Spallation Neutrons and Pressure – SNAP – DE-FG02-03ER46085

CLOSE-OUT MAY 2009

John B. Parise, Stony Brook

The major achievements of the award were:

- 1) The Instrument Design Team and Executive Committee met in five annual meetings at Los Alamos and at the SNS. These meetings included significant outreach to other stakeholders and communities likely to be interested in neutrons and pressure. For example joint meetings were held April 12-14, 2008 with the COMPRES (Consortium for Mineral Physics Research in the Earth Sciences) group and highlighted complementary studies using high energy x-ray and neutron scattering, the 4th meeting in April 1-4, 2007 concentrated on science results with the NOMAD group and highlighted studies of poorly crystalline materials at non-ambient conditions.

The outstanding product of these meetings was the redesign of the beamline to accommodate focusing optics. This revolutionary concept, championed by Gene Ice of ORNL, was discussed at the first SNAP meeting in Los Alamos in 2004. The IDT at that meeting recommended “It is vital final decisions, based on real world experience with cells, be made on detector and focusing options”. This resulted in whirlwind tests over 3-weeks at the Chalk river facility. Upon learning of this in 2004 we immediately set about testing this optic for suitability for high-pressure research. We also initiated a travel program for Tulk, Parise and postdoc Darren Locke, to engage in research to discover the limitations and advantages of focusing optics and detectors. The results of these efforts,

Fig. 1 Images of the K-B mirrors installed on the engineering beamline of the Chalk river reactor, ON, Canada. In the upper left image the gem cell is installed with an imaging plate detector at 90° to the incident beam. A close-up of the mirrors, shown in the lower right.

summarized below, have been used to update the beamline design, to accommodate the new optic. Since we began testing, outreach to a broader community of potential SNS users has resulted in enthusiastic adoption. A similar optic is being constructed for the single crystal beamline.

Fig. 2 Focusing in the horizontal (110 μm) and vertical (90 μm) with polychromatic neutrons at Chalk River

- 2) Outreach to the Geosciences community: Parise is co-editing an issue of the popular magazine "Elements" concentrating on the Deep Earth. Pressure plays a major role in this research. The issue will appear in June 2008. The web site where this will be available as Vol.4, issue 3 (June 2008) located at <http://www.elementsmagazine.org/>. Two chapters are published as part of a volume of instructional materials resulting from the December 3, 2006, short course "Neutrons in the Earth Sciences". One on "Theory of neutron scattering" and a second on "High pressure studies"; the latter includes the latest SNAP design and features.
- 3) Outreach to universities, industry and national labs: Parise gave the following presentations related to the SNAP outreach efforts:

2006 "In situ studies of "crystallographically challenged" materials" one of the Sandia National Laboratory distinguished lecture series, Sept 14

"SNAP: high pressure at SNS" Gordon Research Conference on High Pressure; June 25-30

"Local and intermediate range structure in crystalline, nanocrystalline and amorphous materials" Denver Diffraction Conference, Denver, CO; 7-11 August

"Neutron scattering of frameworks under high pressure" Goldschmidt Conference, Melbourne Australia, August 30.

"Introduction to properties and applications of neutrons" Part of the MSA/GSA short course "Neutron Scattering in Earth Sciences" Berkeley, Dec 2006

"High pressure applications" Part of the MSA/GSA short course "Neutron Scattering in Earth Sciences" Berkeley, Dec 2006

2007: "New structural mineralogy from analysis of total X-ray/neutron scattering" University of Chicago, Jan 5

"Structural studies of nano-minerals" Lamont-Doherty Observatory, Feb 7

"Crystallography at non-ambient conditions using access to the APS-XOR beamlines" XOR SAC review June 27, 2007

"Neutron focusing and the promise for high pressure Laue scattering" Laue 2007, Grenoble France Jan 23 – 27

"Time resolved studies using high energy X-rays and neutrons" APS user meeting, May 10 2007

- 4) Outreach to universities, industry and national labs: Parise gave the following presentations

related to the SNAP outreach efforts:

2007 Neutron focusing and the promise for high pressure Laue scattering” Laue 2007, Grenoble France Jan 23 – 27

"Time resolved studies using high energy X-rays and neutrons" APS user meeting, May 10 2007

"Mineralogy on small length scales" GSA symposium honoring Gordon Brown, October 29, 2007

"Crystallography at non-ambient conditions" XOR SAC review June 27, 2007

2008 "Beyond Rocks: Geology and Planetary Sciences with X-rays and neutrons" Cornell University American Association of Physics Teachers, April 18 - 19

"Crystallography" Associated Scottish Universities Summer School in Physics, May 27-28, Skye

- 5) Presentations at conferences involving SNAP personnel including postdocs Locke and Ehm
American Crystallographic Association Meeting Oak Ridge TN

Structural Changes in Nano-crystalline Mackinawite (n-FeS) at High-pressure. Lars Ehm, Marc Michel, SytleAntao, Peter Chupas, Peter Lee, David Martin, Sarvjit Shastri, John Parise.

Disorder in sII and sH Krypton Clathrate Hydrates. Bryan Chakoumakos, LingYang, D.D.Klug, C.A.Tulk,D.Martin, Lars Ehm, J.B. Parise.

A New Hexagonal Phase for Pressure-Quenched Xe Clathrate Hydrate. L. Yang, C. A. Tulk, D.D. Klug, L. Ehm, D. Martin, B.C. Chakoumakos, J.J. Molaison, J. B. Parise

- 6) Articles appearing or in press

McIntyre, G. J., Melesi, L., Guthrie, M., Tulk, C. A., Xu, J. and Parise, J. B. (2005) One picture says it all—high-pressure cells for neutron Laue diffraction on VIVALDI J. Phys.: Condens. Matter 17, S3017–S3024 doi:10.1088/0953-8984/17/40/004

Chapman, K. W., Chupas, P. J., Kurtz, C. A., Locke, D. R., Parise, J. B. and Hriljac, J. A. (2007) Hydrostatic low-range pressure applications of the Paris–Edinburgh cell utilizing polymer gaskets for diffuse X-ray scattering measurements J. Appl. Cryst., 40, 196-198

Martin, C. D., Chupas, P. J., Chapman, K. W. and Parise, J. B. (2007) Local vs. average structure: A study of Neighborite (NaMgF₃) utilizing the pair-distribution function method for structure determination. J. Appl. Crystallog. 40, 441-448

Michel, F. M., Ehm, L., Antao, S. M., Chupas, P. J., Lee, P., Liu, G., Strongin, Schoonen, M. A. A., Phillips, B. L. and Parise, J. B. (2007) The structure of ferrihydrite, a nanocrystalline material, Science, 316, 1726-1729

Ehm, L., Antao, S. M., Chen, J., Locke, D. R., Michel, M. F., Martin, C. D., Yu, T., Parise, J. B., Chupas, P. J., Lee, P. L., Shastri, S. D., Guo, Q. (2007) Studies of local and intermediate range structure in crystalline and amorphous materials at high pressure using high-energy X-rays, Powder Diffraction, 22, 108-112

Martin, C. D., Meng, Y., Prakapenka, V. B., Parise, J. B. (2008) Gasketing optimized for large sample volume in the diamond anvil cell: first application to MgGeO₃ and implications for structural systematics of the perovskite to post-perovskite transition. J. Appl. Crystallog. 41, 38-43

Antao, S. M., Benmore, C. J., Li, B., Wang, L., Bychkov, E. and John B. Parise, J. B. (2008)

Network Rigidity in GeSe₂ Glass at High Pressure, Phys. Rev. Lett., 100, 115501
Ehm, L., F. M. Michel, S. M. Antao, C. D. Martin, P. L. Lee, S. D. Shastri, P. J. Chupas and J. B. Parise (2009) Structural changes in nanocrystalline mackinawite (FeS) at high pressure, J. Appl. Crystallog. 42, 15-22

Graduate student theses and postdoctoral fellows trained

Graduate Students, year, prizes, present position

Marc Michel, PhD, 2008, *Awarded President's award for PhD thesis 2008 (one of two awarded per year)* now at SLAC/Stanford as beamline scientist.

David Martin, PhD, 2007, now director's post doctoral fellow, APS, Argonne National Laboratory

Sytle Antao, PhD 2006, now Assistant Professor, Calgary, Ca

Aaron Celestian, PhD 2005, Assistant Professor Western Kentucky University

SNAP Post docs and present positions

Lars Ehm, joint appointment, research assistant professor/scientist Stony Brook/BNL National Synchrotron Light Source, 2008 -

Darren Locke, postdoctoral fellow, APS, Argonne National Laboratory, 2007 -

Paul Forster, Assistant Professor of Chemistry, UNLV, 2007-

1st Annual Meeting Report

The 1st annual meeting of the Spallation Neutrons and Pressure (SNAP) executive and advisory committee was held April 6 and 7, 2004 at the LANSCE conference center in Los Alamos. Future meetings will be held in Oak Ridge and planning for the second meeting in June 2005 is advanced with Emily Vance meeting with potential local organizers in Oak Ridge in July 2004. Well over 45 people, including members of the LANSCE high pressure group and other LANSCE facility notables, attended the spring '04 meeting in Los Alamos. The two-day meeting concentrated on the vital steps to be taken in the next year to coordinate with the SING effort, cement the instrument design, resolve outstanding detector and focusing issues and coordinate with other efforts in the condensed matter community in order to make SNAP and establishing a high pressure culture at SNS a reality. Attendees from major neutron scattering facilities, including ISIS, Los Alamos, Oak Ridge and IPNS were in attendance as were the high-pressure cell experts. Considerable progress in cell design, vital to providing SNAP with the next generation of high-pressure devices, was reported. A brief summary of action items arising from the meeting is as follows (presentations from the participants are available and included in a separate CD ROM along with initial planning documentation)

- 1) Beamline design: The IDT/advisory and executive committees were supportive of the broad goals in the beamline design, and agreed this aspect was on track. They encouraged some flexibility in the beamline design, so that detector distances and sample distances might be more easily varied. This would give the beamline a more synchrotron feel. It was important that the design not compromise the core mission of providing high quality high-pressure data. It was felt that the opportunity to build a new instrument, one reflecting the lessons learnt from previous generations of design, should not be missed.
- 2) Detectors and focusing: It is vital final decisions, based on real world experience with cells, be made on detector and focusing options. Following up on this advice from the meeting recent tests at IPNS with gems cells have narrowed the choice of detectors to those being developed at Oak Ridge. A breakthrough by Gene Ice at Oak Ridge in focusing could revolutionize single crystal diffraction and the meeting urged quick decisions on the suitability of the optic. Provision has been made in the beamline design and ground-truth tests are planned in mid-July 2004 at Chalk River. These detector tests at IPNS were carried out by Chris Tulk and the decision was made to proceed with detectors which would be made at Oak Ridge. This provides maximum detector coverage while not compromising, resolution, cost, sensitivity or peak-t-background discrimination.
- 3) Outreach: A well-organized effort by Earth Scientists was described and the outreach by SNAP to that community is ensured by Parise/Hemley/Mao involvement in the geosciences program of the Consortium for Mineral Physics Research in the Earth Sciences (COMPRES). We recently appointed Darren Locke as a postdoctoral scientist. Darren has close ties to this community, is an expert in high-pressure apparatus and has attended a number of COMPRES sponsored neutron scattering workshops. Prof. Nancy Ross is in charge of the COMPRES neutron effort and she is working closely with the SNAP PIs.

One important aspect of the SNAP meeting is feedback and advice from the advisory committee. To complement this aspect we invited individuals from groups to join us in our annual IDT meetings; the invitees at The Los Alamos meeting included Prof. B. Fultz of Caltech, and this outreach activity will continue in the future. With recent advances in focusing and an increase in interest in single crystal work possible invitees for the next SNAP meeting at Los Alamos will include representatives from the single crystal effort, Gene Ice who is heading the focusing effort and representatives from the X-ray energy dispersive high pressure single crystal interest group and from the corresponding neutron community (Garry McIntyre from ILL, for example). Attendees at SNAP-I agreed that software development is key to success. We believe one of the most effective ways to bring new ideas into the high pressure community, and to project these outwards, is to include a range on new people and ideas at our annual meeting. Of course, we will continue to support travel and attendance at meeting. We believe for the future, a "point person" that can help potential users with high-pressure experiments at existing sources is vital. We are moving ahead with this and hope to report

significant new initiatives at SNAP-II at Aok Ridge in June 2005..

Spallation Neutrons and Pressure – SNAP - Outreach 2004-05

John B. Parise, Stony Brook

Summary: The major out reach efforts of the IDT during this year were

- 1) The testing of the suitability of the “ice” mirrors” for single crystal neutron scattering
- 2) Planning for the second SNAP executive and advisory committee at SNS, Oakridge July 17-19 2004
- 3) Analysis of single crystal data collected from high-pressure cells using “pink beams”
- 4) Planning for and participation in two thematic sessions on neutrons at the Vienna meeting of the European Geological Union (EGU) in April 2005 and at the ACS meeting in Washington, DC to be held August 2005.
- 5) Participation in a number of Earth Science for a including the COMPRES (Consortium for Materials Property Research in Earth Sciences)

1). *The original SNAP beamline design included a focusing optic.* The optic under consideration at that time (2002) was a capillary bundle. Gene Ice, at Oakridge meanwhile, was developing Kirkpatrick-Baez mirrors for neutron focusing. Upon learning of this in 2004 we immediately set about testing this optic for suitability for high-pressure research. We also initiated a travel program for Tulk, Parise and postdoc Darren Locke, to engage in research to discover the limitations and advantages of focusing optics and detectors. The results of these efforts, summarized below, have been used to update the beamline design, to accommodate the new optic. Since we began testing, outreach to a broader community of potential SNS users has resulted in enthusiastic adoption. A similar optic is being constructed for the single crystal beamline.

2). *Following a very successful initial meeting at Los Alamos National Laboratory, it was decided future meetings will be held in Oak Ridge.* The next meeting will be held July 17-19 with the program and list of attendees given in the description section below. A full report of the meeting will be included as an update following the meeting. This meeting was delayed from the “usual” Spring meeting time since it was preempted by the Neutrons in Earth Science Symposium in Vienna in April. Over 50 people are expected for the July 17 meeting, including a number of beamline scientists from the SNS who are interested in incorporating high pressure into their programs. We regard this as an essential part of the SNAP outreach effort. The two-day meeting is planned around formal presentations from advisory committee members as well as invited guests who are important to the SNAP mission. Most important amongst these attendees are those involved in the cutting edge aspects of the instrument, including focusing (Ice) cell development (Klotz) and detector construction (Cooper). Several science talks are planned with recent innovations in collecting and analyzing data from small single crystals by members of the SNAP team (Tulk, Parise, Mao, Hemley, Xu and Locke) and by those in the European theatre (McIntyre and Loveday).

3) *The collaboration of McIntyre* at the ILL on the collection and analysis of single crystal data has allowed us to jump-start the program. Garry will be joining us at the SNS for the 2nd SNAP meeting in July, and he is already scheduled to talk not only to the SNAP team and collaborators but also to those on the single crystal instrument interested in his expertise.

Many of these out reach efforts are of general interest to the condensed matter community and will help in establishing a high-pressure culture at SNS.

4) *Other outreach efforts* over the past year have included talks by the PIs at national meetings and coordination with specific interest groups. For example Parise was a co-convenor of the Symposium on Neutrons at the Frontier of Earth Sciences and Environment – NESE – part of the 2nd General Assembly of the European Geosciences Union EGU. This meeting was held in Vienna, Austria, 25 - 26 April 2005.

NESE is one of a series of conferences organized jointly by European, American and Japanese scientific institutions with a strong affiliation to Neutron Scattering. Two days of interesting talks showing the most recent experiments carried out with neutrons in the areas of Earth Science and Environment. 32 Speakers from the States, Japan and Europe reported about the advances on the effectiveness of the neutron techniques providing information about atoms arrangements and dynamics of minerals.

The presentations, including those given by SNAP PIs Tulk and Parise are available on the NESE web-site, http://neutron.neutron-eu.net/n_nmi3/n_networking_activities/n_nese/860.

One picture says it all – high-pressure cells for neutron Laue diffraction

Possible applications of the neutron single-crystal Laue diffraction technique with a large image-plate detector to high-pressure studies were examined during a series of experiments at the ILL in the Fall of 2004 and Spring 2005. Experiments are continuing during summer 2005 and there are several very encouraging results.

One opposed-piston cell with a Ti-Zr casing is shown to be acceptable for medium pressures. For higher pressures a moissanite-anvil cell with reasonably large accessibility is shown to offer impressive gains in data collection rate as compared to the monochromatic technique. The moissanite anvil cell is the device being constructed at the Geophysical laboratory under the supervision of SNAP PIs Hemley and Mao and SNAP technical officer Xu.

An unanticipated but very useful result is that the projected forms of the reflections from the sample and anvils facilitate alignment, and the wide wavelength band of the Laue technique allows recovery of reflections masked by the cell pillars, simply by rotation of the cell. The time resolved

Fig. 9 (left) Magnetic structure of hematite at room temperature. The rhombohedral unit cell containing four unique iron atoms is shown. (right) Low angle diffraction pattern of hematite (Fe₂O₃) showing disappearance of the magnetic only (111) peak as the moment reorients from perpendicular to parallel to the [111]_{rhombohedral} direction.

Laue technique will be used at SNA and so these initial results suggest the GEM cells will be useful not only at SNAP but at other beamlines as well.

4. Application of pressure cells for powder diffraction at Reactor sources

One outgrowth of the SNAP program at Chalk river (where focusing efforts have been worked on over the past year) has been the use of high pressure cells with pink beams. However, monochromatic diffraction is also possible if the cells is carefully shielded, to prevent parasitic scattering, if neutron transparent TiZr is used as a gasket material, and if cubic boron nitride (c-BN) anvils are used. This later innovation allows high pressures to be achieved while allowing self collimation by the neutron absorbing c-BN. We have applied these techniques to study the magnetic transitions in $\alpha\text{-Fe}_2\text{O}_3$ (hematite).

At room pressure and temperature hematite is antiferromagnetically ordered with moments aligned in the basal plane, perpendicular to the rhombohedral axis (Fig 9). Below about 250 K (the Morin transition [1]) the magnetic moments reorient along [111]. This transition is easily followed by measuring the relative intensities of the magnetic only reflections, 111 and 100 (Fig. 10); below the transition the 111 reflection disappears. The effect of pressure on has been studied by several workers, including those in references [2-4]. Pressure induced antiferromagnetic ordering and moment reorientation is well known, and so it is surprising that the pressure equivalent of the Morin transition in hematite is apparently difficult to induce [3]. Several reasons are cited, including the deviatoric stress induced by pressurizing solid media.

We have carried out studies to 7 GPa nominal pressure, using fluorinert as a pressure transmitting medium, in a Paris-Edinburgh cell at the DUAL-spec powder diffractometer at Chalk River. Although originally adapted to spallation sources, we find the PE cell, especially when properly shielded to prevent parasitic scattering from overwhelming signal from the sample, to be quite suitable for studies of magnetic phase transitions at high pressure [5]. For the hematite study, we pressurized both bulk and nano-crystalline materials and observe a near disappearance of the 111

Fig. 10 Angle of the moment on the Fe atom to the hexagonal c-axis (rhombohedral [111]) as a function of nominal pressure.

reflection at 7 GPa nominal pressure. Rietveld refinement using data collected between room pressure and 7 GPa nominal allowed the moment reorientation to be followed (Fig. 3).

1. FJ Morin, Phys Rev 78 (1950) 819
2. CG Shull and EO Wollan Phys Rev 83 (1951) 333
3. IN Goncharenko, J-M Mignot, G Andre, OA Lavrova, I Mirebeau and VA Somenkov High Press Res 14 (1995) 41
4. N Kawai, F Ono and KH Hirooka J Appl Phys 39 (1968) 712
5. http://www.ill.fr/AR-03/site/02_scientific/021_magnetic/02_magn_10.htm

5. Preliminary program for SNAP-II July 17-19 SNS, Oakridge National Laboratory

***** **DRAFT 7** *****

**Spallation Neutrons and Pressure
2nd Annual Meeting
July 18 - 19, 2005
Oak Ridge, Tennessee**

Sunday, July 17, 2005 **Arrival and Check-in**
The Oak Ridge Comfort Inn
433 S. Rutgers Avenue
Oak Ridge, Tennessee 37830
Tel: 865-481-8200

Monday, July 18, 2005

8:00 am	BADGING (See Instructions on Greetings page)
9:00 – 11:00am	Tour of the Facility
11:00 – 11:15pm	BREAK
11:15 – 11:20am	Welcome John B. Parise, State University of New York, Stony Brook
11:20 – 11:40pm	Overall SNS Status Ian Anderson, Oak Ridge National Laboratory
11:40 – 12:00pm	SING → SNS → SNAP John Haines, Oak Ridge National Laboratory
12:10 – 12:20pm	Discussion
12:20 – 1:30pm	LUNCH

BEAMLINE

1:30 – 1:55pm	Beamline Status Specifics Christopher A. Tulk, Oak Ridge National Laboratory
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1:55 – 2:00pm	Discussion
2:00 – 2:20pm	Detectors for SNAP Ronald G. Cooper, Oak Ridge National Laboratory
2:20 – 2:30pm	Discussion
2:30 – 2:50pm	Science at Vivaldi Garry McIntyre, ILL
2:50 – 3:00noon	Discussion
3:00 – 3:15pm	<i>BREAK</i>

POSTER SESSION

3:15 – 3:45pm	Oral Presentations
3:45 – 5:15pm	Poster Presentations

HIGH PRESSURE CELLS

5:15 – 5:35pm	Development at Los Alamos Yusheng Zhao, Los Alamos National Laboratory
5:35 – 5:45pm	Discussion
5:45 – 6:05pm	Progress in Moissanite Anvil Cells for Neutron Diffraction Jian Xu, Carnegie Institute of Washington
6:05 – 6:15pm	Discussion
6:30 – 8:30pm	<i>DINNER</i>

Tuesday, July 19, 2005

8:00 – 8:30am	Coffee and Mingle
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8:30 – 8:50am	High-pressure beamline at the new neutron source, J-PARC, in Japan Hiroyuki Kagi, Tokyo University
8:50 – 9:00am	Discussion
9:00 – 9:20am	What's new in the PE world? Stefan Klotz, University of Paris
9:20 – 9:30pm	Discussion
9:30 – 9:50pm	New Cell Designs for ISIS Martin Dove, University of Cambridge
9:50 – 10:00pm	Discussion
<i>10:00 – 10:15am</i>	<i>BREAK</i>

NEW NEUTRONICS

10:15 – 10:35am	Next-generation High-Performance Neutron Focusing Optics Gene Ice, Oak Ridge National Laboratory
10:35 – 10:45am	Discussion
10:45 – 11:05pm	Progress on SXD John Loveday, University of Edinburgh
11:05 – 11:15am	Discussion
11:15 – 11:35pm	Something on Diffuse Scattering Dave Keen, ISIS
11:35 – 11:45pm	Discussion
<i>11:45 – 12:45pm</i>	<i>LUNCH</i>

POSTER SESSION

12:45 – 1:00pm	Oral Presentations
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1:00 – 2:00pm Poster Presentations

OUTREACH & COMMUNITY DEVELOPMENT/NEW SCIENCE

2:00 – 2:20pm Neutron Clathrate
Ian Swainson, Chalk River National Labs

2:20 – 2:30pm Discussion

2:30 – 2:50am Amorphous Ices
Christopher A. Tulk, Oak Ridge National Laboratory

2:50 – 3:00pm Discussion

3:00 – 3:15pm BREAK

3:15 – 3:35pm

3:35 – 3:45pm Discussion

3:45 – 4:05pm CLOSING REMARKS
Russell J. Hemley, Carnegie Institution of Washington

4:05 – 4:15pm Discussion

4:15 – 5:45pm Executive Committee Meeting Wrap-up

Spallation Neutrons and Pressure – SNAP - Outreach 2005-06
John B. Parise, Stony Brook

Summary: The major outreach efforts during this year were

- 5) Continued testing of the suitability of the “ice” mirrors” for single crystal neutron scattering at Chalk River reactor and adaptation of cells to orientation hardware.
- 6) New mounts for new generation PE cells were built for studies at Chalk River, Los Alamos, IPNS and APS. This involved considerable work to adapt the cells but resulted in new science and the introduction of several new users to high pressure techniques. These mounts were vital for testing cells and collecting data to test strategies for powder structure refinement etc and study of magnetic and amorphous materials. With the SNAP cells soon to be deployed to SNS, this exciting science can continue if new cells are procured for the facilities, especially the APS. The cell adaptors, not useful at SNS, will stay in place at APS, IPNS, LANSCE. A lasting legacy of this outreach from SNAP. These mounts will be used by new users.
- 7) Discussions with HIFR at Oak Ridge, during Sept 28, 2005 review of SING instruments, with Brian Chakomakous on possibility of putting new generation PE cell on powder instrument. Requires some design modification but there is pressure from users to start using (mainly Ross at VPI with the COMPRES group). Cells were tested successfully at the Oak Ridge HIFR reactor. The success of these experiments prompted us to move all cells to Oak Ridge 12 months ahead of schedule and to relocate post doc Darren Locke to take advantage of lab space to store cells and begin the integration of high pressure into the SNS culture.
- 8) The second executive and advisory committee meeting (SNAP 2005) at SNS, Oak Ridge, July 17-19 2005 (delayed from Spring because of the NESE neutrons in Earth Science meeting in Vienna, Austria) details are attached.
- 9) The third executive and advisory committee meeting (SNAP 2006) at SNS, Oak Ridge, April 10-11 concentrated on science results; details are attached.
- 10) Planning for SNAP 2007 has begun.
- 11) Further analysis of single crystal data collected from high-pressure cells using “pink beams”.
- 12) A paper was prepared on "New opportunities at emerging facilities" and published in the Elements magazine (a copy of the article and the full magazine are attached).
- 13) Program committee assignments and participation in a number of Earth Science and Materials Science forums including the COMPRES (Consortium for Materials Property Research in Earth Sciences) meetings, American Chemical Society special session on neutron scattering, Washington, DC, International Mineralogical Association, Kobe (program committee).
- 14) Workshops on high pressure included a September 13, 2005, gathering at Los Alamos to discuss the high pressure program and requirements for a large volume high pressure device to be installed at Los Alamos and perhaps, eventually, at SNAP, SNS.
- 15) Outreach to industry: Parise gave a talk at the Gillette Company R&D facility "Directed high pressure synthesis and time resolved neutron scattering" host Paul Christian.
- 16) Outreach to the Environmental community: Parise presented a talk to Synchrotron Environmental Science-III held at NSLS "Complementary use of neutrons and synchrotron radiation for environmental studies".
- 17) Outreach to the Geosciences community. Two chapters are being prepared for the December 3, 2006, short course "Neutrons in the Earth Sciences". One on "Theory of neutron scattering" and a second on "High pressure studies" which will include the latest SNAP design and features.

Expansion on specific points

1). Further tests on the focusing optic and orienting single crystals in the beam. SNAP like all SNS lines has challenges built into the design for focusing, as the importance of imaging on the sample was not realized in the early design. Specifically there is a limited path length available for adjustable slits and other essential optical elements (2.5 m). There is also the need for about 250 mm of clearance between the end of the optical elements and the sample. Finally the imaging system must be removable to allow for other optical approaches. The optic under consideration at that time (2002) was a capillary bundle. Gene Ice, at Oak Ridge meanwhile, developed Kirkpatrick-Baez mirrors for neutron focusing, which we successfully tested (see last year's report) with crystals both inside and outside the gem cells. The initial results are used to update the beamline design. Since we began testing, outreach to a broader community of potential SNS users has resulted in enthusiastic adoption. A similar optic is being constructed for the single crystal beamline and TOPAZ. IDT members are invited speakers to our IDT meeting where common concerns on sample location, software and optics are regularly discussed.

Figure 1. (left) SNAP 1st generation and (middle) 2nd generation mirrors (see text). A recently tested hexapod positioning table is shown right.

The 2nd generation SNAP mirror design is based on the use of nested mirrors. This design is much more compact than a traditional KB mirror system which makes it ideal for the SNAP beamline. It also allows for a larger sample to optics distance and because both mirrors are relatively large, can work well up to a larger beam size when needed. As can be seen, in a normal KB mirror system (Fig. 1 left), if a second mirror is placed before its paired partner, it must be very long and very perfect (expensive and difficult to make). If it is placed after its partner mirror, then it restricts the clearance to the sample. The beamline optic, now a nested design (Fig. 1 middle), will be more compact, provide a larger distance between optic and sample and a larger acceptance with uncompromised beams up to ~ 2 mm. It is believed that, with improved optics, the $90 \mu\text{m}$ beams we have already obtained at Chalk River in 2004 can be reduced to $25 \mu\text{m}$. The possibility of obtaining usable data on laboratory X-ray sized crystals is now very real and must be tested. We are making plans to do this at Los Alamos this summer. Provided we obtain the agreement of LANSCE administrators we will construct a mock-up of SNAP (detectors, focusing optic, positioning table

(Fig 1 right). Not only will the SNAP mirrors be good for SNAP, but we believe they will be widely adopted and will be the first wave of a revolution in neutron science. Our vision is that this form of nondispersive focusing optics will become essential for all samples less than 2 mm in size. Sample positioning stages compatible with the focusing optic are also being considered and will be tested.

2). *New mounts and outreach to a new generation of high pressure researchers.* The nation's high pressure capabilities are now at synchrotron sources. The best in the country is the APS and it is situated close to IPNS where we do much of our testing. This is an excellent opportunity to engage some of the nation's brightest synchrotron researchers and to at the same time test our hardware. We therefore began a program of high pressure work at the XOR high energy beamlines at APS where we engaged Peter Lee, Pete Chupas, Karina Chapman, Dean Haefner, Sarjvit Shastri and others. This collaboration has been spectacularly successful not only in enabling us to test hardware that will eventually be at SNS (it is much easier to measure pressure and test line-of-sight optics at a synchrotron source) but it has introduced young enthusiastic scientists to pressure research. Here is a list of publications resulting from collaborations over the past year. Several manuscripts in preparation are not listed.

- Martin, C. D., Antao, S. M., Chupas, P. J., Lee, P. L., Shastri, S. D. and Parise, J. B. (2005) Quantitative High Pressure Pair Distribution Function Analysis of Nanocrystalline Gold, *Appl. Phys. Lett.* 86, 061910-1 - 061910-3.
- Colligan, M., Lee, Y., Vogt, T., Celestian, A. J. Parise, J. B., Marshall, W. G. and Hriljac, J. A. (2005) High Pressure neutron diffraction study of superhydrated natrolite *J. Phys. Chem.* 109, 18223-18225.
- McIntyre, G. J., Melesi, L., Guthrie, M., Tulk, C. A., Xu, J. and Parise, J. B. (2005) One picture says it all—high-pressure cells for neutron Laue diffraction on VIVALDIJ. *Phys.: Condens. Matter* 17, S3017–S3024 doi:10.1088/0953-8984/17/40/004.
- Parise, J. B., Antao, S. M., Michel, F. M., Martin, C. D., Chupas, P. J. and Lee, P. L. (2005) Quantitative high-pressure pair distribution function analysis, *J. Synchrotron Rad.* 12, 554-559.
- Michel, F. M. Antao, S. M. Chupas, P. J. Lee, P. L. Parise, J. B. and Schoonen, M. A. A. (2005) Short- to Medium-Range Atomic Order and Crystallite Size of the Initial FeS Precipitate from Pair Distribution Function Analysis *Chem. Materials* 17, 6246-6255.
- Michel, F. M. Schoonen, M. A. A., Zhang, X. V., Martin, S. T. and Parise, J. B. (2006) Hydrothermal Synthesis of Pure -Phase Manganese(II) Sulfide without the Use of Organic Reagent, *Chem. Materials*, ASAP Article; DOI: 10.1021/cm048320v.
- Parise J. B., Brown G. E. (2006). New opportunities at emerging facilities, *ELEMENTS* 2, 37-42.

Not listed here are publications resulting from users such as Jim Martin (North Carolina) Chupas and Chapman (ANL) Benmore (ANL) Tulk (SNS) and others who used the cell independently.

An added bonus to this outreach is the training of young scientists in the use of the high pressure apparatus so that they will be independent of the SNAP group when the cells move to Oak Ridge this year. The downside of course, is that a vital and growing research program in high pressure at XOR will be left without cells and expertise. We are moving to redress this problem by submitting a proposal to DOE call **DE-FG02-06ER06-13**. The provision of funds to continue this

excellent work is a priority since some further testing of cells, and their continued improvement will benefit SNAP. For example, although the powder scattering technique is now well integrated we still have to adapt software. This is more easily done at synchrotron sources; despite the heavy competition for time there are still more X-rays than neutrons, and those X-rays are close to the IPNS.

The following photo collage shows some of the experiments carried out over the past year with the captions explaining where the experiments were carried out and identifying key players in the enterprise.

2a. Experiments at 11-ID with SNAP Paris-Edinburgh cells and gem cells

Figure 2. (left) The PE cell designed for the SNAP instrument mounted (with the breach removed) on the 11-ID Huber diffractometer. Peter Chupas (XOR) contemplates the next move (middle) while Paul Forster (Stony Brook) and Karena Chapman (ANL Materials) contemplate data (right)

2b Experiments at IPNS and LANSCE to test collimation and perform high pressure experiments on GeSe_2 glass and other glasses and framework materials.

Figure 3. (left to right). Adapter built at Stony Brook with outreach funds to allow the next generation VX SNAP PE cells to be mounted for use at IPNS. Tulk (SNS) and Benmore (IPNS/APS) loading the cell into the GLAD instrument. Post-doc Darren Locke (Stony Brook) preparing sample and loading into the HIPD instrument at LANSCE where Anna Lobert (LANSCE, LANL) is instrument responsible and has now used cells with her own program and that of users.

2c. Experiments to adapt the large volume cells and gem cells to cryostats and to sample positioners.

Figure 4. (left) The VX PE cell mounted on the hexapod positioner. The adapter is built to protect the high pressure tubing extending from the bottom of the cell. This device also fits the powder diffractometer at the HiFR reactor at ORNL where initial and successful tests were carried out (point 3 above) by painting the anvils with Gd paint (the white above) which provided good collimation as an alternative to the BN anvils we used in previous tests. (Right) The VX-5 PE cell (from the Paris lab of Stefan Klotz, the group leader of the PE project) shown mounted on an adapted "orange cryostat. This is a viable solution for cooling the cell.

2d. Experiments at the 1-ID beamline using gem (diamond and SiC-moissonite) anvil cells

Figure 5. (left) diamond anvil cell mounted on the 1-ID beamline at APS showing the GE detector (red handle). Some of the data collected on sample are used to justify a preproposal by colleague Clare Grey from Stony Brook to purchase such a detector. This preproposal is separate from one being submitted for high pressure devices. While the GE detector is useful for high pressure

research it is not necessary and so we decided to submit the high pressure work as a separate request. (Middle) Lars Ehm (Stony Brook) is a post doc on the SNAP project overseeing the single crystal and DAC adaptation work. Sytle Antao (graduate student, Stony Brook) will be joining the XOR group as a postdoc in June 2006. She is well versed in all aspects of high pressure work and work with the GE detector of pair distribution function work. (right) The Geophysical lab "gem" cell for use at SNAP mounted in the through anvil geometry with the beam entering right and scattering being detected on the black (GE) detector left.

3) HIFR and the ORNL "high pressure laboratory". Test at HIFR in the winter of 2005-2006

produced patterns comparable to those obtained at ILL and Chalk River [Parise, 2006 #218; Kernavanois, 2002 #238] as part of this outreach effort. Based on these results and the needs of users we have decided to move all cells to ORNL and to establish a high pressure laboratory to service the needs of the SNAP beamline. Some funds from the outreach grant will be needed for this as postdocs Darren Locke will have to establish the lab. Darren (Fig. 3) will be leaving for ORNL in May to search for housing and to establish a presence in the Geochemistry group who have offered space for the lab. Eventually we will be looking for office and lab space in SNS and having "boots on the ground" will help this. The upside of this move is the availability of cells for ORNL projects. The downside is the depletion of capabilities at ANL just as the user base at XOR for high pressure is expanding. We hope to redress this gap with DE funding for HP instrumentation for the personnel trained in high pressure by the SNAP outreach grant (Figs. 2, 3 and 5).

4) *Other outreach efforts* over the past year have included talks by the PIs at national meetings and coordination with specific interest groups. Several review type articles have been written and copies are attached.

4. SNAP 2005 July 17-19 SNS, Oak Ridge National Laboratory. The 2nd annual meeting of the Spallation Neutrons and Pressure (SNAP) executive and advisory committee was held July 18-19 2005 at the SNS office conference center in Oak Ridge. Action items arising from discussions at the meeting included:

- 1) Exploration of the possibility for a large volume device, of similar design to that currently being considered at Los Alamos at the LANSCE facility, and making sure the design accommodates this possibility.
- 2) Final decisions of the detector-focusing package are in place and decisions to proceed with ORNL designs were endorsed by the IDT.
- 3) Software design is a concern of the IDT and SNAP 2006 will include presentations from appropriate SNS staff coordinating these efforts.
- 4) Software being used by the TOPAZ team should be aired at SNAP 2006 and Art Shultz and Christina Hoffman will be invited to speak.
- 5) The COMPRES community executive committee and facilities committees should be invited to SNAP 2006.

Following up on these recommendations lead to many of the points summarized above including adaptation of cells, cell testing and decisions to move operations early to ORNL/SNS/HIFR.

5. SNAP 2006 April 10-11, 2006, SNS, Oak Ridge National Laboratory. The 3rd annual meeting of the Spallation Neutrons and Pressure (SNAP 2006) executive and advisory committee was held April 10-11 at the SNS office conference center in Oak Ridge.

Science Highlights SNAP 2006

SNAP 2006 continued the tradition established in Los Alamos in 2004 of having IDT members, the executive committee and ad hoc invitees add technical advice on the mix of innovative ideas for constructing the world's first optimized neutron high pressure beamline. The technical suggestions included several ideas for the location of small samples in focused beams that we will be following up on. The X-ray community again provided excellent ideas including possible adoption of YAG imaging optics. It was suggested that Gd doping YAG might be a suitable imaging plate for neutrons coupled to a CCD. The YAG is of order magnitude \$1K and we will purchase one to test as IPNS. If this works of course a more serious effort will be mounted. We chose this exact route when starting to develop the focusing optic option.

Other technical contributions included tours of the software suit and hooks for the automatic pumping station to be integrated into the SNS software philosophy. Rick Riedel provided an excellent overview of the software operation.

Heating was initially omitted from the hardware package but at the urging of the IDT we will have to find a way to partner with users and potential users to at least deliver components to include the possibility of heating one of the SNAP PE cells from day 1.

Apart from excellent technical discussions, we purposely included a large amount of exciting science in SNAP 2006. Several IDT members had recent exciting results to share and a number of post-docs and student resented on going research. A brief summary of highlights follows.

Hydrogen storage in molecular compounds: Recent graduate Wendy Mao, now a post-doc at Los Alamos presented a summary of recent efforts to study molecular compounds for hydrogen storage. These materials, which include $\text{H}_2\text{-H}_2\text{O}$, $\text{H}_2\text{-CH}_4$, and other clathrates and ices are stabilized at high P and low T. Particularly interesting were very recent experiments with multi-components and adding promoter molecules (e.g. CHCl_3 , CCl_4 , SF_6 , Xe, THF) or CH_4 to bring hydrogen storage materials to more favorable conditions and allow formation at lower P. Other contributors to this theme were Kim Tait, a graduate student at Arizona stationed at LANSCE. Kim described nice work on clathrate systems using the gas pressure apparatus similar to that destined to be installed at SNAP.

James Martin (NC State) gave a "chemist's perspective" on how high P might influence glass structure. Starting with his own recent work on liquid crystal inorganic-organic hybrid glass and metal halide networks James described the rich glass structural chemistry accompanying changes in cations as network modifiers. It was clear that changes in structure due to changes in cation size were targets for work at high pressures.

Bjoern Winkler of the Institut für Geowissenschaften, Goethe Universität Frankfurt Germany described recent experiments at ISIS using HP-HT apparatus. Winkler described the technical aspects of temperature measurement using resonance broadening (right) and how this was used to measure the thermal equation of state of graphite. He then described fascinating new experiments and calculations related the phonon dispersion measurements under pressure and European efforts to make large samples for neutron scattering studies

John Finney of UCL, UK gave two excellent short presentations on order in ices As part of a series of short (10 min) presentations John presented his latest results (2 weeks old!). Most ices are orientationally disordered, but disordered phases should have an ordered ground state and the ordered equivalents of ice-IV, V, VI and XII are unknown. By acid doping, the preparation and structures of H-ordered phases of ice have been produced. Neutron scattering data collected at ISIS and the structures of the ordered forms were solved. John pointed out the following were critical in solving these very exciting new structures

- High flux
- Temperature variation for phase transition
- High resolution

In a second short presentation Finney described very recent experiments on doping water with THF. This is a topic of current interest since THF is proposed as a promoter for H-storage (see Mao presentation). His tentative conclusions included that THF strongly perturbs water structure while most other solutes don't! THF has an apparent compressive effect and that THF enhances interactions near lone pair regions. Interestingly pressure leads to occupying sites between hydrogens.

Garry MacIntyre (ILL) described the Laue instrument on which many of the SNAP cells were tested and where the initial trials of Laue diffraction software were carried out. Data collected on the VIVALDI Laue instrument on a sample in a Geophysical Laboratory moissanite-anvil cell: 60° vertical access 1 mm³ natrolite crystal, with steel gasket at 1 bar.

There were many other exciting talks and these are included as an appendix in the form of a CD ROM for both SNAP 2005 and SNAP 2006.

Spallation Neutrons and Pressure
2nd Annual Meeting
July 18 - 19, 2005
Oak Ridge, Tennessee

Sunday, July 17, 2005 **Arrival and Check-in**
 The Oak Ridge Comfort Inn
 433 S. Rutgers Avenue
 Oak Ridge, Tennessee 37830
 Tel: 865-481-8200

Monday, July 18, 2005

8:00 am	BADGING (See Instructions on Greetings page)
9:00 – 9:10am	Welcome Russell J. Hemley, Carnegie Institution of Washington
9:10 – 9:40am	Overall SNS Status Ian Anderson, Oak Ridge National Laboratory
9:40 – 10:00am	SING → SNS → SNAP John Haines, Oak Ridge National Laboratory
10:00 – 10:10am	Discussion
<i>10:10 – 10:30am</i>	<i>BREAK</i>

BEAMLINE: Chair – Rus Hemley

10:30 – 10:50am	Beamline Status Specifics Christopher A. Tulk, Oak Ridge National Laboratory
10:50 – 11:00am	Discussion
11:00 – 11:20am	Detectors for SNAP Ronald G. Cooper, Oak Ridge National Laboratory
11:20 – 11:30am	Discussion
11:30 – 11:50pm	High-pressure beamline at the new neutron

source, J-PARC, in Japan
Hiroyuki Kagi, Tokyo University

11:50 – 12:00noon Discussion

12:00 – 1:30pm LUNCH

Lunch Presentation:
Update on Neutron Scattering Opportunities and High Pressure
John B. Parise, State University of New York, Stony Brook

HIGH PRESSURE CELLS: Chair – John Parise

1:30 – 1:50pm Development at Los Alamos
Luke Daemen, Los Alamos National Laboratory

1:50 – 2:00pm Discussion

2:00 – 2:20pm Progress in Moissanite Anvil Cells for Neutron Diffraction
Jian Xu, Carnegie Institute of Washington

2:20 – 2:30pm Discussion

FACILITY TOURS AND INFORMAL DISCUSSION ([Coffee Break](#))

3:15 – 4:15pm Group 1 Tour / Informal Discussions

4:30 – 5:30pm Group 2 Tour / Informal Discussions

6:00 – 8:30pm DINNER
Outback Steakhouse
402 S. Illinois Avenue
Oak Ridge, TN 37830
(865) 220-0783

Dinner Presentation:
Neutron Focusing and X-Ray Focusing
Gene Ice, Oak Ridge National Laboratory

Tuesday, July 19, 2005

8:00 – 8:30am Coffee and Mingle

HIGH PRESSURE CELLS (Continued): Chair – Chris Tulk

8:30 – 8:50am Application of Recent Advancements in Neutron Techniques: An Earth Sciences Perspective

Darren Locke, State University of New York, Stony Brook

8:50 – 9:00am Discussion

9:00 – 9:20am What's new in the PE world?
Stefan Klotz, University of Paris

9:20 – 9:30am Discussion

9:30 – 9:50am High-P/T Work at ISIS
Martin Dove, University of Cambridge

9:50 – 10:00am Discussion

10:00 – 10:15am BREAK

NEW NEUTRONICS

**10:15 – 10:35am Next-generation High-Performance Neutron
Focusing Optics**
Gene Ice, Oak Ridge National Laboratory

10:35 – 10:45am Discussion

10:45 – 11:05am Progress on SXD
John Loveday, University of Edinburgh

11:05 – 11:15am Discussion
11:15 – 11:35am Experimental Tests of a Supermirror Compact Focusing Device
Dave Keen, ISIS

11:35 – 11:45am Discussion

11:45 – 1:00pm LUNCH

Lunch Presentation:
SNAP Beam Line Installation
Steve Chae, Oak Ridge National Laboratory

NEW SCIENCE

1:00 – 1:20pm Freezing in Natural Porous Materials
Ian Swainson, Chalk River National Labs

1:20 – 1:30pm Discussion

1:30 – 1:50pm	Recent Experiments with Microfocusing and SNAP Mirrors
Christopher A. Tulk, Oak Ridge National Laboratory
1:50 – 2:00pm	Discussion
2:00 – 2:15pm	CLOSING REMARKS
John B. Parise, State University of New York, Stony Brook

MEETING WRAP-UP

2:15 – 2:45pm	Open Meeting Wrap-up
<i>2:45 – 3:00pm</i>	<i>BREAK</i>
3:00 – 4:30pm	Executive Committee Meeting Wrap-up

5. SNAP 2006 April 10-11 SNS, Oak Ridge National Laboratory.

SNAP 2006

**Spallation Neutrons and Pressure
3rd Annual Meeting**

April 10-11, 2006

Oak Ridge, Tennessee

Saturday/Sunday, April 8-9, 2006

The Oak Ridge Comfort Inn
433 S. Rutgers Avenue
Oak Ridge, Tennessee 37830
Tel: 865-481-8200

Arrival and Check-in

Sunday, April 9, 2006 BBQ (Information is in your meeting packets)

Monday, April 10, 2006

8:00 am BADGING (See Instructions on Greetings page)

INTRODUCTIONS: SNS, SING, SNAP UPDATES – Chair: John Parise

- 9:00 – 9:10am** **Welcome**
John B. Parise, Stony Brook University
- 9:10 – 9:30am** **Overall SNS Status**
Ian Anderson, Oak Ridge National Laboratory
- 9:30 – 9:50am** **SING → SNS → SNAP**
John Haines, Oak Ridge National Laboratory
- 9:50 – 10:00am** **Discussion**
- 10:00 – 10:30am** **Beamline Status Specifics**
Christopher A. Tulk, Oak Ridge National Laboratory
- 10:30 – 10:40am** **Discussion**
- 10:40 – 11:00am* ***BREAK***

SOFTWARE: Chair – Chris Tulk

- 11:00 – 11:20am** **Data Acquisition Overview**
Richard Riedel, Oak Ridge National Labs
- 11:20 – 11:30am** **Discussion**
- 11:30 – 11:50pm** **SNS Analysis Software Overview**
Steve Miller, Oak Ridge National Labs
- 11:50 – 12:00noon** **Discussion**
- 12:00 – 12:10pm** **What is COMPRES?**
Robert Liebermann, COMPRES,
Stony Brook University

12:15 – 1:15pm ***LUNCH***

Lunch Presentation:
**Update on Neutron Scattering Opportunities and
High Pressure**
John B. Parise, Stony Brook University

POWER POINT POSTER SESSION: Chair – John Parise

- 1:15 – 1:20pm** **Ordering Ices Through Acid Doping: Ices XIII and XIV**

John Finney, University College-London

- 1:20 – 1:25pm** **Dynamics and Structural Properties of Clathrate Hydrates studied with Neutron and X-Ray Scattering**
Dennis D. Klug and John S. Tse
- 1:25 – 1:30pm** **Inelastic Neutron Scattering on Hydrogen Clathrates**
Tim Jenkins, Carnegie Institution of Washington
- 1:30 – 1:35pm** **Neutron Diffraction Under Hydrostatic Pressure: Clathrate Hydrates Investigation**
Konstantin Lokshin, University of Tennessee
- 1:35 – 1:40pm** **Gas Hydrate Experiments at the Los Alamos Neutron Scattering Center (LANSCE)**
Kim Tait, Los Alamos National Laboratory
- 1:40 – 1:45pm** **Hydrogen Bonding of Goethite (α -FeOOD) at High Pressure**
Hiroyki Kagi, Tokyo University
- 1:45 – 1:50pm** **Pressure response of Methylammonium Lead Bromide**
Ian Swainson, Chalk River National Labs
- 1:50 – 1:55pm** **The Dynamic Structure of Cyanide-Bridged Molecular Framework Materials: The Effects of Temperature and Pressure**
Karena Chapman, Argonne National Laboratory
- 1:55 – 2:00pm** **Crystallography Without a Lattice: Understanding Structure and Interactions in Solutions**
John Finney, University College-London
- 2:00 – 2:05pm** **In-situ Measurements of the Pressure-induced Amorphization of Siliceous Chabazite**
Paul Forster, Stony Brook University
- 2:05 – 2:10pm** **Effectiveness of K-B Mirrors on Neutron Micro-Focusing for Single-Crystal Diffraction Studies**
Darren Locke, Stony Brook University
- 2:10 – 2:15pm** **Recent Examples of High Pressure and High Temperature Neutron Research in the Earth Sciences**
Husin Sitepu, Virginia Tech

SINGLE CRYSTAL NEUTRON DIFFRACTION: Chair – John Finney

- 2:20 – 2:40pm** **Display and Analysis of Single Crystal TOF Laue Data Using ISAW**

Arthur J. Schultz, IPNS, Argonne National Laboratory

- 2:40 – 2:50pm Discussion**
- 2:50 – 3:10pm High-pressure Cells for Neutron Laue Diffraction on VIVALDI**
Garry J. McIntyre, Institut Laue-Langevin
- 3:10 – 3:20pm Discussion**
- 3:20 – 3:40pm Latest Experiences with "Online" Polarized Incident Neutron Single Crystal Diffraction**
Christina Hoffman, Oak Ridge National Lab
- 3:40 – 3:50pm Discussion**
- 3:50 – 4:10pm BREAK*

HIGH PRESSURE MATERIALS/EARTH SCIENCE: Chair – Dave Mao

- 4:10 – 4:30pm Hydrogen Storage in Molecular Compounds**
Wendy Mao, Los Alamos National Laboratory
- 4:30 – 4:40pm Discussion**
- 4:40 – 5:00pm High Pressure and Temperature Measurements with the PE-cell: Thermoelastic Behaviour of Graphite**
Bjoern Winkler, Universitaet Frankfurt
- 5:00 – 5:10pm Discussion**

FACILITY TOURS: Chris Tulk

- 5:15 – 6:30pm Tours**
- 7:00 – 9:00pm DINNER*
Comfort Inn Meeting Room
433 S. Rutgers Avenue
Oak Ridge, Tennessee 37830
Tel: 865-481-8200

Dinner Presentation: Recent Photographs of the Construction Activities at the SNS.

Tuesday, April 11, 2006

- 8:00 – 8:30am Coffee and Mingle*

GLASSES AND AMORPHOUS MATERIALS AT HP: Chair - Dennis Klug

- 8:30 – 8:50am** **Application of Paris-Edinburgh and Diamond Anvil Cells to High-Pressure Neutron and X-ray Scattering from Calcium-Silicate and -Germanate Glasses**
Darren Locke, Stony Brook University
- 8:50 – 9:00am** **Discussion**
- 9:00 – 9:20am** **Combination of High Energy X-rays with a Paris-Edinburgh cell for Pair Distribution Function Studies of the High Pressure Behavior of the Cuprite Structure**
Peter Chupus, Argonne National Laboratory
- 9:20 – 9:30am** **Discussion**
- 9:30 – 9:50am** **Amorphous Materials Engineering: Designing Structure in Glasses and Liquids**
James Martin, North Carolina State University
- 9:50 – 10:00am** **Discussion**
- 10:00 – 10:20am** **Recent Diffraction Results in Disordered Systems**
Christopher Tulk, Oak Ridge National Laboratory
- 10:20 – 10:30am** **Discussion**
- 10:30 – 10:50am** ***BREAK***

RECENT TECHNICAL DEVELOPMENTS: Chair – Hiroyuki Kagi

- 10:50 – 11:10am** **Moissanite Anvils and Anvil Pressure Cells Used in Neutron Diffraction**
Jian Xu, Carnegie Institute of Washington
- 11:10 – 11:20am** **Discussion**
- 11:20 – 11:40am** **Second-generation Kirkpatrick-Baez Supermirror Optics for SNAP**
Gene Ice, Oak Ridge National Laboratory
- 11:40 – 11:50am** **Discussion**
- 11:45 – 1:00pm** ***LUNCH***
- [Lunch Presentation:](#)**

Update on Construction Specifics
Steve Chae, Oak Ridge National Laboratory

1:00 – 1:20pm **CLOSING REMARKS**
John B. Parise, Stony Brook University

MEETING WRAP-UP

1:20 – 1:50pm **Open Meeting Wrap-up**

1:50 – 2:10pm ***BREAK***

2:10 – 3:30pm **Executive Committee Meeting Wrap-up**

Spallation Neutrons and Pressure – SNAP - Outreach 2006-07
John B. Parise, Stony Brook

Summary: The major SNAP out reach efforts 2006-07 were

- 18) The 4th executive and advisory committee meeting (SNAP 2007) at SNS, Oak Ridge, April 1-4 concentrated on science results; details are attached. This was a joint meeting with the NOMAD group and highlighted studies of poorly crystalline materials at non-ambient conditions. The outcome was positive – although expensive in terms of resources.
- 19) Planning for SNAP 2008 has begun.
- 20) Efforts continue to integrate HIFR into a general HP program at Oak Ridge. Working with Chris Tulk and Bryan Chakoumakos we placed a cell on HIFR and successfully collected data on hematite (Fe₂O₃) at high pressure, observing the pressure-mediated spin-flip transition.
- 21) Further tests on components, including focusing optic, orienting single crystals in the beam and an auto-pumping station.
- 22) New mounts and beamline optics for high energy X-ray studies. This included adapting detectors/cells and data collection strategies or use with focused beams at 1-ID. This SNAP effort has inspired groups from ASU, APS and HP_CAT to take up the study of glasses at high pressures using strategies developed. Without SNAP outreach, these efforts would have been delayed. Now several groups compete for scarce beamtime at 1-ID for these measurements: we will propose duplicating and improving efforts at 1-ID by adapting optics and HP cells at 11-ID-B, a dedicated PDF beamline in a proposal this October, 2007. This will greatly enhance access for the community.
- 23) Further analysis of single crystal data collected from high-pressure cells using “pink beams”. These new studies suggest new dual-use (High energy X-ray and neutron) cell designs such as those discussed by Goncharenko at SNAP-2007 will need further investigation. This will be a priority
- 24) Outreach to the Geosciences community. Two chapters are published as part of a volume of instructional materials resulting from the December 3, 2006, short course "Neutrons in the Earth Sciences". One on "Theory of neutron scattering" and a second on "High pressure studies"; the latter includes the latest SNAP design and features. Copies of the chapters are included in the DVD accompanying this report
- 25) Outreach to universities, industry and national labs: Parise gave the following presentations related to the SNAP outreach efforts:
 - 2006** “In situ studies of “crystallographically challenged” materials” one of the Sandia National Laboratory distinguished lecture series, Sept 14
 - “SNAP: high pressure at SNS” Gordon Research Conference on High Pressure; June 25-30
 - “Local and intermediate range structure in crystalline, nanocrystalline and amorphous materials” Denver Diffraction Conference, Denver, CO; 7-11 August
 - “Neutron scattering of frameworks under high pressure” Goldschmidt Conference, Melbourne Australia, August 30.
 - "Introduction to properties and applications of neutrons" Part of the MSA/GSA short course "Neutron Scattering in Earth Sciences" Berkeley, Dec 2006
 - "High pressure applications" Part of the MSA/GSA short course "Neutron Scattering in Earth Sciences" Berkeley, Dec 2006

2007: "New structural mineralogy from analysis of total X-ray/neutron scattering" University of Chicago, Jan 5
"Structural studies of nano-minerals" Lamont-Doherty Observatory, Feb 7
"Crystallography at non-ambient conditions using access to the APS-XOR beamlines" XOR SAC review June 27, 2007
"Neutron focusing and the promise for high pressure Laue scattering" Laue 2007, Grenoble France Jan 23 – 27
"Time resolved studies using high energy X-rays and neutrons" APS user meeting, May 10 2007

The study of materials research under extreme conditions has been steadily growing at Oak Ridge National Laboratory for a number of years now and this situation is poised for substantial growth in the coming years, particularly with the soon to be operating SNAP instrument. To carry on the momentum generated we would like to propose the formation of a ***national center of scientific excellence*** to be seeded at Oak Ridge National Laboratory with the broadly stated mission of understanding ***materials properties and processes under extreme conditions***. We envision such a center as being more broadly defined than just neutron diffraction on the SNAP beamline, and indeed of broader scope than neutron diffraction itself, and including other complimentary characterization techniques such as laboratory based x-ray diffraction, Raman and IR spectroscopy, Calorimetry, DTA, NMR. Such an extreme environments effort could be supported by a suite of highly specialized laboratory equipment located along with SNAP at ORNL. We note that there currently exists the nucleus of such a group with 10 core researchers currently conducting research aimed at extreme environment properties at ORNL.

A number of flag-ship experiments are currently underway and could form the basis upon which to build the center of scientific excellence. These include first and foremost the study of high pressure phenomenon in ***water clathrates*** and other guest-host systems. At high pressure water clathrates form new as of yet very poorly understood crystalline and amorphous phases. The guest clustering and interactions with the host water framework in such phases has not been addressed at all. Furthermore, water clathrates form the largest known store of hydrocarbon gas resources known on Earth, and in addition are becoming known as a potential framework for storing hydrogen gas, and as a sequestration medium for man generated green-house gasses such as carbon dioxide. As another example, ***magnetic systems under extreme environmental conditions*** are a classic application of neutron scattering only now beginning to be studied and this has largely nucleated a research effort at ORNL and has resulted directly from the SNAP effort. Particularly the monoxide systems such as MnO, CoO, and FeO are ideally suited for neutron scattering and magnetic properties studies under extreme conditions.

It was with this broader agenda in mind that we decided to highlight science at the annual SNAP meeting and to bring on board the NOMAD group. We believe the meeting was very successful – identifying many common points of interest and first experiments for the instrument. It also identified several of the areas identified above as potential growth areas over the next year. As a newly appointed member of the SNSNS, Parise will be looking for opportunities for extreme conditions research at SNS, ORNL and beyond.

1. Highlights: 4th executive and advisory committee meeting (SNAP 2007) joint with NOMAD at SNS, Oak Ridge, April 1-4.

We decided after the 2006 meeting that it was appropriate to highlight science results that are now resulting from outreach efforts. Many of these results come from the high energy beamlines that were used to test the high pressure cells. The program of the meeting and the list of attendees is attached to this document as an appendix and the full talks are on the DVD accompanying the report.

A tremendous synergy has been established between high energy X-rays and neutrons with results from one source complementing the other. Excellent examples were provided by *several young researchers include:*

a) Marc Michel (graduate student Stony Brook)

Marc described work soon to appear in *Science* on the elucidation of the structure of ferrihydrite (iron-oxide-hydroxide). This represents a collaboration between Stony Brook-Temple and the XOR group at beamlines 1-ID and 11-ID-B at APS Argonne. These beamlines have been the focus of intense developmental efforts for large volume gem and PE cells to be used at SNS. Marc's presentation is in the accompanying materials

New single phase model (Polyhedral representation) of the hexagonal unit cell for ferrihydrite. The bonded atoms (shown in yellow) define a cubane-like moiety that connects the basic structural motif of the model. This nano-crystalline material has defied structural characterization for over 30 years and succumbed to characterization with the help of new beamline capabilities at XOR, APS.

b) Chupas and Chapman (junior researchers, XOR, APS Argonne)

Pete and Karena, in two separate talks, emphasized the synergy between high pressure neutron and X-ray work in the study of framework materials. Several exciting phase transitions in negative thermal expansion (NTE) materials based on metal cyano-chemistry ($\text{Zn}(\text{CN})_2$) were described and the fascinating array of phenomena associated with the study of NTE at high pressure reveals

c) Chrystèle Sanloup (junior researcher, Edinburgh) described recent exciting results on the phase diagram of sulfur.

d) Lars Ehm (postdocs, SNAP) described recent fascinating experiments on the phase transitions associated with nano-crystalline FeS at high pressures using the PDF techniques. Further talks are on the enclosed DVD

With an eye to the start-up of SNAP commissioning in 2008, more senior researchers who will provide the first experiments were also highlighted. Magnetism is an obvious first target and two world renowned experts *Tappan Chatterji (ILL)* and *Igor Goncharenko (Saclay)* laid out the likely directions for future work on magnetism at high pressure. Both discussions of new cell designs and the most important systems for study were included in their talks. These will form a very nice basis for direction in 2008 as we explore science opportunities.

Work on clathrates (*Klug and Tulk*) continued the theme of grand challenge-type opportunities for the SNAP-NOMAD efforts at SNS. The work on Kr-clathrates at high pressure and from quench samples pointed out several fascinating structural variations and the relationship between loading and the location of Kr within the clathrate.

The point of holding a joint meeting with the NOMAD group was to explore common interests and identify early experiments that take advantage of SNS capabilities. This was pointed out in several cross-cutting talks by *Benmore (IPNS/APS Argonne)* and *Wilding (Wales)* who described work on glasses and melts respectively (see presentations). Particularly exciting was a presentation by *Poulsen (Riso)* who described means of measuring strain in amorphous materials, following up on his ground breaking ESRF work on strain in crystalline and composite materials.

The three day meeting also included updates from SNS management (*Anderson and Thibadeau*) and updates on progress on the beamline (on budget and ahead of schedule). These talks are enclosed, and they provide a flavor of the excitement generated by the three-day meeting.

2. Planning for SNAP 2008 has begun.

The 2008 meeting will be the final meeting prior to commissioning experiments. We found the joint SNAP-NOMAD meeting useful but expensive (much MORE expensive than previous standalone meetings) since many of the resources had to be used to cover costs from NOMAD-related expenses.

3. Continued efforts at HIFR

Efforts continue to integrate HIFR into a general HP program at Oak Ridge. Working with Chris Tulk and Bryan Chakomakous we placed a VX-5 HP cell on HIFR beamline WANDS. This Required some design modification and adaptation that was documented (see figures below). The Cells were tested successfully to 10 GPa. The success of these experiments prompted us to move all cells to Oak Ridge 12 months ahead of schedule and to relocate post doc Darren Locke to take advantage of lab space to store cells and begin the integration of high pressure into the SNS culture. This phase of the project is now drawing to an end and we still anticipate delivering all cells ahead

SNAP staff scientist Chris Tulk beside the VX-5 Paris-Edinburgh cell on the WAND beamline at HIFR (left). The data collected from hematite clearly shows the spin flip transition beginning at low pressure and almost complete at 7 GPa.

of schedule and to have them tested by the end of the project.

4. Further tests on components, including focusing optic, orienting single crystals in the beam and an auto-pumping station

The 2nd generation SNAP mirror design based on the use of nested mirrors is approaching completion. Mirrors are coated and are being tested at ILL prior to delivery to ORNL. Automated pumping stations are approaching completion and will be included in the hutch design. The photo-collage below presents some recent work with the pumping station and the mirror design.

Pumping station (left) showing two VX cells (one behind the protective shield under pressure. The computer controlled pumping station model, which will be used to remotely control pressure, is shown behind the cell with the lap-top. (right) The neted mirror design for the SNAP mirrors capable of focusing to sub-100 micron.

5). Further development of the HP capabilities at 11-ID and 1-ID at the APS. We have taken advantage of the synergy between high energy X-ray work and neutrons, and in particular the proximity of the APS to IPNS where we do much of our testing of cells and optics. In this way we have engaged Peter Lee, Pete Chupas, Karina Chapman, Dean Haeffner, Sarjvit Shastri and in the HP enterprise and they are performing cutting-edge experiments. As the SNAP project draws to an end, SNAP postdocs Darren Locke has applied for a position with the XOR group to carry out HP experiments on oil shales. Further graduate student Dave Martin was encouraged to apply for a directors post doc at the APS, and has received an offer. Dave will be joining this burgeoning effort in July 2007. The SNAP outreach has been responsible for kick starting the careers of Darren and Dave and we expect their presence at APS will be a positive influence on the high pressure programs there.

Articles appearing, or in press (06 – 07) reporting results supported by SNAP outreach funds (PDFs of those in **bold** included in DVD)

Parise J. B., Brown G. E. (2006) New opportunities at emerging facilities, *ELEMENTS* 2, 37-42

Mei, Q., C. J. Benmore, R. T. Hart, E. Bychkov, P. S. Salmon, C. D. Martin, F. M. Michel, S. M. Antao, P. J. Chupas, P. L. Lee, S. D. Shastri, J. B. Parise, K. Leinenweber, S. Amin, and J. L. Yarger (2006) Topological changes in glassy GeSe₂ at pressures up to 9.3 GPa determined by high-energy x-ray, and neutron diffraction measurements *Phys. Rev. B* 74 014203

Parise, J. B., Locke, D. R., Tulk, C. A., Swainson, I. and Cranswick, L. (2006) The effect of pressure on the Morin transition in hematite (α -Fe₂O₃) *Physica B*, 385-386, 391-393

Parise, J. B. (2006)) Introduction to Neutron Properties and Applications in *Neutron Studies in Earth Sciences* H-R. Wenk, Ed. Reviews in Mineralogy and Geochemistry series editor JJ Rosso (Ed) Vol 63, pp 1 - 27

Parise, J. B. (2006) High Pressure Studies in *Neutron Studies in Earth Sciences* H-R. Wenk, Ed. Reviews in Mineralogy and Geochemistry series editor JJ Rosso (Ed) Vol 63, pp 205-233

Chapman, K. W., Chupas, P. J., Kurtz, C. A., Locke, D. R., Parise, J. B. and Hriljac, J. A. (2007) Hydrostatic low-range pressure applications of the Paris-Edinburgh cell utilizing polymer gaskets for diffuse X-ray scattering measurements *J. Appl. Cryst.*, 40, 196-198

Michel, F. M., Ehm, L., Liu, G., Han, W. Q., Antao, S. M., Chupas, P. J., Lee, P. L., Knorr, K., Eulert, H., Kim, J., Grey, C. P., Celestian, A. J., Gillow, J., Schoonen, M. A. A., Strongin, D. R. and Parise, J. B. (2007) Similarities in 2- and 6-Line Ferrihydrite Based on Pair Distribution Function Analysis of X-ray Total Scattering, Chem. Materials, Web Release Date: 03-Feb-2007; (Article) DOI: 10.1021/cm062585n)

In press

Michel, F. M., Ehm, L., Antao, S. M., Chupas, P. J., Lee, P., Liu, G., Strongin, D. R., Schoonen, M. A. A., Phillips, B. L. and Parise, J. B. (2007) The structure of ferrihydrite, a nanocrystalline material, Science (in press)

Not all publications resulting from users such as Jim Martin (North Carolina) Chupas and Chapman (ANL) Benmore (ANL) Tulk (SNS) and others who used the cell independently.

An added bonus to this outreach is the training of young scientists in the use of the high pressure apparatus so that they will be independent of the SNAP group now that the cells have moved to Oak Ridge. The downside of course, is that a vital and growing research program in high pressure at XOR will be left without cells and expertise. We are moving to redress this problem by submitting a proposal to DOE this Fall. In the meantime, funds have been scrapped together from the APS budget to purchase a small PE cell for 11-ID-B. This along with Dave Martin's arrival (Dave will complete his PhD in June, 2007) will provide a boost to efforts at 11-ID-B.

SNAP/NOMAD 2007

Spallation Neutrons and Pressure 4th Annual Meeting

April 1-4, 2007

Oak Ridge, Tennessee

Saturday, March 31 – Sunday, April 1, 2007 Arrival and Check-in

The Oak Ridge Comfort Inn
433 S. Rutgers Avenue
Oak Ridge, Tennessee 37830
Tel: 865-481-8200

Sunday, April 1, 2007 “Meet & Greet” Informal Gathering

5:00 – 9:00pm *Jalisco Mexican Restaurant*
(Walking Directions in Meeting Packet)

Monday, April 2, 2007

8:00 am BADGING (See Instructions on Greetings page)

INTRODUCTIONS: SNS, SING, SNAP UPDATES – Chair: Rus Hemley

9:00 – 9:05am Welcome
John B. Parise, Stony Brook University

9:05 – 9:20am Overall SNS Status
Ian Anderson, Oak Ridge National Laboratory

- 9:20 – 9:35am** **SING → SNS → SNAP**
Barbara Thibadeau, Oak Ridge National Laboratory
- 9:35 – 9:45am** **Discussion**
- 9:45 – 10:00am** **Beamline Status Specifics**
Christopher A. Tulk, Oak Ridge National Laboratory

EMERGING OPPORTUNITIES: Chair – Chris Tulk

- 10:00 – 10:30am** **Opportunities for Studies of Magnetism at High Pressures**
Tapan Chatterji, Institut Laue-Langevin
- 10:30 – 10:40am** **Discussion**
- 10:40 – 11:10pm** **Inelastic Neutron Scattering at Low Temperatures: Melting
and Amorphisation of Ice**
Stefan Klotz, Paris VI
- 11:10 – 11:30noon** **Discussion**
- 11:30 – 11:50am** **BREAK**
- 11:50 – 12:20pm** **Combined Neutron & X-ray Measurements on Powder and Single
Crystals at High Pressures and Low Temperatures**
Igor Goncharenko, Laboratoire Léon Brillouin C.E.A.-C.N.R.S.
- 12:20 – 12:30pm** **Discussion**
- 12:30 – 1:00pm** **New Opportunities for High Pressure Research at the
ESRF**
Wilson Crichton, European Synchrotron Radiation Facility
- 1:00 – 1:10pm** **Discussion**
- 1:10 – 2:10pm** **LUNCH**
- Lunch Presentation:**
SNAP Beamline Installation
Steve Chae/Chris Tulk, Oak Ridge National Laboratory

2:10 – 2:40pm Rings, Chains, Liquid-like States, Tc and CDW in the Elemental Sulphur
Eugene Gregoryanz, University of Edinburgh

2:40 – 2:50pm Discussion

SHORT PRESENTATIONS – More Emerging Opportunities Part I.
(10 minutes + 5 minutes discussion): Chair – John Parise

Ices/Ice Related:

2:50– 3:00pm Molecular Dynamic Simulations at HP of Clathrate Hydrate Systems
Dennis Klug, National Research Council of Canada

3:00– 3:15pm High Energy X-ray Scattering Studies at HP of Clathrate Hydrate Systems
Chris Tulk, Oak Ridge National Laboratory

3:15 – 3:30pm Precise Measurements on Compressibilities of Delta-AIOOH and Phase D and the Relationship to the H-bond Symmetrization
Hiroyuki Kagi, University of Tokyo

3:30 – 3:45pm High Pressure, Low Temperature Clathrate Hydrates Examined with Inelastic Neutron Scattering, X-ray Diffraction, and Raman Scattering
Tim Jenkins, Carnegie Institution of Washington

3:45 – 4:15pm *BREAK*

Nano-Materials:

4:15 – 4:30pm High-pressure Behavior of Nanocrystalline Mackinawite (FeS)
Lars Ehm, Stony Brook University

4:30 – 4:45pm Time-resolved Pair Distribution Function as a Method to Understand the Kinetics of Nano-Particle Formation
Peter Chupas, Argonne National Laboratory

4:45 – 5:00pm Structural Studies of Functional Coordination Framework Materials
Karena Chapman, Argonne National Laboratory

5:00 – 5:15pm Theoretical Techniques in the Analysis of Neutron Static and Dynamics Structures
John S. Tse, University of Saskatchewan

7:00 – 9:00pm ***DINNER: Bus to Peerless Steakhouse***

**Dinner Presentation: Gene Ice: SNAP Song and
Recent Photographs of the Construction Activities at the SNS.**

Tuesday, April 3, 2007

8:00 – 8:30am ***Coffee and Mingle***

8:30 – 8:50am **Strain in Amorphous Samples**
Henning Poulsen, Risoe

8:50 – 9:00am **Discussion**

GLASSES AND AMORPHOUS MATERIALS AT HP: Chair - Dennis Klug

9:00 – 9:30am **Network and Molecular Glasses at High Pressure**
Chris Benmore, Argonne National Laboratory

9:30 – 9:40am **Discussion**

9:40 – 10:10am **Changes in the Structure of MgO-SiO₂ Glasses with
Pressure: In Situ Neutron Diffraction Results**
Martin Wilding, University of Wales, Aberystwyth

10:10 – 10:20am **Discussion**

10:20 – 10:50pm **Density of Amorphous Sulfur Measured from X-ray
Diffraction Data in Diamond-anvil Cell at Extreme Pressure
Conditions**
Chrystèle Sanloup, Pierre & Marie Curie University

10:50 – 11:00am **Discussion**

11:00 – 11:30am ***BREAK***

SHORT PRESENTATIONS – More Emerging Opportunities Part II.
(10 minutes + 5 minutes discussion): Chair – Dave Mao

11:30 – 11:45am Probing the Structure of a Protein-coated Iron Mineral
F. Marc Michel, Stony Brook University

11:45 – 12:00pm Competition in Perovskites
Despina Louca, The University of Virginia

**12:15 – 12:30am Diffraction from Magnetic Thin Films and Multilayers: a
Challenge for SNS, an Opportunity for HIFR**
Garry J. McIntyre, Institut Laue-Langevin

**12:30 – 12:45pm Highlight of the Current Instruments and Future
Instrument Development at the HFIR that would be Amenable to
High Pressure Research**
Bryan Chakoumakos, Oak Ridge National Laboratory

12:45 – 1:00 pm SNAP CLOSING REMARKS - NOMAD TRANSITION
Russell Hemley, Carnegie Institution of Washington

1:00 – 2:00pm LUNCH

SNAP IDT Executive Committee Meeting Wrap-Up
(Working Lunch Session)

FACILITY TOURS: Chris Tulk (Available upon request)

START NOMAD SECTION: Chair – J. Michael Simonson

**2:00 – 2:30pm Resources for Neutron Science in the Center for Nanophase Materials
Sciences**
J. Michael Simonson, Oak Ridge National Laboratory

2:30 – 2:40pm Discussion

2:40 – 2:55pm Q-resolution and the PDF Analysis

Takeshi Egami, University of Tennessee

2:55– 3:00pm

Discussion

3:00 – 3:15pm

Structure of Glasses and Templated Materials

James Martin, North Carolina State University

3:15 – 3:20pm

Discussion

3:20 – 3:35pm

Atomic-scale Theory for Nanoscale Structure and Interactions (to be confirmed)

Ariel A. Chialvo, Oak Ridge National Laboratory

3:35 – 3:40pm

Discussion

3:40 – 3:55pm

Local and Nanoscale Structure in Polymer Systems, Including Effects of Applied Fields

Volker Urban, Oak Ridge National Laboratory

3:55 – 4:00pm

Discussion

4:00 – 4:20pm

BREAK

**4:20 – 4:35pm
Environment**

In Situ Determinations of Structural Change with Sample

Andrew Payzant, Oak Ridge National Laboratory

4:35 – 4:40pm

Discussion

4:40 – 4:55pm

Structure Measurements at Extreme Temperatures and Under Non-equilibrium Conditions

Rick Weber, Materials Development, Inc.

4:55 – 5:00pm

**Discussion and Adjourn for the Day
Dinner on your own**

Wednesday, April 4, 2007

8:30 – 9:00am	<i>Coffee and Mingle</i>
9:00 – 9:15am	Structural Studies of Molecular Fluids John Turner, University of Tennessee
9:15 – 9:20am	Discussion
9:20 – 9:35am	Scattering Studies of Condensed Systems Jacob Urquidi, New Mexico State University
9:35 – 9:40am	Discussion
9:40 – 9:55am	Status of the NIMROD Diffractometer Alan Soper, ISIS, UK
9:55 – 10:00am	Discussion
10:00 – 10:30am	Break and Teleconference Connections
10:30 – 12:30pm	TELECONFERENCE: Instrument Performance Chair: Joerg Neufeind (Henry Fischer and Alan Hewat, ILL to participate)
12:30 – 1:30pm	<i>WORKING LUNCH</i>
1:30 – 3:00pm	Additional Discussions (including early experiments)
3:00 – 3:30pm	NOMAD CLOSING REMARKS (Summary - Action Items)

FACILITY TOURS: Mike Simonson (Available upon request)

Spallation Neutrons and Pressure – SNAP - Outreach 2007 - 08
John B. Parise, Stony Brook

Summary: The major SNAP out reach efforts 2007 - 08 were

- 26) The 5th and final executive and advisory committee meeting (SNAP 2008) at SNS, Oak Ridge, April 12-14 concentrated on science results; details are attached. This was a joint meeting with the COMPRES (Consortium for Mineral Physics Research in the Earth Sciences) group and highlighted complementary studies using high energy x-ray and neutron scattering.
- 27) Planning for the start-up of the SNAP beamline has begun
- 28) Outreach to the Geosciences community: Parise is co-editing an issue of the popular magazine "Elements" concentrating on the Deep Earth. Pressure plays a major role in this research. The issue will appear in June 2008. The web site where this will be available as Vol.4, issue 3 (June 2008) located at <http://www.elementsmagazine.org/>
- 29) Outreach to universities, industry and national labs: Parise gave the following presentations related to the SNAP outreach efforts:
 - 2007 Neutron focusing and the promise for high pressure Laue scattering” Laue 2007, Grenoble France Jan 23 – 27
 - "Time resolved studies using high energy X-rays and neutrons" APS user meeting, May 10 2007
 - "Mineralogy on small length scales" GSA symposium honoring Gordon Brown, October 29, 2007
 - "Crystallography at non-ambient conditions" XOR SAC review June 27, 2007
 - 2008 "Beyond Rocks: Geology and Planetary Sciences with X-rays and neutrons" Cornell University American Association of Physics Teachers, April 18 - 19
 - "Crystallography" Associated Scottish Universities Summer School in Physics, May 27-28, Skye
- 5) Presentations at conferences involving SNAP personnel including postdocs Locke and Ehm
 - American Crystallographic Association Meeting Oak Ridge TN
 - Structural Changes in Nano-crystalline Mackinawite (n-FeS) at High-pressure. Lars Ehm, Marc Michel, SytleAntao, Peter Chupas, Peter Lee, David Martin, Sarvjit Shastri, John Parise.
 - Disorder in sII and sH Krypton Clathrate Hydrates. Bryan Chakoumakos, LingYang, D.D.Klug, C.A.Tulk,D.Martin, Lars Ehm, J.B. Parise.
 - A New Hexagonal Phase for Pressure-Quenched Xe Clathrate Hydrate. L. Yang, C. A. Tulk, D.D. Klug, L. Ehm, D. Martin, B.C. Chakoumakos, J.J. Molaison, J. B. Parise

The SNAP instrument has taken its first neutrons and now awaits commissioning of the detectors and a small amount of fit-out. Tulk and colleagues are now independently performing high pressure cell loading with all cells delivered and operating to specifications.

A number of flag-ship experiments are currently underway and could form the basis upon which to build the center of scientific excellence. These include first and foremost the study of high pressure phenomenon in *water clathrates* and other guest-host systems. Three abstracts are submitted for the Oak Ridge American Crystallographic Association Meeting in July

Structural Changes in Nano-crystalline Mackinawite (n-FeS) at High-pressure. Lars Ehm, Marc Michel, SytleAntao, Peter Chupas, Peter Lee, David Martin, Sarvjit Shastri, John Parise.

Disorder in sII and sH Krypton Clathrate Hydrates. Bryan Chakoumakos, LingYang, D.D.Klug, C.A.Tulk,D.Martin, Lars Ehm, J.B. Parise.

A New Hexagonal Phase for Pressure-Quenched Xe Clathrate Hydrate. L. Yang, C. A. Tulk, D.D. Klug, L. Ehm, D. Martin, B.C. Chakoumakos, J.J. Molaison, J. B. Parise

It was with the broader implications for high pressure research that we decided to highlight science at the annual SNAP meeting and to bring on board the COMPRES group, and a broad agenda to include high energy x-ray scattering. We believe the meeting was very successful – identifying many common points of interest and first experiments for the instrument.

1. Highlights: 5th executive and advisory committee meeting (SNAP 2008) joint with COMPRES and products of the outreach grant.

We decided after the 2007 meeting that it was appropriate to highlight science results that are now resulting from outreach efforts, including those resulting from use of the HP cells at the high energy lines at APS. *The program of the meeting and the list of attendees is attached to this document as an appendix and the full talks are on the DVD accompanying the report.*

Articles appearing, or in press (07-08) reporting results supported by SNAP outreach funds
Chapman, K. W., Chupas, P. J., Kurtz, C. A., Locke, D. R., Parise, J. B. and Hriljac, J. A. (2007) Hydrostatic low-range pressure applications of the Paris–Edinburgh cell utilizing polymer gaskets for diffuse X-ray scattering measurements J. Appl. Cryst., 40, 196-198
Martin, C. D., Chupas, P. J., Chapman, K. W. and Parise, J. B. (2007) Local vs. average structure: A study of Neighborite (NaMgF₃) utilizing the pair-distribution function method for structure determination. J. Appl. Crystallog. 40, 441-448
Michel, F. M., Ehm, L., Antao, S. M., Chupas, P. J., Lee, P., Liu, G., Strongin, Schoonen, M. A. A., Phillips, B. L. and Parise, J. B. (2007) The structure of ferrihydrite, a nanocrystalline material, Science, 316, 1726-1729
Ehm, L., Antao, S. M., Chen, J., Locke, D. R., Michel, M. F., Martin, C. D., Yu, T., Parise, J. B., Chupas, P. J., Lee, P. L., Shastri, S. D., Guo, Q. (2007) Studies of local and intermediate range structure in crystalline and amorphous materials at high pressure using high-energy X-rays, Powder Diffraction, 22, 108-112

Martin, C. D., Meng, Y., Prakapenka, V. B., Parise, J. B. (2008) Gasketing optimized for large sample volume in the diamond anvil cell: first application to MgGeO₃ and implications for structural systematics of the perovskite to post-perovskite transition. J. Appl. Crystallog. 41, 38-43

Antao, S. M., Benmore, C. J., Li, B., Wang, L., Bychkov, E. and John B. Parise, J. B. (2008) Network Rigidity in GeSe₂ Glass at High Pressure, Phys. Rev. Lett., 100, 115501

Graduate student theses and postdoctoral fellows trained

Graduate Students, year, prizes, present position

Marc Michel, PhD, 2008, *Awarded President's award for PhD thesis 2008 (one of two awarded per year)* now at SLAC/Stanford as beamline scientist.

David Martin, PhD, 2007, now director's post doctoral fellow, APS, Argonne National Laboratory

Sytle Antao, PhD 2006, now beamline scientist Argonne National Laboratory

Aaron Celestian, PhD 2005, Assistant Professor Western Kentucky University, intending to begin program at ORNL/SNS

SNAP Post docs

Lars Ehm, joint appointment, research assistant professor/scientist Stony Brook/BNL National Synchrotron Light Source, 2008 -

Darren Locke, postdoctoral fellow, APS, Argonne National Laboratory, 2007 -

Paul Forster, Assistant Professor of Chemistry, UNLV, 2007-

Presentations at conferences involving SNAP personnel including postdocs Ehm

American Crystallographic Association Meeting Oak Ridge TN

Powder Challenges

02:15 - 03:00 PM 13.12.02

(W0260) Lars Ehm.

Structural Changes in Nano- crystalline Mackinawite (n-FeS) at High-pressure. (IL)

et al: Marc Michel, Sytle Antao, Peter Chupas, Peter Lee, David Martin, Sarvjit Shastri, John Parise.

03:00 - 03:30 PM

Coffee Break. ()

03:30 - 03:50 PM 13.12.03

(W0281) Bryan Chakoumakos.

Guest Atom Disorder in sII and sH Krypton Clathrate Hydrates. (P)

et al: Ling Yang, D. D. Klug, C. A. Tulk, D. Martin, Lars Ehm, J. B. Parise.

03:50 - 04:10 PM 13.12.04

(W0316) L. Yang.

A New Hexagonal Phase for Pressure- Quenched Xe Clathrate Hydrate. (CL)

et al: C. A. Tulk, D. D. Klug, L. Ehm, D. Martin, B. C. Chakoumakos, J. J. Molaison, J. B. Parise

SNAP/COMPRES 2008

A Joint Meeting Exploring Opportunities for Scattering using Neutrons and HE X-rays

Spallation Neutrons and Pressure 5th Annual Meeting

April 13-15, 2008

Oak Ridge, Tennessee

Monday, April 14, 2008

8:00am BADGING (See Instructions on Greetings page)

INTRODUCTIONS: SNS, SING, SNAP UPDATES – Chair: John Parise

9:00 – 9:05am	Welcome John B. Parise, Stony Brook University
9:05 – 9:20am	Overall SNS Status Ken Herwig, Oak Ridge National Laboratory
9:20 – 9:35am	SING → SNS → SNAP Barbara Thibadeau, Oak Ridge National Laboratory
9:35 – 9: 50am	Beamline Status Specifics Christopher A. Tulk, Oak Ridge National Laboratory
9:50 – 10:00am	Discussion
10:00 – 10:10am	COMPRES and User Facilities for Mineral Physics Research Robert C. Liebermann, COMPRES
10:10 – 10:15am	Discussion

Emerging Capabilities for Neutron and High Energy X-ray Research:

Chair – Chris Tulk

- 10:15 – 10:35am** **Changes in the Structure of MgO-SiO₂ Glasses with Pressure: In Situ Neutron Diffraction Results**
Martin Wilding, University of Wales, Aberystwyth
- 10:35 – 10:40am** **Discussion**
- 10:40 – 10:55am** ***BREAK***
- 10:55 – 11:15am** **High Pressure High Temperature**
Stefan Klotz, IMPMC
- 11:15 – 11:20am** **Discussion**
- 11:20 – 11:40am** **Complementing IXS and LH-DAC Experiments with DFPT Modeling**
Bjoern Winkler, Universitaet Frankfurt
- 11:40 – 11:45am** **Discussion**
- 11:45 – 11:55pm** **IMAGINE Quasi-Laue Diffractometer at the HFIR**
Bryan Chakoumakos, ORNL
- 11:55 – 12:00pm** **Discussion**
- 12:00 – 1:00pm** ***LUNCH***

High Pressure Single Crystal Techniques: Chair – Chris Tulk

- 1:00 – 1:20pm** **Software Requirements for Single Crystal Diffraction**
Ross Angel, Virginia Tech
- 1:20 – 1:25pm** **Discussion**
- 1:25 – 1:45pm** **Data Visualization and Reduction for Single-Crystal Diffraction at the ILL**
Garry McIntyre, Institut Laue-Langevin
- 1:45 – 1:50pm** **Discussion**
- 1:50 – 2:10pm** **Studying High-pressure Transformations of Possible Light-element Phases in the Core and Development of Novel Single-crystal Diffraction Techniques for High-pressure Crystallography**
Przemek Dera, University of Chicago
- 2:10 – 2:15pm** **Discussion**
- 2:15 – 2:30pm** **The Structural Diversity of Sodium**
Eugene Gregoryanz, University of Edinburgh
- 2:30 – 2:35pm** **Discussion**

Facilities Updates:

- 2:35 – 2:50pm** **Update on JPARC**
Wataru (Sho) Utsumi, Japan Atomic Energy Agency
- 2:50 – 2:55pm** **Discussion**
- 2:55 – 3:10pm** **Plans and Updates on the High-pressure Beamline Project in Japan**
Hiroyuki Kagi, University of Tokyo
- 3:10 – 3:15pm** **Discussion**
- 3:15 – 3:30pm** **Plans for High Energy Scattering at X17A, NSLS**
Lars Ehm, Mineral Physics Institute, Stony Brook University
- 3:30 – 3:35pm** **Discussion**
- 3:35 – 3:45pm** ***BREAK***

SHORT PRESENTATIONS – Hydrogen in Minerals and Ices:

Chair – John Parise

3:45 – 3:55pm	Nucleation and Growth of Ice XI Hiroshi Fukazawa, Japanese Atomic Energy Agency
3:55 – 4:00pm	Discussion
4:00 – 4:15pm	Molecular Dynamic Simulations at HP of Clathrate Hydrate Systems Dennis Klug, National Research Council of Canada
4:15 – 4:20pm	Discussion
4:20 – 4:35pm	High Energy X-ray Scattering Studies at HP of Clathrate Hydrate Systems Chris Tulk, Oak Ridge National Laboratory
4:35 – 4:40pm	Discussion
4:40 – 4:55pm	Joint X-N Studies of Hydrogenous Materials John Loveday, University of Edinburgh
4:55 – 5:00pm	Discussion
5:00 – 5:10pm	High Energy X-ray Diffraction Studies of Filled Ice (Ar-H₂O at High Pressure) Ling Yang, Oak Ridge National Laboratory
5:10 – 5:15pm	Discussion
7:00 – 9:00pm	DINNER: Peerless Steakhouse

Tuesday, April 15, 2008

8:00 – 8:30am *Coffee and Mingle*

Novel Hardware – Novel Science: Chair – Robert Liebermann, COMPRES

8:30 – 8:50am	Temperature at Pressure: Novel Studies David Dobson, University College London
8:50 – 8:55am	Discussion
8:55 – 9:15am	Density Measurement at High-Pressure and Applications to the Deep Earth David Walker, Columbia University
9:15 – 9:20am	Discussion
9:20 – 9:35am	Topaz Update Christina Hoffman, Oak Ridge National Laboratory
9:35 – 9:40am	Discussion
9:40 – 9:55am	Multi-anvils PE Cell for Ex Situ and Angle X-ray Diffraction Studies Yann LeGodec, IMPMC
9:55 – 10:00am	Discussion
10:00 – 10:15pm	Inelastic Neutron Scattering Studies of Adsorbed Water on Oxide Nanoparticles Nancy Ross, Virginia Tech
10:15 – 10:20pm	Discussion

10:20 – 10:30am *BREAK*

EMERGING OPPORTUNITIES I: Glasses, Melts, Liquids and Networks: Chair – Rus Hemley

10:30 – 10:50am	Network and Molecular Glasses at High Pressure Chris Benmore, Argonne National Laboratory
10:50 – 10:55am	Discussion
10:55 – 11:15am	Structure Measurements at Extreme Temperatures and Under Non-equilibrium Conditions Richard Weber, Materials Development, Inc.
11:15 – 11:20am	Discussion
11:20 – 11:35am	High Energy XRD Studies Under High Pressure for Metallic Glasses and Other Amorphous Materials Haozhe Liu, Harbin Institute of Technology, China

11:35 – 11:40am	Discussion
11:40 – 11:55pm	High-pressure Studies of Novel Perovskite Compounds Changqing Jin, Inst of Physics, Chinese Academy of Sciences
11:55 – 12:00pm	Discussion
12:00 - 12:10 pm	High Energy X-ray Diffraction Studies of High Pressure Crystalline and Amorphous Germanium Based Clathrates Anotonio Moreira Dos Santos, Oak Ridge National Laboratory
12:10 – 12:15 pm	Discussion
12:15 – 12:25 pm	Compressed CaSiO₃ Glass Cathy Tarabrella, Stony Brook University
12:25 – 12:30 am	Discussion
12:30 – 1:30 pm	LUNCH
<u>EMERGING OPPORTUNITIES II: Nanoparticles and Networks:</u>	
Chair – John Parise	
1:30 – 1:45pm	What it Takes to Make the Best PDF Measurements with High Energy X-rays and Area Detectors Peter Chupas, Argonne National Laboratory
1:45 – 1:50pm	Discussion
1:50 – 2:05pm	Structural Studies of Functional Coordination Framework Materials Karena Chapman, Argonne National Laboratory
2:05 – 2:10pm	Discussion
2:10– 2:20pm	Pressure-Induced Polymerization of Diiodobutadiyne in Assembled Co-Crystals Chris Wilhelm, Stony Brook University
2:20 – 2:25pm	Discussion
2:25 – 240pm	High Pressure Study of Magnetization of Organometallic Complexes John Tse, University of Saskatchewan
2:40 – 2:45pm	Discussion
2:45 – 3:15pm	Frameworks under high pressure: recent results Lauren Borkowski UNLV

3:15 – 3:30pm **BREAK**

FACILITY TOURS: Chris Tulk