Year 1 Annual Progress Report of the Consortium for Materials Properties Research in Earth Sciences (COMPRES)

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EXECUTIVE SUMMARY

This first year of the COMPRES consortium has been one of building and putting into action the ideas and plans that were described in the original COMPRES proposal. We have become defined as an organization with a set of community sanctioned set of by-laws that govern us. We have put into place a management structure, with specific committees that have clearly defined purposes, responsibilities, and members who have voted into their positions by the electorate. We have undertaken significant reorganization of the operations of centralized facilities, in particular some synchrotron beamlines at national laboratories, and have made great progress in making those facilities more useful and readily available to the high-pressure Earth and planetary science community. We have interacted with the NSF, in particular the Instrumentation and Facilities Program and its advisory panel, on how COMPRES should focus its efforts in order to achieve the maximum effect. We have sponsored and helped to organize international and domestic scientific meetings, educational workshops, outreach to inform the broader scientific community about COMPRES, and workshops to advance the technology used in our research. Over the first nine months of its existence, COMPRES has been the transformed from a set of ideas put forward by the small nucleus of people that wrote the COMPRES proposal, into an organization that is defined and guided by its members. In short, we have turned the COMPRES concepts into reality. While we feel a great number of tangible accomplishments have been achieved thus far, we have also identified many areas in which we need to focus our effort over the years ahead. The paragraphs below briefly outline our activities since May 2002. Greater detail on the activities of specific COMPRES projects is given in subsequent sections.

Relationships to National Facilities:

This is one of the most important, and in some ways challenging categories of COMPRES activities. The nature of our relationship is highly varied. We now provide direct support for beamline operations at the National Synchrotron Light Source (NSLS, Brookhaven Nat. Lab.) and for the Advanced Light Source (ALS, Lawrence Berkeley National Lab). Projects for developing novel infrastructure are being pursued in collaboration with GeoSoilEnviroCars at the Advanced Photon Source (GSECARS, APS, Argonne National Lab). With all of these facilities, COMPRES is taking part in evaluating the experiences of users via user evaluation forms. We are in turn providing feedback to beamline managers on problems experienced by users, and ways in which beamline operations could be improved. For those facilities obtaining support for beamline operations we have had to define a set of operational principles defining the relationship between COMPRES and the beamlines. These operational principles must address: the distribution of beamtime, the proposal evaluation process, actual beamtime allocation, the role of COMPRES in deciding how beamlines will be instrumented, how the costs of upgrades and new instrumentation will be distributed. With partial operational support now coming through COMPRES, we are having to redefine how these beamlines carry out their business, and this must be done in a way which improves the efficiency and quality of beamline services. In the coming year, we will need to formalize the agreements with beamlines and initiate new memoranda of understandings, where appropriate.

Enormous progress has been made in the formation of a West Coast Synchrotron Facility. Unlike the high-pressure facilities at NSLS and APS, the West Coast high-pressure effort is being built from essentially nothing into a major resource for the COMPRES community. COMPRES is now a major partner in the California High Pressure Observatory (CALIPSO). We are helping to define the ultimate form of the high- pressure facility at ALS through co-sponsoring of design workshops. Formal agreements have been negotiated with higher-level management at the ALS for beamtime dedicated to COMPRES, and the ALS is already providing x-ray and spectroscopy facilities to our members. We are greatly encouraged at how quickly the West Coast facility has progressed and have every expectation that it will become a highly subscribed first class facility. Finally, major strides have been made in organizing the user base for neutron scattering facilities. We view this as an area with high growth potential. Historically, activity in neutron scattering has been relatively small within the US Earth sciences community. Our view is that neutrons are a largely untapped resource, and with construction of the Spallation Neutron Source, which is now being built at Oak Ridge and will be the most intense neutron source in the world. We are continuing to work on informing our community on the use of neutron scattering and organizing to define a long term scientific agenda in this area (see outreach, below).

Administrative Structure:

The committee structure of COMPRES has changed significantly from that given in the original proposal submitted to NSF. It is now more streamlined. We now have two standing committees: A Facilities committee (Q Williams (chair), M Rivers, JM Brown, Y Fei, H Green), and an Infrastructure Development committee (J Tyburczy (chair), P Burnley, R Jeanloz, D Walker, Y Wang). The committee members were elected at our first annual meeting. The Executive Committee is now a five-person committee instead of the initial 17-members. Documents defining the purpose, responsibilities, and authority of each committee have been written and approved by the membership (see by-laws, attached). We have put in place a system with checks-and-balances to serve the community in a fair and open way. The members of an external advisory committee have also been chosen, and consists of R O'Connell (Harvard), Guy Masters (Scripps), C-C Kao (NSLS), B Buffett (U Chicago), and P Silver (Carnegie).

The standing and executive committees are extremely active. All committees met in December 2002 to evaluate progress on all COMPRES projects, to discuss possible changes in procedures and policies, to recommend budgets, etc. Thus, the progress and budget for each Facilities or Infrastructure Development project was discussed and evaluated in by two committees. An organizational chart of COMPRES follows this summary.

President Search:

At the first annual COMPRES meeting, Jay Bass was elected as the first President, to serve as Interim President through August 2003. A search committee was set up to recommend a permanent President to the Executive Committee. The search committee includes D. Walker (Chair, Columbia), R. Jeanloz (Berkeley), C. Prewitt

(Carnegie), N. Ross (Virginia Tech.), D. Weidner (Stony Brook). Advertisements for this position appeared in EOS and Science.

Annual COMPRES Meetings:

The first Annual Meeting was held on September 14-15, 2002 at Stony Brook, NY. This was the first opportunity of the initial COMPRES membership to openly discuss issues related to the administrative structure, organization, scope of activities, officers, and policies of COMPRES. This meeting was absolutely essential to refine the definition of COMPRES in a way that is consistent with the views of the community and to identify community priorities. Each committee defined its mission and began to carry out its work. Much time was devoted to discussing the new relationship that would exist between COMPRES and supported facilities. Many of the outcomes of this meeting were presented to NSF at an October 17, 2002 presentation. Discussions were continued at committee meetings held during the Fall AGU meeting in San Francisco.

The 2003 Annual COMPRES Meeting will be held on June 18-20 in Santa Cruz, CA. Now that the basic organizational structure of COMPRES is in place, this meeting will include a healthy scientific component, including presentations by those who have used COMPRES facilities. As a result, we are will encourage the participation of students as well as COMPRES institutional representatives.

Meetings and Workshops:

COMPRES has supported a number of meetings with different formats and purposes. The international conference “High Pressure Mineral Physics Seminar” was held in August 2002 inVerbania, Italy, with COMPRES support for US participants. An interdisciplinary conference titled “Workshop on Mantle Composition, Structure, and Phase Transitions” will be held in Frejus, France on April 2003 with joint partial support from COMPRES and the CNRS in France (http://www.ens-lyon.fr/LST/WorkShop/frejus/)

Workshops with a narrower focus have been held or are planned for 2003. An informational-planning workshop on the West Coast synchrotron facility was held in Dec. 2002 following the Fall AGU meeting. A follow-up workshop to obtain community input on the design of the laser-heating system to be built there is planned for Feb. 2003. A workshop for users of multi-anvil devices was held at the Fall AGU and was well attended by senior researchers and students alike. Workshops are planned for the coming year on advancing laser-heating technology for diamond cell work (T Duffy, organizer), “Neutrons in Solid-State Chemistry and the Earth Sciences” (March, N. Ross organizer), and pressure-temperature calibration (Y Fei, organizer). Student will be participating in these workshops. In addition, COMPRES is holding a workshop on present and future research directions in high –pressure mineral physics, March 22-23 in Miami, Florida. A professional writer (Ellen Kappel) has been contracted to help produce a report on that meeting for the NSF.

Education and Outreach:

As noted above, COMPRES is involving students in a majority of its workshops. We have provided a substantial amount of financial support for students to participate in meetings, and we plan to continue this policy to the extent possible in the coming year. We will place emphasis on instructional workshops in which students can obtain familiarity with new theory and techniques. The neutron workshop listed scheduled for March 2003 is one example of this.

In its written review of the COMPRES Oct. 17 meeting at NSF, the IF panel stated "One of the strong recommendations of the IF panel was that the educational aspects, including workshops, be funded." and "COMPRES should move forward with the educational aspects and the workshops in the initial proposal". We agree wholeheartedly with that recommendation and are trying to act on it, as indicated above. In addition, we have requested funds in the current budget for a beamline internship program. This would place two graduate students at synchrotron facilities to work full-time as beamline scientists for one year. We believe that this would be an extremely valuable hands-on experience for the students, and it is worth investing in for a 1-2 year trial period.

During the past year, COMPRES has been building partnerships with national organizations including the Digital Library for Earth System Education (DLESE) and EarthScope, in order to perform education and outreach through a nationwide network of scientists, educators and other stakeholders. Glenn Richard, of COMPRES, has become a vital member of the planning committees of both organizations. Templates for generating nationally based education programs are being developed in these organizations and some of the models are being piloted in the COMPRES program.

Membership:

We have launched a membership drive with detailed information sent by email, in the COMPRES newsletter, and described on our web site. We have made an effort to make the broader community aware of opportunities available through COMPRES by holding meetings and presenting posters at major conferences (for example, a town-hall style meeting at the Fall AGU meeting).

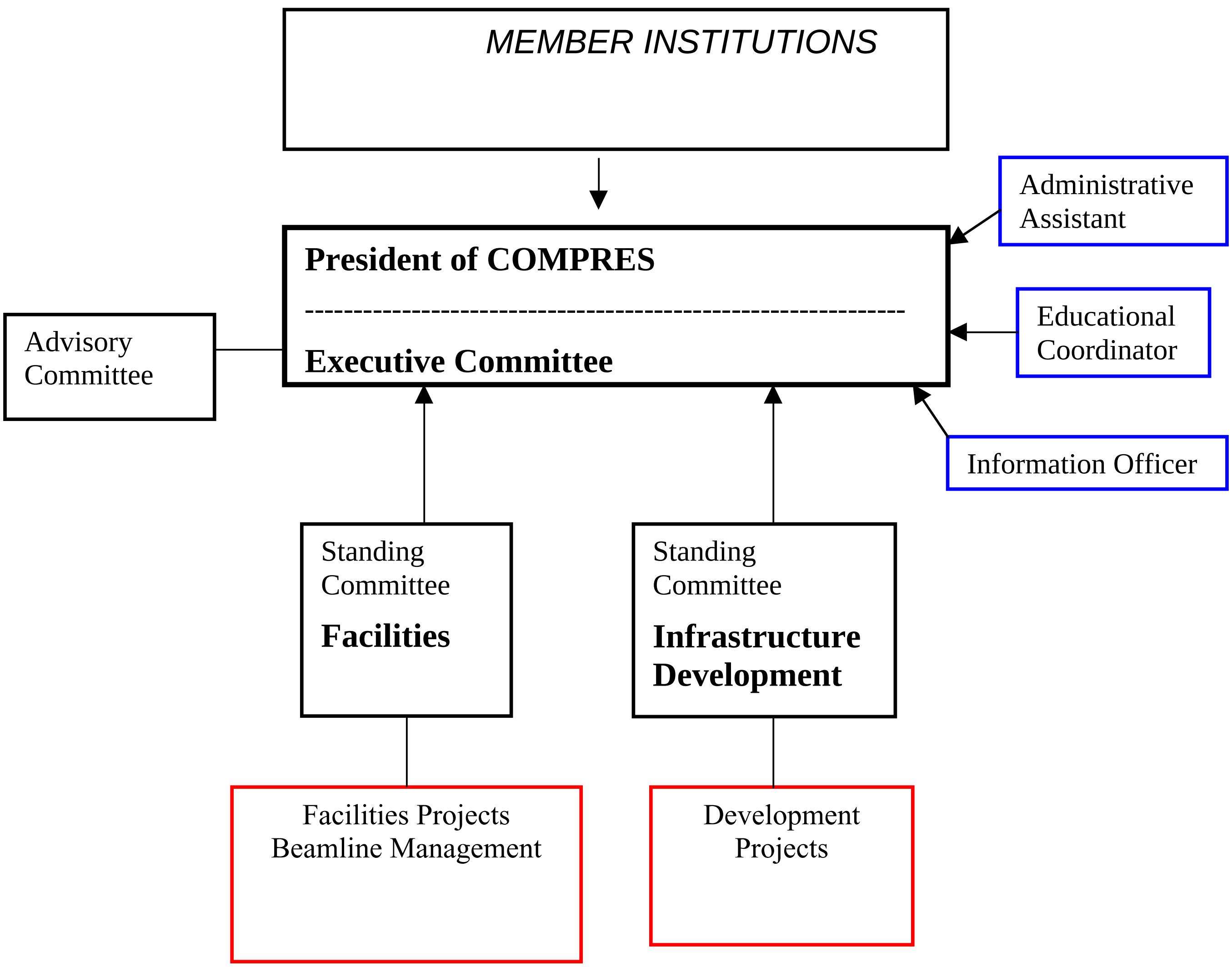
Communications and Media: (videoconferencing, website, and newsletter).

Internet technology presents COMPRES with numerous options for implementing organizational services for its members and for developing an attractive and useful interface with the educational and public communities. A website is up and operational (www.compres.stonybrook.edu). We have begun testing videoconferencing tools for conducting coast-to-coast meetings and feel that this is the best way to keep informed without expensive travel budgets. We have found that frequent meetings of the Executive Committee (EC) are necessary to keep pace with issues requiring their attention. The EC is now holding videoconferences every two weeks. A monthly newsletter is prepared and sent out electronically. All back issues are available on the website.

Liaisons with Other Agencies:

COMPRES plans to expand its visibility within the broader high-pressure community, act follow the recommendations of the IF panel in this regard. We have initiated contact with program officers (Kerch and Montano) at the Department of Energy to visit their offices and discuss ways in which DOE and COMPRES could coordinate on issues of common interest (beamline management, workshops, etc.) Similar contacts will be made with the Materials Sciences Program at NSF. Forming working relationships with other agencies and NSF programs is a major goal for the remainder of year 1 and for year 2.

COMPRES Organizational Chart


MEMBER INSTITUTIONS
President of COMPRES
Executive Committee
Advisory Committee
Administrative Assistant
Educational Coordinator
Information Officer
Standing Committee
Facilities
Standing Committee
Infrastructure Development
Facilities Projects
Beamline Management
Development Projects

Education and Outreach

During the past year, COMPRES has been building partnerships with national organizations, such as the Digital Library for Earth System Education (DLESE) and EarthScope, in order to perform education and outreach through a nationwide network of scientists, educators and other stakeholders. Glenn Richard became a member of the Planning Committee for the 2002 annual meeting of DLESE and organized a set of skills workshops that were offered during the first day of the meeting. These workshops introduced educators and scientists to data and visualization tools that can be used in formal and informal educational settings. Mr. Richard also organized a group of several dozen people to offer presentations of digital educational tools at the share fair event that formed part of the activities during the second day of the DLESE meeting. He is serving on the Planning Committee again this year.

Mr. Richard also worked with the EarthScope Education and Outreach Committee to develop an Education and Outreach Program Plan, that calls for the establishment of a distributed network comprised of a set of partners and local alliances throughout the United States that will work together to develop and disseminate educational resources.

This plan, which was published in November, 2002, includes COMPRES as a partner. Through this partnership, COMPRES plans to make data on materials properties available for educational use and to work with others to develop interactive modeling and visualization tools that will enable students and educators to explore properties of Earth materials. These will be disseminated on the web and through meetings of DLESE, EarthScope, and other organizations.

In 2002, we worked to develop an education and outreach plan for a proposal to NSF that established a Center for Environmental Molecular Science (CEMS). The proposal, which was subsequently awarded funding, provides for a full-time Educational Specialist to link with Glenn Richard in the COMPRES education office. This will enable COMPRES and CEMS to collaborate on the development of educational programs and to cooperate on dissemination of resources.

COMPRES manages its education and outreach programs in conformance with the goals of the emerging EarthScope Education and Outreach Network. These allow us to pilot many programs through our Central Office at Stony Brook University. The guiding principals include the following:

- Provide students, researchers and members of the general public opportunities to investigate the Earth, its materials, and phenomena that relate to these materials.
- Promote the experience of science as an inquiry-based process of discovery.through the integration of research and education.
- Stimulate young people to consider careers in the sciences
- Promote the utilization of our planet as a hands-on learning laboratory where students and educators can conduct scientific research.
- Facilitate an interdisciplinary approach that integrates the other sciences and mathematics into Earth Science education.

- Integrate the use of computer technology into science education.
- Promote science literacy among the general public.

COMPRES accomplishes these goals through cooperation with national organizations, direct interaction with pre-K through 12^{th} grade and college students, creation and posting of interactive educational material on the World Wide Web, teacher workshops, university credit courses for teachers, and collaborations with other educational units on campus and in the surrounding community.

Since its initiation, COMPRES has cultivated partnerships with local educators and students in order to pilot a model for local alliances, crafted as components of a national network, as formulated in the EarthScope Education and Outreach Program Plan.

An important function of local alliances is that of developing digital tools for education. Project Java (http://www.journey.sunysb.edu/ProjectJava/) is a program designed to engage undergraduate computer science majors to create interactive educational material for the World Wide Web. It was initiated in 1996 and is now administered by COMPRES, the Department of Technology and Society, and the Department of Chemistry at Stony Brook. This program provides dual educational rewards as it offers undergraduate students tangible computer programming challenges and it provides teaching tools in the form of interactive programs that focus on specific educational topics.

Local alliances also need to create opportunities for underserved groups to experience science as a process of inquiry. COMPRES collaborates with the Project WISE (Women in Science and Engineering) program to offer young women opportunities to experience science as a process of inquiry. A pre-existing project is the “Lets Make Diamonds!” program, which offers students research experience that focuses on the nature of our planet's interior, paralleling COMPRES’s research mission. Participants, guided by researchers and COMPRES educational staff become familiar with background information and concepts, and devise and conduct an experiment designed to convert graphite to diamond, using high-pressure equipment. In the spring of 2002, COMPRES offered a new program, “Investigating Earthquakes” to groups of pre-college and undergraduate Project WISE students. Participants used data with mapping and modeling tools to explore relationships of seismicity to mantle processes, Earth materials and tectonic plates as well as effects of earthquakes on human society.

Another important function of local alliances is the development of integration between university research and pre-college education. COMPRES and the Department of Geosciences offer an honors Earth science course to students at Sayville High School that is equivalent to Stony Brook’s undergraduate introduction to physical geology. During the first year of the course, which runs over a two year cycle, lecture and laboratory components of the undergraduate course are incorporated into the course at Sayville. During the second year students complete a major research project that is carried out over the course of the academic year.

COMPRES also works with the Science and Technology Entry Program (STEP) and the Department of Geosciences on the Stony Brook Campus to offer a Brentwood Honors Earth Science program modeled after Stony Brook’s introductory environmental geology undergraduate course. During the summers, students engage in a four week residential program, emphasizing scientific methodology, research techniques and data collection in the field. During the following academic year, students work in teams to conduct research projects.

Educational networks need to leverage their resources by working with teachers in order to reach large numbers of students. CEN 514: Long Island Geology is a professional development-level course designed for teachers, offered each fall, that explores processes that have governed the geological development of Long Island and other parts of New York State. Topics include mantle processes and their relation to plate tectonics, the tectonic history of New York State, local seismicity, the origin of local rocks, and a brief overview of current research in mineral physics and its relation to processes that have played a role in the geologic history of New York. GEO 514 is taught by Glenn Richard and Steven Englebright, a University Adjunct who is a New York State Assemblyman.. Each participant in CEN 514 is required to develop a lesson plan that is designed to familiarize secondary school students with Earth science as an investigative process.

Future Plans

During the coming year, COMPRES will continue to work with the EarthScope Education and Outreach Network and other groups to integrate the study of seismic and tectonic phenomena with the study of properties of Earth materials, and to evaluate and disseminate the resulting tools and programs to national audiences, including pre-college and college students as well as the general public. COMPRES also plans to seek means to make mineral physics data available to students for analysis in inquiry-driven investigations of Earth materials. Through DLESE, COMPRES will be able to disseminate data and data tools to researchers and educators on a national basis. This can be accomplished through their search engine, as well as the skills sessions and share fair at their meetings. Work on new tools, such as the following will be initiated:

- Interactive models of fault zones that enable users to specify a composition and vary temperature, pressure, stress and other conditions to observe resulting elastic strain and fault movement
- Interactive models that demonstrate the relationship of rheological properties of materials to plate motions under user-controlled pressures, temperatures and chemistry.
- An upgrade of tr660, which enables users to specify possible compositions of the mantle transition zone and observe graphs of resulting P and S-wave velocities for educational purposes, or utilize it as a template for developing a similar model that applies to the asthenosphere.

Budget:

Domestic travel	$4000
Supplies	2000
Equipment	4000
Total	$10,000

COMPRES Information Technology

Web Site

Internet technology presents COMPRES with numerous options for implementing organizational services for its members and for developing an attractive and useful interface with the educational and public communities. For the mineral physics community, it can provide a centralized location for information on important events, job openings, detailed information on the organization and management of COMPRES, and streamlined systems for finding information, applying for facilities time and registering for events. It projects our organization to the world and is one of the first impressions we will make on people who are not familiar with COMPRES and its work.

In order to realize the benefits that Internet technology makes possible, COMPRES has established a Web site at http://www/compres.stonybrook.edu, which is being maintained by Glenn Richard and Michael Vaughan. At present, the site provides the following information:

- A general overview of COMPRES
- COMPRES staff contact information
- Contact information for COMPRES the Facilities, Infrastructure Development and Executive Committees.
- Information about institutional and affiliate membership with application forms
- Results from experiments on crystal structure, equations of state, elasticity, phase relations and rheology.
- An overview of the currently funded Grand Challenges.
- Links to synchrotron and neutron source web sites, including instructions for applications for beam time.
- Links to information on past and upcoming meetings.
- Publication lists for GSECars and the Mineral Physics Institute
- The COMPRES Newsletter.
- Education and Outreach.

The COMPRES Central Office envisions the future role of the web site as that of an electronic Central Office that supports all the functionality necessary to enable the Consortium to serve the community's research and educational needs. This includes automation of the entire process needed to apply to perform an experiment at a facility and for reporting on the experiment afterwards as well as the sharing of experimental results.

Other Electronic Information Technology Services

- **List servers:** The initial list server is at. Additional lists will be established during the coming months.

- **People database:** Contact information for people involved in COMPRES. This will be made available online through a browser-based form
- **Threaded discussion board:** The Central Office is currently seeking means to offer threaded discussion bulletin board service to the COMPRES community.
- **Online Forms for meeting registration:** Plans are in place to offer online registration for meetings and workshops beginning in the summer of 2003.
- **Videoconferencing:** The Central Office plans to host a bridge to support videoconferenced meetings.

FACILITIES

Activity Report for Beamline X17-DAC at the National Synchrotron Light Source October 1, 2001 – September 30, 2002

Hemley and Mao

X17-DAC facility is located at the superconducting beamline X17 of National Synchrotron Light Source (NSLS). With the EAR00-04084 grant (EAR-IF, Feb 2001-Feb 2003), partial COMPRS support and CIW cost-sharing, X17C has been developed for diamond-anvil cell (DAC) high-pressure research, and X17B, for additional high-pressure laser-heating and low-temperature research, which extends beyond the spatial constraints of the X17C hutch.

Scientific highlight

- Stishovite is known to transform to orthorhombic $CaCl_2$-type structure at 50±3 GPa. The study of elastic instabilities is important for understanding phase transformations, and the stishovite–$CaCl_2$-type transition, which is driven by an instability of an elastic shear modulus, has attracted much attention. Princeton University Group used lattice strain measurements under nonhydrostatic compression in a diamond anvil cell to examine dense SiO_2 pressure up to 60 GPa. The ratio of differential stress to shear modulus t/G is 0.019(3) to 0.037(5) at pressure from 15 to 60 GPa. The ratio for octahedrally coordinated stishovite is lower by a factor of about 2 than observed in four-coordinated silicates. Using a theoretical model for the shear modulus, the differential stress of stishovite is found to be 4.5(1.5) GPa below 40 GPa and decrease sharply as the stishovite to $CaCl_2$-type phase transition boundary is approached. The differential stress then recovers rapidly to values of 5±2 GPa at 52-55 GPa in the $CaCl_2$-type phase. The inversion of measured lattice strains provides direct experimental evidence for softening of $C11$-$C12$.
- Compressibility (reciprocal of the bulk modulus) is an important physical property of a material. Strongly bonded materials have short interatomic distances and correspondingly strong repulsive interatomic forces, leading to high bulk moduli. The bulk modulus has been correlated empirically with the interstitial electron density , cohesive energy , and mechanical hardness . LLNL group studied compressibilities of 5d trasition metals Ru, Ir, and Os to 60 GPa by energy dispersive and angle dispersive x-ray diffraction. Using third order Birch- Murnaghan equation to fit experimental data yields the bulk modulus of Os, Ir and Ru as 462±12 GPa, 383±14 GPa and 348±18 GPa, respectively. They note that the bulk modulus of Os is higher than the diamond bulk modulus 443 GPa that has known the highest one. Their experimental results are also compared with the results obtained by first principles electronic structure calculations of equation of state for C, Os, Ir, Re, Ru and W. The transition metals compressibility decreases in the order W-Ru-Re-Ir-Os. This result provides impetus for a continued search for superhard materials, including transition metal carbides, nitrides, and oxides.

- Using a Mao-Bell type diamond anvil cell with an external Mo-wire resistance heater, Mark Frank et al (GL CIW) studied the equation of state and phase relation of MgO-H_2O system to 80 GPa. The diffraction pattern of ice VII, MgO and gold (internal pressure calibrant) were monitored during the experiment. The appearance of brucite would always proceed the disappearance of Ice VII diffraction lines. The data suggest that brucite formed from MgO as soon as fluid H_2O became available whereas Ice VII melted over a small temperature range (>50 K). These results will be used further to constrain the *PVT* properties of fluid H_2O at elevated pressures and temperatures by taking the pressure derivative of the Gibbs free energy difference between ice VII and fluid H_2O along the Ice VII melting curve. Comparison of these results suggests that previously reported equations of state of fluid H_2O underestimate the molar density of fluid H_2O at pressures > 35 GPa.
- D. Errandonea et al. studied phase transitions in scheelite ($CaWO_4$) by *in-situ* x-ray diffraction up to 65 GPa. At 12 GPa the high-pressure phase was characterized as a wolframite-type structure (SG: P2/c, No. 13). On pressure release $CaWO_4$ reverts to its ambient pressure scheelite structure (SG: I41/a, No. 88). The wolframite phase remains stable up to 40 GPa. Under further compression the diffraction peaks disappeared and a pronounced broad scattering corresponding to amorphous solids was observed. Clear peaks did not reappear simply by releasing pressure, until the quenched amorphous sample at low pressure was heated to 477 K for 2 hours, then a pattern corresponding to the ambient pressure phase is obtained. When the amorphized sample was heated to 477 K at 45 GPa, new diffraction lines were observed, indicating the existence of a new high-pressure phase stable at high temperature. This phase is found to be monoclinic (SG: C2/m, No. 12) which is 13.2% denser than the scheelite phase. This new high-pressure high-temperature phase gives additional support to the idea that amorphization takes place because of the frustration of a phase transition which is kinetically favored by the heating.
- B. Chen et al (Univ. of Missouri) have studied the particle-size dependence of the compressibility of nanocrystalline alumina (γ-Al_2O_3). X-ray diffraction data up to 60 GPa yield bulk moduli of B_{67}=238±3 GPa and B_{37}=172 ±3 GPa for γ-Al_2O_3 with particle sizes of 67 and 37 nm, respectively. Combined with the results of previous high-pressure x-ray studies of 20 and 6 nm nanocrystalline γ-Al_2O_3, the relation between compressibility and particle size is established. In addition, four new diffraction peaks are observed at 51 GPa and 56 GPa for γ-Al_2O_3 of 67 and 37 nm, respectively. These lines are indexed as a previously unknown high-pressure cubic phase.

Current Beamline Technique

1. High- pressure diamond anvil cell
2. Energy dispersive x-ray diffraction
3. Simultaneous high pressure and high temperature with external resistance heating
4. Quenching high temperature with YAG laser heating and high pressure
5. Simultaneous high pressure and low temperature with liquid-helium cryostat

6. X-ray microprobe with K-B focusing mirrors

7. Pressure calibration with ruby fluorescence spectroscopy

Research Program

X-ray diffraction studies of crystallography, melting, phase transitions, equations of state, elasticity, and rheology at high *P-T* conditions of the Earth's and planetary deep interiors.

Beamline upgrades in 10/1/01 to 9/30/02

1. Fixed-size cleanup slit was replaced by an adjustable slit at X17C

2. Cryogenic system for DAC for low temperature (liquid-nitrogen or liquid-helium temperature) and high-pressure x-ray diffraction.

3. Developed a PC program for single crystal high-pressure experiment.

Upgrade plans in the near future:

- Saggital Laue monochromator that provides high-intensity, high-energy (up to 60 keV) monochromator is available at X17C, but we do not have detector to utilize the powerful beam. We propose to acquire a CCD detector at a cost of **$190 K**.
- The X17B-DAC facility, currently sharing the X17B1 hutch with three other programs, will be permanently set up in X17B3 hutch in early 2003. DAC and multianvil (MA) research programs will have separate permanent hutches, thus eliminating the extensive moving and setup time and allowing sophisticated instrumentation such as integrated laser-heating system, Raman optical system and liquid-helium cryogenic system, that was prohibited by the temporal and spatial constraints previously in X17B1 hutch. VME based operating system with related hardware and software for operating the new X17B3 hutch needs **$140K.**
- Replacing the current multimode laser, which is the first prototype of double-side laser heating system acquired in 1996, with a YLF laser and improved optical system will need **$160K**.

Users (10/1/01 to 9/30/02)

Advanced Photon Source -- D. Errandonea, Y. Meng, M. Somayazulu

U. Alabama -- R. Paterson, N. Velisavievic, Y. Vohra

Caltech – I. Halevy, A. Papandrew, E. Üstündag

Carnegie Institution -- N. Boctor, Y. Fei, M. Frank, A. Goncharov, R. Hemley, Y. Ma, H. Mao, C. Sanloup, J. Shu, Y. Song, S. Stewart, V. Struzhkin, O. Tschauner, J. Xu

U. Chicago -- J. Lin, W. Mao

Columbia U.-- A. Kavner

Geochem. Inst. CAS-- M. Chen, X. Xie

U. Hawaii -- L. Ming

Lawrence Livermore Nat. Lab. – J. Akella, A. Baer, H. Cynn, B. Farber, W. Evans, J. Park

Los Alamos Nat. Lab. – G. Chesnut, D. He, K. Lokshin, J. Qian, B. Svile, Y. Zhao

U. Michigan-- W. Panero

U. Missouri – A. Chen, A. Goedken, U. Hemamala, K. Kalita, M. Kruger, S. Muthu

National Synchrontron Light Source -- Q. Guo, J. Hu, H. Liu, Z. Liu

Oak Ridge Nat. Lab. — C. Rawn, A. Rondinone

Princeton U. -- T. Duffy, S. Hugsresawat, S. Shieh, S. Speziale

Publications

B. Chen, D. Penwell, L. R. Benedetti, R. Jeanloz, and M. B. Kruger, Particle-size effect on the compressibility of nanocrystalline alumina , Phys Rev. B, 66, 144101, Oct. 4, 2002

M. Croft, I. Zakharchenko, Z. Zhong, Y. Gurlak, J. Hastings, J. Hu, R. Holtz, M. DaSilva and T. Tsakalakos, Strain field and scattered intensity profiling with energy dispersive x-ray scattering , Journal of Applied Physics, 92, 578-586, July 1, 2002

H. Cynn, J. Klepeis, C. Yoo, and D. Young, Osmium has the lowest experimentally determined compressibility , Phys. Rev. Lett. 138701—4, April 1, 2002

D. Errandonea, M. Somayazulu, and D. Hausermann $CaWO_4$: A new high-pressure and high-temperature phase , Phys. Stat. Sol. (b) 231, R1-R3, April, 8, 2002

Halevy, I., D. Dragoi, E. st ndag, A. F. Yue, E. H. Arredondo, J. Hu, and M. S. Somayazulu, The effect of pressure on the structure of $NiAl_2O_4$, J. Phys.: Condens. Matter 14, 10511-10516, 2002.

Haozhe Liu, Changqing Jin, and Yonghao Zhao, Pressure induced structural transitions in nanocrystalline grained selenium , Physica B, 315, 210-214, 2002

W. L. Mao, H. K. Mao, A. F. Goncharov, V. V. Struzhkin, Q. Guo, J. Hu, J. Shu, R. J. Hemley, M. Somayazulu, and Y. Zhao, Hydrogen clusters in clathrate hydrate , Science, 297, 2247-2249, Sept. 27, 2002.

S. Merkel, A. P. Jephcoat, J. Shu, H. K. Mao, P. Gillet, and R. J. Hemley, Equation of state, elasticity, and shear strength of pyrite under high pressure , Phys. Chem. Minerals, 29, 1-9, 2002.

N. Velisavljevic, G. N. Chesnut, Y. K. Vohra, S. T. Weir, Vince Malba, and J. Akella, Structural and electrical properties of beryllium metal to 66 GPa studies using designer diamond anvils , Phys. Rev. B, 65, 172107, May 8, 2002

J. R. Patterson, A. Kudryavtsev, and Y. K. Vohra, X-ray diffraction and nanoindentation studies of nano-crystalline graphite at high pressures , Applied Physics Letters, 81, no. 12, 16 Sept. 2002.

C. Sanloup, H. K Mao, R. Hemley, High pressure transformations of xenon hydrates , PNAS, 99, 25—28, January 8, 2002

C.˚Sanloup, R Hemley, H Mao. Evidence for xenon silicates at high pressure and temperature , Geophys. Res. Lett. , 29,1029. 2002

S. R. Shieh, and T. S. Duffy, Raman spectroscopy of $Co(OH)_2$ to 30 GPa: Implications for amorphization and structural frustration , Phys. Rev. B, 66, 134301, 30 Oct. , 2002

S. R. Shieh, and T. S. Duffy, Strength and elasticity of SiO2 across the stishovite—CaCl2-type structural phase boundary , Phys. Rev. Lett. , 89, 25507(4), 16 Dec. 2002.

X˚Xie, M Minitti, M Chen, H Mao, D Wang, J Shu, Y Fei. Natural high-pressure polymorph of merrillite in the shock veins of the Suizhou meteorite , Geochim. Cosmochim. Acta. 66, 2439-2444. 2002

NSLS Abstracts of 10/1/01-9/30/2

Abstract No. Ches0028, X-Ray Diffraction Study of Cerium, Zirconium, and Teflon at High Pressure, G Chesnut, X17C, N/A

Abstract No. Erra0059, Phase Transitions and Amorphization of CaWO4 at High Pressure, D Errandonea, M Somayazulu, D Hausermann, X17C, N/A

Abstract No. Liu0453, High pressure EDXD and Raman studies on nanoscale 3C-SiC, H Liu, J Hu, H Mao, X17C, X17B1

Abstract No. He0456, Bulk metallic glass gasket for high pressure, in situ x-ray diffraction , D He, Y Zhao, T Sheng, R Schwarz, J Qian, L Daemen, H Mao, J Hu, J Shu, J Xu, X17C, N/A

Abstract No. Song0490, High pressure structure and equation of state of nitrosonium nitrate from synchrotron x-ray diffraction, Y Song, M Somayazulu, H Mao, R Hemley, D Herschbach, X17C, N/A

Abstract No. Ma0506, Sysnthsis of Superhard Materials of Boron Nitride in A Rotation Diamond Anvil Cell, V Levitas, J Hashemi, W Mathis, M Holtz, Y Ma, X17C, X17B1

Abstract No. Ma0507, X-ray Diffraction Study of BN Superhard Material from A Rotation Diamond Anvil Cell, Y Ma, V Levitas, J Hashemi, W Mathis, M Holtz, X17C, X17B1

Abstract No. Fran0539, The melting curve, bulk modulus and thermal expansivity of Ice VII to 65 GPa., M Frank, Y Fei, J Hu, X17C, N/A

Abstract No. Fran0540, Constraining the equation of state of fluid H2O to 80 GPa using the MgO-H2O system., M Frank, Y Fei, J Hu, X17C, N/A

Abstract No. Kavn0559, Strength and Elasticity of Grossular Garnet at High Pressure, A Kavner, X17C, N/A

Abstract No. Hu0576, High-Pressure X-Ray Diffraction Studies of Novel Nanostructured Transparent Vitroceramic Medium, K Lipinska-Kalita , X17C, N/A

Summary of U2A activity in 2002

Hemley and Mao

U2A beamline is an integrated and dedicated facility with abilities to do measurement of far- to near-infrared spectra of a wide variety of materials from ambient to ultrahigh pressures at variable temperatures by coupling synchrotron infrared microspectroscopic techniques with new diamond-anvil cell methods. Users performed a wide variety of research using this facility during the period 10/01/2001-09/30/2002.

Scientific Highlights:

- A broad range of studies of hydrous minerals under pressure was conducted. First, far-IR absorption spectra of ($CaAl_2Si_2OH_2(H_2O)$)-Lawsonite and $Mg_6[SiO_{20}](OH)_4$-talc up to 30 GPa at room temperature have been measured and the vibrational frequencies combined with all other available data used to determine mode Grüneisen parameters for Lawsonite. These studies are important for understanding the phenomenon that substantially more water is current subducted into the Earth's mantle than is released volcanically. We found that the weighted average Grüneisen parameter is ~1.17 which is almost 20% below the value determined for the thermodynamic Grüneisen parameter (1.44), and indicate that anharmonic contributions to the thermal expansion and heat capacity are important.
- Synthetic Fe-bearing hydrous magnesium silicates at high temperature and pressure have been studied with combined single-crystal x-ray diffraction and infrared spectroscopy. We found theses silicates have two possible structures corresponding to $R\bar{3}m$ and $P6_3/mmc$.
- High-pressure infrared absorption measurements of a polysomatic series of hydrous magnesium silicates, humtes, have been performed up to 40 GPa at room temperature. Our results indicate that the crystal structures of both clinohumite and chondrodite are preserved up to 40 GPa. In the OH stretching region, all vibrational modes shift to higher frequency with pressure. The results are compared with the behavior of other hydrous magnesium silicates, where the OH vibrational frequency decreases with pressure, implying an increase in hydrogen bond strength as O-H…O distance shorten at high pressure.
- We performed a synchrotron far-IR study of moissanite (SiC) to assess the utility of these crystals as alternative of diamond anvils for high-pressure IR spectroscopic measurements. Although moissanite exhibits absorption in the far-IR around 240 cm^{-1}, the intensity of this band is too low to cause substantial interference with far-IR measurements, thus the anvils made of moissanite work very well for conducting high-pressure measurements in this spectral region.
- Superhard nanocrystallites embedded in a strong amorphous matrix are currently the most promising concept for the synthesis of novel superhard material in bulk form. It is well know that the achievable strength and hardness of engineering materials are usually orders-of-magnitude lower than the theoretical values due to deformation and fracture occur through the multiplication and propagation of vacancies and dislocations in crystalline materials. The nanocrystalline and amorphous could greatly

minimize these drawbacks and enhance their mechanical performance substantially. Synchrotron IR technique is used to characterize the synthesized B-C-N superhard ternary phase and to confirm the existence of the ternary BC_2N single phase based on the changes of vibrational spectra between starting materials and the synthesized composites. The IR spectra of BC_2N show clear new bands at 892, 1442, and 3233 cm^{-1}, which correspond to neither diamond nor cBN. These new modes are presumably C-N and B-C vibrational modes and this synchrotron IR absorption spectroscopic study provides evidence for the existence of the ternary BC_2N single phase.

- Nitrosonium nitrate ($NO^+NO_3^-$) was synthesized by laser heating of N_2O under high pressures in a diamond anvil cell. We performed a combined Raman/infrared synchrotron study of $NO^+NO_3^-$ to 32 GPa at room temperature and low-T Raman to14 GPa at temperature down to 80 K in the spectra region from 100 cm^{-1} to 2500 cm^{-1}. Major IR-active modes are identified in the lattice and intramolecular regions as well as their combinations. The pressure dependence of all IR active modes was examined at room temperature. We found that in the high-pressure region, $NO^+NO_3^-$ is a crystalline phase. Our room temperature and low-T Raman measures both agree with the previous studies of the transformation of N_2O_4 to $NO^+NO_3^-$. The low-T measurements provide a positive evidence of a phase transition for $NO^+NO_3^-$ near 5 GPa.

Beamline Changes/Upgrades:

Three IR microscope systems have been totally modified in order to improve the IR performance and accommodate to diverse requirements of the general users. Three vacuum and two dry nitrogen gas purged boxes have been adapted to the Bruker IFS66v FTIR spectrometer and covered whole optical components. This is crucial to remove the vapor absorption bands from infrared, especially for far-IR, spectra. The high efficient far-IR microscope system is the first one running in the vacuum condition at VUV ring of the NSLS. All IR microscope systems have been arranged user friendly and feasible to general users.

Ongoing and future Beamline Upgrade Plans:

U2A beamline has been built in an optical hutch in order to carry out both synchrotron IR and Raman/fluorescence experiments. However, the distance between beamline end station (spectrometer/microscope) and synchrotron source spot is about 15 meters, 3-5 times longer compared to other five IR beamlines at NSLS. As the synchrotron beam is collimated with a parabolic mirror (f=30in) and delivered with several flat mirrors (fl=1.5in) trough a thin vacuum pipe system (OD=2in), we found that beam divergence, scattering, and distortion becomes significant after the so-called collimated beam traveled more than 8 meters. As a result, IR performance at U2A is about two times worse in mid-IR and five times worse in far-IR ranges compared to other IR beamlines. Supported by COMPRES and NSLS, we are going to replace current small pipe system with a larger one (OD=4in). Based on Larry Carr’s calculations, far-IR throughput could be improved by factor of 5 for an upgraded U2A beamline (same length but with 2X larger optics). NSLS will commit labor, resource and a space close to the exit of U2 port.

As first step, we will install new pipe system at the end of February. Second step, we propose to build a side station close to the U2 port. The distance from the spot of synchrotron source to the IR system will be only about 3 meters. This will avoid any potential beam divergence and image distortion. Significant improvement of spatial resolution is expected (as U10B) and experiments at extremely high pressure with very small sample (5 micron) can be done at this side station.

Budget for the side station:

IR spectrometer: NEXUS™ 670 FT-IR spectrometer, $55, 410

Or: EQUINOX 55 FT-IR spectrometer, ~$60,000

Custom IR/Raman microscope system: ~$10,000

Optical table and vacuum pipe: ~$5000

Publications: 2001-2002

Hemley, R. J. and H. K. Mao, "Progress in cryocrystals to megabar pressures", J. Low Temp. Phys., **122**, 331-344, 2001.

Williams, Q., and R. J. Hemley, "Hydrogen in the deep Earth", Ann. Rev. Earth Planet. Sci., **29**, 365-418, 2001.

Nakano, S., R. J. Hemley, O. Tschauner and H. K. Mao, "High-pressure/high-temperature transformations of boron hydride", Ultra-High Pressure Research --Novel Process for New Materials, edited by M. Akaishi, T. Sekine, K. Takemura, T. Taniguchi, T. Kobayashi, K. Tajima and N. Aida, pp. 49-50, Nat. Inst. Res. Inorg. Materials, Tsukuba, Japan, 2001.

E. Gregoryanz, A. F. Goncharov, R. J. Hemley, and H. K. Mao, "High-pressure amorphous nitrogen", Phys. Rev. **B 64**, 052103-1, 2001.

M. Somayazulu, A. Madduri, A.F. Goncharov, O. Tschauner, P.F. McMillan, H.K. Mao, and R.J. Hemlley, "Novel broken symmetry phase from N_2O at high pressure and high temperatures", Phys. Rev. Lett. **87**,˚ 135504-1, 2001.

M. Koch-M ller , Y. Fei , E. Hauri and Z. Liu, "Location and quantitative analysis of OH in coesite", Phys. Chem. Minerals **28**, 693, 2001.

B. Chen, D.V.S. Muthu, Z. Liu, A.W. Sleight, and M.B. Kruger, "High-pressure Raman and infrared study of HfW_2O_8", Phys. Rev. **B 64**, 214111, 2001.

A.F. Goncharov, E. Gregoryanz, H. K. Mao, and R. J. Hemley, " Vibrational dynamics of solid molecular nitrogen to megabar pressures", Low Temp. Phys. **27**, 866, 2001.

M. Koch-Mueller, Y. Fei, A. Hofmeister and Z. Liu, "High-pressure IR-spectra and the thermodynamic properties of chloritoid", Am. Mineral. **87**, 609, 2002.

Z. Liu, J. Hu, H. Yang, H.K. Mao, and R.J. Hemley, "High-pressure synchrotron x-ray diffraction and infrared microspectroscopy: applications to dense hydrous phases", J. Phys: Condens. Matter **14**, 10641-10646, 2002.

D.D. Klug, M.Z. Zgierski, J.S. Tse, Z. Liu, J.R. Kincaid, K. Czarnecki and R.J. Hemley, "Doming modes and dynamics of model heme compounds", Proc. Nat. Acad. Sci. **99**, 1256, 2002.

S. Nakano, R.J. Hemley, E.A. Gregoryanz, A.F. Goncharov, and H.K. Mao, "Pressure-induced transformations of molecular boron hydride", J. Phys: Condens. Matter **14**, 10453-10456, 2002.

B. Chen, D.V.S. Muthu, Z. Liu, A.W. Sleight, and M.B. Kruger, "High-pressure optical study of HfW_2O_8", J. Phys: Condens. Matter **14**, 13911-13916, 2002.

Z. Liu, G.A. Lager, R.J. Hemley and N.L. Ross, "Synchrotron infrared spectroscopic study of OH-chondrodite and OH-clinohumite at high pressure", Am. Mineral, submitted.

H. Yang, C.T. Prewitt and Z. Liu, "Crystal structures and infrared spectra of two Fe-bearing hydrous magnesium silicates synthesized at high temperature and pressure", J. Mineral. Petro. Sci., in press.

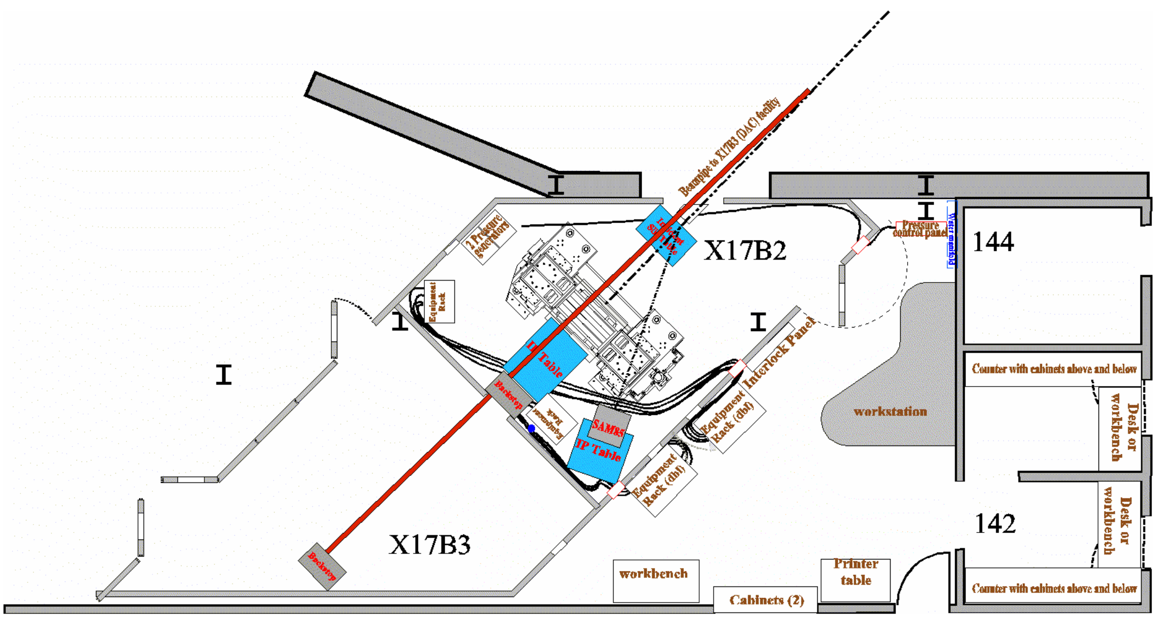
Y. Song, R.J. Hemley, Z. Liu, M. Somayazulu, H.K. Mao, and D.R. Herschbach, High-pressure stability, transformation and vibrational dynamics of nitrosonium nitrate from synchrotron infrared and Raman spectroscopy , J. Chem. Phys., submitted.

Multianvil Press Facility at the NSLS

Weidner and Vaughan

The 2002-2003 fiscal year, has seen major changes and upgrades to the LVP facility on the superconducting wiggler beamline at X17B. Two new radiation hutches were built downstream from X17B1. These hutches are dedicated to the Multianvil Press (X17B2) and the Diamond Anvil Cell (X17B2), which used to time-share the X17B1 hutch. The previous arrangement created great scheduling difficulties, because each group had to physically move in and out of the hutch at the beginning and end of its assigned time. Although the same beam will be shared between X17B1, X17B2, and X17B3, the removal of the need to move frequently enhances the scheduling flexibility and should increase the total beam time available to each group. The beam into the hutches will be split into two or three parts. One part will be enclosed in a beam pipe which will pass through the Multianvil Press in X17B2, providing simultaneous x-rays to the DAC facility in X17B3. We are also planning to put an in-hutch, single-bounce monochromator in the front of X17B2 to provide a secondary beam to a second press in the same hutch.

Proposed equipment purchases for year 2 include \$49,000 for a Deformation DIA and \$5500 toward the purchase of the pumping system.



Science Highlights

- ***Deformation experimental technique breakthrough and scientific research:*** A new high pressure deformation apparatus D-DIA has been married to the synchrotron x-ray source. The new apparatus has typical cubic-anvil geometry with independent control of top and bottom rams. Therefore under high pressure and temperature, the top and bottom ram can advance or retreat independently to deform the sample. In conjunction with synchrotron x-ray, the sample stress and strain can be measured by x-ray diffraction and radiograph imaging. Multiple x-ray diffractions along different direction relative to the principal stress axis yield an accurate measurement of stress in the sample to 10-100 MPa, and correlation of strain-mark images on the radiograph provides a precise strain measurement to $10^{-4} – 10^{-3}$. More remarkably, the technique avoids the uncertainty introduced during the deformation and friction modeling of the pistons in the high pressure cell. Therefore the deformation experiments can be carried out far beyond the pressure limits (3GPa) of conventional deformation apparatus. Deformation study on olivine at 10GPa has revealed revolutionary information about the activation volume of this mineral, which is significantly less than what people had believed. This result will have important impact on the understanding of mantle rheology.

- ***Understanding the strength of perovskite:*** Result of strength measurement of dominant lower mantle mineral, $Mg_{0.9}Fe_{0.1}SiO_3$, at high pressure and temperature has been published in Nature. The study indicates that perovskite is the strongest mineral among the mantle minerals. It also has unique characteristics during stress relaxation, insensitive to temperature. These findings help us to allocate the viscosity jump boundary in the mantle, to understand the deflection of the subduction slab at the boundary of transition zone and lower mantle, and to illustrate why on earthquakes start from lower mantle.
- ***Ultrasonic measurements of non-quenchable phases at high pressure and temperatures:*** Study of acoustic velocity in minerals has move forward to measure lower mantle minerals and non-quenchable phases at the beamline. Challenges have been made to obtain acoustic data of $MgSiO_3$ perovskite, Al doping $MgSiO_3$ perovskite, non-quenchable $CaSiO_3$ perovskite and high pressure pyroxene phase. Weakening effect of Al in perovskite has been confirmed. Data on the non-quenchable phases supply important information for understanding the tectonic structure of mantle.
- ***Melt property study at high pressure:*** A technique has been developed to measure the melt density at high pressures using x-ray radiograph and absorption simulation. Measurements have been carried out on FeS, a possible source of light element in the core. Melting volume of this material has been measured at 4GPa, and the data is used to calculate the slop of melting cure. Acoustic measurement of molten material at high pressure has also been pioneered at the beamline. Characteristic signals of P-wave and S-wave are observed when phase transition and melting happens.

System capabilities - Pressure and Temperature:

The DIA operates routinely to 10 GPa, and 2000K with cylindrical samples about 2 mm length and 1 mm diameter. This system uses cubic cell assemblies, but can operate to very large values of two theta.

The T-cup operates routinely to 20 GPa and 2000K with cylindrical samples about 1 mm length and 1 mm diameter. The cell is an octahedron and pressure is generated through the 6-8 geometry. 7/2 cells are used for the highest pressures, but 10/4 have been used as well with lower pressures (15 GPa) but more uniform temperatures and larger samples. Cubic boron nitride (CBN) anvils can be used to higher pressure (29

GPa current peak pressure). The CBN anvils are also transparent to x-rays, opening many new experimental possibilities.

All of these conditions are generated by a 200 ton hydraulic press.

x-ray characteristics

The super-conducting wiggler at X-17 provides a white x-ray flux to the sample that is at the useful limit of the system. The diffraction detection electronics, which are state-of-the-art, are saturated by the white beam. Thus, in energy dispersive mode, for the standard sample and assembly, there is no advantage from the x-ray point of view, to use a third generation source. In fact, the flux at X-17 is about an order of magnitude greater than that at the bending magnet port of APS while it is about a factor of 15 less than a white beam on the undulator port. In white mode, a complete diffraction spectra requires about 30 seconds for a fairly robust signal.

In monochromatic mode, angle dispersive data requires a longer time. With area detectors, which we use, 1 to 4 minutes is required to gather a robust spectra. The disadvantage of an area detector is that everything that is along the path of the x-ray contributes to the recorded signal. Only with point counting can one collimate the recorded signal. Point counting requires much longer – hours – for a robust spectra.

Types of experiments

Equation of state. P-V-T experiments are routine on this system. Pressure is monitored with a diffraction standard such as NaCl.

Phase transformations. X-ray diffraction provides a signature of the sample phase. Phase boundaries can be reversed *in situ* and observed during the process.

Crystal structure. With angle dispersive data, Rietveld refinements can be made to refine crystallographic properties.

Stress. We have developed metrics for defining the state of the deviatoric stress in the sample. With the CBN anvils, we can measure diffraction as a function of angle around the direct beam. This can yield differential stress with a precision of about 200 bars.

Imaging. We analyze the magnified image of the sample as projected on a fluorescence screen. This can define length of sample (1 micron accuracy), or strain (10^{-4} accuracy) by comparing two images. Falling spheres in a liquid can be used to define viscosity.

Absorption. X-ray absorption is sensitive to density. With reference materials, we can measure density of amorphous materials or liquids to about 1% accuracy.

Ultrasonics. We can measure simultaneously P-wave velocity, S-wave velocity, x-ray density (from diffraction), length, stress, along with pressure (from a reference) and temperature from the thermocouple. These can be conducted in the DIA or with the large truncation of the T-cup throughout their P, T range.

Time resolved. With 30 second time scale for collecting data, one can observe time dependence of phenomena. Kinetics of phase transformations, time dependence of order-disorder, rheology, diffusion, are all candidates of these experiments.

Budget for Multi-anvil facility at NSLS — Direct costs

Salaries + Fringe Benefits	Beamline Scientist and Machine/Electronics Technician	$134415
Equipment*		$55454
Supplies/Anvils		$40000
Office space NSLS (other)		$6500
Travel/lodging for users		$20000
Totals		$256369

*Primary equipment item for year 2 is a D-DIA (Deformation DIA) and items needed in order to occupy the new hutch including electronics, motors and drivers, and computers.

Users

Since October 1, 2002, the LVP program supported 33 experimental runs, performed by 13 different Principle Investigators. The results from these runs have not all been completely analyzed, but one paper has been submitted, eight are in process, and eight abstracts have been submitted to either the fall, 2002 or the spring, 2003 meeting of the American Geophysical Union. These are listed in the References:

Publications

Articles in Professional Journals

Chen, J., D. J. Weidner, and M. T. Vaughan, Strength of $Mg_{0.9}Fe_{0.1}SiO_3$ Perovskite at High Pressure and Temperature, Nature,419, 824-826 (2002)

Li., B., M. T. Vaughan, and D. J. Weidner, Direct length measurement using X-radiography for in-situ velocity measurements at high pressure and high temperature, Rev. Sci. Instr., 2002 (submitted)

Li, B., Sound Velocity of Mg_2SiO_4 Ringwoodite to 12 GPa, American. Mineralogist, 2002(submitted).

Li, B., K. Chen, J. Kung, R. C. Liebermann, D. J. Weidner, Ultrasonic velocity measurement using transfer function method, J. of Physics:Condensed Matter, 14, 11337-11342, 2002.

Li, L., D. J. Weidner, P. Raterron, J. Chen and M. T. Vaughan, Pressure dependence of olivine rheology, submitted to PEPI, Nov. 2002

Li, L., P. Raterron, D. J. Weidner, and J. Chen, olivine flow mechanism at 8 GPa, submitted to PEPI, July 2002.

Kung, J., B. Li, D. J. Weidner, J. Zhang, and R. C. Liebermann, Elasticity of $(Mg_{0.83},Fe_{0.17})O$ ferropericlase at high pressure: ultrasonic measurements in conjunction with X-radiation techniques, Earth and Planetary Science Letters, 203, 557-566, 2002.

Shieh, S. R., T. S. Duffy, and B. Li, Strength and elasticity of SiO_2 across the stishovite - $CaCl_2$ phase boundary, Physics Rev. Lett., 89 (25), DEC 16 2002.

Voronin, G. A., C. Pantea, T. W. Zerda, J. Zhang, L. Wang, and Y. Zhao (2002) In situ X-ray diffraction study of phase diagram of germanium at pressures up to 11 GPa and temperatures up to 950 K. Submitted to Physical Rev. B

Abstracts:

Chen, J., L. Li, D. J. Weidner, P. Raterron, and M. T. Vaughan, Plastic deformation of San Carlos olivine at high pressure, EOS. Trans. Amer. Geophys. Un. 83(47), F605, 2002.

Chen. J., L. Li, D. J. Weidner, and M. T. Vaughan, Rheology of fayalite: In situ stress and strain measurements at high pressure and temperature by using synchrotron X-rays, High Pressure Mineral Physics Seminar, August 25-30, 2002.

Chen, J., P. Raterron, D. J. Weidner, J. B. Parise, and M. T. Vaughan, Pseudomartensitic transformation at high pressures, XIX Congress of the International Union of Crystallography, August 6-15, 2002.

Chen, J., T. Inoue, H. Yurimoto, and D. Weidner, Water and 410-km Seismic Discontinuity: Experimental Results of Water Effect on Olivine-Wadsleyite Phase Transition in $(Mg,Fe)_2SiO_4$ System, 12th Annual VM Goldschmidt Conference, August 18-23, 2002.

Chen, J., L. Li, D. Weidner, and M.T. Vaughan, Rheology study of fayalite at high pressures and temperatures: In situ measurements of strain and stress by using synchrotron x-rays, EOS, Trans, AGU, 83 (19), S334, 2002.

Gwanmesia, G. D., J. Zhang, B. Li, K. Darling, J. Kung, D. Neuville, P. Raterron, S. Sullivan, and R. C. Liebermann Elastic Properties of Synthetic Pyrope $(Mg_3Al_2Si_3O_{12})$ to 9 GPa and 1000°C, abstract submitted to EGU, 2003, Nice

Kung, J., B. Li, T. Uchida and Y. Wang. Elasticity measurements of phase transition in pyroxene and the upper mantle reflectors. EOS. Trans. Amer. Geophys. Un., 83 (47), F977

Higo, Y., B. Li, R.C. Liebermann, T. Inoue, and T. Irifune, Elastic wave velocities of iron-bearing Ringwoodite $(Mg_{0.8}Fe_{0.2})_2SiO_4$ to 12GPa at room temperature, Eos Trans. AGU, 83(47), Fall Meet. Suppl., Abstract MR62B-1072, 2002

Li, B., J. Kung, R. C. Liebermann, M. T. Vaughan, D. J. Weidner, and T. Uchida, Advancement in acoustic velocity measurement in conjunction with synchrotron radiation at high pressure and temperature, EOS. Trans. Amer. Geophys. Un., 83.(19), S333.

Li, B., Ultrasonic velocity measurements in conjunction with synchrotron X-radiation at high pressure and high temperature: A great tool for probing the Earth s interior, Proceedings of Rigi Workshop on Crystal Physics, Switzerland, August, 2002.

Li, B., J. Kung, R. Liebermann, D. Weidner, T. Uchida, and Y Wang, Ultrasonic Velocity Measurements in Conjunction With Synchrotron Radiation to the Conditions of the Earth s Interior, Eos Trans. AGU, 83(47), Fall Meet. Suppl., Abstract U11A-0021, 2002

Li, B., Ultrasonic Measurements of Elasticity of Mantle Phases in Conjunction with Synchrotron Radiation, GeoForschungs Zentrum Potsdam, Germany, 2002.

Li, L., D. J. Weidner, J. Chen, and P. Raterron, Flow Mechanisms of San Carlos olivine at high pressure, EOS. Trans. Amer. Geophys. Un., 83 S341(2002)

Li, L., D. J. Weidner, J. Chen, and P. Raterron, Rheology constraints on deep earthquakes, EOS. Trans. Amer. Geophys. Un., 83, F1004(2002)

Li, L., D. J. Weidner, S. Mei, P. Raterron, J. Chen, and W. B. Durham, Search for Olivine Activation Volume, abstract submitted to EGU, 2003, Nice. (Invited talk)

Liebermann, R. C., B. Li, J. Kung, F. Decremps, D. J. Weidner, and M. T. Vaughan, Advances in studies of the elasticity of mantle minerals at simultaneous high pressures and temperatures in conjunction with synchrotron X-radiation techniques, 6th High-Pressure Mineral Physics Seminar, Verbania, Italy, August 26-31.

Voronin, G. A., C. Pantea, T. W. Zerda, J. Zhang, L. Wang, and Y. Zhao Study of phase diagrams of silicon and germanium at pressures up to 16 GPa and temperatures up to 1100 K by in situ X-ray diffraction method. American Physical Society Meeting. (2003)

Wang, L., P. Raterron, J. Chen, D. J. Weidner, M. T. Vaughan, Y. Wang, and T. Uchida (2002) Kinetics of the phase transformation of olivine to its high-pressure polymorphs: implication for subduction zone processes. 6th High Pressure Mineral Physics Seminar (Verbania, Italy).

Weidner, D. J., P. Raterron, J. Chen, and L Li, Rheological Limitations on Deep Earthquakes, M04, EOS. Trans. Amer. Geophys. Un., 83, S338(2002)

Mei, S., L. Li, W. B. Durham, Y. Wang, T. Uchita, I. C. Getting, M. T. Vaughan, D. J. Weidner, and P. Burnley, Preliminary deformation results to 12 GPa pressure using Deformation -DIA, EOS. Trans. Amer. Geophys. Un., 83 603 (2002)

Mei, S., W. B. Durham, L. Li, D. J. Weidner, and Y. Wang, Experimental studies on deformation at high pressures using the Deformation-DIA, abstract submitted to EGU, 2003, Nice. (Invited talk)

Mistler, G. W., M. Ishikawa, and B. Li, Elastic Wave velocity measurements on mantle peridotite at high pressure and temperature, Eos Trans. AGU, 83(47), Fall Meet. Suppl., Abstract MR72B-1045, 2002

Raterron, P., L. Li, D. J. Weidner, and J. Chen: Olivine Deformation in the Mantle: Mechanism and Rheological law, abstract submitted to EGU, 2003.

Vaughan, M. T., L. Li, D.J. Weidner, and J. Chen: Two-dimensional strain measurements using X-rays in high pressure - high temperature Rheology experiments, High Pressure Mineral Physics Seminar, August 25-30, 2002.

Reports in the NSLS Activity Report for 2002

Chen, J., L Li, D. J. Weidner, and M. T. Vaughan: In situ stress and strain measurements at high pressure and temperature

Chen, J., D. J. Weidner, and M. T. Vaughan: Density of FeS at High Pressure

Chen, J., M. T. Vaughan, D. J. Weidner, and L. Li: Macroscopic stress measurements by X-ray diffraction

Chen, J., P. Raterron, D. J. Weidner, and M. T. Vaughan: Observation of pressure-induced nanostructure formation

Chen, J. and J. Zhang: X-ray diffraction study of $NaAl_2O_4$ at high pressure

Solozhenko, V., Solozhenko, E., P. Zinin, L. Ming, J. Chen, and J. B. Parise: Equation of state and phase stability of turbostratic carbon nitride

Li, L., D Weidner, P Raterron, J Chen, M Vaughan: Plastic deformation of San Carlos olivine at high pressure

Ishikawa, M. And B. Li: Elastic wave velocity of mantle peridotite at high P and T

Mei, S., Y. Wang, T. Uchida, D. J. Weidner, L. Li, M. T. Vaughan, J. Chen, and I. C. Getting: The deformation-DIA: Deformation of materials to 15 GPa pressure

Kung, J., B. Li, and M. T. Vaughan: Elasticity of mantle composition (Mg,Fe)O at high pressure and temperature

Zhang, J.: The olivine-wadsleyite phase transformation in mantle peridotite

Zhang, J. and M. Zelinkas: Further studies of phase relations at high pressure and temperature in $LiAl_2SiO_4$ eucryptite

Martin, C., J. Kung, D. J. Weidner, and J. P. Parise: Elasticity of $NaMgF_3$ at high pressure and temperature

Voronin, G., C. Pantea, T. Zerda, J. Zhang, L. Wang, and Y. Zhao: In situ X-ray diffraction study of phase diagram of germanium at pressures up to 11 GPa and temperatures up to 950 K

Wang, L., J. Chen, D. J. Weidner, and M. T. Vaughan: Kinetics of the phase transformation of olivine to wadsleyite: a comparison study between natural Fe-bearing olivine and synthetic forsterite

Ishikawa, M., M., J. Kung, and B. Li: Elastic wave velocity of mantle peridotite

Wang, L., J. Zhang, and D. J. Weidner: In situ X-ray determination of the phase boundary between forsterite and wadsleyite

Annual Report of West Coast COMPRES Effort

Raymond Jeanloz, January, 2003

The first year s effort for the West Coast component of COMPRES have focused on activities at Lawrence Berkeley National Laboratory s Advanced Light Source (LBNL ALS), as was proposed. The reason for this emphasis, with correspondingly little effort at the Stanford Synchrotron Radiation Laboratory (SSRL), was that the primary beamline used at SSRL (10-2) suffered two breakdowns that put it out of commission for most of the past year. Also, the SPEAR-III upgrade is expected to make SSRL unavailable for much of the coming year. Therefore, initial investments on behalf of the NSF-funded Earth science high-pressure community have been made primarily at ALS.

Current Status of ALS Activity

Approval and funding have been secured to establish a superbend beamline (12.2.2) dedicated to high-pressure research at ALS. Construction is underway, and initial experiments are expected in 2003 (Fig. 1). COMPRES is part of a larger effort centered at ALS, the California High-Pressure Observatory (CALIPSO), which has secured most of its funding from the DOE. The entire cost of the beamline has not yet been raised, so further funding is currently being sought. Based on a recent agreement reached with ALS, the level of access will ultimately be proportional to the fraction of annual support being provided. The amount of beamtime directly available to COMPRES will therefore depend on the amount of funding provided by COMPRES, relative to other funding sources. Specifically, the $175 K of support currently being provided translates to nearly 20% access to the beamtime for COMPRES, excluding the 25% made available by ALS to general users (for which COMPRES members also have access).

Dedication of a second superbend beamline to high-pressure research has been approved by the ALS Scientific Advisory Committee, but the status of such a develop is uncertain until funding has been raised.

The single most important action this past year was the appointment of Dr. Simon Clark in a permanent staff member of the ALS. He will serve as the Manager for the CALIPSO Project, and will therefore be the lead contact person for COMPRES at the ALS. His position represents a significant contribution by LBNL to the high-pressure research effort at ALS.

An offer has been made to an experienced beamline scientist, a position to be fully supported by COMPRES. We expect negotiations to come to closure within the next 4 weeks. Another position supported by COMPRES will then be filled, within the next 6-8 weeks, for an associate to work under the beamline scientist. In addition, we expect to make one other staff appointment this year, to be funded from other sources.

Another significant contribution made by the ALS to the COMPRES community is the commitment of 20% of the time on beamline 11.3 (until 12.2.2 becomes available) for X-ray absorption spectroscopy and diffraction, and 20% of the time on beamline 1.4 for infrared spectroscopy. This has just been agreed to (01/03) and, amounting to about 40 days on each beamline per year, is a major increase in access for the high-pressure Earth-science community in the United States. Requests for this beamtime will be reviewed by the new COMPRES Committee within CALIPSO, augmented as needed by technical commentary from relevant ALS beamline scientists (e.g., to ensure feasibility of experiments and optimal use of facilities).

An interim COMPRES Steering Committee (Q. Williams, Chair) had been established in order to initiate the project’s first year of effort on the West Coast. This has now been superceded by a new COMPRES Committee, which includes representation from Arizona, California, Hawaii and Washington (see listing below). It is chaired by Simon Clark, and serves to make recommendations both to the ALS and the local PI for COMPRES (Raymond Jeanloz). It will also overlap with the broader Scientific Advisory Committee for CALIPSO.

Workshops

A workshop held on December 11, 2002 (immediately after the Fall AGU Meeting), was attended by over 50 participants from the United States and abroad. The program is shown below, and talks can be viewed at http://www-library.lbl.gov/teid/tmVideo/aboutus/VideoDefault.htm (select Online Presentations). The objective of introducing the ALS high-pressure effort to the COMPRES community was therefore well met. One of the major recommendations that emerged from this workshop is for the ALS to vigorously develop a capability for single-crystal X-ray diffraction at high pressures (and variable temperatures), as well as to enhance its current capability in infrared absorption and reflectance spectroscopy at high pressures. Both of these efforts are viewed as highly complementary to existing programs at NSLS, APS and CHESS, and would significantly enhance the high-pressure Earth-science research community's capabilities.

The development of single-crystal capability represents a significant enhancement of the program originally proposed for COMPRES, so will require additional funding if the community considers it of high enough priority to implement in the near future.

A laser-heating workshop was also announced, with the dual objective of helping i) the research community identify key research to be pursued in order to advance research with the laser-heated diamond-cell, and ii) the ALS identify the optimal hardware (and configuration) it should put in place for such research. The workshop is scheduled for Saturday, February 22, 2003, and will be followed up by a workshop to be sponsored by GSECARS at the APS.

Current-Year and Future Plan

In addition to the development of the new high-pressure x-ray beamline, and providing access to existing x-ray and infrared beamlines, we plan to establish a laser-heating laboratory during 2003. The 02/03 workshop will help define the characteristics of the laser-heating system to be installed for use by the research community at large. In addition, the ALS has purchased about $100 K of equipment, including laser, monochromator and optics, that will allow development work and ongoing research. Based on this development work and the community's input, we plan to use COMPRES funds to purchase laser-heating equipment for use by the community. It would be installed in the x-ray hutch in the second year of the COMPRES grant, and designed to be remotely operated with as much automation as possible. In order to implement this plan, we have arranged for Prof. Mike Walter (Okayama University) to spend 2003 (full calendar year) helping us establish this laser-heating capability. His support comes from other funds, and represents a major contribution to the COMPRES community's infrastructure.

Starting in the second year of the grant, we anticipate detailed planning efforts to determine the best use of SSRL by the COMPRES community. Initial planning suggests that little built-in infrastructure may be required at beamlines in the near future, but instead that portable and user-friendly equipment be placed at SSRL for general use by the high-pressure community (*e.g.*, ruby fluorescence and sample-loading capability). This plan needs to be updated in the light of SSRL's developing plans for the allocation of beamlines.

Looking further into the future, we anticipate initial planning for laser-spectroscopy capability at the ALS, including both Raman and Brillouin. This will be possible because of the arrival of Sergio Speziale (upon completion of his Ph. D.), who has considerable experience in this domain. His position is also supported by other funds, again representing a significant contribution to the COMPRES program.

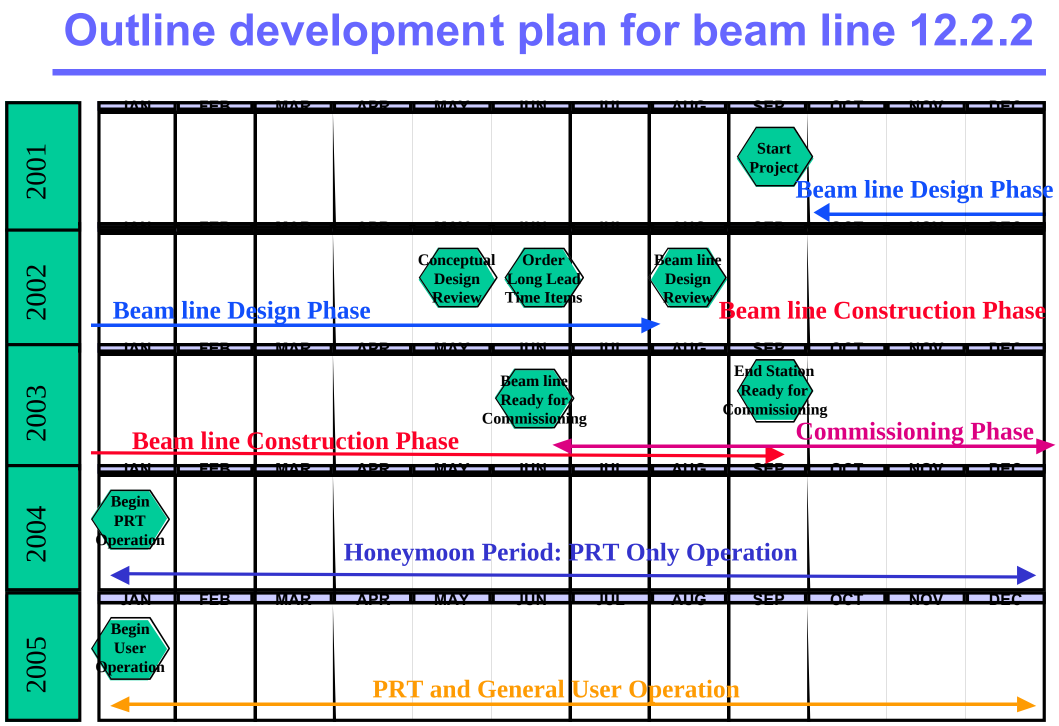


Figure 1

West Coast COMPRES Committee

Simon Clark, Chairman	Advanced Light Source
Abby Kavner	UCLA
Raymond Jeanloz (*ex officio*)	UC Berkeley
Elise Knittle	UC Santa Cruz
Michael Martin (Beamline Liaison)	Advanced Light Source
Matt McCluskey	University of Washington
Li-ching Ming	University of Hawaii
Tom Sharpe	Arizona State University
Quentin Williams	UC Santa Cruz

COMPRES at the ALS: 11 Dec., 20002 Workshop

Introduction (Quentin Williams)

8:50-9:00	Welcome	Daniel Chemla
9:00-9:20	High-pressure science: a world perspective	Ho-kwang Mao
9:20-9:40	CALIPSO: Overview	Raymond Jeanloz
9:40-10:10	Plans for High-Pressure beam lines at the ALS	Simon Clark
10:10-10:20	Discussion and coffee	

Session I: Laser Heated dac, powder diffraction (Raymond Jeanloz)

10:20-10:50	Overview of laser heated dac work	Dion Heinz
10:50-11:20	High-P laser heating at Spring-8	Kei Hirose
11:20-11:30	Discussion	

Session II: Multianvil, powder diffraction (Yanbin Wang)

11:30-12:00	The challenges for Multianvil research	Don Weidner
12:00-12:30	Experiences of Multi-anvil work at DL?	Dave Walker
12:30-12:40	Discussion	
12:40-1:40	Sandwich lunch and tours of the ALS facilities	

Session III: Infra-red (Alexander Goncharov)

1:40-2:10	Overview of high-pressure infrared research	Russ Hemley
2:10-2:30	Facilities for IR at the ALS from synchrotron to super radiant	Michael Martin
2:30-2:40	Discussion	

Session IV: Single Crystal Diffraction (Mark Rivers)

2:40-3:10	High-Pressure single crystal diffraction	Joe Smyth
3:10-3:30	Single-crystal facility at the ALS	Howard Padmore
3:30-3:40	Discussion	
3:40-4:00	Coffee	

Conclusions and close

COMPRES Neutrons: Year 1 Report

Activities: July – December 2002

- Construction of website (http://www.crystal.vt.edu/compres/) that includes information on COMPRES, an educational component describing neutron scattering and potential applications, links to neutron facilities around world, existing projects such as HIPPO and SNAP, and application information for Earth scientists requiring support to travel to neutron facilities to perform experiments.
- Contributions to travel/subsistence of U.S.-based earth scientists doing neutron scattering experiments at facilities in the U.S. and abroad (funds became available in 8/02)

  Sponsored scientists:

  R.J. Angel (Va. Tech), P. Chupas (Stony Brook), A. Celestian (Stony Brook)
- Organized special session on "Neutron Scattering in Earth Sciences" at the 2002 Fall AGU Meeting (Dec. 6-10, 2002; N.L. Ross and J.B. Parise, co-convenors). The session was well attended (standing room only) and helped raise the profile of neutron scattering.

Support provided for invited speakers: S.A.T. Redfern (Univ. Cambridge), M.D. Welch (NHM, London), B. Winkler (U. Frankfurt) and participating students: W. Mao (U. Chicago).

- Joint Institute of Neutron Scattering Workshop: **Neutrons In Solid State Chemistry and the Earth Sciences Today and Tomorrow,** (March 12-16,2003) a **"**hands-on" workshop aimed at graduate students who may apply for support through COMPRES.

Advertised at AGU and attracted much interest. Anticipate supporting ~10 graduate students.

Future Activities:

Continue financial assistance for U.S.-based Earth scientists doing neutron scattering experiments at facilities in the U.S. and abroad.

- Organize short course on "Neutron Scattering in Earth Science" (possibly with Mineralogical Society of America accompanied by a volume in the Reviews in Mineralogy Series).
- Support graduate student "internships" at both laboratory facilities (for training, synthesis of samples, etc.) and at neutron facilities.
- Hire a Postdoctoral Research Associate who will utilize existing high-pressure neutron facilities in order to test and modify high-P cell designs, optimize detectors, carry out comparisons between neutron facilities, develop calibrations at high P and T and optimize data collections at high P and T (Years 3-5).

INFRASTRUCTURE DEVELOPMENT PROJECTS

A Brillouin spectrometer at GSECARS, APS

This project involves the design and construction of a Brillouin spectrometer at GeoSoilEnviroCARS (GSECARS) at the Advanced Photon source, Argonne National Laboratory. The final product will be a Brillouin spectrometer which can be used with synchrotron x-radiation for simultaneous measurements of sound velocities (by Brillouin) and density (by x-rays). Ultimately we intend to do such simulatneous measurements at high pressures (using diamond anvil cells) and high temperatures.

In year one of this project, we have been primarily involved with design aspects of the system, preparing the lab space at University of Illinois, making minor renovations, and ordering the main pieces of equipment needed to build the Brillouin spectrometer. We have ordered a six-pass Sandercock interferometer and made partial payment. This instrument is the heart of the Brillouin system and also the equipment which has the longest required lead time for delivery. We have also ordered some optics and done some designing of the new system. It will have a smaller optical table and footprint than the system we use here. Space has been identified for the new system (which will be built at UIUC and moved to APS) and renovations are arranged at UIUC to make space for the new Brillouin system, to get additional power installed, etc. In short, we have been moving ahead with designing, and acquiring the needed equipment, parts, and space for the new system. This is a big part of the job.

The second year of the project will involve continued equipment acquisition and the actual construction of the Brillouin spectrometer. We will experiment with a variety of configurations for the optical system in an attempt to find the optimize it for operation at GSECARS. Space is a main concern because it is limited inside the x-ray hutch. While the system must be economical spacewise, we cannot compromise on the quality of the external optical system (that is, the optics outside of the interferometer housing). We will also do extensive experimentation with motorizing the various adjustments that must be made during a Brillouin experiment so that they can be performed remotely outside of a synchrotron hutch. For achieving the final design we may enlist the services of John Sandercock, the designer or the interferometer, as a consultant.

A suggestion that was made by the infrastructure Development Committee is to hold a workshop at GSECARS on the practical aspects of Brillouin scattering and how to operate the system. This workshop would be held when the project is completed or near completion. We feel this is an excellent idea for building an initial user base for the spectrometer, and we will likely request funds for this purpose in Year 3 or 4 of COMPRES.

In order to meet the COMPRES budget target for the first year of the consortium, $60K was deferred from the first year budget for this project. It was not possible to restore these funds during the second year. As a result, it will not be possible to buy the solid state laser which will be used as a light source in the completed system at GSECARS. This problem can be overcome during the initial construction phase at UIUC, because we can likely share the laser being used for our ongoing research. However, completion of the project and installation of the spectrometer at APS will not be possible until the deferred funds are received, probably in year 3 of COMPRES.

Laser Heated Diamond Cell Facility at the Advanced Photon Source

Thomas Duffy, Guoyin Shen, and Dion Heinz, Goals

The overall goals of the project have not changed. They are:

1) development of a dedicated CO_2 laser heating system to be installed at the GSECARS sector of the APS;

2) a comprehensive interlaboratory effort to characterize and eliminate major error sources in laser heating;

3) micro-engineering of the diamond cell sample enviroment;

4) pioneering efforts to develop a new generation of heating methods;

5) integration of laser heating technology with new x-ray techniques including radial diffraction, x-ray spectroscopy, inelastic scattering, etc.

Progress

1. Modeling. We have begun performing finite element computer modeling of heat flow in the laser-heated diamond cell using the software package SEPRAN. This will be an important aid in testing some of our development ideas and will also enable us to answer some interesting scientific questions. The work that we are doing will build on previous semi-analytical models (e.g. Panero and Jeanloz, 2001; Bodea and Jeanloz, 1989) and finite element calculations (Dewaele et al., 1998; Morishima and Yusa, 1998). An example of the calculation results is shown below (Figure 1). These preliminary results show that we can reproduce the calculations of Dewaele et al. (1998). We expect to go well beyond these earlier calculations, however, and effectively test a wide range of parameters and the effects of changing such factors as anvil design, gasket design, use of a microfurnace, etc. These calculations were carried out by Dr. Boris Kiefer, a post-doctoral fellow in Duffy's research group.

2. Hardware Development. We are continuing to develop design ideas for the system. We are investigating CO_2 laser systems (CW and pulsed) to see which will best meet our goals. Dion Heinz and Tom Duffy plan to set-up visits to several CO_2 heating laboratories around the country in the Winter/Spring of 2003 including Chicago, Illinois, and the Geophysical Laboratory. No actual equipment purchases have been made as yet, however. Due to the first year budget cut, we have decided to stockpile our resources to allow for major equipment purchases in the second year.

3. Personnel. Dr. Sean Shieh, a post-doctoral Associate in Duffy's group had been lined up to work on the laser heating development project. However, very recently, Dr. Shieh has accepted a position as an assistant professor at National Cheng Kung University in Taiwan. This is a set-back as we will now need to seek a new post-doc for this position. We plan to run ads for this in the near future. Also, Dr. Shieh is planning to spend summers in Princeton (his family will remain here) and will be able to work on the project during the summer and break periods. I expect he will dedicate 50% of his time to this project during these times.

4. Community Involvement. As the design program evolves and solidifies over the next six months, we plan to consult extensively with community members who have expressed interest in this project. At a later stage, we plan to hold a workshop/meeting on laser heating at the Advanced Photon Source.

Budget

Our first year budget allotment was $113,000 out of a requested $180,000. It is our expectation that this shortfall will be made up in years 2 and 3. There are no changes in our budget needs.

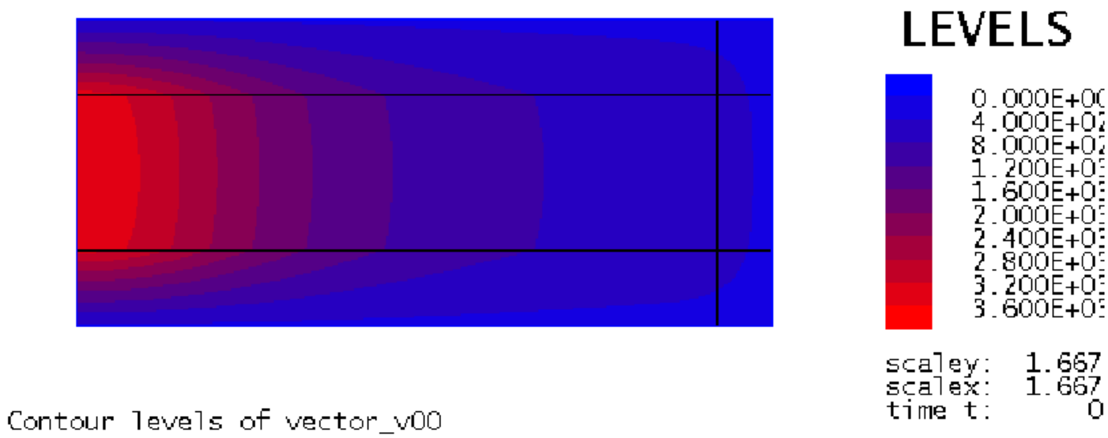


Figure 1. Temperature distribution in laser heated diamond cell from finite element calculations. The sample radius is 90 microns and the thickness is 40 microns. The laser beam is Gaussian with power of 19 Watts. The insulation medium is argon.

References

Bodea, S., and R. Jeanloz, J. Appl. Phys., 65, 4688, 1989.

Dewaele et al., Res. Sci. Instrum., 69, 2421, 1998.

Morishima and Yusa, J. Appl. Phys., 83, 4572, 1998.

Panero, W. R., and R. Jeanloz, Rev. Sci. Instrum., 72, 1306, 2001.

COMPRES- Pressure Calibration at High Temperatures (Y. Fei)

(1) The goals of the project

a) Establishing a self-consistent pressure scale at high temperature

b) Establishment of a Spectroscopically-based Pressure Scale at High Temperature

c) Establishing an absolute pressure scale at high temperature

2) Progress and future plan

Our strategy to solve the pressure scale problem at high temperature is first to establish a self-consistent pressure scale through in situ X-ray diffraction measurements of the primary pressure standards such as NaCl, MgO, Au, Pt, W, Mo, Cu, Ag, and Pd in a multi-anvil apparatus up to 28 GPa and 2300 K and in an externally-heated diamond-anvil cell up to 100 GPa and 1100 K. We have directly compared the MgO, Au, and Pt scales using the multi-anvil apparatus installed at a beamline of the SPring-8 synchrotron facility up to 28 GPa and over a temperature range of 1473-2273 K. Our results demonstrated that the calculated pressures can vary as large as 4 GPa at 2173 K depending on the pressure standards and the thermal equations of state used in the calculations. The Au scale of *Jamieson et al*. [1982] predicted the highest pressure whereas the Pt scale of *Jamieson et al*. [1982] predicted the lowest pressure. We are in favor of using the MgO scale of *Speziale et al*. [2001] as the pressure scale for consistency and inter-laboratory comparison. The MgO scale of *Speziale et al*. [2001] needs to be further verified by redundant equation-of-state measurements (e.g., simultaneous X-ray diffraction and acoustic measurements).

In collaboration with the Berkeley group (Sang-Heon Shim and Raymond Jeanloz), we have conducted one set of experiments to compare the MgO, Au, and Pt pressure scales at APS using an externally-heated diamond-anvil cell. More experiments are planed before June 2003.

For the second year, we will continue to perform in-situ X-ray diffraction measurements of the commonly used pressure standards and provide an critical evaluation of these pressure scales. We will establish a spectroscopically-based pressure scale at high temperature. Isotopic pure ^{13}C diamond and cubic BN will be used as pressure calibrants. The Raman shifts of ^{13}C diamond and cubic BN as a function of pressure (up to 100 GPa) at high temperatures (up to 1173 K) will be established by simultaneous Raman spectroscopic measurements and X-ray diffraction.

3) Community involvement

We will actively collaborate with people who are interested in the pressure scale problem and can provide unique expertise to enhance our program. We are already collaborating with the Berkeley group on in-situ X-ray diffraction measurements in the diamond anvil cell. We are seeking collaborators on redundant equation-of-state measurements to establish an absolute pressure scale at high temperature based on MgO as an internal standard. We plan to organize a workshop to assess our progress and the needs of our community. In order to establish a mutually consistent pressure scale, we will need continuous feedback from our colleagues who will be the users of this proposed scale.

4) Budget outline for year 2

Personnel	
Prodoctoral fellow (12 months)	
Plus Fringe Benefits	$55,255
Equipment	
Diamond anvils ($2,900/pair)	$5,800
Materials and Supplies	
Carbide, gasket, Ar-H_2 gas, etc.	$8,000
Travel	
Trips to APS ($1,000/trip)	$6,000
Trips to SPring-8 ($1,500/trip)	$4,500
One trip to national meeting	$1,500
Total Direct Costs	$81,055
TMDC	*$75,255*
Total Indirect Costs (59%TMDC)	$44,401
Total Requested	$125,456

Budget Justification

Salary support. Synchrotron experiments require team work. I typically bring two postdoctoral associates to synchrotron facility for a scheduled experiment. Participation of postdoctoral associate in this project is crucial. I request 12-month salary per year for a postdoctoral associate.

Travel In situ/ex situ X-ray measurements will be carried out by using the synchrotron diffraction techniques at SPring-8 (Japan) and APS (Chicago). I request $4,500 per year ($1,500 per scientist) for travel to SPring-8 (Japan). The travel expenses are substantially low because SPring-8 covers our domestic travel expenses within Japan. The multi-anvil system in synchrotron facility is well established at SPring-8. APS is building a similar multi-anvil system. It has a well-supported facility for diamond cell experiments. We will perform the proposed diamond cell experiments and possibly some of the multi-anvil experiments at APS. I request $6,000 for two trips to APS for three scientists. Funds ($1500/year) are also requested to attend one national conference each year.

Equipment. The externally-heated high-temperature diamond-anvil cell is the principal tool for the proposed diamond cell experiments at high pressure and temperature. Replacement of diamonds is required in some of the experiments. Funds for two pairs of diamond anvils each year ($2,900/pair) are requested.

Miscellaneous. Funds ($8,000/year) are requested for miscellaneous supplies. These include tungsten carbide cubes and cell assemblies for the multi-anvil experiments, Re gaskets, capsules, and furnace materials, Ar-1%H_2 gas, thermocouple materials, and other materials related to the high-temperature set up. For experiments at pressures above 23 GPa, tungsten carbide consumption is relatively high (typical cost for a finished Toshiba carbide cube is about $120 and eight cubes is required for each experiment).

Absolute Pressure and Temperature Calibration

Ivan C. Getting

This component of the COMPRES Infrastructure Development program seeks to establish and offer to the community accurate temperature and pressure scales based on NIST traceable measurements and sound metrological practice.

Temperature

Temperature measurements have proven very difficult in high pressure environments. Decades of consideration have failed to yield realistic calibration for thermocouples as used at high pressure. Temperature can be measured accurately in a high pressure environment by Johnson noise thermometry, however. Johnson noise is the very small voltage noise generated across any resistor at temperature above absolute zero. The mean square noise voltage across a resistor R is $\langle E^2 \rangle = 4kBRT$, where k is Boltzmann's constant, T is the absolute temperature, and B is the electrical bandwidth in which the noise voltage is observed. This Gaussian-distributed, random fluctuating voltage has zero mean, but a definite quadratic-average value. All of the effects of pressure, strain, and any chemical reactions on the resistor sensor are cast into the resistance term. The resistance is measured separately for each reading thereby accounting for all such effects. Very subtle electrical measurements are required to determine the value of $\langle E^2 \rangle$. John Hall, an invaluable metrologist colleague at the University of Colorado, and I have constructed all the circuits for an automated Johnson noise thermometer to be used at high pressure. In the last few months these components have been assembled and tested successfully. A new computer sufficient to the task of sampling and reducing this data has been acquired. Computer code and instrument networking have just been complete.

The value of $\langle E^2 \rangle$ depends on the product RT. We are now making fully automated readings of all the relevant electrical parameters simulating high temperatures by changing resistance. One million samples of this random noise requires 10 seconds sampling time and results in a temperature resolution of better than 2 K at 1000 K. The time to sweep through a complete set of all the required measurements is 15 seconds. Higher resolution is achieved by additional sampling. A one atmosphere furnace is under construction. This furnace will be used in the next few weeks to begin testing of the Johnson noise thermometer at high temperature.

It would be very difficult to make an absolute Johnson noise thermometer in and of itself due to slight non-linearity and ambiguous definition of band width in the electronic amplifiers involved. This thermometer will be calibrated against NIST traceable thermocouples at one atmosphere to establish its intrinsic calibration. An high quality, NIST traceable digital voltmeter and NIST traceable thermocouples for this purpose are on hand. The final result will be NIST traceable determinations of absolute temperature at high pressure. Measurements of the effect of hydrostatic pressure will be made on several thermocouple types in the gas piston cylinder apparatus. This does not directly solve the problem of temperature measurements in the various solid medium devices, however, as thermocouples are additionally affected by strain and chemical reactions in those instruments. The beauty of the Johnson noise thermometer is that it can be applied in any large volume high pressure device to the maximum pressure and temperature attainable. All that is required is a resistor and four electrical leads and appropriate electrical insulation between the critical components. Using thermoelements for the leads yields a direct calibration of thermocouples. The major effort in the next two years will be concentrated on this Johnson noise thermometry.

Pressure

Pressure refers to uniform, isotropic, compressive stress. The gas piston cylinder apparatus at Colorado offers unique capability in producing very nearly hydrostatic pressure of know magnitude. It uses fluid/solid argon as its pressure medium and has a sliding piston seal with extremely low friction. Absolute, NIST traceable pressure uncertainty ranges from 0.2 % at 2 GPa to an anticipated value of 0.5 % at 6 GPa.

In response to the COMPRES committee response to my preliminary first year report, pressure calibration efforts will be significantly reduced in favor of temperature calibration. At this time, I do not plan to try to characterize the Ba I-II transition circa 5.5 GPa. If I am able to reach ~8 GPa in the gas piston cylinder apparatus I will try to characterize the high Bi transition in detail with a large sample by monitoring volume change as per my recent characterization of the Bi I-II transition circa 2.5 GPa [*Getting*, 1998].

Realistic estimates of the piston seal area are required in any study in the gas piston cylinder apparatus. This dilation was calculated in the Bi I-II study by analytic elastic means and by linear finite element analysis (FEA). For higher pressure work non-linear FEA is required as the tungsten carbide core of the pressure vessel suffers increasing plastic strain at pressures above about 3 GPa. Algor FEA software capable of the subtle, non-linear analysis has been purchased. I am presently collaborating with Prof. Kaspar Willam and his students in the Civil Engineering Department at Colorado and with the Algor company itself to assure realistic calculations of bore dilation at the pressure seal.

Materials are on hand to introduce optical fibers into the gas piston cylinder apparatus. Arrangements have been made with Mike Brown at the University of Washington to perform a very detailed calibration of the ruby fluorescence pressure scale with accurate internal temperature measurements if I can make the optical fiber feed-throughs work. This would be a virtually continuous calibration by direct, NIST traceable force/area measurements to the highest pressures attainable, conceivable approaching 8 GPa.

Plans

My post-retirement employment at the University of Colorado is limited to five months per year. I anticipate working from November through March each of the first 3 years of COMPRES. During the remaining 2.5 months of my employment this first year of COMPRES I plan to complete one atmosphere development, testing, and calibration of the Johnson noise thermometer. As time permits I will also address the following projects:

1.) Design an 8 GPa modification of the gas piston cylinder apparatus.

2.) Design new mitre ring seals to reach pressures of 6-8 GPa in the gas piston cylinder apparatus without seal extrusion.

3.) Analyze the elastic-plastic bore dilation by non-linear FEA to accurately determine the piston seal area in the gas piston cylinder apparatus at pressure above 3 GPa.

4.) Develop and test feed-throughs to bring optical fibers into the gas piston cylinder apparatus.

My original work plan called for 18 months collaboration with a post-doc. The loss of this component of the project means a large reduction in total effort during the 2nd and 3rd years. In light of this I will concentrate during the second year on installing the Johnson noise thermometer in the gas piston cylinder apparatus at Colorado. This will permit me to determine the effect of truly hydrostatic pressure on thermocouples. The pressure-temperature objective is 6 GPa and 2000 K. As time permits I will also address the following projects:

1.) Assemble and test the 8 GPa version of the gas piston cylinder apparatus.

2.) Calibrate the ruby fluorescence pressure scale in the gas piston cylinder.

3.) Characterize the high Bi transition in the gas piston cylinder.

During the third year Johnson noise thermometry should be migrated to solid media, large-volume high pressure devices. The best, in fact the only reasonable way to begin this process will be to bring multi-anvil tooling sets to Colorado. Issues of electrical noise pollution from the ambient will all ready have been well solved in the course of running the Johnson noise thermometer in the gas piston cylinder apparatus. For instance, a specialized power supply may be required for cells in which the Johnson noise thermometer is used as the electrical noise from typical SCR power supplies may be intolerable. I anticipate the further assistance of my colleague Jan Hall as we explore these issues during the second year.

There are tooling sets presently on hand in various labs for both cubic and octahedral multi-anvils which can be run in my 7 MN (~700 ton) capacity press. Thermocouple calibration depends on both apparatus type and the specific cell utilized, but is independent of the lab in which the tests are made. A wide variety of cells could be calibrated in Colorado with the results applied in many labs elsewhere. If the COMPRES effort to develop standardized cells has been effective by that time, the Johnson noise thermometer should surely be run in those cells. This would constitute a very efficient means of migration accurate temperature measurement to many other labs simultaneously as the thermocouple calibration procedure need not be repeated in the labs using those cells.

The (cubic) D-DIA tooling set at Livermore National Laboratory is self-contained and can be run in my press to its full capacity. The results would be applicable to the X-ray transparent, quasi-hydrostatic DIA devices at APS, SUNY, and elsewhere. The unique D-DIA device would enable us to calibrate thermocouples not only at quasi-hydrostatic pressure, but also in the deviatoric stress fields associated with controlled sample deformation.

The “T-cup” apparatus at SUNY and the “T-10” apparatus at APS are X-ray transparent, miniature octahedral multi-anvils. They also can be run to their full capacity at Colorado.

The “Walker Module” is a “full size” octahedral device with one inch carbide cubes. This apparatus can be run at Colorado to a large fraction of it maximum load capacity. This type of apparatus is in wide use. Thermocouple calibration transferal, especially with standard COMPRES cells, would be very efficient in this device.

This strategy will require others to commit their tooling sets, cells parts, personnel, and skills to the migration effort. They will have to come to Colorado for requisite periods of time with their equipment. They can reasonably hope to leave with the best temperature measurements ever made at high pressure. No workshops are anticipated in the first two years of this lab-intensive effort. This will be made up for in the third year with collaborative thermocouple calibration sessions at Colorado involving a wide range of devices and cells.

Some types of high pressure devices and cells would be best calibrated in their home labs. This might include large octahedral multi-anvils with bear loads in excess of 7 MN and large opposed anvil cell currently under development. My lab is not equipped to handle these types of device. Eventually the Johnson noise thermometer should be carried to other labs.

Reference

Getting, I. C., New determination of the bismuth I-II equilibrium pressure - A proposed modification to the practical pressure scale, *Metrologia*, 35, 119-132, 1998.

Budget Justification

The budget for the second year is reduced essentially to my salary alone. I will undertake the second year tasks as describe above unassisted. Resources from the first year will be applied as required and available to support the second year's work.

Multi-anvil Cell Assembly Development Project, COMPRES

K. Leinenweber and J. Tyburczy, Arizona State University

The multi-anvil cell assembly development project is a community project to develop high-pressure cell assemblies for multi-anvil laboratories in the United States (members of COMPRES). The project was initiated at Arizona State University (ASU) on May 1, 2002. In the first year, the project has been proceeding with several lines of activity.

Creating a set of standard cell assemblies using proven designs

The first goal of the effort is to decide upon, create, and distribute a standard set of cell assemblies to the user community. These will be the first "COMPRES cell assemblies." Members of the community will be provided small batches of assemblies on request, and in return will be asked to provide information on the performance of the assemblies, calibration data, problems, successes, etc. This information will be part of the characterization of the performance of the cell. Also, for previously used designs, information from prior work using the assemblies can be included in the characterization.

The purpose of providing only small batches at first is to enable distribution to the largest number of users possible and to enable flexibility should design parameters change. The goal is to develop a rapid delivery system so that assemblies can be rapidly produced and distributed.

Cell assembly design group meeting, Dec. 8, 2002

In order to steer the development of the cell assemblies for this project, members of the COMPRES community met at the Fall 2002 AGU meeting (at a 2-hour special event on Sunday, Dec. 8, 2002). There were approximately 35 attendees at this event. Three designs were arrived at, and a plan for their fabrication and testing was made. Particular interest was shown in the rapid development of a Fei-style 8 mm assembly, so that many labs can extend their research to higher pressure. The cell assemblies will be fabricated at ASU using the COMPRES Machining Facility (see below), and also at outside companies when feasible. The distribution if assemblies will begin as soon as possible. The goal of this section of the project is not to pursue striking new developments, but to make a first group of cell assemblies rapidly available to laboratories that desire them and who will contribute to the group collection of performance data.

The three designs will be for three different ranges of P and T, and assemblies for which a great deal of prior experience exists (such as the Bayreuth and Stony Brook 14/8 assemblies, and the Fei 8/3 assembly) received top consideration in the choice of starting projects.

Development of new assemblies and concepts

A medium-term goal of the cell assembly design project is to work on new assembly designs – to make new and better assemblies that increase performance (in terms of temperature gradient, lower cost, P-range, T-range, reduced failure rate, enabling new types of experimentation, etc). Improved design, new materials, and improved production techniques will be brought into play. First, thermal modeling will be used to predict thermal gradients in assemblies ahead of time, allowing us to optimize the assembly materials and dimensions to reduce thermal gradients. Secondly, both new sources of old cell assembly materials and entirely new materials will be sought out and tested.

A cell assembly design program, tentatively called "Cell Assembly Designer," is being developed in order to fulfill the thermal modeling part of the project (J. Hernlund numerical code, K. Leinenweber graphic design "front-end.") The program has been written and is undergoing testing. This program will allow new cell assembly designs to be tested for good thermal characteristics theoretically, before they are fabricated and tested experimentally. Ultimately the program will be released to participating COMPRES institutions.

The new assembly design program will begin after the first 3 standard assemblies are underway. For this purpose, a cell assembly design workshop will be convened in Arizona during Spring 2003, using funds in the project budget to host perhaps 10 visitors to plan new cell assembly designs that increase performance in terms of temperature gradient, P-range, T-range, reduced failure rate, lower cost, enabling new types of experimentation, etc.

Machining facility

A Haas Computerized Numerical Control (CNC) mini-lathe has been ordered, and is expected to arrive in December 2002. The electrical connections, air-handling systems, and floor space have already been prepared for the lathe. Following the arrival of the lathe, a small group of machinists from the ASU Mechanical Shop and K. Leinenweber will be trained on its operation. This lathe is an important part of the COMPRES cell assembly development effort, as it will enable the automated production of parts for the COMPRES cell assemblies, which is needed to allow cell assemblies to be efficiently produced for the community.

The lathe is equipped with live tooling, which means that not only can cylindrical parts be machined with automation, but slotting and milling can also be done on the parts while they are on the lathe. Thus, thermocouple slots and other features can be included when desired.

A search for a CNC-skilled machinist will be conducted in February 2003, after the lathe is installed. In the meantime, a machinist, Eric Johnson, has been hired on a 4-month "emergency hire" position (Nov. 18-March 18) to help set the device up, make drawings and preparations for the standardized designs, and test other techniques such as the milling of pyrophyllite gaskets.

Industrial contacts/ New fabrication techniques

An ultimate goal of this project is to find companies that will make many or all of the parts for the standardized cell assemblies. Many of the newest breakthroughs in cell assembly development may be expected to come from interaction with specialty companies, for example in the ceramics or the high-tech industries, that have new types of capabilities for making parts for multi-anvil assemblies.

So far this year a number of new techniques were pursued. Several of them showed promises; these are outlined below.

Gasket Machining

The machining and cutting of pyrophyllite gaskets is one of the most time-consuming steps in preparing multi-anvil assemblies. It is a bland detail, to be sure, but a technique for rapid, economical fabrication of these pieces would create a "silent breakthrough" in our field, enabling many more runs to be done. Improved means of production is being pursued through test runs with St. Gobain (Norton) ceramics company. They have purchased a 5-axis water jet cutter, which can cut 50 feet of ceramic per minute and make straight cuts up to 6 inches deep. The cut is only a few thousandths of an inch wide, so little material is wasted, and a computer-driven table allows pieces of high tolerance and complex shapes to be made. The applicability of this technique to fabrication of pyrophyllite gasket pieces for multi-anvil experiments is currently being tested on sample pieces of pyrophyllite that we have sent to the company.

Paper backing for gaskets

In combination with the making of the gaskets, the backing for the gaskets is also under development using outside sources. Many assemblies use paper backing for the pyrophyllite gaskets. We have been testing paper backing cut by a commercial firm using a high-precision laser technique (commonly used in architecture). The primary benefits of this technique are better reproducibility and that the paper itself may be used to place the gasket (the reverse of the normal order), which removes the user-dependence on gasket placement and which eliminates the task of teaching new users the art of putting on gaskets.

Injection-molding

Innovations in fabrication of ceramic parts are also being sought. Injection molding is a technique that can provide both new types of ceramics and a new fabrication technique for them. Under the COMPRES project, a set of test pieces of various ceramics has been fabricated at an outside company. One ceramic type is an alumina ceramic, and the other is a spinel + MgO ceramic. Others will also be investigated. To avoid the cost of making a mold for each test project, the test boules are being produced in a blank shape, which will initially be machined (milled) for testing. When the ceramic is found to be suitable for a certain purpose, molds will be ordered and the ceramic will be produced to final shape.

EDM on metals

Parts made of metal foils, such as rhenium furnaces, are difficult to prepare reproducibly using conventional cutting technologies. One of the possible three first COMPRES assemblies, the Fei-Geophysical Lab 8/3 assembly, uses a rhenium furnace that is small, thick and difficult to cut. (The Geophysical Laboratory uses a heavy-duty cutter that was found in their basement, perfect for the job but not available on the market!) It is hard to imagine teaching the preparation of this part to the community in a distributed community effort. As part of the COMPRES effort, a test batch of Re furnaces was made by computerized Electron Discharge Milling (EDM) cutting at a Phoenix-area company. The result is very encouraging. The process is inexpensive, and the furnaces are highly reproducible. Yingwei Fei is currently working up revised dimensions for the furnaces, to closely match those he uses himself, and EDM cutting may become a frequently used approach when metal foils are involved.

Teaching

Perhaps the hardest part of a community effort such as this is the transfer of knowledge to the end users of the technology. The use of a cell assembly that is developed by a community group, and fabricated at one place, needs to be taught to others who desire to use it. For this reason, the designs are geared towards easy use from the beginning. Several of the design principles - the exactness of dimensions, the pre-slotting of parts using the CNC lathe, the pre-cutting of metal foils, precut paper backing, etc. – are intended not only for reproducibility, but also to reduce the teaching load when new users are trained or when the designs are distributed to end-users.

There is no specific teaching component in our current plans, but it is suggested that a teaching workshop for students and other end-users would be in order at a later date, using the travel budget from year 2.

Budget

The Year 2 budget given below is identical to that in the initial proposal. In the original proposal, this project was proposed as a 3-year project.

Budget request for Year 2 – May 2003-May 2004

(Unchanged from the original proposal)

Machinist	$37,440
Fringe 30%	$11,232
Materials, Supplies, Analysis	$40,000
Tooling	$5,000
Travel	$10,000
IDC (50%)	$51,900
Total Project	$155,600

COMPRES BUDGET SUMMARY FOR YEAR 1

Category	Current Budget	Actual Expenditures	Projected Estimates	Total Expenditures
Salary & Wages	$151,000.00	$63,354.22	$87,645.78	$151,000.00
Fringe	$44,545.00	$18,959.05	$25,585.95	$44,545.00
Equipment	$40,273.00	$11,437.75	$28,835.25	$40,273.00
Travel	$17,556.00	$2,588.62	$14,967.38	$17,556.00
Participant Support	$65,000.00	$44,005.80	$5,399.78	$49,405.58
Materials & Supplies	$31,000.00	$46,594.42	$0.00	$46,594.42
Publications	$1,000.00	$0.00	$1,000.00	$1,000.00
Other	$5,500.00	$0.00	$5,500.00	$5,500.00
Indirect costs	$214,928.00	$214,928.00	$0.00	$214,928.00
Subawards	$1,229,198.00	$1,229,198.00	$0.00	$1,229,198.00
Totals	$1,800,000.00	$1,631,065.86	$168,934.14	$1,800,000.00

BUDGET JUSTIFICATION FOR YEAR 2

The budget as reported on the 1030 forms includes the following:

Stony Brook Budget

- Central Office
- Multi-anvil lab at the NSLS
- Subcontracts

Each subcontract has individual 1030 forms.

Here we give budget justifications for the Stony Brook part and for other parts where the budget itself was not self evident.

Stony Brook

The Stony Brook budget consists of the Central Office budget which supports the President (Aug, 2003 – April 2004), administrative staff, the educational and outreach program (total budget for direct cost is $10,000) and the student intern program along with the Multi-anvil user program at the National Synchrotron Light Source. These two programs are detailed below.

Central Office

The Central office supports two full time personnel, the president and an administrative assistant. Part of the year's salary for the president is indicated here, and part is contained in the U. of Illinois budget (for Jay Bass until the end of his term as President in August).

The Educational and Outreach program supported by COMPRES is also part of this budget. Direct funds for education amount to $10,000 and are distributed among travel, supplies, and publication costs. The support of Glenn Richard is completely born by the University and his entire effort is devoted to the Education and Outreach effort and to the Information Technology effort of COMPRES.

The specific equipment item is for a video conferencing facility. This will enable committee communication within COMPRES with no travel needed. We have piloted such a system and find that it is an excellent substitute for face to face meetings and will greatly increase the communication within the COMPRES system.

The Student Intern program is included in the Central Office Budget and is responsible for the total COMPRES budget exceeding $2.1M for year two.

Budget for Central Office — Direct Costs

Salaries	President	
	Administ. Assist	
	Student Interns	
Salaries + Fringe		$289880
Equipment*		$32000
Supplies		$24000
Travel		$24000
Participant support for meetings		$51700
Publications		$2000
Totals		$423580

Multianvil Press Facility at the NSLS

Weidner and Vaughan

This budget is folded into the Stony Brook University budget along with the Central Office and subcontracts.

Budget for Multi-anvil facility at NSLS — Direct costs

Salaries	Beamline Scientist	
	Machine/Electronics Technician	
Salaries + Fringe Total		$134415
Equipment*		$55454
Supplies/Anvils		$40000
Office space NSLS (other)		$6500
Travel/lodging for users		$20000

Totals	$256369

*Primary equipment item for year 2 is a D-DIA (Deformation DIA) and items needed in order to occupy the new hutch including electronics, motors and drivers, and computers.

Carnegie Institution of Washington

Megabar Synchrotron Center

Hemley and Mao

X17B/C are currently supported by EAR00-04084 at $381,288 for two years (ending Feb 2003), and U2A is supported by EAR00-79510 at $70,968 for the current year (ending July 31, 2001). The integration brought about by the new center will enhance the operation of all facilities. Funds are requested in year 1 for support of the IR beamline, the creation of the infrastructure for the Megabar Synchrotron Center, and user travel/lodging. Notably, the annual requests for the Center itself in years 2-5 (including inflationary adjustments but excluding travel) are comparable to the current total for the individual beamlines (initially 9% more than this year). The additional amount is for important user travel and lodging.

Salary Support Full-time technical support is crucial for the success of this general user facility. Sixmonths/ yr salary support for a Ph.D. level beamline scientist to manage the facility and serve the user community is requested (plus fringe benefits). The remaining 6 months salary will be obtained from Carnegie or other grants. The position is currently occupied by Zhenxian Liu. An excellent spectroscopist, he is skilled in micro-IR, Raman, and high-pressure techniques, and has been working on geoscience applications with partial support from CHiPR. The success of the x-ray beam line program at X17C has come about in large measure because of the strong technical support provided by its on-site personnel (Jingzhu Hu), as indicated by enthusiastic user reviews of the beam line. Following the current funding model in place for X17B and C, in the years 2-5, we request continued funds for the X17C beamline scientist and partial support for the X17B technical associate. As is currently done, the remaining 6 mo/yr for the latter will be obtained from other members of the principal research team (PRT) of the beam line.

Equipment Funds are requested in the first year for the initial permanent equipment needed to create the fully operational three-beam line diamond-anvil cell center at the NSLS. This request includes a pair versatile diamond-anvil cells for general users ($10,000 total), together with replacement anvils (type Ia diamond for x-ray and far IR experiments, type II diamond for x-ray and mid-IR, and new larger single-crystal moisonite anvils; $10,000). A high-quality stereo microscope (Olympus MZ11; $25,000 including imaging system and computer), a major deficiency of the current facility, is also needed. A gas-loading system is needed so that users can carry out truly hydrostatic experiments over a broad range of pressures ($20,000). Also in the first year, we need to upgrade and integrate the computer facilities. This includes LAN network utilizing a unix server that links X17B, X17C, and U2A and experimental control through WinNT/Win2000 Workstations operating as X-terminals. The unix data server will be accessible off-site and serves as a data archiving facility for all x-ray and IR beamline experiments ($10,000). Associated software (e.g., IDL, Exceed, NFS Maestro, VxWorks) is required as well ($5,000). In addition, the beamline control at X17C is over ten years old; we will upgrade the current CAMAC based data acquisition and control to a more versatile and reliable VME based system. The initial upgrade will involve phasing out the E500 motor controllers and the CAMAC crates with the following: purchase of VME controllers ($10,000). The total equipment cost is therefore $90,000, of which the Carnegie Institution is committing $27,000; the matching request covers the microscope and imaging system described above ($25,000) and $2,000 for anvils, bringing the total request from NSF to $63,000. Funds for more modest permanent equipment are requested for years 2-5. These figures are based on our experience over the past decade for maintaining and continuing to upgrade the diamond-cell beamlines at the NSLS. Initially during this period, we finish the upgrade of the electronics with the acquisition of MVME CPUs and motor control cards for VME with transition boards ($20,000), stepper and micro-stepper power packs and motor drive modules (20 for a total of $20,000), and replacing the existing AIM and MCA modules to operate fully via EPICS ($10,000). Other equipment needs during this period include an ample supply of anvils 2 for users (estimated to be $5,000/yr), upgraded drilling and gasket preparation instrumentation ($5,000), glove box ($15,000), cryostats and furnace assemblies ($10,000) as well as general upgrades and replacements for optics (laser spectroscopy/IR spectroscopy/laser heating; $10,000). Additional components will include dedicated detectors (*e.g.*, CCDs), x-ray optics, and laser/IR components. Each of these will be acquired or phased in as dictated by user needs. As mentioned above, we expect 30% cost-sharing by Carnegie for permanent equipment for these remaining years of the project ($13,000 each year for years 2 through 5). Over the course of 5 years, it is expected that additional equipment and instrumentation will be sought by the user community. As some of this technology is still developing, and user needs will surely evolve during this period, it is prudent to consider these acquisitions as needs arise through separate proposal requests to the COMPRES organization, directly to NSF, or to other agencies.

Travel Funds are requested in the first year for general users to stay at the facility for experiments (an average of 100 person-days at $50/day or $5,000 plus $5,000 for travel). Note that our current NSF grant provides support for first-time users and student users of X17, a resource that many are taking advantage of. When this grant terminates (in year 2), we anticipate that the total user travel/lodging support will need to increase to $25,000/yr when such funds are required for both the x-ray and IR components of the Center. Because of the shortage of COMPRES funding, the first year support of IR related travel is not included in this budget but delayed to October in anticipation of a $16,000 (travel plus IC) supplement application.

Miscellaneous Expendable supplies total about $7,000 in the first year. This includes l-He for detectors for far-IR measurements and sample cryostats (20 storage dewar at $160/dewar). Other needs include miscellaneous IR windows (typically 4 per year at $500/each), computer supplies and data storage ($800/year), and mounts, microscope supplies, and sample preparation materials ($1,000). In the subsequent years, an additional $3,000/yr is needed for materials and supplies for the x-ray beamlines (gaskets, mounts, furnaces, microscope supplies), based on the existing budget for

X17B/C. Beamline publications (not covered by users) are expected to cost $1000/year in the first year; increasing to $3,000 in years 2-5. Finally, charges for space, communications, and small maintenance by the NSLS are expected to be $4,000 in the first year, increasing to $10,000 in years 2-5.

COMPRES- Pressure Calibration at High Temperatures

Y. Fei

Salary support.

Participation of postdoctoral associate in this project is crucial. I request 12-month salary per year for a postdoctoral associate.

Equipment.

The externally-heated high-temperature diamond-anvil cell is the principal tool for the proposed diamond cell experiments at high pressure and temperature. Replacement of diamonds is required in some of the experiments. Funds for two pairs of diamond anvils each year ($2,900/pair) are requested.

Travel

In situ/ex situ X-ray measurements will be carried out by using the synchrotron diffraction techniques at SPring-8 (Japan) and APS (Chicago). I request $4,500 per year ($1,500 per scientist) for travel to SPring-8 (Japan). We will perform the proposed diamond cell experiments and some of the multi-anvil experiments at APS. I request $6,000 for two trips to APS for three scientists. Funds ($1500/year) are also requested to attend one national conference each year.

Miscellaneous.

For the pressure calibration project (the first two years), funds ($8,000/year) are requested for miscellaneous supplies. These include tungsten carbide cubes and cell assemblies for the multi-anvil experiments, Re gaskets, capsules, and furnace materials, Ar-1%H2 gas, thermocouple materials, and other materials related to the high-temperature set up. For experiments at pressures above 23 GPa, tungsten carbide consumption is relatively high (typical cost for a finished Toshiba carbide cube is about $120 and eight cubes is required for each experiment).

Budget Summary for Carnegie Institution of Washington

BUDGET SUMMARY

	PRESSURE CALIBRATION		COMPRES NSLS MEGABAR CENTER				
	Year 1	Year 2	Year 1	Year 2	Year 3	Year 4	Year 5

Personnel							
Post Doctoral Associate							
X17C Beamline Scientist							
X17B Technical Associate (6 mo/yr)							
U2A Beamline Scientist (6 mo/yr)							
Total Salaries							
Fringe Benefits							
Total Personnel	53,970	55,255	$28,270	$120,790	$125,930	$131,070	$136,210
Equipment							
Total Equipment	$5,800	$5,800	$63,000	$29,324	$30,000	$30,000	$30,000
Travel							
Trips to APS @ $1,000/person-trip	$6,000	6,000					
Trips to SPring-8 @ $1,500/person-trip	$4,500	4,500					
Scientific meetings	$1,500	1,500					
General User Travel/Lodging				25,000	25,000	25,000	25,000
Total Travel	$12,000	$12,000		$25,000	$25,000	$25,000	$25,000
Other Direct Costs							
Materials and Supplies	$7,741	$7,714	$5,095	$10,000	$10,000	$10,000	$10,000
Publications			1,000	3,000	3,000	3,000	3,000
NSLS charges (communication, space, etc.)			4,000	10,000	10,000	10,000	10,000
Total Materials and Supplies	$7,741	$7,714	$10,095	$23,000	$23,000	$23,000	$23,000
Total Direct Costs	$79,511	$80,769	$101,365	$198,114	$203,930	$209,070	$214,210
TMDC	$73,711	$74,969	$38,365	$168,790	$173,930	$179,070	$184,210
Indirect Costs (59%TMDC)	$43,489	$44,232	$22,635	$99,586	$102,619	$105,651	$108,684
Total Request	$123,000	$125,001	$124,000	$297,700	$306,549	$314,721	$322,894

Princeton University

Laser Heated Diamond Cell Facility at the Advanced Photon Source

Thomas Duffy, Guoyin Shen, and Dion Heinz, Goals

Personnel

Funds are requested for 50% support of a post-doctoral research associate in year 2 of this project. The research associate will assist in the construction and testing of the CO_2 heating system, and for conducting the synchrotron x-ray diffraction experiments directed at improvements to the sample environment.

Equipment

The major equipment purchases required this year are a CO_2 Laser (e.g., Coherent), spectrometer (Acton Research), CCD detector (Roper Scientific) and optics for the heating system.

Travel

Travel costs are included for the PIs and post-doctoral fellow to make several trips to synchrotrons in the US to conduct experiments. The cost for a single experiment (4 days) at the Advanced Photon Source in Chicago is roughly $2000 for three person, including transportation, hotel, and meals). We are also planning visits to several laboratories around the country that currently have laser-heating facilities, in order to interact with and exchange ideas with colleagues. We also expect to present results at one or two national meetings during year two of this project.

Other Direct Costs

Funds are requested for expendable materials and supplies including compressed gases, gasket materials, microdrills, EDM supplies, sample materials, chemicals, occasional machining needs, and miscellaneous tools.

Indirect Costs

Indirect costs are included at the negotiated rate of 58% of all direct costs except for permanent equipment and tuition.

University of Colorado

Absolute Pressure and Temperature Calibration

Ivan C. Getting

The budget for the second year is reduced essentially to my salary alone. I will undertake the second year tasks as describe above unassisted. Resources from the first year will be applied as required and available to support the second year's work.

Arizona State University

Multi-anvil Cell Assembly Development Project, COMPRES

K. Leinenweber and J. Tyburczy, Arizona State University

The second-year budget for the multi-anvil cell assembly development project of COMPRES will be used to run the program that was launched in the first year. Part of the equipment for the project was purchased up front in the first year: a numerical CNC lathe provided by COMPRES for fabrication of cell assembly parts is on the floor at Arizona State University (Haas Mini Lathe). The second-year plan is based primarily on the discussion that was made during the special event entitled “Multi-anvil Working Group” at fall 2002 AGU. The budget breakdown for the second year is as follows:

Line item G1: Materials and Supplies. The materials and supplies budget will be used to purchase and prepare the ceramics that will be made into cell assemblies for the project. We plan to provide everything except carbide and precious metal capsules for each of the cell assemblies during the development stage: this means octahedra, pregaskets, and all the parts outside the sample capsule. We will at first introduce 3 different cell assemblies for testing by the community, as discussed in San Francisco. This requires the purchasing of ceramics and metals, their further preparation by cutting and other processes at outside companies, and the fabrication of parts for the assemblies wither outside or at ASU. New preparation techniques, including EDM cutting of metals, injection molding of new ceramics, and others will continue to be tested and utilized in the second year with the Materials and Supplies budget. The resulting assemblies will be supplied to members of COMPRES, who have agreed in return to provide information about the performance and calibration of the assemblies.

Line Item G6: Other. This is listed as “tooling” in the original budget page submitted with the COMPRES proposal; it is for the specialized tooling needed for the work, in particular diamond cutting tools for automated machining of ceramics (as used at Coors ceramics and other companies).

Section B: Personnel. The technician salary will be used to hire a CNC-experienced machinist. Now that the machine is in place, and the first runs are scheduled for late January, this hire will be made as soon as possible. This person will be in charge of machining all the parts for the COMPRES assemblies, and also will be expected to contribute to the development of techniques for making parts, including adding slots and grooves for thermocouples, and other geometrically complex work, and will help in identifying outside sources for the future fabrication of parts.

Section E: Travel. The travel budget will be used to fund trips between laboratories for the purpose of enabling the goals of this project to be met. On the one hand, the PI’s will travel to laboratories to create technology transfer (this was agreed upon at the December meeting) and to calibrate assemblies, and on the other hand, workshops is planned in Arizona, and the travel money will be used to help fund the travel of the attendees of the workshop. Depending on the nature of the workshops (both a cell assembly development workshop and a teaching workshop are desired during the lifetime of this project), the travel funding will be used to fund either the primary designers of assemblies, or student attendees.

NSF BUDGET FORM 1030 FOR ALL INSTITUTIONS