COMPRES

Newsletter

An NSF funded Consortium for Materials Properties Research in Earth Sciences
http://www.compres.us

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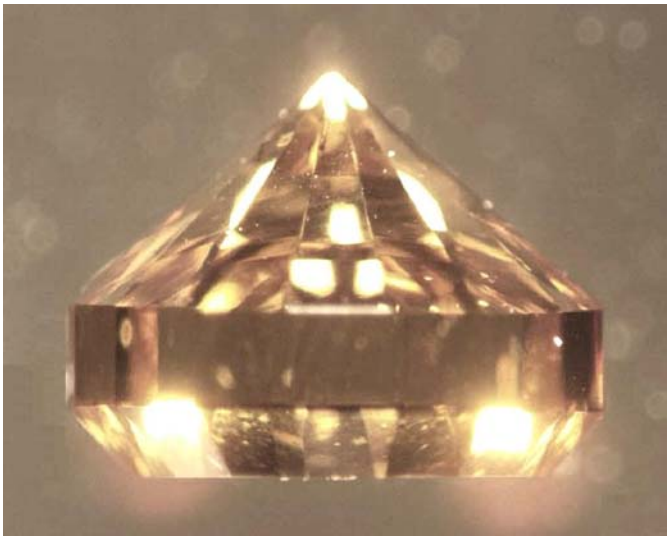
Project Report

Breakthroughs in Chemical Vapor Deposition of Diamond: Fast, Large, Hard, and Tough

Producing a material that is harder than natural diamond has been a goal of materials science for decades. The Geophysical Laboratory, Carnegie Institution of Washington has produced gem-sized diamonds that are harder than any other crystals (Yan et al. 2004). The diamonds were grown directly from a gas mixture by the microwave plasma chemical vapor deposition (CVD) process at a rate that is up to 100 times faster than other microwave plasma CVD methods used to date (Yan et al. 2002).



Modified brilliant cut single-crystal diamond grown by chemical vapor deposition (CVD). About 2.5 mm high, this crystal was grown in one day at Carnegie Institution. The very bottom (table) of the crystal is a type 1b seed; hence the yellow tint is due to internal reflection (the CVD diamond is transparent) (Yan et al. 2004).

The aim of this project is create new classes of devices for high-pressure geoscience research. As such, the early work on this began in the late 1990s under the auspices of the NSF Center for High Pressure Research (CHiPR). The recent results were made possible by an NSF Grand Challenge Grant. The goal is produce single crystal diamonds in the 10-100 carats range for new classes of high-pressure experiments. This is not feasible using current high-pressure/high-temperature (HPHT) synthetic diamond technology. The CVD process has another advantage; it highly flexible and can more easily control impurities in the material.

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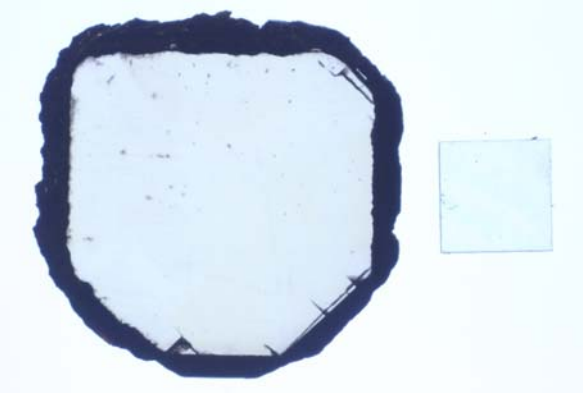
IN THIS ISSUE:

Project Report (diamond) *(Cont'd)*



A 5 x 5 x 0.5 mm single crystal plate grown in 5 hours at Carnegie; right: a HPHT plate available from De-Beers (Element Six).

The Carnegie researchers grew the crystals using microwave plasma CVD system to deposit CVD diamond on synthetic {100} seed diamond, typically with areas of 4x4 mm^2 to 10x10 mm^2; a standard microwave; and an infrared detector for determining the temperature inside the growing chamber. There are two particularly unique features of the device, a special holder for the seed diamond and an infrared temperature gauge.

Once the seed is placed in the chamber, a mixture of methane and hydrogen and nitrogen gas (10-20% CH_4/H_2 and 0.5-5% N_2/CH_4 at total pressure 150-250 torr) flows into the unit; then the microwave is turned on to create plasma to heat the seed to temperatures in the 1200 ^oC range. The carbon is deposited homoepitaxially on the diamond seed to produce coherent single crystal diamond growth. The group has grown single crystals of diamonds up to 10 millimeters across and over 5 millimeters in thickness by this method. Polished CVD anvils and plates can be produced right now for new classes of shock wave experiments, including dynamic compression experiments on precompressed samples (Figures on the front page and above). CVD anvils have already been used to reach 200 GPa in static compression experiments (Mao et al. 2003).

The CVD crystals are subjected to high-pressures and high-temperatures (HPHT), which enhance the color and further harden the material. Although this annealing has been used on synthetic diamonds made via high-pressure processes, the Carnegie scientists are the first to anneal single-crystal diamonds made by CVD. The annealed diamonds are heated to 2000° C at 5-7 GPa for ten minutes. This process renders the diamonds at least 50% harder than 'conventional' diamond (Figures below and on next page)

This high fracture toughness seems to be associated with residual sp^2 carbon defects in the as-grown single-crystal CVD diamonds, which have no grain boundaries and fewer twins than polycrystalline CVD diamond films. The mechanism of the extraordinary hardness enhancement by HPHT annealing is still under investigation. It might be associated with the transformation of amorphous carbon bonds and nitrogen-vacancy to denser sp^3 hybridized fine crystalline structures during the HPHT annealing.

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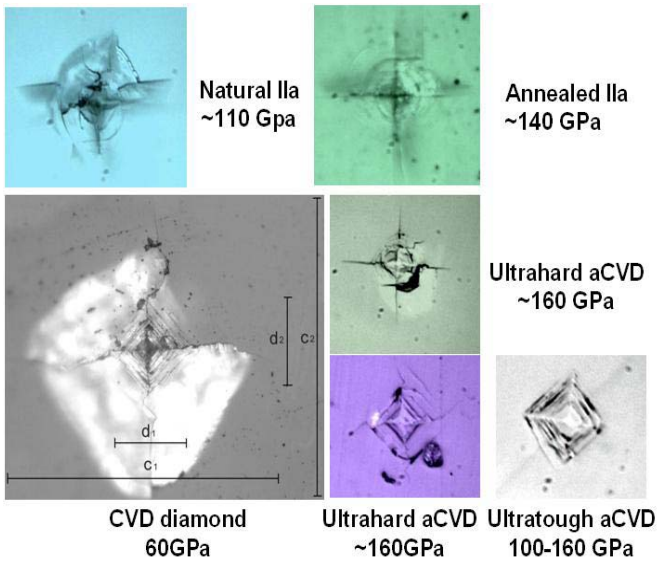


Photomicrographs of natural diamond, CVD diamond, and annealed natural diamond and annealed CVD diamond (aCVD) after indentation, together with a sketch of the cracked lines, d_1, d_2, c_1 and c_2 used to determine the hardness (H_v) and toughness (K_c) using standard procedures (Drory et al. 1995). $H_v = 1.854 \times L/d^2$, $K_c = (0.016 \pm 0.004)(E/H_v)^{1/2}(P/C^{3/2})$, $d = (d_1+d_2)/2$, $c = (c_1+c_2)/4$, P = Load, E = Young's modulus [assumed to be 1000 GPa (Prelas et al. 1998)].

Project Report (diamond) *(Cont'd)*

The primary goal of this project is to produce a new generation of high-pressure devices based on CVD diamond technology for use by the entire high-pressure community. The Carnegie group plans to install a second chamber for producing anvils for immediate use and for scale up to the larger sizes. The group's ability to make these ultra-strong crystals very fast has opened up an entirely new way of producing single-crystal diamonds for a variety of other applications that utilize their transparency, strength, insulating and thermal properties. Different types of diamonds are sought after to use in electronics, optics, and precision cutting tools.

—— By ***Chih-Shiue Yan*** and ***Russell J. Hemley***

Reference

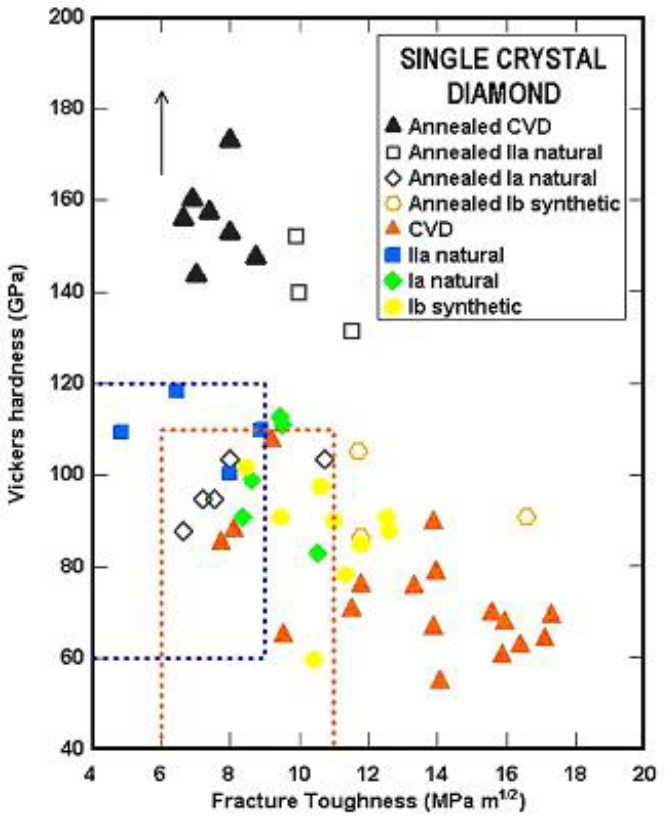
Drory MD, Dauskardt RH, Kant A, Ritchie RO (1995) Fracture of synthetic diamond. Journal of Applied Physics 78: 3083-3088.

Prelas MA, Popovici G, Bigelow LK (1998) Handbook of industrial diamonds and diamond films, Marcel Dekker, New York

Mao W., Mao H. K., Yan CS., Shu J., Hu J. & Hemley R J (2003) *Appl. Phys. Lett*. **83**, 5190-5192.

Yan CS, Mao HK, Li W, Qian J, Zhao Y, Hemley RJ (2004) Ultrahard diamond single crystals from chemical vapor deposition. Physica Status Solidi (a) 201: R24-R-27.

Yan CS, Vohra YK, Mao HK, Hemley RJ (2002) Very high growth rate chemical vapor deposition of single-crystal diamond. Proceedings of the National Academy of Sciences 99: R25-27.



Vickers hardness and fracture toughness on the {100} faces of various diamonds in the <100> direction. Reported ranges for natural IIa diamond are enclosed by the blue dotted square, and for polycrystalline CVD diamond films and conventional polycrystalline diamond by the red dotted square (Prelas et al. 1998). The arrow indicates this is a lower bound. The scatter in all of the data is a measured of the estimated uncertainty.

COMPRES users start using beamline 12.2.2 at the ALS: Although commissioning of the new high-pressure beamline at the ALS is not due to be completed until the end of the year COMPRES users have already started collecting diffraction data. Below are pictures Abby Kavner and Nathalie Conil from UCLA collecting radial diffraction data on water samples. End station 1, equipped with resistive heating dacs, is now fully commissioned for diffraction experiments. End station 2, equipped with laser heating, will be commissioned for diffraction next month. Updates and details of how to obtain beam time on this new facility can be found on the beam line website:

http://xraysweb.lbl.gov/bl1222/home.htm

President's Message

Bob Liebermann

COMPRES has now entered its third year. Following are some of the highlights of activities of the COMPRES and related scientific communities during the period July to September 2004.

The biennial Gordon Research Conference on High Pressure was held at the Kimball Union Academy in Meriden, New Hampshire from June 27 to July 2, 2004. Ann Chopelas of the University of Washington was the Chair of this year's conference with Reinhard Boehler serving as Vice-Chair [he will chair the 2006 meeting]; both Ann and Reini are card-carrying members of the COMPRES community. Of the 109 attendees, 25 were from mineral physics/geosciences, including many of the invited speakers: Yue Meng, Agnes Dewaele, Danielle Antonangeli, Przemek Dera, Ross Angel, Jennifer Jackson, Jonathan Crowhurst, and Yusheng Zhao. In addition, many of the poster presentations reported work done under the auspices of COMPRES support. The John Jamieson Award for the most outstanding lecture by a young post-graduate student was won by Jennifer Jackson of the University of Illinois at Urbana-Champaign for her excellent lecture on "High-pressure investigations of deep Earth materials using Brillouin and X-ray spectroscopy."

On August 13-14, SCEC and COMPRES jointly sponsored a Workshop on the Science, Status and Future Needs of Experimental Rock Deformation at Mt. Holyoke College in Massachusetts. This workshop was organized by Terry Tullis, Tom Jordan and Bob Liebermann and was attended by 38 scientists from around the world, including 80% of the members of the COMPRES Executive Committee. This workshop was tacked on to the end of a very successful Gordon Research Conference on Rock Deformation chaired by Andreas Kronenberg and Mark Jessell.

The new report on "Current and Future Research Directions in High-Pressure Mineral Physics" is now available in a glossy version which can be obtained by sending Ann Lattimore an email using the link on the Home Page at: www.compres.us. The report can also be downloaded from the Home Page at: http://www.compres.stonybrook.edu/Publications/BassReport/Bass_Report_8_31_04.pdf
This report is an outgrowth of the discussions and results of a Workshop on "A Vision for High Pressure Earth and Planetary Sciences Research: The Planets from the Surface to the Center" which was held on March 22-23, 2003 in Miami, Florida. The NSF Division of Earth Sciences commissioned and supported this workshop, and COMPRES organized it. Fifty-six scientists attended the Workshop, which was convened by Jay Bass and Donald Weidner. It was edited by Jay Bass using the materials presented at the Workshop or submitted soon thereafter. He collaborated with Ellen Kappel of Geosciences Professional Services, Inc. for the editing and design.

On behalf of the COMPRES and Mineral Physics community, I would like to thank Jay for his extraordinary and splendid efforts in bringing this important report to fruition. In recognition of his work, I expect that this report will henceforth be known as the Bass Report. See separate article in this issue of the newsletter with highlights from the report.

The process of reviewing and revising the ByLaws of COMPRES, initiated at the 2004 Annual Meeting in Santa Cruz, has now been completed. The Electorate [36 U. S. institutions eligible to vote] has cast its ballots, with 33 institutions voting. All of the proposed changes received more than the necessary number of votes [requires 2/3 of the Electorate, or 24 votes to approve changes]. The revised ByLaws are posted on the COMPRES website at:http://www.compres.stonybrook.edu/AboutUs/Bylaws/index.html. The Executive Committee has already instituted the changes in its procedures which are required by the revised ByLaws. On behalf of the Executive Committee, I would like to thank the ByLaws Committee, the Electors and Alternates, and the wider COMPRES community for their participation and contributions to this important process.

During the past three months, applications from the following institutions have been

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President's Message *(Cont'd)*

unanimously approved by the Executive Committee for membership in COMPRES:

Royal Institution of Great Britain in London
Representative: Paul McMillan

Massachusetts Institute of Technology:
Elector: Sang-Heon (Dan) Shim
Alternate Elector: Rob van der Hilst

Please note that, according to the revised ByLaws: "Educational or governmental institutions that are chartered in the United States with research and educational programs in high-pressure research in the science of Earth materials are eligible to apply to become Members of the Organization." Consequently, the three U. S. governmental institutions which are currently non-voting affiliates, now have full voting membership: (1) Argonne National Laboratory; (2) Astromaterials Research and Exploration Science, NASA; and (3) Lawrence Livermore National Laboratory.

These additions bring the total number of U. S. institutions to 41, with 15 foreign affiliates. You can find the Royal Institution [and other European member institutions on the new member map for Europe, which is now on the website along with the U. S. and World maps, see page 10]. Our thanks to Glenn Richard for creating and maintaining these maps.

The annual call for proposals for Infrastructure Development Projects for Year #4 of COMPRES [5/1/05-4/30/06] was sent to the COMPRES community on September 7. Proposals for new Infrastructure Development projects are due on 1 November 2004 and should be submitted electronically to the Committee Chair, Nancy Ross (nross@vt.edu) and also to COMPRES Central (alattimore@notes.cc.sunysb.edu). Interested persons can find a link to this Call for Proposals on the COMPRES Home Page at www.compres.us (or see page 7).

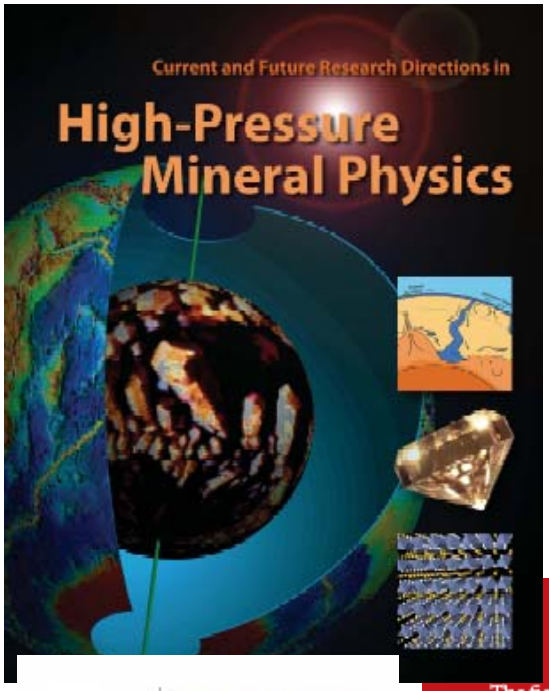
On September 28, a call for pre-proposals for MRI initiatives from the COMPRES community was distributed with a deadline of November 18, 2004. Interested persons can find a link to this Call for Pre-proposals on the COMPRES Home Page at www.compres.us (or see page 8).

In accordance with the revised ByLaws, Minutes of the meetings of the Executive Committee are now published on the COMPRES website at: http://www.compres.stonybrook.edu/Publications/Minutes%20for%20Web%20site%20posting/ ExCom%20Minutes%20Links%20page.htm

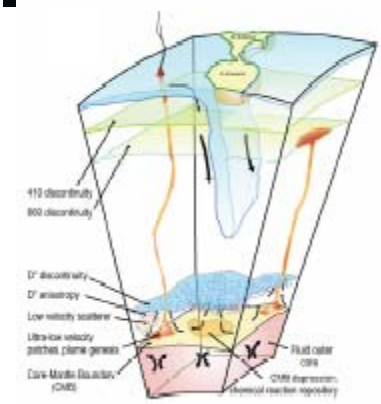
At its meeting on September 14, the Executive Committee of COMPRES tentatively set the dates for our 2005 Annual Meeting as June 16 [Thursday] to June 19[Sunday], with alternative dates as June 18 [Saturday] to June 21 [Tuesday]. If any of you know of conflicts with other professional meetings, please let me know as soon as possible.The location is under investigation and review. At this time, we ask that you pencil in and reserve these dates on your 2005 calendars.

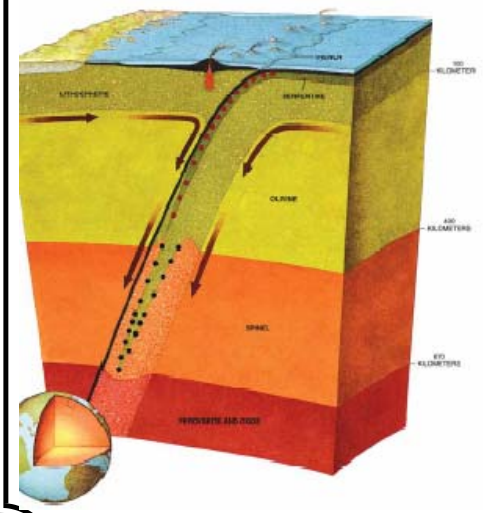
On September 21, Harry Green, Chair of the Executive Committee, and I visited the Division of Earth Sciences at the NSF for discussions with David Lambert and his colleagues. Our morning meeting with Dr. Herman Zimmerman, Division Director, centered on official presentation of glossy copies of the new report on "Current and Future Research Directions in High-Pressure Mineral Physics" from the 2003 Miami Workshop, which had been commissioned by Dr. Zimmerman; he had already downloaded it from the website and used it in presentations at NSF. In the afternoon in a meeting with Dr. Lambert and other EAR program directors [Russell Kelz, Robin Reichlin, Sonia Esperanca, and David Fountain]. I gave a presentation on "COMPRES at Age 2", followed by questions and discussion. Also attending was Nicholas Woodward from DOE-BES, who discussed the implications of new changes in DOE beamline operations at national laboratories and potential implications for beamlines operated by COMPRES and related programs [e.g., GSECARS]. In our separate meetings with Dr. Lambert, we discussed a number of budget issues, including the outlook for FY06 and funding for COMPRES in Year #4 [May 2005 to April 2006].

Current and Future Rsearch Direction in High-Pressure Mineral Physics



A report of the COMPRES Workshop on "A Vision for High Pressure Earth and Planetary Sciences Research: The Planets from the Surface to the Center" (March 22-23, 2003, Miami, Florida): *Current and Future Research Direction in High-Pressure Mineral Physics* has become available to public. The report summarizes the discussions of fifty six workshop participants, and addresses the frontiers in mineral physics research from four prospects: the core and core-mantle boundary, subduction and mantle processes, near-surface processes, and planetary processes. Developments of technology to promote the mineral physics research are highlighted in the report. The workshop was funded by NSF and convened by Jay Bass and Donald Weidner. The report was edited by Jay Bass in collaboration with Ellen Kappel of Geosciences Professional Services, Inc.





High-Pressure Mineral Physics

The field of high-pressure mineral physics is highly interdisciplinary, encompassing the full range of chemical, physical, and biological processes that take place at high pressures beneath the surfaces of planets. These processes influence magnetism in Earth's core at over 1.3 million atmospheres of pressure, and methane production by microbes at high pressure in ocean sediments. In the broadest sense, the goals of mineral physics studies are to understand how planetary systems operate, from the center to the surface, and to understand the processes involved in planetary evolution. This is done by examining the properties of minerals under extreme high pressure and temperature conditions, by performing computer simulations to understand the behavior of planetary materials at the most fundamental atomistic level, and by studying the interactions among the components of the entire Earth system—be they chemical reactions among minerals or biologically mediated interactions.

This field has witnessed numerous discoveries and breakthroughs during the past decade. Along with breakthroughs come not only the ability to understand more-complex phenomena, but also the ability to confront exciting challenges. In light of these recent achievements, the challenges for the future, and, consequently, the immense prospects for discovery in the field of mineral physics, it seems timely to describe them in a single document. This report is organized to take the reader on a journey from Earth's center to its surface, and then beyond to the other planets and moons in our solar system, all from the perspective of high-pressure mineral physics.

Finally, no description of high-pressure mineral physics would be complete without mentioning the role that technology plays in our research. This is a field that has distinguished itself through technological innovation and invention. In trying to understand the interiors of planetary bodies, Earth and planetary scientists have been the leaders in pushing forward the boundaries of extreme conditions that can be attained in the laboratory. They have been central players in the use of synchrotron radiation and neutron scattering for understanding the states of matter at high pressures and temperatures. High-pressure mineral physics is an area that by its very nature thrives on inventing new tools to understand Earth, and to see deeper and deeper into the interiors of planetary bodies.

Call for COMPRES Infrastructure Development Projects

Due: 1 November 2004

This is the annual call for proposals for Infrastructure Development Projects for Year #4 of COMPRES [5/1/05-4/30/06]. Infrastructure Development Projects should include one or several of the following attributes: development of frontier-level tools that require high degrees of innovation; making new capabilities easily accessible to the community; fostering connections and sharing of expertise within the COMPRES community; fostering connections and sharing of expertise with other scientific communities.

- **Each new project is expected to develop strong, active community outreach and collaboration.**
- **Projects should normally be for 1 to 2 years duration. Proposals for 3 years duration should be accompanied by special justification.**
- **Budgets are typically in the range of $50K/yr to $150K/yr, but will depend on overall COMPRES needs and resources.**

New proposals shall consist of:
1) 3-5 pages of text (including figures, tables and references),
2) NSF-style 2-page curricula vitae of project directors
3) NSF budget forms.

Proposals for new Infrastructure Development projects are due on **1 November 2004** and should be submitted electronically to the Committee Chair, Nancy Ross (nross@vt.edu) and also to COMPRES Central (alattimore@notes.cc.sunysb.edu).

All proposals will be evaluated by the COMPRES Infrastructure Development Committee and, in cases where additional expertise is required, by external reviewer(s). The committee's recommendations will be forwarded to the Executive Committee, which will make the final decisions and will prepare the master COMPRES budget proposal for Year #4.

Every Infrastructure Development project shall, generally within the first year of its initiation, obtain community input and educate the community on the directions, goals and capabilities of the project. One means of accomplishing this may be through one or more workshops. We also encourage the use of any effective means of involving the community in the project, including electronic communications, targeted collaborations or other innovative approaches. Funding for this activity, including workshops, shall come from within the project's budget, and may be supplemented by other (non-COMPRES) sources of support. The specific form, venue and timing of any workshop shall be determined by the project's directors, who may benefit from consultation with members of the Infrastructure Development Committee. The intent is to develop strong, active and early community interaction and collaboration for each project.

At the COMPRES Annual Meeting, each current Infrastructure Development project PI has the responsibility to make a presentation on current progress and results. A written annual project progress report will be submitted each year by a date determined by the Committee (currently set at November 1).

For further information contact Nancy Ross (Infrastructure Development Committee Chair) or COMPRES President Robert Liebermann. The current members of the Infrastructure Development Committee are: Pamela Burnley (Georgia St. Univ.), Yanbin Wang (Univ. of Chicago), Kevin Righter (NASA) and Dan Shim (MIT).

Call For Preproposals For MRI Initiatives From COMPRES Community

To: COMPRES Community
From: Bob Liebermann
Subject: Announcement of COMPRES Eligibility to Submit a Proposal to the NSF's Major Instrumentation Program [MRI] and Call for Pre-proposals

The next annual deadline for full proposals to the NSF's MRI program is January 27, 2005.

According to the Program Solicitation [NSF 04-511] at url:
http://www.nsf.gov/pubs/2004/nsf04511/nsf04511.htm
proposals may be submitted by institutions of higher education, independent nonprofit research institutions, research museums, and legally documented incorporated consortia of eligible institutions. [Note: Although COMPRES is not incorporated, neither is CARS-Consortium for Advanced Radiation Sources and CARS has been eligible to submit MRI proposals in the past].

Therefore, the Executive Committee has concluded that COMPRES is eligible for this MRI competition and approved this call for Pre-proposals for distribution to the COMPRES community.

Final proposals will be submitted to the NSF by the home institution of the Principal Investigator, with a clear indication in the proposal's title that it is being submitted from an institution that is a member of COMPRES and that it is being submitted under COMPRES eligibility. This proposal will NOT count against the regular institutional quota of proposals [2 per institution or 3 if one is for instrument development].

The minimum award from NSF will be $100,000 and the maximum award will be $2 million. The minimum and maximum award amounts represent NSF's contribution to the project and do not include the institution's cost sharing where it is required and allowed. For Equipment Acquisition proposals, the proposing institution is required to cost share at a level of exactly 30% of the total eligible project costs.

In the case of COMPRES, of course, the matching funds [cost-sharing] for the MRI proposal would have to come from the home institution (s) of the Principal Investigator (s), because COMPRES has no non-federal funds.

Brief pre-proposals for review by the COMPRES Executive and Standing Committees are due by Thursday, November 18, 2004 at 8:00 AM [East Coast time].

The pre-proposal should contain the following elements:

- Description of the need for the equipment (3 pages maximum)
- List of Co-PIs and major users, with 2-page CVs and current and pending support for each.
- Proposed cost-sharing arrangements, including clear evidence of willingness of the home institution to provide matching funds for the equipment acquisition. This item should indicate that the PI has had discussions at his/her institution with the relevant administrator who will be potentially responsible for providing matching funds and that the administrator has indicated a willingness to provide the matching funds if the pre-proposal from the PI is approved for submission from COMPRES.
- A description of how this new equipment will address the needs and advance the agenda of the COMPRES community.

These materials should be sent to Ms. Ann Lattimore at:
Email: alattimore@notes.cc.sunysb.edu
And must be received by 8:00 AM [East Coast time] on Thursday, Nov 18, 2004.

From COMPRES Beamline Interns

Arianna Gleason-Holbrook:

As the COMPRES intern at the Advanced Light Source at the Lawrence-Berkeley National Laboratory, I am involved with a variety of projects and management tasks. I currently organize and maintain the High Pressure Lab at the ALS under the direction of Simon Clark and Martin Kunz. This involves helping beamline users with diamond-anvil cell experiment preparation, high pressure equipment training and safety. These tasks not only facilitate refinement of my own skills, but also expose me to the wide variety of current synchrotron research. I also maintain our new website for ALS High Pressure research at: http://xraysweb.lbl.gov/bl1222/home.htm

My research focuses on development and implementation of a resistively heated diamond-anvil cell system. Our pilot study involves the phase transitions of talc. I will be giving a poster presentation at the 2004 ALS Users' Meeting on the progress of this talc project. A resistively heated diamond-anvil cell [DAC] system for high pressure and high temperature diffraction measurements has been developed at the ALS and tested on Beamlines 11.3.1 and 12.2.2. This system is placed in a controlled environment and consists of a commercial diamond cell supplied by Diacell Products heated by resistive elements surrounding the diamonds. Temperature control is through PID feedback on a K thermocouple in contact with the diamonds and the gasket. To date, the system has operated to 450 ^{o}C and 24 GPa. Achieving these P/T conditions is important as we seek to understand more about physico-chemical properties of minerals as a function of pressure and temperature. Measurements at these temperatures are particularly crucial to study the behavior of minerals at subduction zones within the Earth where water content and phase stability are of great interest. Improvements to the system include the substitution of more thermally stable

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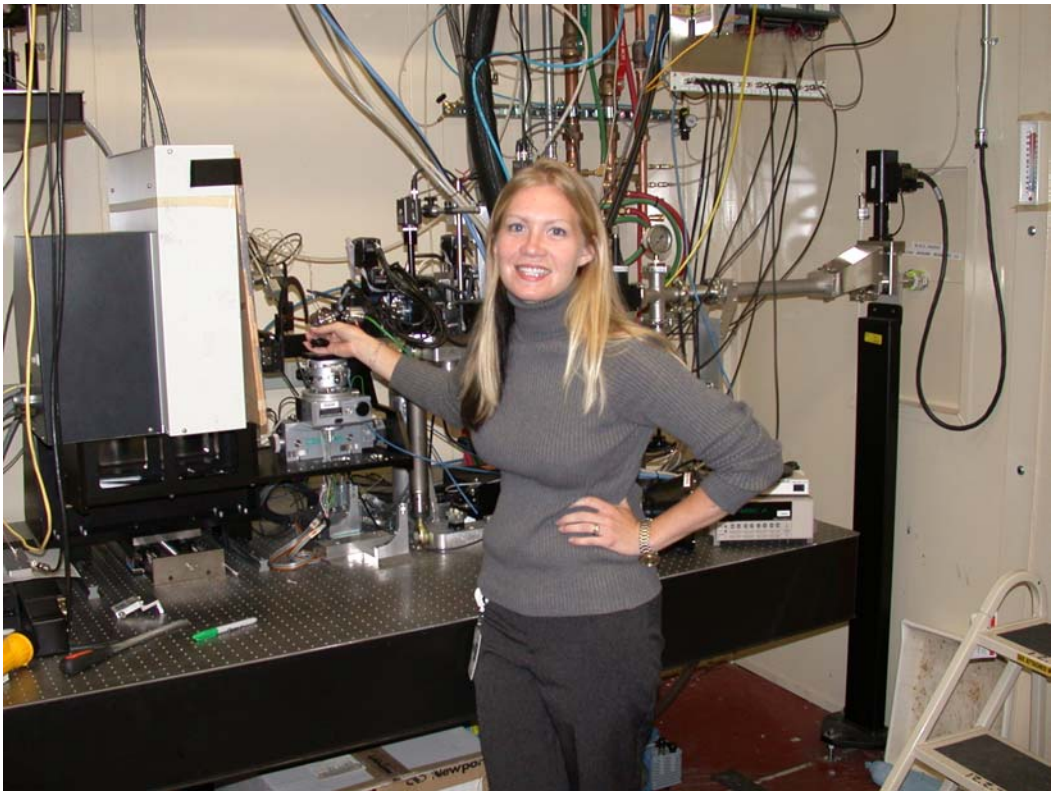


The COMPRES beamline intern, Arianna Gleason-Holbrook, is ready to load her DAC at the 12.2.2 beamline of ALS

COMPRES Interns *(Cont'd)*

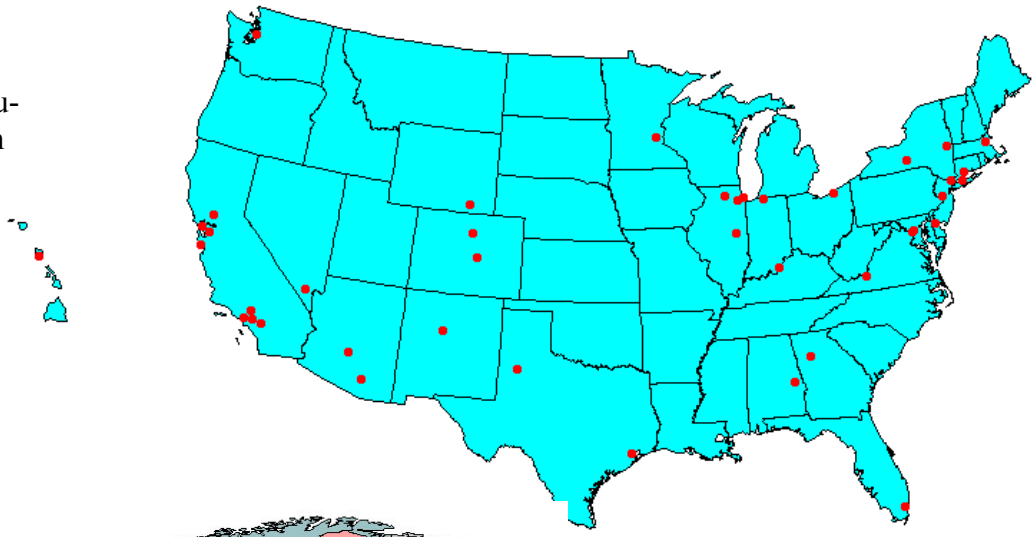
steels for the DAC body which will substantially increase the service temperatures achievable.

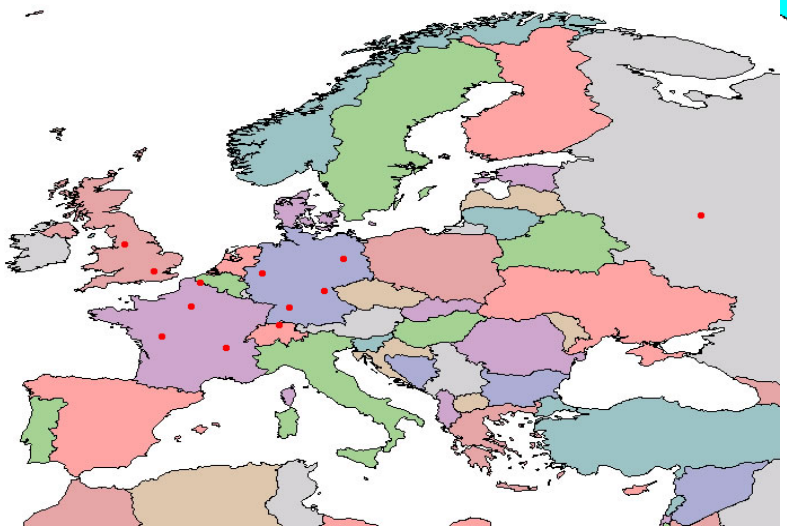
I will integrate the techniques I have learned and the data I collect into a PhD thesis when I attend University of California, Berkeley in fall 2005. So far, my experience here has been one of invaluable scientific involvement and immense opportunity. I feel honored to be apart of this group and look forward to the coming challenges.

COMPRES Member Update

The COMPRES membership has grown significantly within the nation and around the world. Currently, COMPRES members include 41 US institutions and 15 foreign institutions. Newly joined member US institutions are: California Institute of Technology and Massachusetts Institute of Technology.

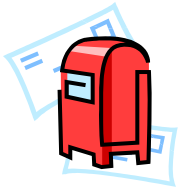
Locations of COMPRES Institutional Members in the United States.





Locations of COMPRES Institutional Members in Europe.

COMPRES has rapidly gained its visibility in European Mineral Physics community in the past year. The number of Foreign Affiliations in Europe has reached 12.



COMPRES Contacts:
President, Robert C. Liebermann, (631)632-1968, Robert.Liebermann@stonybrook.edu
Chair of Executive Committee, Harry Green, (951) 827-4505 (hgreen@mail.ucr.edu),
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Newsletter, Jiuhua Chen, (631)632-8058, Jiuhua.Chen@sunysb.edu