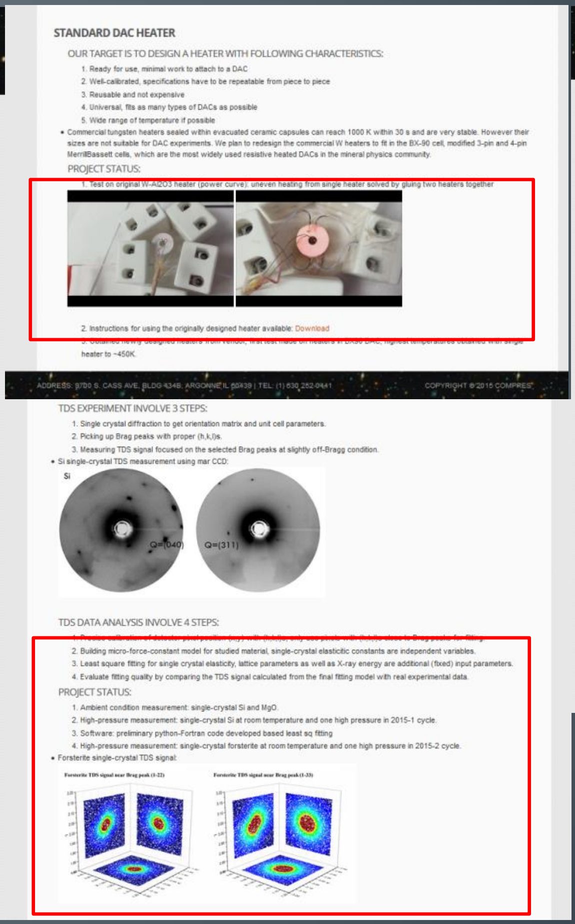


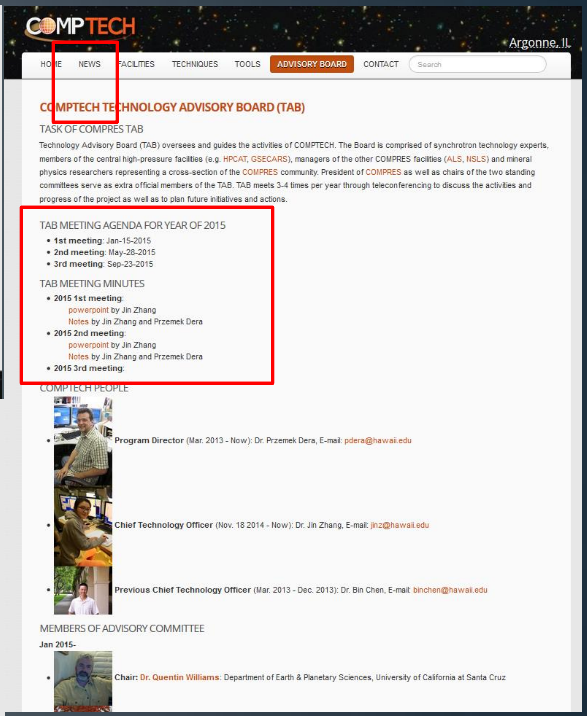
- Project status:
  - 1st test with DAC: with **single** heater – sample chamber reached highest T 450K
  - 1st supply to COMPRES users: (Dan Shim and Kurt Leinenweber)

Website

http://comptech.compres.us/







Thanks! Questions?