COMPTECH 2015 Annual Plan

Jin Zhang
COMPTECH Officer @ APS, ANL

01/15/2015 Argonne, IL

Proposed Projects

- X-ray Thermal Diffuse Scattering (On going now)
- Multi-grain X-ray diffraction (initiated since 2013)
- Universal Membrane cap for most DACs (On going now)
- Standard “cheap” resistive heater for DAC (initiated since 2013)

X-ray thermal diffuse scattering (TDS)

- X-ray thermal diffuse scattering (TDS) is caused by lattice thermal vibrations (phonons). In contrast to sharp Bragg diffraction peaks, TDS is diffusely distributed, due to the continuous distribution of phonon modes in the reciprocal space.
- Historically it has been used for determine phonon dispersion relation in crystals. In 1960s TDS experiments were performed using laboratory X-ray sources and on crystals of several cubic millimeters in size. Data collection is slow.
- With 3rd generation undulator X-ray source, TDS experiments for samples were carried out successfully at ambient conditions. Serious efforts to implement TDS analysis at high pressure are currently under way at PhotonFactory and ESRF (Ohtsu et al. 2008; Wehinger et al. 2014), yet in USA, besides the single study by Ding et al. in 2006 on **vanadium** , we are not aware of any attempts in this direction.

X-ray thermal diffuse scattering (TDS)

- The advantages of using TDS for measuring sound velocities of Earth materials include but are not limited to:
  - 1. TDS can be applied for work with diamond anvil cell (DAC) apparatus easily.
  - 2. TDS is highly suitable for measurements on opaque materials.
  - 3. Single-crystal elastic properties can be determined through TDS.
  - 4. The method easily applies to lower symmetry materials.
  - 5. The method does not require special sample preparation (polishing), is not sensitive to surface quality, and is much less sensitive to sample orientation than e.g. Brillouin.
  - 6. Data acquisition is fast with 3rd generation undulator X-ray source (e.g. APS).
  - 7. Experimental setup is essentially identical to what is used for routine X-ray diffraction experiments.

X-ray thermal diffuse scattering (TDS)

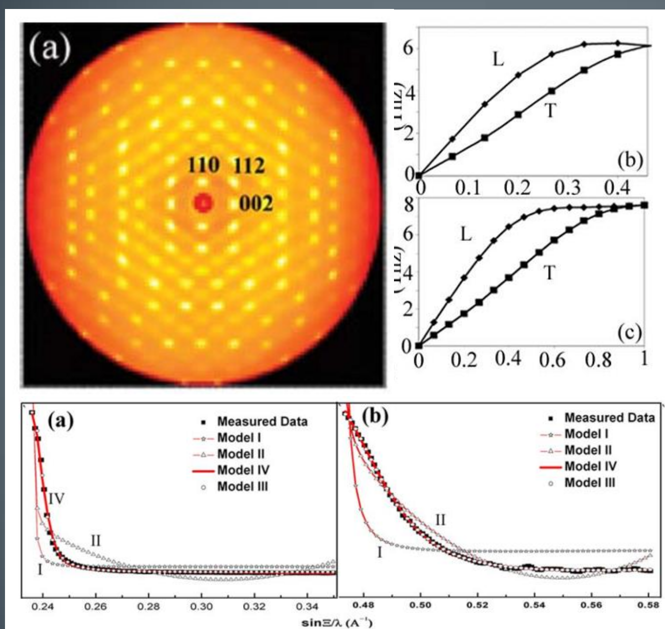
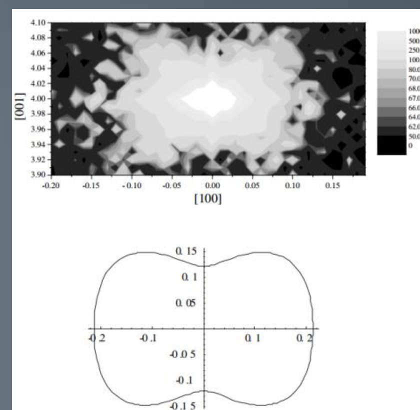
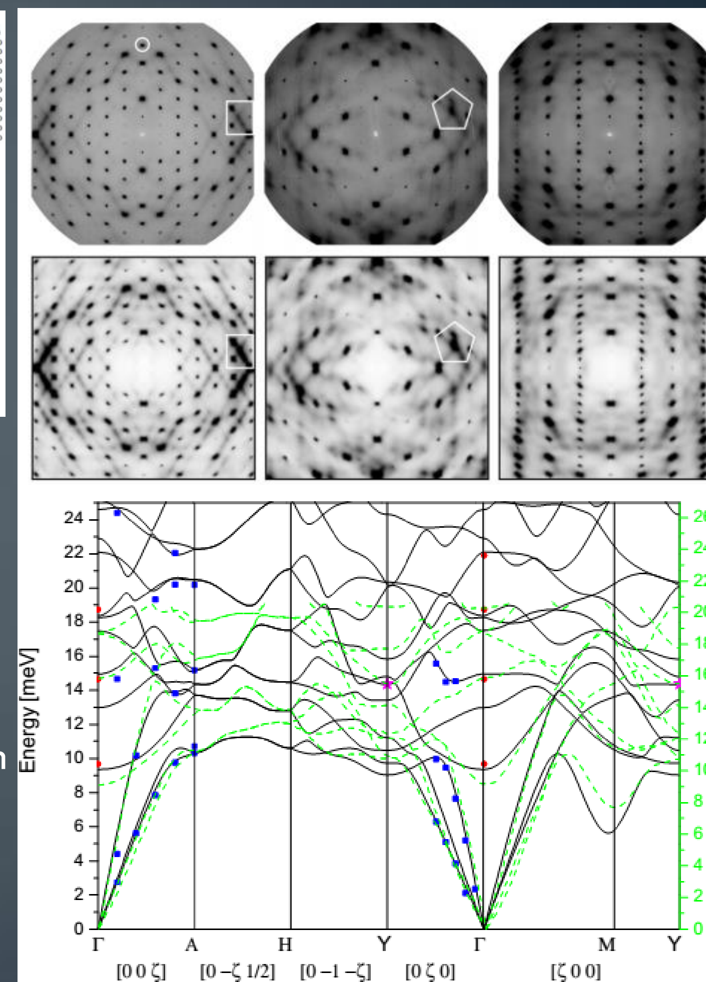


FIG. 2. (a) Fits of measured intensity of vanadium from sin θ/λ =0.22 (Å^{-1}) to 0.36 (Å^{-1}) along [110] with four models. (b) The fits of measured intensity of vanadium from sin θ/λ=0.46 (Å^{-1}) to 0.58 (Å^{-1}) along [110] with four models.

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



Ohtsu et al. 2008 CdTe
Phase transition at 3.79 GPa



Wehinger et al. 2013 Coesite
Phonon dispersion

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)

1st order – obtain ratios between Cijs

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

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

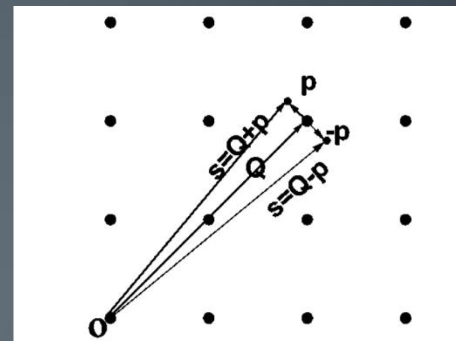


Fig. 1. Schematic diagram showing the relation between diffraction vector **s**, the reciprocal lattice vector **Q**, and lattice wave vector **p**: **s** = **Q** ± **p**.

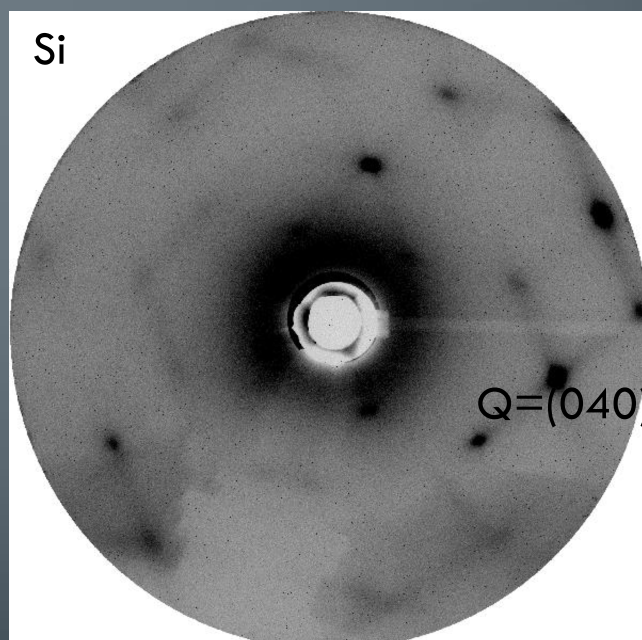
Wang et al. 2004

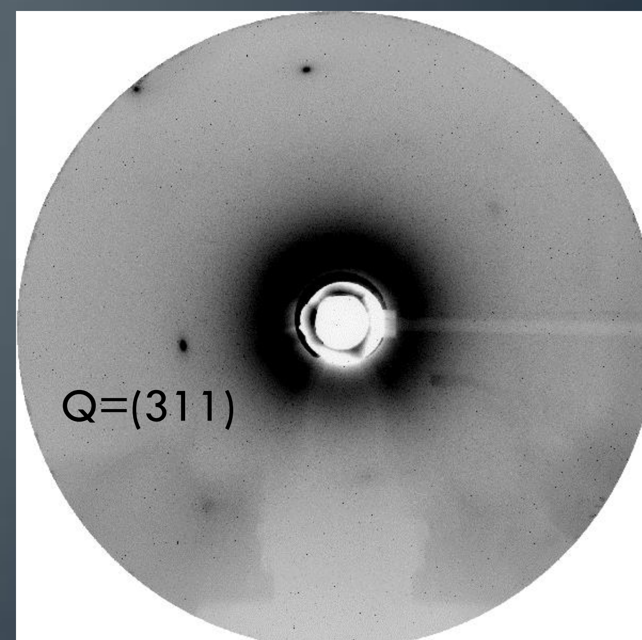
Vc is the volume of an unit cell, F(Q) is the structure factor of the unit cell and $e^{-2W(Q)}$ is the Debye–Waller factor.

Our primative test in December 2014

1. Good data on 15um thick Si
2. Time in BM
3. Signal intensity is strongly **Q** dependent

Pre-knowledge of Cij





X-ray thermal diffuse scattering (TDS)

- We devised a plan in which preliminary tests and methodological developments will be carried out at PX^2, while in the future, we will seek a PUP partnership with APS Sector 34 where undulator source and sub-micrometer focusing are available. In December we initiated a collaboration with sector 34 beamline scientist Dr. Ruqing Xu, an expert on TDS analysis at ambient pressure. First testing experiments were conducted at 13BMC.

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

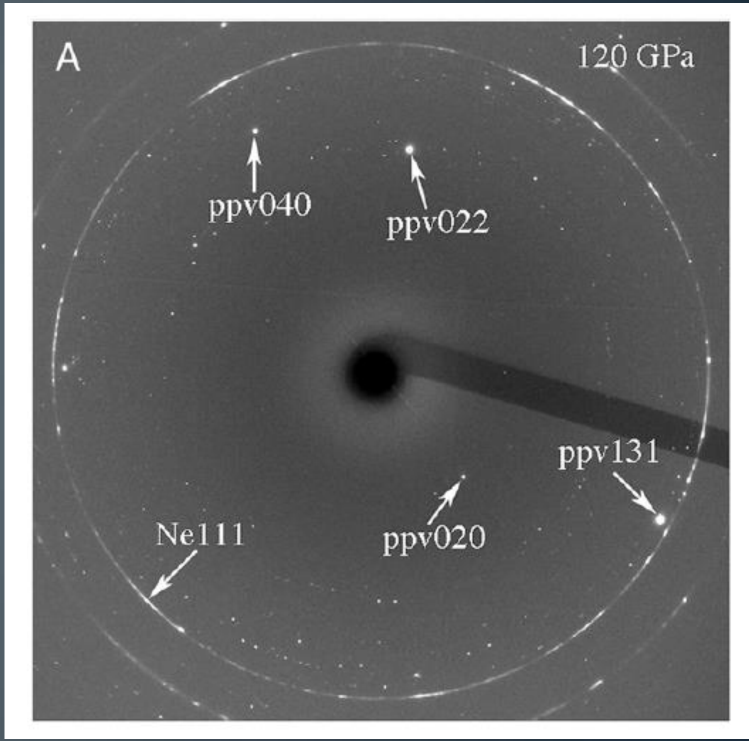
Well-defined samples: Si, MgO etc. → Any materials

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.

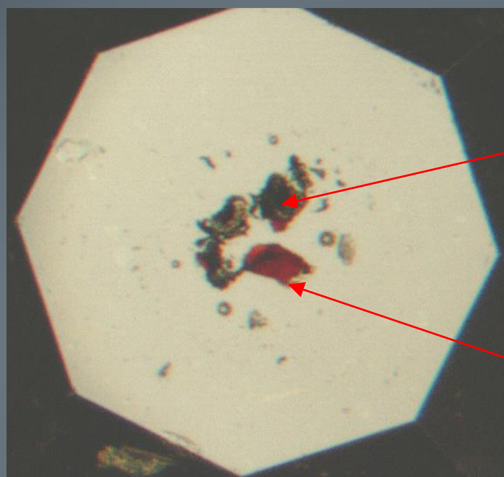
- There has been a number of recent very exciting high-pressure discoveries that resulted from laser heating experiments and produced coarse powder samples of new unquenchable phases, e.g. Fe_4O_5 (Lavina et al. 2009, PNAS), ppv (Zhang et al. 2013, PNAS), H-phase (Zhang et al. 2014, Science 2014), carbonates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

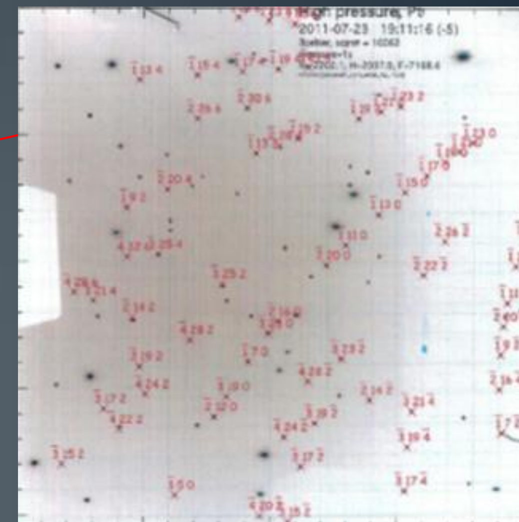
- The same approach has also been applied to study lattice preferred orientation development in rheological experiments is DACs (NISR et al. 2014, HPR).

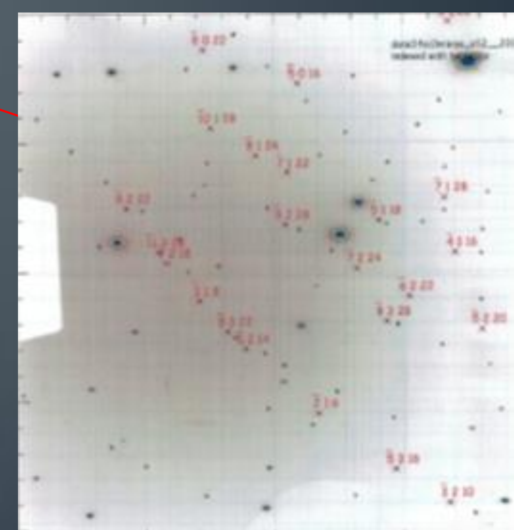


Zhang et al. 2013, PNAS. Data from 16IDB

Multi-grain X-ray diffraction







Typical samples of new phases obtained by laser heating of single crystals contain multiple homogeneous single crystal grains on a micrometer scale (e.g. above laser heated Fe_2O_3), but there is often a mixture of phases present (e.g. untransformed starting material).

Structure and properties (e.g. oxidation state) of these grains can be evaluated using multigrain diffraction (monochromatic or Laue).

Dera et al. in prep. Laue diffraction data from 34IDD

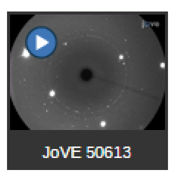
Multi-grain X-ray diffraction

- 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**

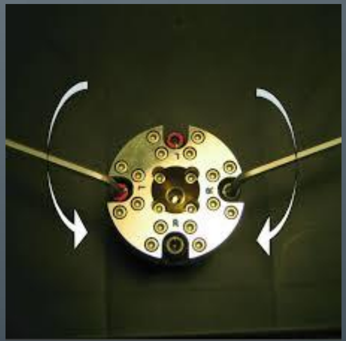


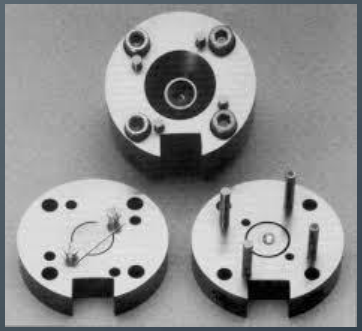
The laser heated diamond anvil cell combined with synchrotron micro-diffraction techniques allows researchers to explore the nature and properties of new phases of matter at extreme pressure and temperature (PT) conditions. Heterogeneous samples can be characterized *in situ* under high pressure by 2D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

Universal Membrane cap for most DACs

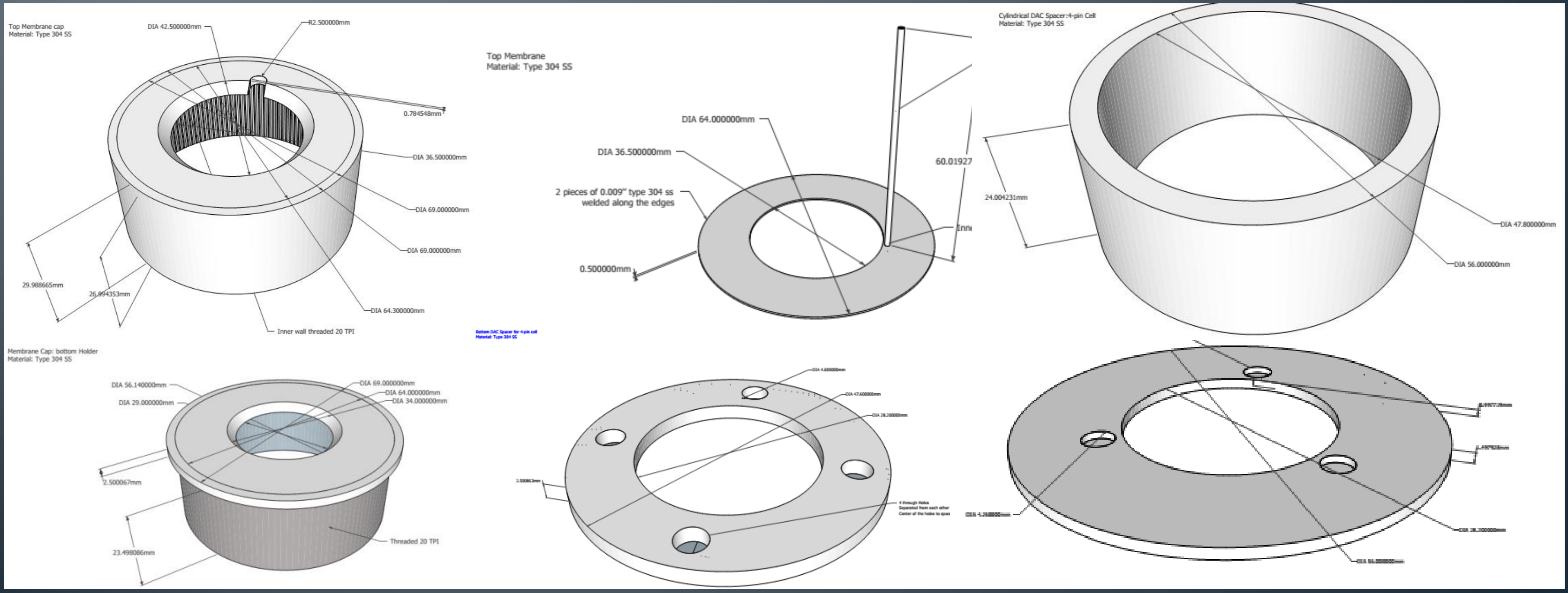
- Advantage of Membrane cap – convert screw-driven DAC into membrane-driven DAC
- Remote precise pressure control
- Currently available membrane caps
  - Too many DACs in different sizes – one cap for each type
  - Limitations to the DAC opening – IXS, Brillouin, single x-stal XRD
  - Most are good only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

- Our strategy
  - Single membrane cap
  - Double membrane cap
  - Online resource: extensive test with perspective users
  - Order available for all COMPRES users through UH machine shop



Standard "cheap" resistive heater for DAC

Limitations of conventional Pt heaters

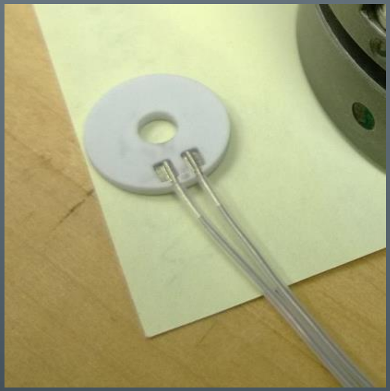
- 1. Lots of work to wrap up a Pt heater
- 2. specifications different from piece to piece
- 3. In general not reusable, very expensive (~$250/pc or even more)
- 4. Temperature limited to 800K-1000K

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, and from time to time: estimation of T on power curve
- 3. Reusable and not expensive
- 4. Universal, fits as many types of DACs as possible
- 5. Wide range of temperature if possible

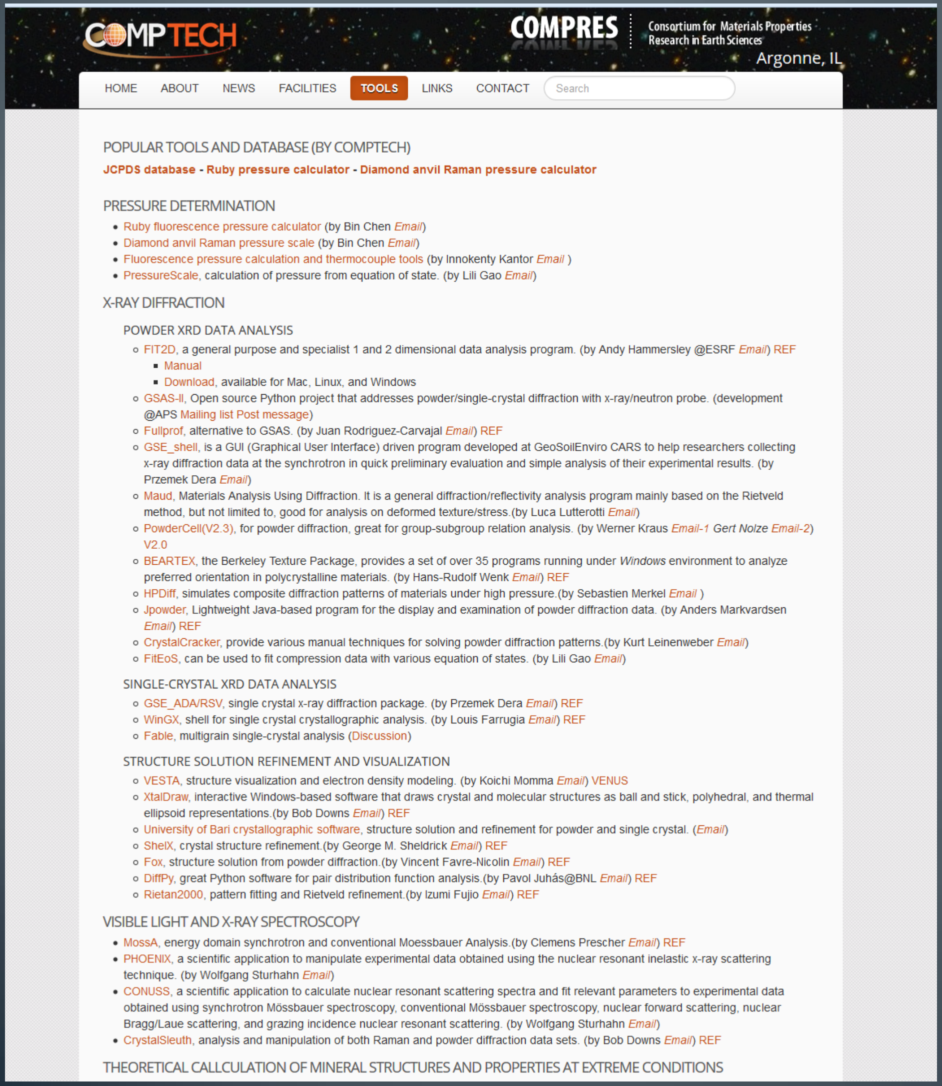
Standard "cheap" resistive heater for DAC

- Commercial W-Al_2O_3 metal ceramics heater can reach 1000 K within 30 s, very stable and cheap (<$10/pc). However their sizes are not suitable for DAC experiments. We plan to modify the dimensions of the commercialized 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.
- Order will be available for all COMPRES users in the future.



Website

http://comptech.compres.us/



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POPULAR TOOLS AND DATABASE (BY COMPTECH)

JCPDS database - Ruby pressure calculator - Diamond anvil Raman pressure calculator

PRESSURE DETERMINATION

- Ruby fluorescence pressure calculator (by Bin Chen *Email*)
- Diamond anvil Raman pressure scale (by Bin Chen *Email*)
- Fluorescence pressure calculation and thermocouple tools (by Innokenty Kantor *Email*)
- PressureScale, calculation of pressure from equation of state. (by Lili Gao *Email*)

X-RAY DIFFRACTION

POWDER XRD DATA ANALYSIS

- FIT2D, a general purpose and specialist 1 and 2 dimensional data analysis program. (by Andy Hammersley @ESRF *Email*) REF
  - Manual
  - Download, available for Mac, Linux, and Windows
- GSAS-II, Open source Python project that addresses powder/single-crystal diffraction with x-ray/neutron probe. (development @APS Mailing list Post message)
- Fullprof, alternative to GSAS. (by Juan Rodriguez-Carvajal *Email*) REF
- GSE_shell, is a GUI (Graphical User Interface) driven program developed at GeoSoilEnviro CARS to help researchers collecting x-ray diffraction data at the synchrotron in quick preliminary evaluation and simple analysis of their experimental results. (by Przemek Dera *Email*)
- Maud, Materials Analysis Using Diffraction. It is a general diffraction/reflectivity analysis program mainly based on the Rietveld method, but not limited to, good for analysis on deformed texture/stress.(by Luca Lutterotti *Email*)
- PowderCell(V2.3), for powder diffraction, great for group-subgroup relation analysis. (by Werner Kraus *Email-1 Gert Nolze Email-2*) V2.0
- BEARTEX, the Berkeley Texture Package, provides a set of over 35 programs running under *Windows* environment to analyze preferred orientation in polycrystalline materials. (by Hans-Rudolf Wenk *Email*) REF
- HPDiff, simulates composite diffraction patterns of materials under high pressure.(by Sebastien Merkel *Email*)
- Jpowder, Lightweight Java-based program for the display and examination of powder diffraction data. (by Anders Markvardsen *Email*) REF
- CrystalCracker, provide various manual techniques for solving powder diffraction patterns.(by Kurt Leinenweber *Email*)
- FitEoS, can be used to fit compression data with various equation of states. (by Lili Gao *Email*)

SINGLE-CRYSTAL XRD DATA ANALYSIS

- GSE_ADA/RSV, single crystal x-ray diffraction package. (by Przemek Dera *Email*) REF
- WinGX, shell for single crystal crystallographic analysis. (by Louis Farrugia *Email*) REF
- Fable, multigrain single-crystal analysis (Discussion)

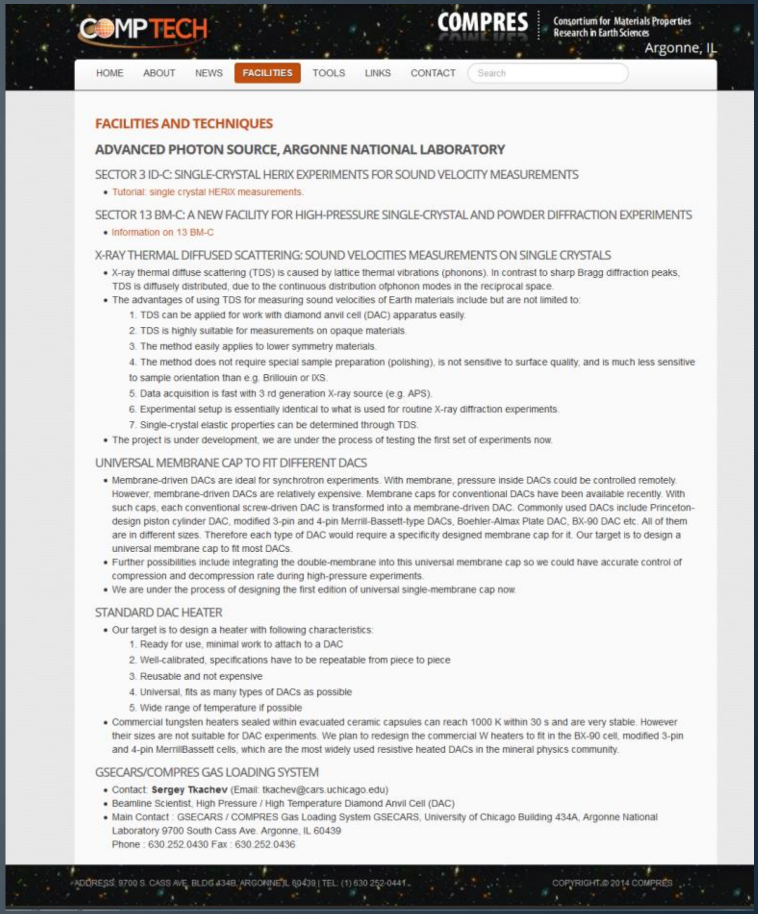
STRUCTURE SOLUTION REFINEMENT AND VISUALIZATION

- VESTA, structure visualization and electron density modeling. (by Koichi Momma *Email*) VENUS
- XtalDraw, interactive Windows-based software that draws crystal and molecular structures as ball and stick, polyhedral, and thermal ellipsoid representations.(by Bob Downs *Email*) REF
- University of Bari crystallographic software, structure solution and refinement for powder and single crystal. (*Email*)
- ShelX, crystal structure refinement.(by George M. Sheldrick *Email*) REF
- Fox, structure solution from powder diffraction.(by Vincent Favre-Nicolin *Email*) REF
- DiffPy, great Python software for pair distribution function analysis.(by Pavol Juhás@BNL *Email*) REF
- Rietan2000, pattern fitting and Rietveld refinement.(by Izumi Fujio *Email*) REF

VISIBLE LIGHT AND X-RAY SPECTROSCOPY

- MossA, energy domain synchrotron and conventional Moessbauer Analysis.(by Clemens Prescher *Email*) REF
- PHOENIX, a scientific application to manipulate experimental data obtained using the nuclear resonant inelastic x-ray scattering technique. (by Wolfgang Sturhahn *Email*)
- CONUSS, a scientific application to calculate nuclear resonant scattering spectra and fit relevant parameters to experimental data obtained using synchrotron Mössbauer spectroscopy, conventional Mössbauer spectroscopy, nuclear forward scattering, nuclear Bragg/Laue scattering, and grazing incidence nuclear resonant scattering. (by Wolfgang Sturhahn *Email*)
- CrystalSleuth, analysis and manipulation of both Raman and powder diffraction data sets. (by Bob Downs *Email*) REF

THEORETICAL CALLCULATION OF MINERAL STRUCTURES AND PROPERTIES AT EXTREME CONDITIONS



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FACILITIES AND TECHNIQUES

ADVANCED PHOTON SOURCE, ARGONNE NATIONAL LABORATORY

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

- Tutorial: single crystal HERIX measurements.

SECTOR 13 BM-C: A NEW FACILITY FOR HIGH-PRESSURE SINGLE-CRYSTAL AND POWDER DIFFRACTION EXPERIMENTS

- Information on 13 BM-C

X-RAY THERMAL DIFFUSED SCATTERING: SOUND VELOCITIES MEASUREMENTS ON SINGLE CRYSTALS

- X-ray thermal diffuse scattering (TDS) is caused by lattice thermal vibrations (phonons). In contrast to sharp Bragg diffraction peaks, TDS is diffusely distributed, due to the continuous distribution ofphonon modes in the reciprocal space.
- The advantages of using TDS for measuring sound velocities of Earth materials include but are not limited to:
  1. TDS can be applied for work with diamond anvil cell (DAC) apparatus easily.
  2. TDS is highly suitable for measurements on opaque materials.
  3. The method easily applies to lower symmetry materials.
  4. The method does not require special sample preparation (polishing), is not sensitive to surface quality, and is much less sensitive to sample orientation than e.g. Brillouin or IXS.
  5. Data acquisition is fast with 3 rd generation X-ray source (e.g. APS).
  6. Experimental setup is essentially identical to what is used for routine X-ray diffraction experiments.
  7. Single-crystal elastic properties can be determined through TDS.
- The project is under development, we are under the process of testing the first set of experiments now.

UNIVERSAL MEMBRANE CAP TO FIT DIFFERENT DACS

- Membrane-driven DACs are ideal for synchrotron experiments. With membrane, pressure inside DACs could be controlled remotely. However, membrane-driven DACs are relatively expensive. Membrane caps for conventional DACs have been available recently. With such caps, each conventional screw-driven DAC is transformed into a membrane-driven DAC. Commonly used DACs include Princeton-design piston cylinder DAC, modified 3-pin and 4-pin Merrill-Bassett-type DACs, Boehler-Almax Plate DAC, BX-90 DAC etc. All of them are in different sizes. Therefore each type of DAC would require a specificity designed membrane cap for it. Our target is to design a universal membrane cap to fit most DACs.
- Further possibilities include integrating the double-membrane into this universal membrane cap so we could have accurate control of compression and decompression rate during high-pressure experiments.
- We are under the process of designing the first edition of universal single-membrane cap now.

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
- 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 MerrillBassett cells, which are the most widely used resistive heated DACs in the mineral physics community.

GSECARS/COMPRES 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

ADDRESS: 9700 S. CASS AVE, BLDG 434B, ARGONNE IL 60439 | TEL: (1) 630 252-0441

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Any suggestions are welcome!