COMPRES

Newsletter

http://www.compres.us

Vol.2 No.4 December 2003, Stony Brook

# Rheology Project Report

## Towards the Experimental Study of Deep Mantle Rheology: A Progress Report

As a part of a collaborative research program [one of several Grand Challenge proposals submitted and funded by the NSF Division of Earth Sciences in 2002 ] focussed on "Experimental Studies of Plastic Deformation under Deep Earth Conditions," we have initiated an experimental study of whole mantle rheology at Yale University. Our emphases are (i) the development of new technique of ultrahigh pressure, large strain deformation apparatus and (ii) the better characterization of defect-related properties in deep mantle minerals. The need for new technology for high-pressure deformation experiments is obvious. The maximum pressure with currently available apparatus is limited to ~3 GPa, and with the most reliable, high-resolution deformation apparatus such as the Paterson gas-medium deformation apparatus, the maximum pressure is only ~0.5 GPa. So the reliable experimental data from the gas-medium apparatus provide data for only ~15 km deep in the Earth. Therefore if one wants to understand whole mantle rheology, one needs a new type of apparatus or techniques to conduct high-pressure deformation experiments. It is also very important to understand the microscopic physics and chemistry of defect-related properties under deep mantle conditions to obtain results that can be applied to Earth. This is particularly true in relation to the influence of chemical environment on kinetic properties.

**IN THIS ISSUE:**

*Photo to the right:* A rotational Drickamer apparatus (RDA) for high-pressure, temperature large-strain deformation experiments developed at Yale University.

The goals of our research are (i) to determine the rheological properties of deep mantle materials and (ii) to investigate the deformation-induced microstructures (such as lattice preferred orientation (LPO) and dynamically recrystallized grain-size) initially for minerals in the mantle transition zone and finally lower mantle and inner core materials. The challenges in this type of research are not only to develop a new technique of high-pressure deformation, but also to formulate a strategy to obtain the results that can be applied to Earth. This latter problem is important particularly in rheological studies, because several deformation mechanisms can operate in a given material and rheological properties are very sensitive to chemical environment (such as water content). For these reasons, the extrapolation of laboratory data of rheological properties to Earth is not straightforward which is very different from other properties such as elastic properties.

The most crucial development in high-pressure rheology is the development of X-ray techniques of stress and strain measurements that have been made in Don Weidner's group at SUNY Stony Brook. In our approach, we plan to use their technique to characterize rheological properties in collaboration with a group at SUNY Stony Brook. As to the apparatus for high-pressure deformation we have chosen to modify the Drickamer apparatus for reasons explained below. We have also initiated a detailed study of defect-related properties of deep mantle minerals. Combining these we hope to make a major progress in understanding the deformation behavior of the whole Earth.

### (1)Progress report on the development of a rotational Drickamer apparatus (RDA)

We have initiated an effort to extend the rheological studies to higher pressures. Our aims are to investigate the rheological properties and microstructural developments under deep Earth conditions (below the transition zone). For these purposes, we chose to modify the Drickamer apparatus to conduct high-pressure deformation experiments to large strains. The advantages of this approach include: (i) with the Drickamer apparatus, anvils have large support due to their geometry (the principles of massive support) and the support by a gasket. Accordingly one can achieve relatively high pressures compared to other apparatus such as a cubic apparatus. Second, by modifying this apparatus by introducing rotation to an anvil, one does not change the support for the anvils too much. Consequently, we expect that we might be able to conduct very high-pressure deformation experiments with this design. In addition, simple shear deformation has lower symmetry than tri-axial compression and is likely to be more important in most part of the Earth's interior. Deformation microstructures such as lattice preferred orientation (LPO) depend on deformation geometry, and therefore results from simple shear deformation will be more useful for the interpretation of seismic anisotropy. Yamazaki and Karato (2001) tested the basic design with a minimal amount of budget (~$10,000) and showed that it works to ~12 GPa at room T. After receiving the funds from the NSF Grand Challenge program, in addition to Karato's start-up money, we started to extend this to higher T and higher P.

The schematic design of the apparatus is shown in Fig.1 (photo in the front page) and the sample assembly in Fig. 2. Briefly, we add a rotation actuator to the Drickamer apparatus using the Harmonic Drive gear box and a dc servo-motor to a conventional Drickamer apparatus. A constant rate of rotation is applied to a specimen by this actuator. An experiment is conducted by first pressuring the sample chamber, and then we anneal the sample at certain T for a certain duration (currently we anneal a sample at 1473 K for 1 hour). The purpose of this annealing is to remove the unwanted defects (dislocations) produced by room T pressurization. After this we rotate the anvil at a constant rate. We have

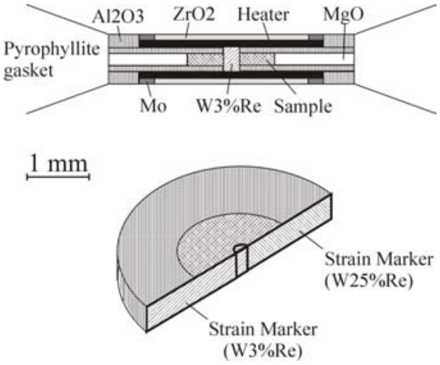
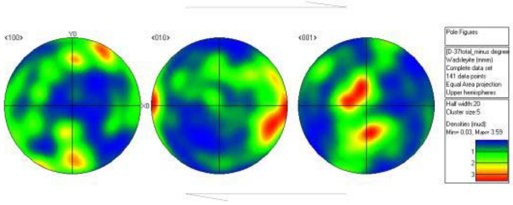


Figure 2. Sample assembly for a RDA deformation experiment

conducted initial tests using Fe, (Mg,Fe)O and wadsleyite as samples. All the tests on Fe were made at room T, but tests on (Mg,Fe)O and wadsleyite were made at high T and P. Early results on Fe showed that this apparatus worked although we noted significant extrusion of samples when sample thickness is large. The first high-T and P deformation experiment was done on (Mg,Fe)O (at ~1500 K, P~12 GPa) that showed that the maximum strain of ~2 was achieved and much of deformation occurred in the sample. After Yu Nishihara joined our team (April, 2003), we started serious efforts on deformation of wadsleyite. By improving the materials and the geometry of sample assembly we were able to deform wadsleyite (in one case wadsleyite was transformed partly to ringwoodite) to large strain (<100% shear strain). All of these experiments were conducted at shear strain-rate of ~$10^{-4}$ $s^{-1}$ or less (strain-rate being zero at the center and linearly increases with distance). The microstructures of samples were studied using mostly SEM (+ TEM) and the lattice preferred orientation was determined (Figure 3). These results are potentially important in interpreting seismic anisotropy in the transition zone. We have determined the LPO of wadsleyite deformed in simple shear at high-T and P (Figure 4). When these results are compared to seismological observations, we will be able to infer the flow pattern in the deep mantle.

Despite these promising results, we also recognize several challenges. (i) Deformation geometry is not ideal in most cases particularly when wadsleyite is deformed. In an ideal case, deformation geometry would be simple shear, but we recognized significant compression components. (ii) The stress levels are still too high judging from the deformation microstructures. Stress levels must be reduced to obtain results that are applicable to Earth's interior. We plan to increase temperature (and perhaps reduce strain-rates) to reduce the stress level (addition of water may also help). (iii) The water contents of our samples have not been controlled.



wadsleyite, $P$ =15 GPa, $T$ = 1673 K, $\gamma \sim 0.7$, $\gamma \sim 10^{-4}\ s^{-1}$

**Figure 3** Pole figures of wadsleyite. The symmetry of this LPO is consistent with simple shear geometry of deformation. The fact that <010> is parallel to the shear direction indicates that the stress was very high during the deformation. $X_0$ is shear direction and $Z_0$ is shear plane normal. All the data points have been corrected to their own shear directions.

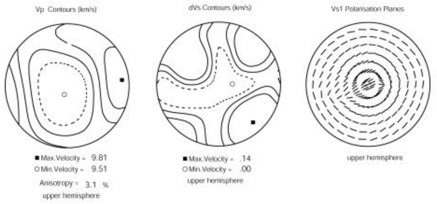


**Figure 4** Seismic anisotropy corresponding to the fabrics of wadsleyite (Figure 5). E-W direction is the shear direction and the center of the plot is the shear plane normal.

The next exciting step is to conduct in-situ X-ray study. Our first experiment is scheduled in late April, 2004 at NSLS synchrotron facility. With this we will be able to characterize the stress levels (as well as strain) during deformation experiments.

Overall, the performance of the RDA is complementary to D-DIA (deformation DIA developed by Bill Durham, Yanbin Wang and Ivan Getting). The D-DIA will be more suitable for characterization of rheological properties (stress/strain is nearly homogeneous in D-DIA. Whereas in a RDA, both stress and strain-rate changes with distance from the center of a sample, and hence the resolution of rheological properties will be limited). However, the maximum pressure at which deformation experiments can be conducted will be somewhat higher for a RDA for reasons discussed above. Also much larger strains can be achieved with a RDA, which is critical to the microstructural studies under high-pressures. Therefore both types of apparatus must be further developed and used in the experimental studies of plastic deformation for whole Earth.

(2) Defects in wadsleyite

We have also made significant progress in understanding the defects in wadsleyite. Wadsleyite is known to be able dissolve a large amount of water (Kawamoto et al. (1996), Kohlstedt et al. (1996)) and there are some reports showing that kinetic processes in wadsleyite are “faster (softer)” than olivine (electrical conductivity: Xu et al. (1998), cation diffusion: Farber et al. (1994), Chakraborty et al. (1999)). Briefly, we found that this conclusion is not really true and when compared at “dry” (water-free) conditions, most of the kinetic processes in wadsleyite are slower than those in olivine. We noticed that wadsleyite dissolves water from environment so easily that one needs to pay special attention to minimize the water content in wadsleyite (a typical so-called “dry” experiment turned out to be pretty much “wet” experiment where water content in wadsleyite exceeds that of olivine in typical “wet” conditions). However, when water is present, the kinetic processes in wadsleyite can be faster than those of olivine due to the higher solubility of water in wadsleyite. In addition, we found that oxygen fugacity has very large effects on some kinetics such as grain-growth (Figure 5). These results show that in investigating the kinetics processes of wadsleyite such as plastic deformation one needs to control or at least characterize the chemical environment carefully.

Related to this issue, we have developed a model of point defects in minerals including wadsleyite. We now have a much clearer understanding of the dissolution mechanism of water in these minerals and how these water-related defects might modify physical/chemical properties.

All of these studies have been supported by NSF, in particular from the Grand Challenge collaborative research program on rheology. These Grand Challenge proposals are officially distinct from the COMPRES Core grant [Consortium for Materials Properties Research in Earth Sciences], but are closely linked on an intellectual level, in that they provide some of the basic scientific rationale for developing and maintaining the experimental facilities overseen and operated under the auspices of COMPRES.

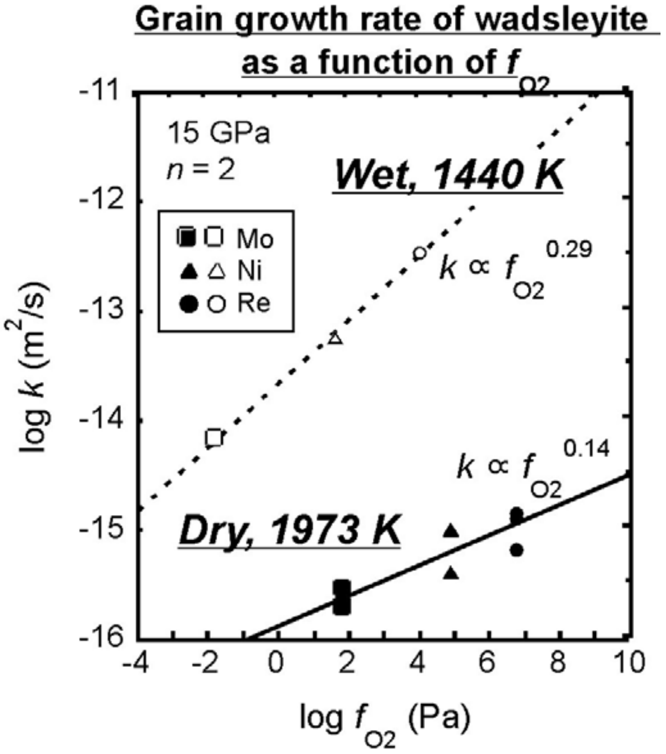


Figure 5. The rate of grain-growth in wadsleyite as a function of oxygen fugacity (fO2). Such a data set provides a strong constraint on the nature of defects that controls the rate of kinetic processes in minerals.

**Correspondence: Shun-ichiro Karato, Yousheng Xu, Yu Nishihara and Toru Shinmei at Department of Geology & Geophysics, Yale University**

## New Address for COMPRES Web Page

The front page of COMPRES web site has a new URL link address: http://www.compres.us.
All the files related to the COMPRES web site are still physically located on www.compres.stonybrook.edu server, therefore all direct links remain unchanged.

# President's Message

As part of my first four months on the job, I paid visits all of the institutions which are responsible for overseeing or operating community facilities for COMPRES or where Infrastructure Development (ID) projects are being conducted under the auspices of COMPRES. The purpose of these visits was to learn about the current state of these operations and to discuss their future plans.

Following is a list of these visits
Sept. 30: GeoSoilEnviro CARS [GSECARS] at the Advanced Photon Source (APS).
Oct. 8: Diamond-anvil cell facilities at the National Synchrotron Light Source (NSLS).
Oct. 16: Geophysical Laboratory (Pressure calibration at high temperature project)
Oct. 20-21: Virginia Tech (Neutron studies program and labs of N. Ross and R. Angel)
Oct. 28: Princeton University (Laser-heating in DAC project)
Oct. 29: Multi-anvil facilities at the NSLS.
Oct. 30: Yale University-Karato lab
Nov 3: University of Washington—labs of Ann Chopelas and Michael Brown
Nov. 4: Synchrotron facilities-Advanced Light Source of the Lawrence Berkeley Lab
Nov. 17: University of Illinois at Urbana-Champaign (Brillouin spectroscopy project)
Nov. 19: University of Colorado (Absolute P & T project)
Nov. 20: Arizona State University (Multi-anvil cell development project)

On September 23, Don Weidner, Chair of the Executive Committee of COMPRES, and I visited the NSF Division of Earth Sciences to discuss with the program directors the current status and our future plans for COMPRES

On Oct. 17, Charlie Prewitt and I represented the Center for High Pressure Research [CHiPR] at a ceremony at the NSF honoring the directors of the Science and Technology Centers Class of 1991. In a brief presentation, we were able to show how the NSF investment in technological developments in the 1990s has led to the formation of the new consortium COMPRES. During that visit to NSF, I met with Dr. Hugh Van Horn, Director of National Facilities in the Division of Materials Research [DMR] to find out more details about a new program for major instrumentation projects within DMR. I had been alerted to this new funding opportunity by David Lambert, Program Director for Instrumentation and Facilties [IF] in the Division of Earth Sciences [EAR] during our visit to EAR on September 23.

On Nov 13, Don Weidner and I visited the Basic Energy Sciences offices of the DOE to meet with Nick Woodward, Helen Kerch and Pedro Montano, at the suggestion of David Lambert. We discussed the new changes in how the DOE proposes to operate their synchrotron and neutron facilities.

Within the past few months, many institutions have been approved by the Executive Committee for membership in COMPRES. With the addition of the University of Wyoming and the Universite de Poitier (France), we now have 38 U.S. institutions and 8 foreign affiliates. A complete list can be found on the COMPRES website: http://www.compres.us. Under People/Member Institutions, where you will also find US and world maps showing the location of these member institutions [thanks to Glenn Richard for creating these maps].

On Nov 1-5, I attended the Annual Meeting of the GSA and MSA in Seattle, Washington. Highlights included a symposium in honor of Charlie Prewitt organized by Nancy Ross, Ross Angel and Russell Hemley, which was a great success. During the meeting, we all took pride in the award of special honors to our colleagues from Mineral Physics and Chemistry, including:
Charles Prewitt—Roebling Medal of the MSA
Guillaume Fiquet—MSA Award
George Harlow—Distinguished Service Award of the MSA
Michael Manga—Donath Medal of the GSA

In addition, at the Seattle meeting, Michael Carpenter of the University of Cambridge was installed as the new President of the Mineralogical Society of America.

The ByLaws Committee [Bruce Buffett, Ronald Cohen, Joseph Smyth, Charles Prewitt, and Lars Stixrude] has elected Ronald Cohen as its chair and has begun its work on behalf of the COMPRES community. As indicated earlier, the ByLaws Committee was encouraged to consult widely the COMPRES community in formulating their recommendations for possible changes in the ByLaws, especially in view of Article XI, which stipulates that changes to the ByLaws require the affirmative vote of two-thirds of the entire Electorate. Following discussions and consideration of the proposed changes by the COMPRES community, the Electorate will be asked to vote (section by section) on the proposed changes, via a confidential balloting system using double sealed envelope, at the 2004 Annual Meeting [which we hope to hold in June]. For those Electors [or their Alternates] who will not be attending the 2004 Annual Meeting, they will be able to submit absentee ballots using the double-sealed envelope balloting system in advance of the Annual Meeting.

The major event of December 2003 for COMPRES was surely the Fall AGU Meeting in San Francisco. Highlights included:

Symposium in honor of Don Anderson's 70th birthday organized by Raymond Jeanloz and Miaki Ishii, which included many interesting oral and poster presentations. I had the pleasure of giving a talk on Don's role in nurturing and encouraging research in mineral physics.

Exhibition booth jointly sponsored by GSECARS and COMPRES, which attracted lots of visitors [despite its awkward location] and seemed to be appreciated. My thanks to Jiuhua Chen, Michael Vaughan and Ann Lattimore for their invaluable work in creating and staffing the booth, as well as to Nancy Lazarz and Mark Rivers of GSECARS for their cooperation.

We all took pride in the award of special honors to our colleagues in Mineral and Rock Physics (MRP), including:

David Kohlstedt—Harry Hess Medal
New AGU Fellows: William Durham, Eiji Ito, Ian Jackson and Taro Takahashi (who was honored for his contributions to ocean chemistry, but whom many know as one of the pioneers in mineral physics research using the diamond-anvil cell).

The Physical Properties of Earth Materials group once again organized a fantastic dinner celebration at Destino's, the critically acclaimed Latin American restaurant in the Castro district. Our congratulations to Brian Bonner and Bill Durham for discovering such a wonderful venue for this special evening.

The Committee on Mineral and Rock Physics hosted a wine and cheese reception, during which Annette Kleppe of Oxford University received the Outstanding Student Award in Mineral and Rock Physics for 2003.

We also had meetings with David Lambert, Program Director for Instrumentation & Facilities in EAR, who oversees the COMPRES initiative, one of which included Harry Green to discuss a proposed plan to acquire and operate a focused ion-beam microscope for the rock and mineral physics community.

The COMPRES Standing Committees held luncheon meetings to discuss the annual reports on the Infrastructure Development projects and Community Facilities. The Executive Committee met for breakfast on Dec 11 to begin the planning process leading to the submission of the Annual Program Plan and Budget request to the NSF on February 1, 2004.

## 2004 Annual Meeting

The dates for the 2004 Annual Meeting have been tentatively set for June 20-21-22 [Sun-Mon-Tues]; alternative dates are June 17-

18-19 [Thurs-Fri-Sat]. We have deliberately tried to avoid conflicts with other known meetings of potential interest to members of the COMPRES community [e.g., the IRIS annual meeting, the Gordon Research Conference on High Pressure, etc]. Details on the site for this meeting will follow shortly.

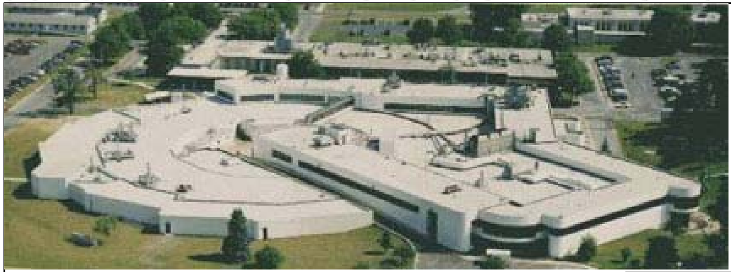
Best regards,
Bob Liebermann

Address:
Robert C, Liebermann
COMPRES
ESS Building
Stony Brook University
Stony Brook, NY 11794-2100
USA
Office Telephone: 631-632-1968
Office FAX: 631-632-8140
email address:
Robert.Liebermann@stonybrook.edu

# COMPRES Booth at AGU Fall 2003 Meeting

At the Fall 2003 Meeting of the American Geophysical Union in San Francisco in December, COMPRES and GeoSoilEnviroCARS co-sponsored an exhibition booth, which included posters and a PowerPoint presentation [first slide of which is shown above with Michael Brown of the Unversity of Washington and Michael Vaughan of Stony Brook Unversity looking on]. Our thanks to Jiuhua Chen and Ann Lattimore for their invaluable work in creating the materials for the booth, as well as to Nancy Lazarz and Mark Rivers of GSECARS for their cooperation. Photo by Ann Chopelas.

# Presentations from the COMPRES Facilities and Projects



## Multi-Anvil High Pressure at National Synchrotron Light Source

**PI: Donald J. Weidner**
**(SUNY Stony Brook)**

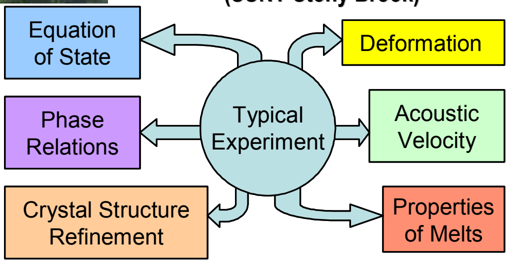
**Facilities:** **Beam line X17B2**

- DIA SAM85, DDIA-type cubic anvil apparatus
- 6-8 double-stage multi-anvil apparatus

**Peak P & T:** ~ 25 GPa, ~ 2000°C

**Measurements:**

EDXD, ADXD, ultrasonic, x-ray radiograph



Typical Experiment

Equation of State

Deformation

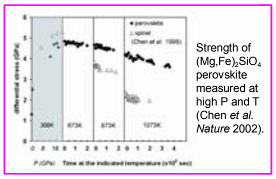
Phase Relations

Acoustic Velocity

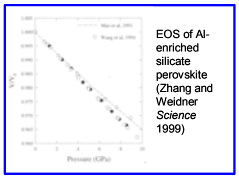
Crystal Structure Refinement

Properties of Melts

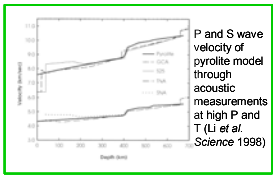
**Examples:**



Strength of $(Mg,Fe)_2SiO_4$ perovskite measured at high P and T (Chen *et al.* *Nature* 2002).



EOS of Al-enriched silicate perovskite (Zhang and Weidner *Science* 1999)

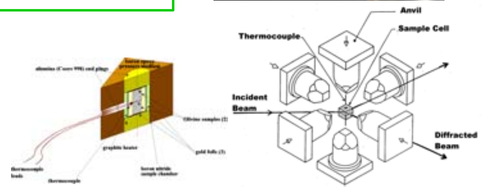


P and S wave velocity of pyrolite model through acoustic measurements at high P and T (Li *et al.* *Science* 1998)

**Beam Time Request (open to all general users):**

Submit a General User Proposal to NSLS User Administration, BNL, Building 725B, Upton, NY 11973-5000, fax: 631-344-7206

| For beam time between: | Jan – April | May – Aug | Sept – Dec |
|---|---|---|---|
| Cycle: | 1 | 2 | 3 |
| Deadline: | **Sept 31** | **Jan 31** | **May 31** |



## West-Coast Synchrotron Facilities

**PI: Raymond Jeanloz**
**(UC Berkeley)**

**Beam line at ALS**

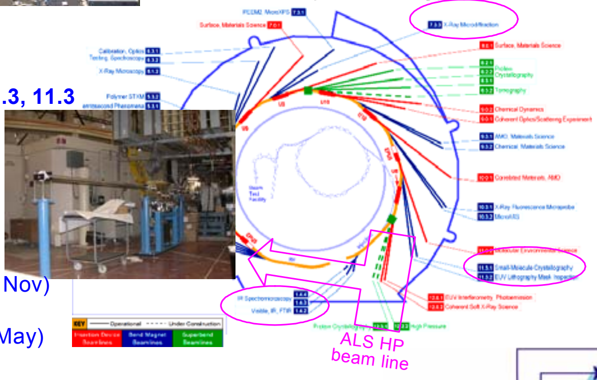
**dedicated: 12.2**
**non-dedicated: 1.4, 7.3, 11.3**

**Beam time request**

http://www.als.lbl.gov/als/quickguide/independinvest.html

**Deadlines**

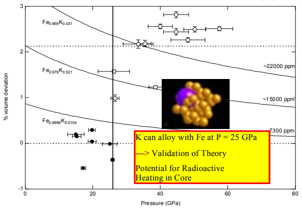
December 1
(*for beam time June* ~ Nov)
June 1
(*for beam time Dec* ~ May)

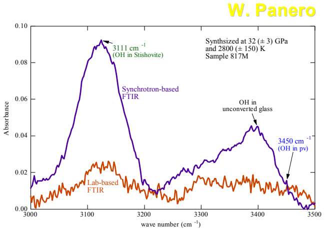


**HP facilities**

Laser-Heating w/Spectroradiometry; Ruby fluorescence

**Example Studies**





**Beam line at SSRL**

**non-dedicated: 10-2**

**Beam time request**

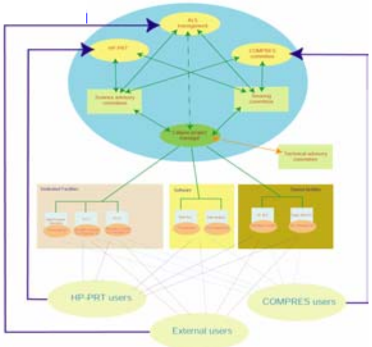
http://www-.ssrl.slac.stanford.edu/users/user_admin/guide.html

**Deadlines**

May 1, *for beam time starting from Oct 1.*

November 1, *for beam time starting from May 1.*

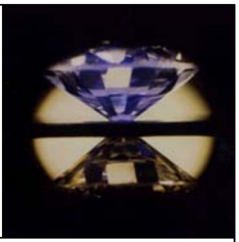
Facility Management Plan (ALS)



# Megabar Synchrotron Center at the NSLS

Beam line X17C, X17B3, U2A

**PI: Ho-kwang Mao and Russell J. Hemley (Carnegie)**



**Facilities:**

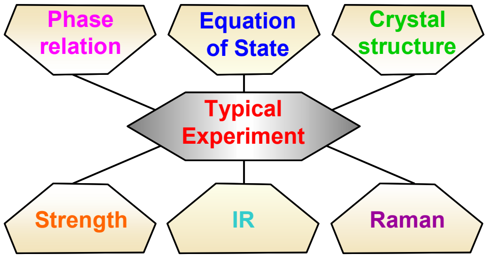
- Diamond Anvil Cells
- On-line laser heating
- Ruby P-calibration

**Peak Pressure:**
multi- megabars

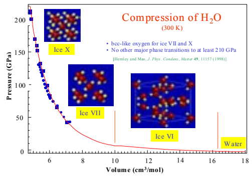
**Peak Temperature:**
multi-kilodegrees

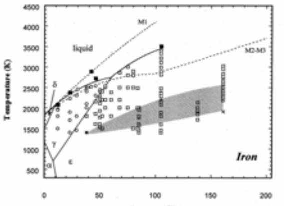
**Measurements:**

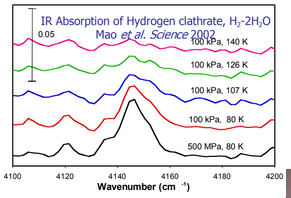
EDXD, ADXD, Raman, IR spectroscopy

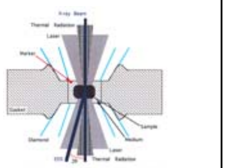

Typical Experiment
Phase relation
Equation of State
Crystal structure
Strength
IR
Raman

**Examples:**









## Beam Time Request :

http://www.nsls.bnl.gov/users/requestingbeamtime

| | | | |
|---|---|---|---|
| Deadline: | **Sept 31** | **Jan 31** | **May 31** |
| For beamtime: | **Jan–April** | **May–Aug** | **Sept–Dec** |

X17C x-ray diffraction hutch

U2A IR/Raman spectroscopy hutch

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# Neutron Facilities

**PI: Nancy Ross**
**( Va. Tech )**

*Chalk River(Canada), FRJ-2 (Germany), GKSS (Germany), HANERO (Korea), HFIR, ORNL (US), ILL (France) , IPNS (US), ISIS (UK), JINR (Russia), KENS (Japan), LANSCE (US), LLB (France), MNRC (US), NIST (US) . . .*

## Goals of the Neutron Facility Project:

- Identify and broaden the EAR neutron scattering community in the U.S.
- Stimulate and promote the use of neutron scattering in the Earth Sciences.
- Carry out educational activities that support the above goals.
- Identify the needs of the communityincluding future requirements for instrumentation and sources

## Financial Assistance

- To carry out neutron scattering experiments and/or to participate in workshops and conferences on neutron scattering.

- See www.crystal.vt.edu or contact Nancy Ross (Va. Tech) at nross @vt.edu for details

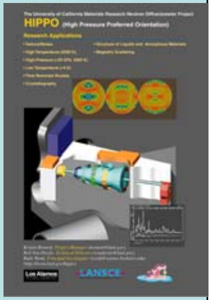
**Spallation Neutron Source (SNS)**
will provide most intense source of neutrons

www.sns.gov

Under construction
in Oak Ridge, TN.
Completion date: 2006

## Why Neutrons?

- *. . . can detect hydrogen, distinguish betw/ Al,Si, . . .*
- *. . can probe lattice vibrations, phonon density of states, electronic transitions*
- *. . . can study magnetism*
- *. . scattering lengths do not change with scattering vector*
- *. . . provide a highly penetrating probe so cryostats, furnaces and pressure cells can be used*
- *allow investigation of texture (preferred orientation) and residual stress analysis:*

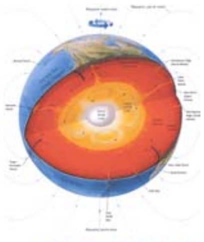


## Neutron News:

**SNAP funded!!**
J. Parise, R. Hemley, H-k Mao and C. Tulk's proposal SNAP **S**pallation **N**eutrons **A**t **P**ressure) at the SNS has been approved for funding by DOE

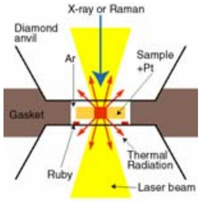
# Development of the Laser-Heated Diamond Anvil Cell

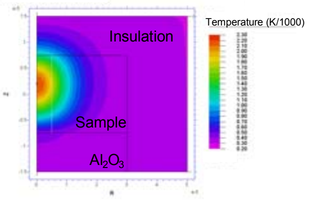
### PI: Thomas Duffy (Princeton), Guoyin Shen (Chicago) Dion Heinz (Chicago)

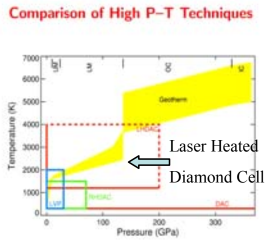


The laser-heated diamond anvil cell is an essential tool for simulation of deep planetary interiors. it is the only device that can access the full range of pressures and temperatures expected to be encountered in the Earth's mantle.

## Thermal Structure of the Laser-Heated Diamond Cell



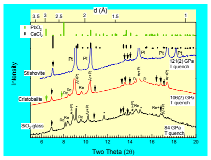


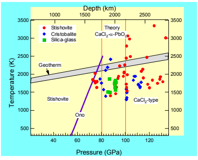


## Laser-Heated Diamond Cell Experiments:

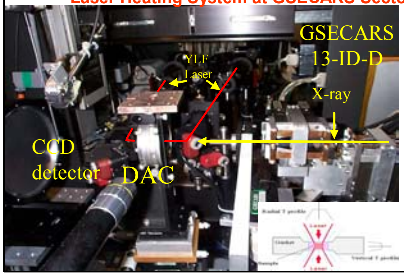
- Crystal structure
- Phase equilibria
- High-temperature EOS
- Element partitioning
- Synthesis
- Melting and melt structure
- Reaction kinetics
- Rheology
- Thermoelasticity
- Solubility
- Annealing
- Transport properties

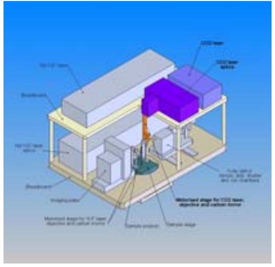
## Ultrahigh Pressure-Temperature Studies of Mantle Minerals



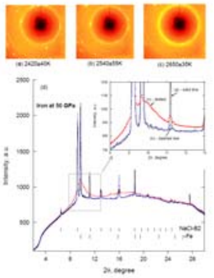


## Laser Heating System at GSECARS Sector of Advanced Photon Source





## Melting and Melt Structure



**Acknowledgements:**

B. Kiefer

(NMSU)

S. Shieh

(NCKU)

S. Shim

(MIT)

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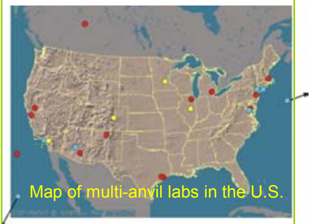
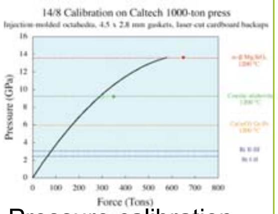
# Multi-anvil Cell Assembly Development

### PI: Kurt Leinenweber and James Tyburczy (Arizona State University)

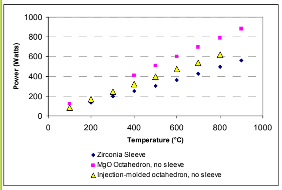
## Purposes of the project:

❖Develop an interlaboratory collaboration throughout the high-pressure community on the use of multi-anvils for research.


Map of multi-anvil labs in the U.S.

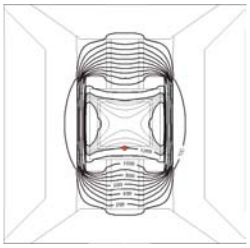


Pressure calibration



Temperature efficiency

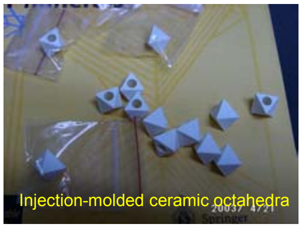
❖Design better pressure cells for multi-anvil experiments.



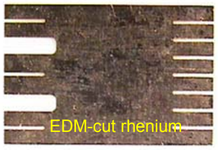
Thermal modeling


Gaskets on an automated mill

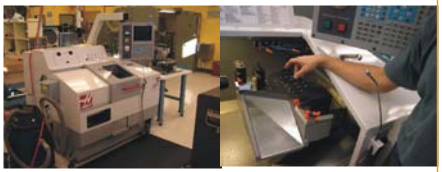
❖Learn better ways to fabricate cell assemblies.


Injection-molded ceramic octahedra

Novel pressure media


EDM-cut rhenium

Novel furnace designs



Use of automated lathe

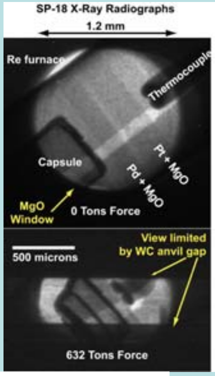
**SPECIAL THANKS TO: Jed Mosenfelder, Jennifer Kung, Jim Van Orman, Robert Liebermann, D.J. Weidner**

# Pressure Calibration at High Temperature

### PI: Yingwei Fei (Carnegie)

## Goals of the Project

1) To establish a self-consistent practical pressure scale.
2) To establish a spectroscopically-based pressure scale at high temperature.

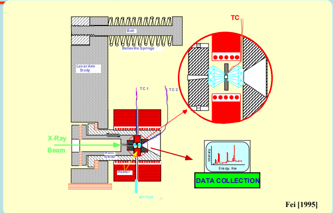


## High-Pressure Techniques

### High-T DAC

**Pressure range:**
**up to 125 GPa**

**Temperature:**
**up to 1100 K**



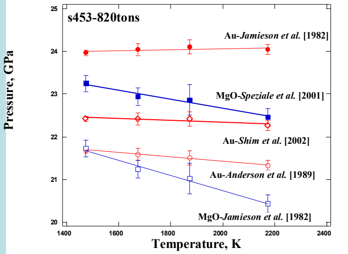
Fei [1995]

### Multi-Anvil Apparatus

**Pressure:**
**up to 28 GPa**

**Temperature:**
**up to 2500 K**

## Comparison of Existing Pressure Scales (Au, Pt, MgO)



Comparison of different pressure scales. The difference in calculated pressures can be as large as 4 GPa at 24 GPa and 2200 K

**The existing thermal equations of state for pressure standards such as Au, MgO, and Pt, predict a range of pressures at high temperatures for samples under the same pressure and temperature conditions. For high P-T experiments, especially at synchrotron facilities, there is an urgent need to establish a reliable and self-consistent pressure scale at high temperature. We evaluated different pressure scales in a series of high P-T experiments with multiple internal pressure standards and concluded that the use of a single practical pressure scale such as the MgO scale of Speziale et al. (2001) would be beneficial for consistency check and data comparison. We determined the relative differences among different pressure scales and established new Au and Pt scales that are consistent with the MgO scale of Speziale et al. (2001). These scales allow us to compare high P-T data that were collected using different internal pressure standards.**

## Future Priority on Pressure Calibration

**The ultimate goal is to establish an absolute pressure scale at high temperature by redundant equation-of-state measurements through simultaneous X-ray diffraction and acoustic measurements. In order to achieve this goal, broad collaborations are required among experts in X-ray diffraction, Brillouin scattering spectroscopy, ultrasonic technique, and inelastic x-ray scattering.**

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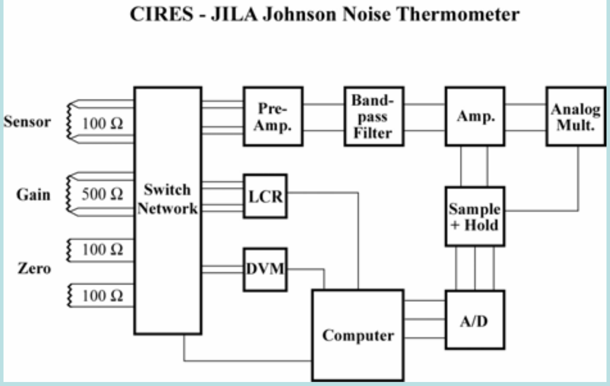
# Johnson Noise Thermometry at High Pressure

### PI: Ivan C. Getting (University of Colorado at Boulder)

## Johnson Noise Thermometry

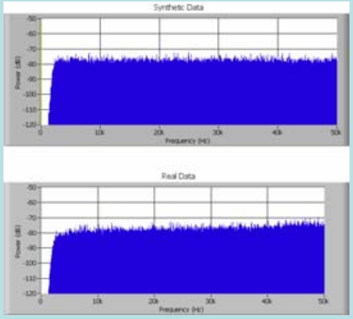
Johnson noise is electrical noise across a resistor associated temperature. A Johnson Noise Thermometer has been developed which makes it possible to measure temperature accurately at high pressure. All effects of pressure, deformation, chemical reaction, etc., which normally plague thermocouples, are accounted for with a simple measurement of resistance each time temperature is to be measured. Thermocouples and other temperature sensors can be calibrated against this absolute thermometer in each specific type of apparatus and cell design of interest. Such calibration studies need be made only once for each experimental configuration with the results applicable to subsequent runs.

**CIRES - JILA Johnson Noise Thermometer**



Sensor
100 Ω
Gain
500 Ω
Zero
100 Ω
100 Ω
Switch Network
Pre-Amp.
Band-pass Filter
Amp.
Analog Mult.
LCR
Sample + Hold
DVM
Computer
A/D

## Synthetic and Real Noise Data Spectra



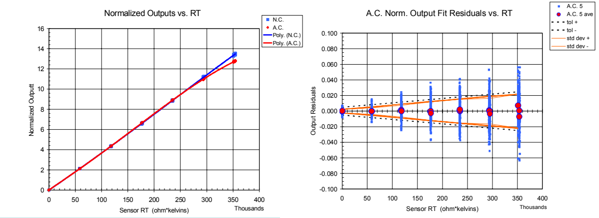
## Johnson Noise and Signal Correlation

For an open circuit resistor in thermal equilibrium, the relation between the mean square noise voltage across the resistor, $\langle E_R^2 \rangle$, the resistance, *R*, and the temperature, *T*, is given by

$$\langle E_R^2 \rangle = 4kTRB$$

where *k* is Boltzmann's constant and *B* is the band width over which the noise voltage in measured.

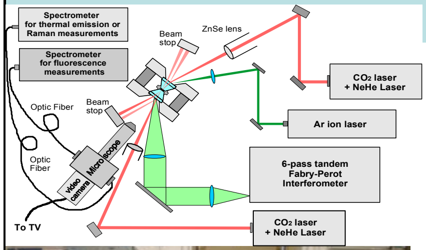
## Johnson Noise System Output Characteristics And Reproducibility



# Brillouin Scattering at the Advanced Photon Source:

## A Community Facility, for Simultaneous Velocity and Density at High P & T

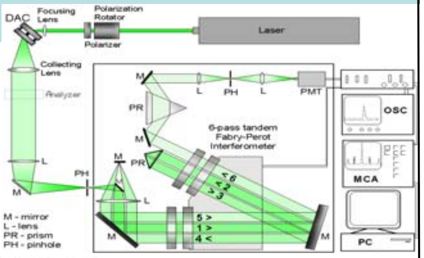
## PI: JD Bass, G Shen



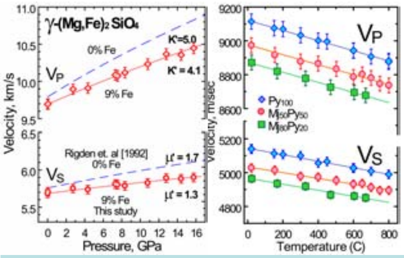
**The Brillouin Spectrometer which will be installed at APS**: A preliminary version of the APS spectrometer has been built at the University of Illinois. This relatively simple spectrometer will now be fully implemented and then transported to GSECARS for installation on beamline 13BM.

**A Brillouin spectrometer will be built on beamline 13BM-D at the APS. Users will be able to perform volume (density) measurements using synchrotron XRD, and velocity measurements using Brillouin, all at high P & T. This will allow determination of absolute pressure scales, one of the community priorities of COMPRES.**

Schematic diagram of planned Brillouin system at APS, for velocity measurements by Brillouin and density measurements by synchrotron XRD. This facility will play a major role in determining an Absolute pressure scale. The initial implementation of the system will utilize the simpler resistance heating only, to about 1000 C. Laser heating can be added later, as an outgrowth of the Laser Heating project of Duffy & Heinz.



The main components of a Brillouin Scattering system for velocity and elastic property measurement on single crystals and polycrystals, at high P&T.



**Velocity measurements at High Pressures** (Note the large effect of Fe on velocities) **and High-Temperatures** ( Note the systematic dependance of velocity on the majorite:garnet ratio)

### New X-ray Methods for Velocity and EOS Measurements

JD Bass, J Parise, G Shen, D Weidner, W Sturhahn

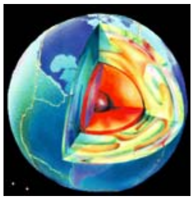
**Inelastic X-ray Scattering (IXS)**: This involves the inelastic scattering of x-rays from acoustic phonons. The technique is conceptually similar to Brillouin scattering with X-rays. It is being developed on: : sector 3 at APS, where energy resolution on the order of several meV are now obtained, making this experiment feasible for accurate velocity measurements. Opaque samples pose no difficulty, and the P-T range for this technique is limited only by the diamond cell design. **Nuclear Resonant Inelastic X-ray Scattering (NRIXS):** Determination of the phonon density of state in samples containing Fe57. It allows the acoustic velocities to be constrained. There is no intrinsic limitation on the P-T range.

# Education Programs

## Glenn A. Richard (COMPRES Central Office at Stony Brook University)

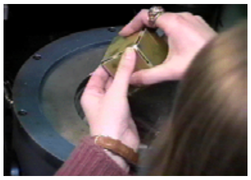
### Earth Science Educational Resource Center

COMPRES conducts education and outreach programs through the Earth Science Educational Resource Center, based at Stony Brook University.



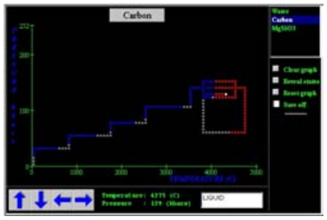
### Let's Make Diamonds!

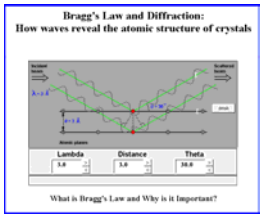
The Let's Make Diamonds program offers high school and undergraduate students an opportunity to engage in an experiment to convert graphite into diamond and to test the resulting product.



### Working with Educators

Through teacher enrichment courses and workshops, and through collaboration with local and national organizations, COMPRES reaches students by working with their teachers.





### Virtual Earth Materials Laboratory

The Virtual Earth Materials Laboratory will offer curriculum materials and related interactive software modules that enable high school and undergraduate students to perform virtual experiments that explore the properties of Earth materials. A portion of this resource will be implemented in conjunction with an NSF-funded EarthScope education project that focuses on strain rate models.

### High School Honors Earth Science Programs

COMPRES collaborates with many other groups to provide opportunities for pre-college students to conduct research projects through high school honors Earth Science courses and other programs.

### Beamline Internships at COMPRES Facilities

Open for applications now.
Send your application to Ann.Lattimore@sunysb.edu

# Recent PhDs

**Haemyeong Jung, Ph. D. 2002,**

*University of Minnesota, Minneapolis, MN*

Dissertation**: Effects of water on the plastic deformation and deformation microstructure of olivine**

Deformation experiments on olivine were conducted in simple shear geometry under both water-poor (“dry”) and water-saturated (“wet”) conditions at high pressures (0.5 - 2.2 GPa) and high temperatures (1400 - 1570 K) using the Griggs apparatus. Hot-pressed synthetic olivine aggregates and single crystals of olivine were used as starting materials. Water was supplied by a mixture of talc and brucite during the experiment and water content of samples was determined by the FT-IR spectroscopy after each experiment. Under these conditions, a wide range of water fugacity can be explored up to ~13 GPa and we have identified significant effects of water both on microstructures and on rheology.

Water affects microstructures of deformed olivine in two ways: (1) the size of dynamically recrystallized grains under water-rich conditions is significantly larger than that under water-poor conditions and (2) the pattern of lattice preferred orientation (LPO) is different under water-rich conditions from those under water-poor conditions. These observations indicate that water enhances grain-boundary migration and also changes the relative easiness of slip systems in olivine. The LPOs found under water-rich conditions are characterized by strong peaks of [001] poles at ~20-30° from the shear direction, although the maxima of poles of other crystallographic axes depend also on the stress level. A diagram showing the relationship between LPO and deformation conditions was constructed. The results indicate that the relationship between seismic anisotropy and flow geometry depends on water content as well as stress level.

The role of water on rheology was investigated through the analyses of stress versus strain-rate relationship under various pressures using dislocation densities as a stress indicator. We found that when the system is saturated with water, the strength of olivine changes with pressure in non-monotonic way: the strength decreases with pressure at low pressures, but strength increases at higher pressures. The results are interpreted using a model in which two parameters (water-fugacity effect and activation volume effect) control the strength. Our results supports a model in which deformation of olivine is controlled by the motion of positively charged jogs through the diffusion of Si via interstitial mechanism.

## Statement

It was my great pleasure to have a thorough guidance for research from my advisor Dr. Shun Karato and to work with my colleagues at the University of Minnesota and Yale University, finding exciting results including “water-induced fabric transitions in olivine”. I am now at the University of California with Dr. Harry Green, studying high pressure faulting mechanisms to understand the basic mechanism of earthquakes. We found an exciting result: dehydration of serpentine (antigorite) under stress results in faults independent of the sign of the net volume change (dV) of dehydration reaction (Jung et al., 2004, Nature, in press). This result confirms that dehydration embrittlement is a viable mechanism for triggering earthquakes independent of depth, so long as there are hydrous minerals breaking down under a differential stress. My current goal of research is to understand the basic mechanism of earthquakes by deforming rocks and minerals at high pressure and high temperature. I am eager to do high pressure faulting experiments using the D-DIA.

# Recent PhDs

**Li Li, PhD 2003**

*State University of New York at Stony Brook*

Dissertation**: Rheology of olivine at mantle pressure**

Knowledge of the rheological properties of mantle materials is critical in modeling the dynamics of the Earth. The high-temperature flow law of olivine defined at mantle conditions is especially important since the pressure dependence of rheology may affect our estimation of the strength of olivine in the Earth's interior. Conventional deformation methods in defining the flow law of olivine have to face factors of large uncertainties of differential stress and/or limited confining pressure for deformation. In this study, high-temperature (up to 1473 K) deformation experiments of polycrystalline olivine (average grain size < 10 μm) at pressure up to 6.5(±0.2) GPa, were conducted *in situ* using a large-volume Deformation DIA (D-DIA) high-pressure apparatus and synchrotron x-ray radiation. The $f_{O2}$ during the run was lower than the Ni/NiO buffer's and higher than the iron-wustite buffer's. More than 30 % strain was generated during the uniaxial compression. Sample lengths during the deformation process were monitored by x-ray radiography. The strain rate was derived with an accuracy of $10^{-6}$ $s^{-1}$. Macroscopic differential stress was measured at constant strain rate (~ $10^{-5}$ $s^{-1}$) using a multi-element solid-state detector combined with a conical slit. This technique was applied for the first time on samples deformed at high pressure at controlled strain rate in the D-DIA. The new data not only consistent with the reported plasticity flow law, but also suggests an activation volume less than 5 $cm^3$/mol for temperatures above 1273K in dislocation creep regime. Recovered specimens were investigated by optical and transmission electron microscopy (TEM). TEM observations are consistent with the rheological data and show that dislocation glide was the dominant deformation mechanism throughout the experiment. Evidences of dislocation cross-slip as an active mechanism at high pressure are also reported.

## Statement

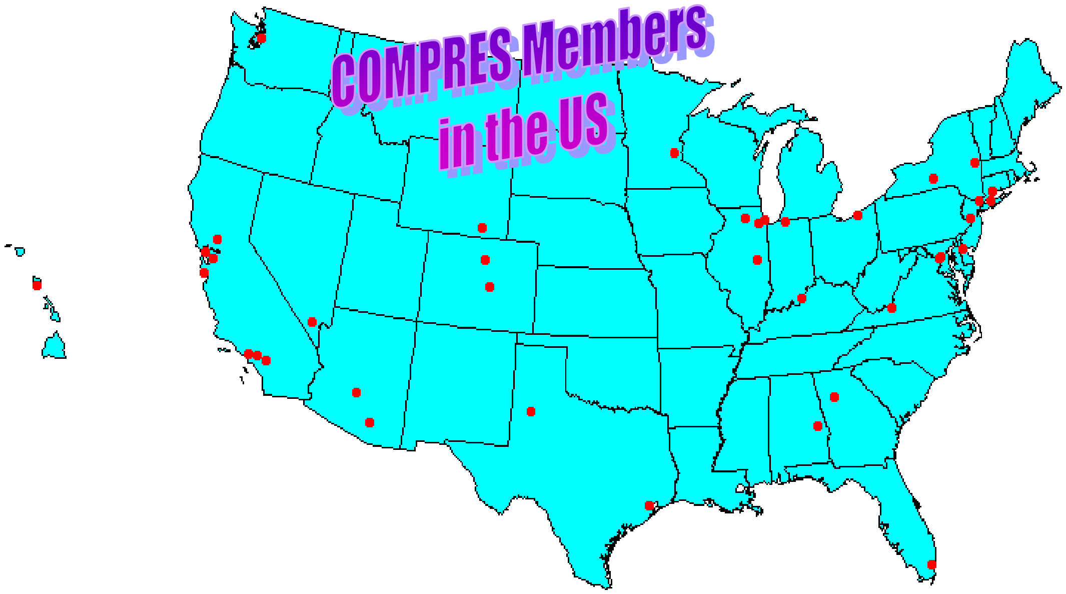
It was a great pleasure to work with my advisor Dr. Don Weidner, and my collegues Dr. Jiuhua Chen and Dr. Mike Vaughan during my Ph. D study. Now I am working as a Post-doc in MPI at Stony Brook, also conducting some collaborative research with Université des Sciences et Technologies de Lille. Our rheology group of MPI at Stony Brook has established a systematic method for mantle rheology research. We also dedicate to optimize the systems for general users. Some of our recent research can be summarize as follows:

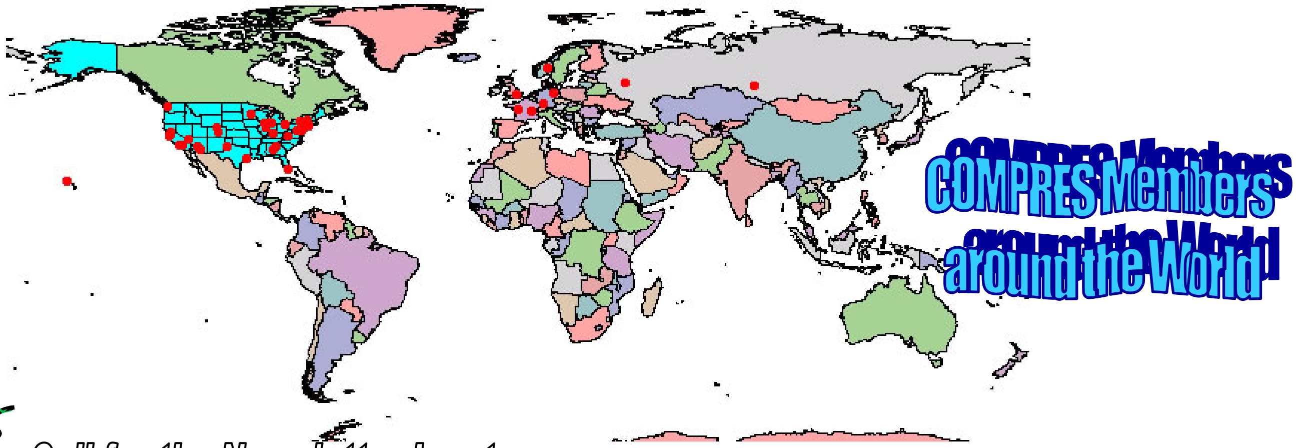
1. The deformation of San Carlos olivine in D-DIA high pressure apparatus demonstrate very compatible power-creep flow laws of "wet" olivine defined by (Karato and Wu, 1993) and (Hirth and Kohlstedt, 1996) and the "dry" flow law of (Chopra and Paterson, 1979), all with an activation volume less than 5 $cm^3$/mol for temperatures above 1273 K. Lower temperature data are compatible with the Pierls plasticity flow law as defined by (Evans and Goetze, 1979), again with an activation volume of zero.
2. We used a self-consistent model to predict the flow-stress during plastic deformation of polycrystalline MgO with slip system of $\{110\}$ $\langle 1\bar{1}0\rangle$, $\{111\}$ $\langle 1\bar{1}0\rangle$, $\{100\}$ $\langle 011\rangle$ at different critical resolved shear stress ratios (CRSS) for the different slip systems. We also conducted deformation experiments on polycrystalline and single crystal MgO samples in D-DIA. The correlation of the data and models suggests that the plastic models need to be used to describe the stress-strain observations with the presence of plasticity, while the Reuss and Voigt models are appropriate for the elastic region of deformation; it also suggests that $\{111\}$ slip system is the most significant slip system in MgO at high pressure and high temperature.
3. Single crystal olivine samples will be deformed in D-DIA to characterize the slip systems as well

as define the flow low for single crystal. We are also interested in the correlation of dynamic dislocation model and self-consistent model with our experimental data.

4. We are interested in the mechanism of deep earthquakes, which may be well characterized by plastic instability mechanism. Thus, it is possible for us couple our experimnetal rheological data with the dynamic condition of the earth to probe the myth of deep earthquakes.

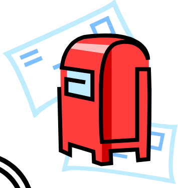
My current goal is to continue the mantle rheology research, both in material properties and technique development. My long term goal is to be able to couple experimental data with computation model to explain the earth's dynamics. Besides my current research, I am also eager to be involved in a wide variety of fields such as elasticity, DAC experiment, inelastic X-ray scattering, etc.





## Call for the Newsletter Input

The Newsletter is designed to report new happenings around the COMPRES, and more importantly, breakthroughs in the facility development, scientific research and education programs of COMPRES. Please send your input to the COMPRES central office.



**COMPRES Contacts:**
President, Robert C. Liebermann, (631)632-1968, Robert.Liebermann@stonybrook.edu
Chair, Donald J. Weidner, (631)632-8211, Donald.Weidner@sunysb.edu
Administration, Ann Lattimore, (631)632-8213, alattimore@notes.cc.sunysb.edu
Newsletter, Jiuhua Chen, (631)632-8058, Jiuhua.Chen@sunysb.edu