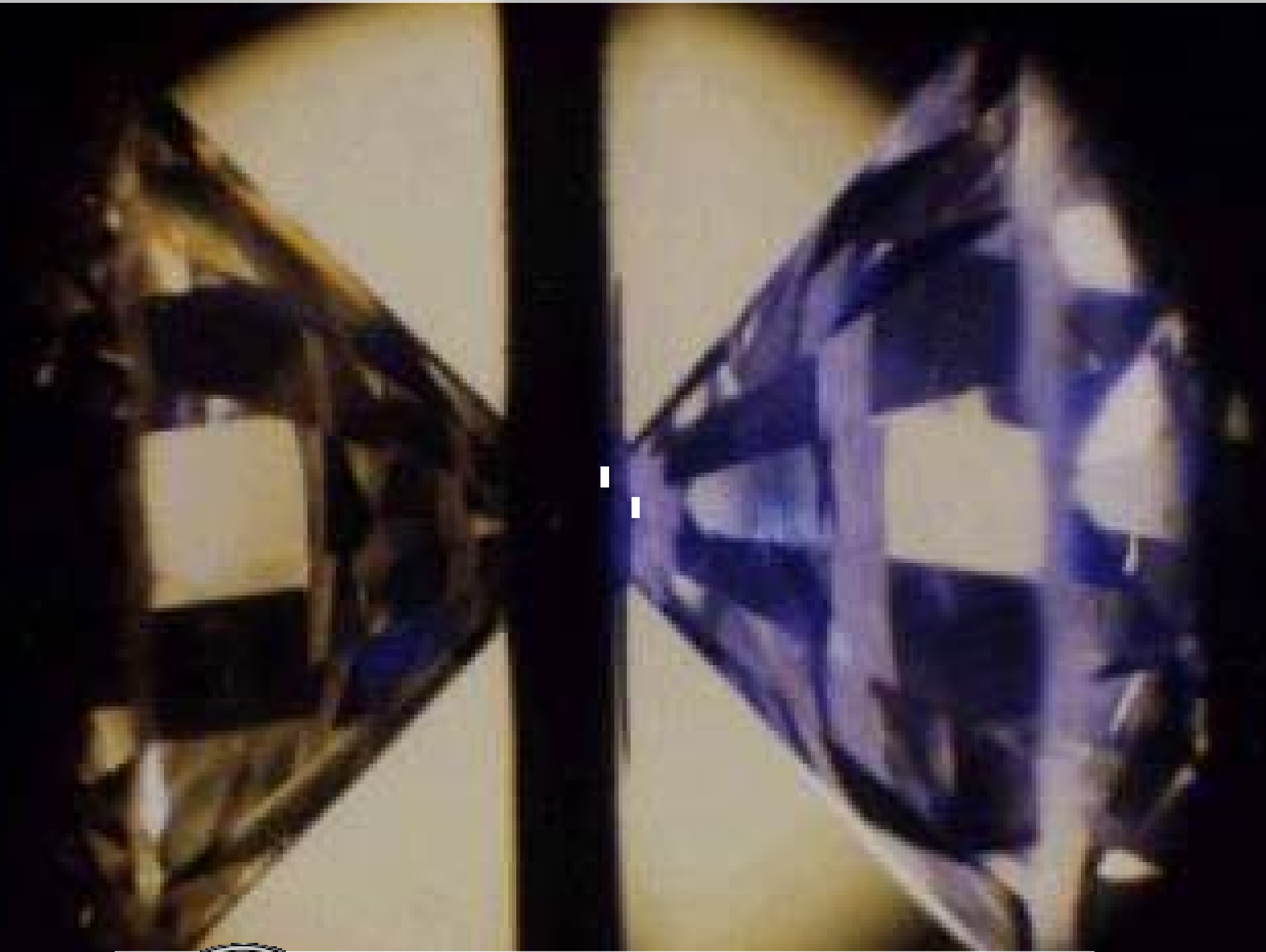
High-Pressure Synergetic Consortium--
A New Approach to HP Research at Synchrotrons

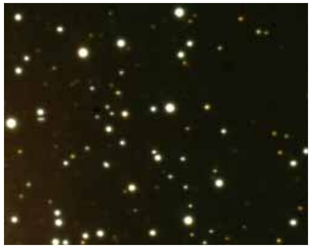


Ho-kwang Mao
Geophysical Lab.
Carnegie Institution

RANGE OF PRESSURE IN THE UNIVERSE

Pressure (Atmospheres)

10^{-32} – Hydrogen gas in intergalactic space



10^{-24}

– Interplanetary space

10^{-16}

– Atmosphere at 300 miles

10^{-8}

1

10^{8} – Center of Jupiter

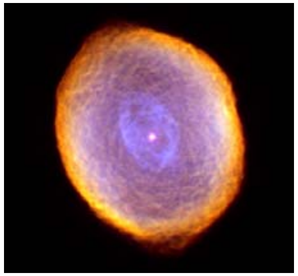
– Center of Sun

10^{16}

– Center of white dwarf

10^{24}

– Center of neutron star



10^{32}

64 orders of magnitude

1 GPa = 10^9N/m^2 = 10^4 bar

Pressure (Atmospheres)

10^{-8}

10^{-6}

– Best mechanical pump vacuum

10^{-4}

– Water vapor at triple point

10^{-2}

1 – Atmospheric pressure (sea level)

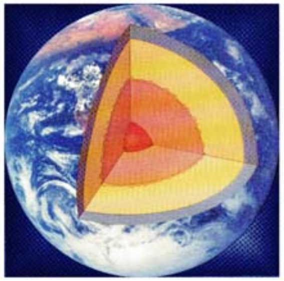
10^{2}

– Deepest ocean

10^{4}

10^{6}

– Center of the Earth



10^{8}

Scientific Challenges at High Pressure

General HP Science

- *Terapascal pressures*
- *Electron volt temperature*
- *Fundamental properties at HP*

Chemistry and Crystallography

- *Chemical reactivity and affinity*
- *Bonding and stereochemistry*
- *Ionic radii and atomic coordination*

Novel incommensurate structures

Geosciences

- *Micro-nano mineralogy*
- *Deep Earth geochemistry*
- *Mission to the core*

Geodynamics and seismology

Physics and Astrophysics

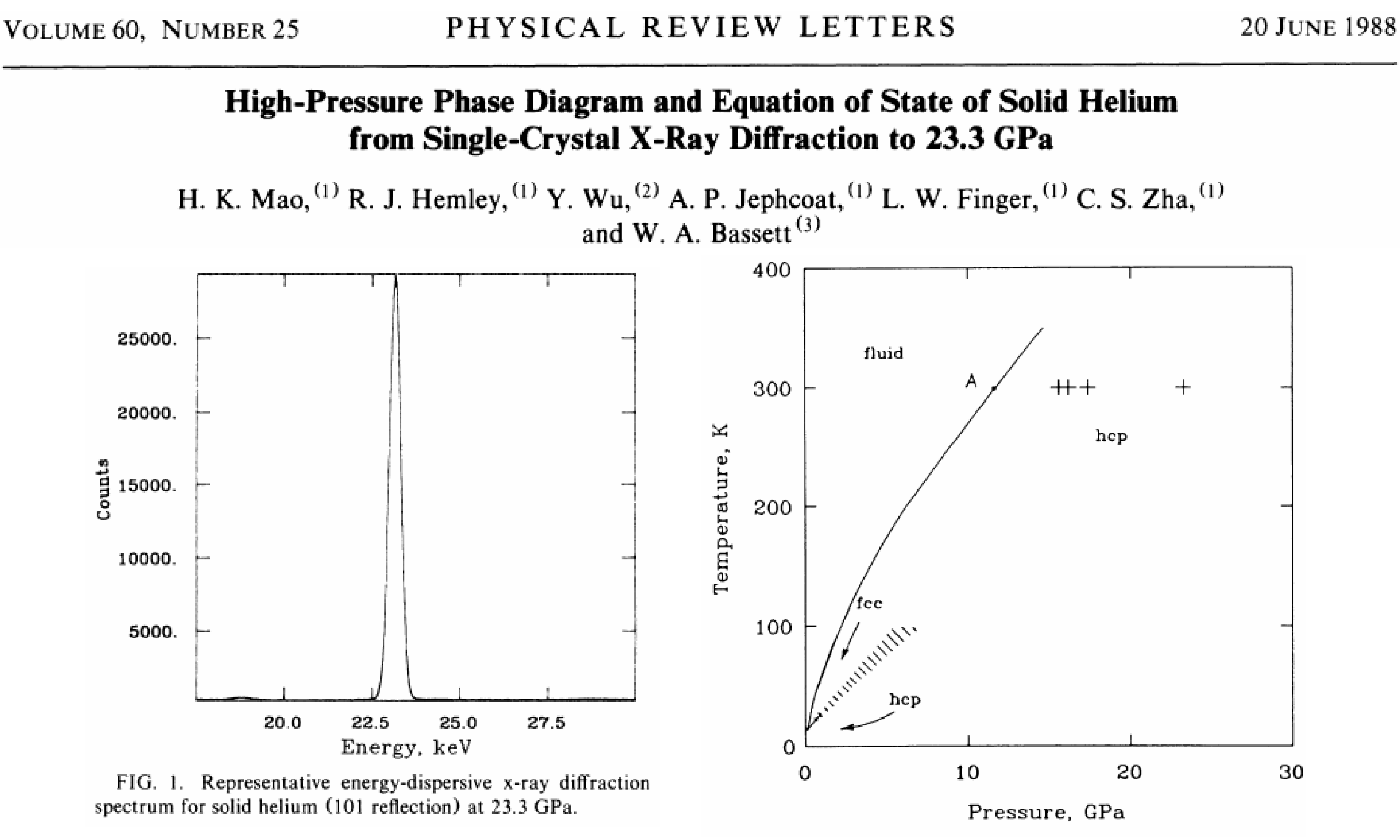
- *Dense, hot hydrogen at high P-T*
- *Metallic hydrogen at low T*
- *Ices in giant planets & satellites*
- *Free electron gas*
- *Metals and superconductivity*
- *Strongly correlated systems*

Materials Science

- *Creating novel materials*
- *Superhard diamond and beyond*
- *Hydrogen storage*
- *Magnetic materials*
- *Stockpile Stewardship Science*
- *Bio-materials*

High-pressure synchrotron research -- Four decades of HP operation modes

1980s	Non-dedicated beamlines for HP users
1990s	Dedicated HP diffraction beamline (e.g., X17c, NSLS)
2000s	Dedicated HP multi-technique beamlines (*e.g.*, HPCAT)
Future	Integrated facility -- optimized HP experiments at facility-wide, specialized beamlines



VOLUME 60, NUMBER 25 PHYSICAL REVIEW LETTERS 20 JUNE 1988

High-Pressure Phase Diagram and Equation of State of Solid Helium from Single-Crystal X-Ray Diffraction to 23.3 GPa

H. K. Mao,(1) R. J. Hemley,(1) Y. Wu,(2) A. P. Jephcoat,(1) L. W. Finger,(1) C. S. Zha,(1) and W. A. Bassett(3)

FIG. 1. Representative energy-dispersive x-ray diffraction spectrum for solid helium (101 reflection) at 23.3 GPa.

- **Four data points took 28 days at A-3 Beamline of CHESS**
- **Showed solid helium was hcp and provided unit cell info.**

1987

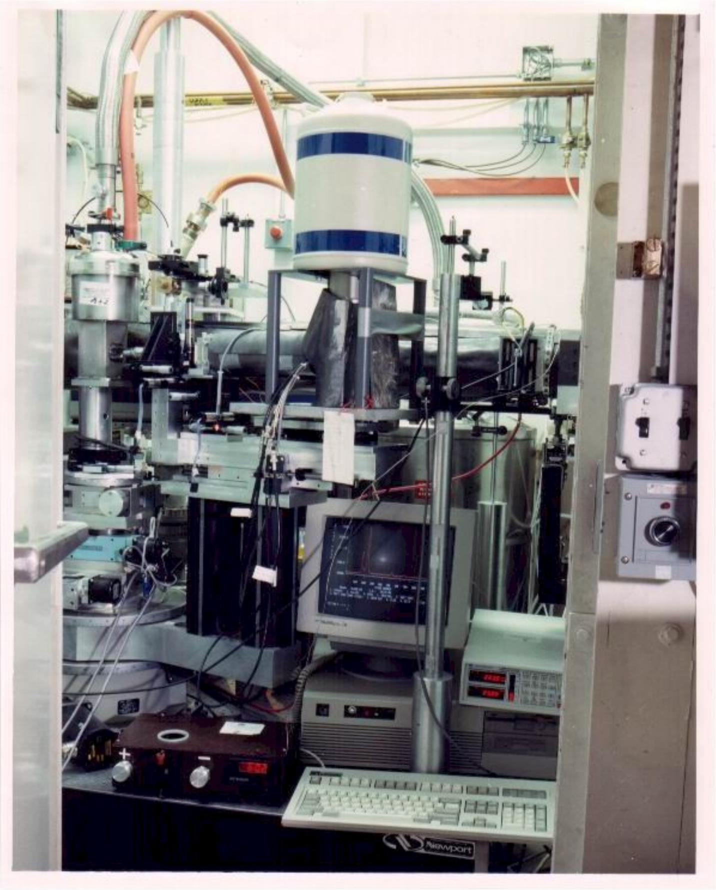
X17c superconductor beamline
Trailblazer for HP Research

- Dedicated high-pressure XRD beamline
- Dedicated high-pressure IR beamline
- Advancing multimegaber XRD
- Integration of XRD and laser-heating at high *P*-*T*
- Double-sided laser heating
- Resistive heating and XRD above 100 GPa-1300 K
- Integration of HP XRD, IR, on-line Raman at cryogenic T
- Radial diffraction for HP elasticity tensor and rheology
- Single crystal diffraction above 60 GPa

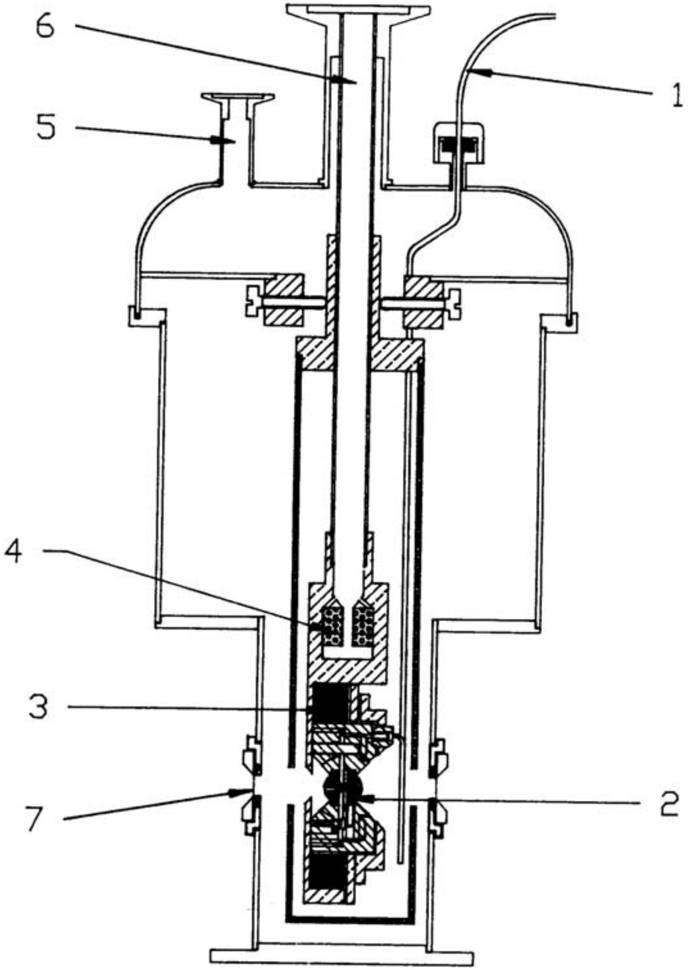
-- but limited to x-ray diffraction

High pressure and low temperature

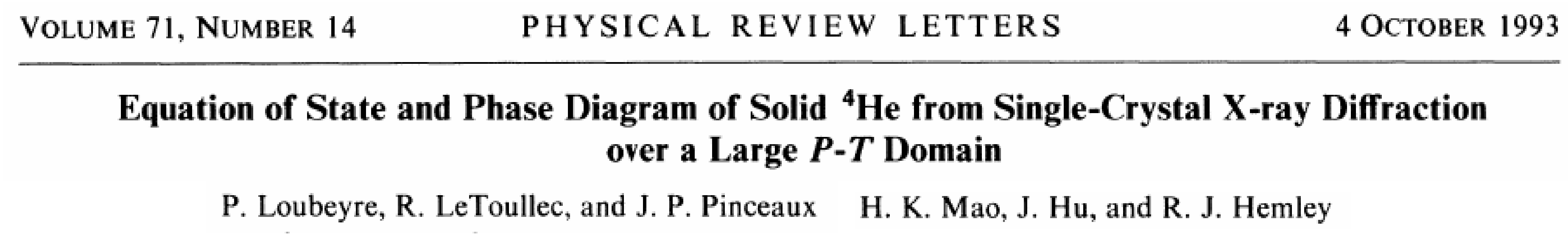
Cryogenic system had been setup at X17C and X17B for high pressure and low temperature experiments.



R. LeToullec et al, High pressure research,6,379-388,1992



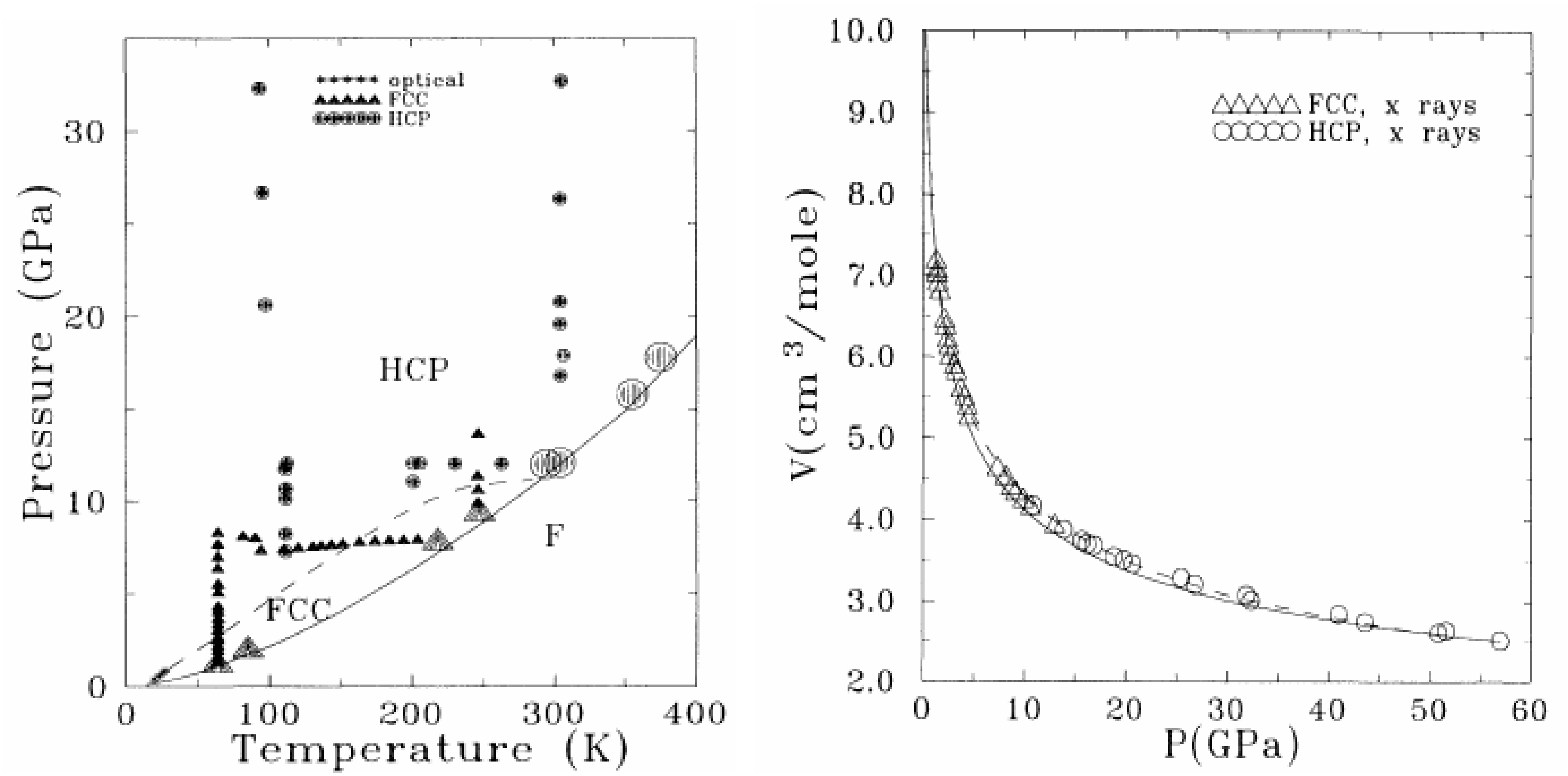
Membrane DAC on a c circle in Dewar



VOLUME 71, NUMBER 14 PHYSICAL REVIEW LETTERS 4 OCTOBER 1993

Equation of State and Phase Diagram of Solid ^{4}He from Single-Crystal X-ray Diffraction over a Large *P-T* Domain

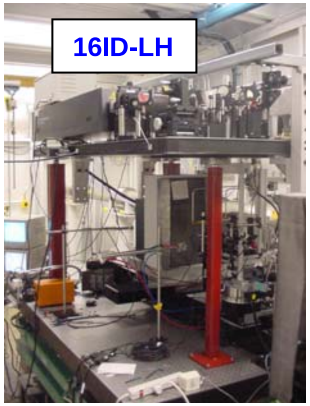
P. Loubeyre, R. LeToullec, and J. P. Pinceaux H. K. Mao, J. Hu, and R. J. Hemley



High P-T x-ray diffraction of He (46-400K, up to 58 GPa) at X17C, NSLS.

1993

2000 -- Dedicated *HP* multi-technique beamlines

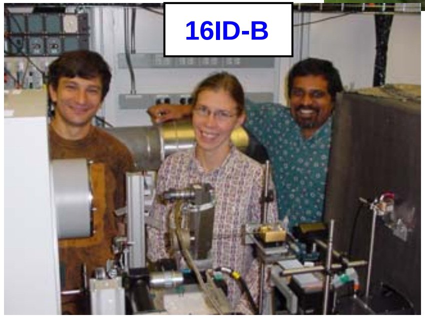


DAC laser-heating and x-ray diffraction

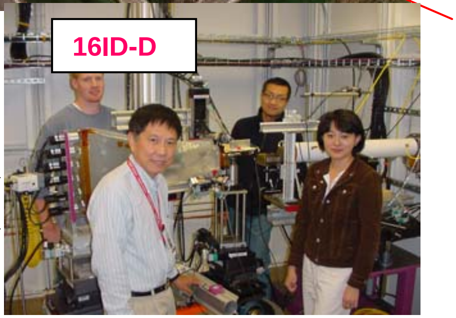
GSECARS Sector 13

HPCAT Sector 16

Advanced Photon Source at Argonne National Lab



DAC X-ray diffraction

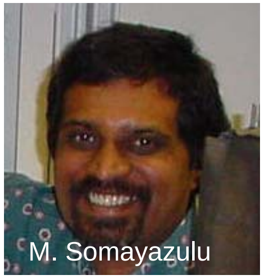


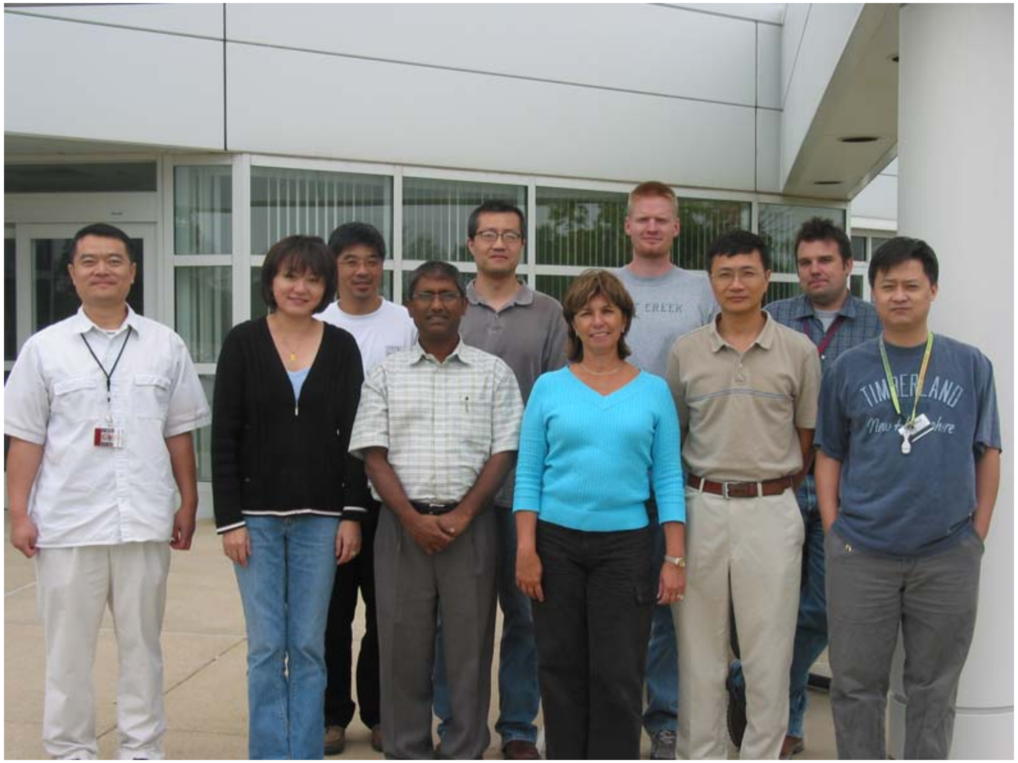
DAC X-ray spectroscopy

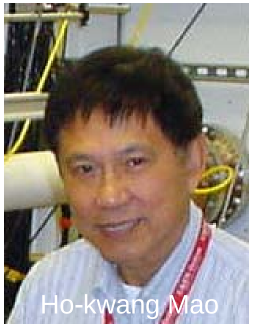
The High Pressure Collaborative Access Team project at the Advanced Photon Source

Beamline scientists enable the HP-SR development


Daniel Hausermann


M. Somayazulu




Ho-kwang Mao

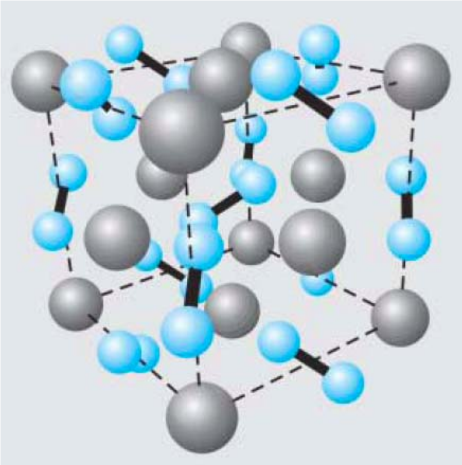
New class of metal nitrides could lead to more durable semiconductors

www.chemie.de/news

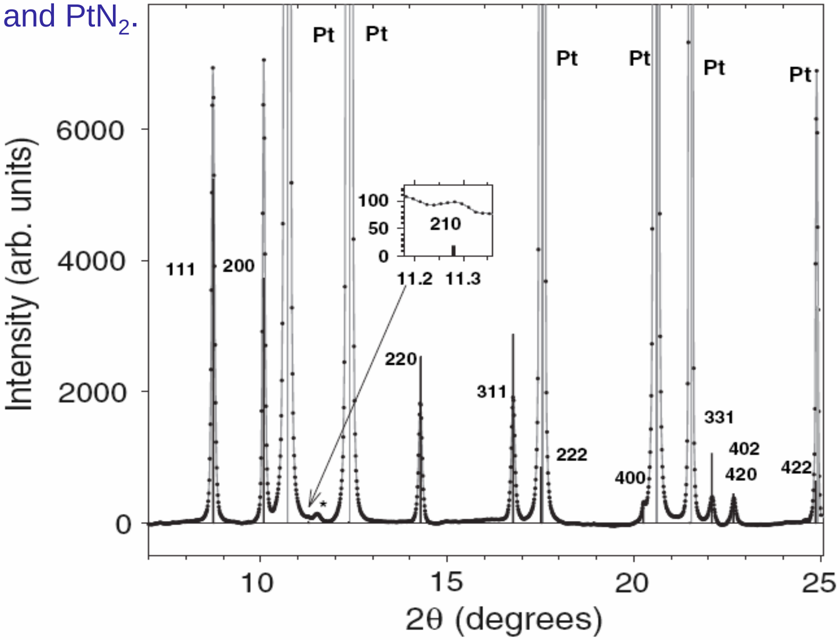
A novel class of nitrides made from noble metals was synthesized under extreme conditions. Using a diamond anvil cell to create high pressures and a laser to create high temperatures, the first bulk nitride of the noble metal iridium was created. By combining x-ray diffraction measurements at HPCAT with first-principle theoretical modeling, the structure and bulk modulus of platinum nitride have been determined. The results could prove useful in semiconductor, superconductor and corrosion-resistant devices by making materials more durable and reliable.

J. C. Crowhurst, *et al.*, Synthesis and characterization of the nitrides of platinum and iridium, *Science.* *311,* 1275 (2006)

PtN_2 in pyrite structure



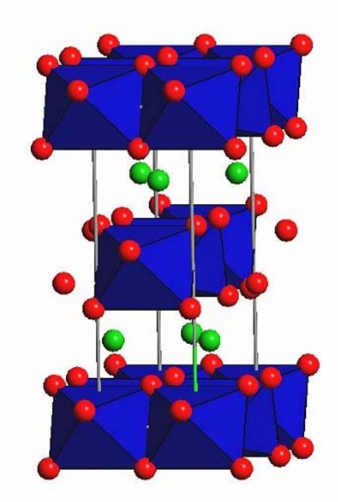
Powder x-ray diffraction pattern of Pt and PtN_2.

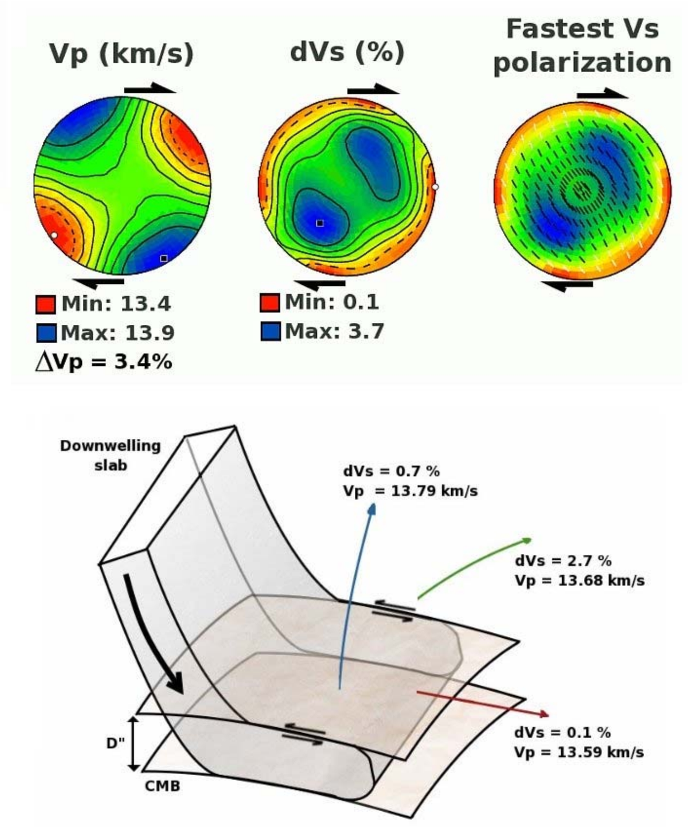


HPCAT 2006 highlight **X-ray diffraction, laser-heating, Raman**

Seismic Anisotropy in D” Layer

Sebastien Merkel and colleagues from U. C. Berkeley, Princeton U., and Carnegie Institution reported plastic behavior of $MgGeO_3$ post-perovskite, an analog for the main constituent in the D” layer deep at the Earth’s core-mantle boundary. They monitored the development of lattice preferred orientations in x-ray diffraction experiment above 100 GPa at HPCAT, and discovered that (100) and (110) slip dominate the plastic deformation of post-perovskite. The results give a new interpretation to the D” seismic anisotropy – the splitting of horizontally and vertically polarized seismic waves.





Contribution of silicate p-Pv to seismic anisotropy in D" after 20% deformation in shear

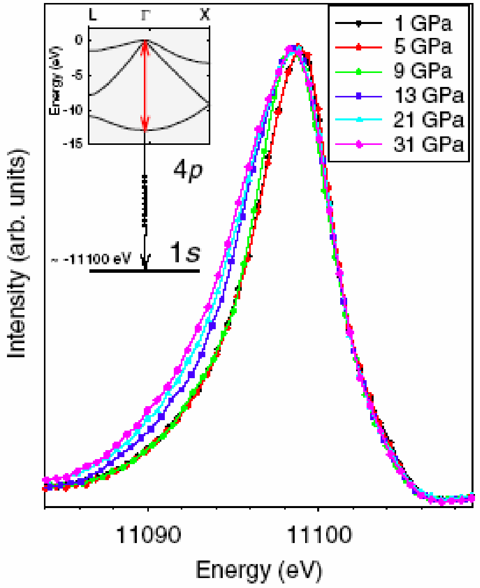
S. Merkel *et al.*, Plastic deformation of $MgGeO_3$ post-perovskite at lower mantle pressures, *Science 311,* 644 (2006)

HPCAT 2006 highlight **Radial x-ray diffraction, laser-heating**

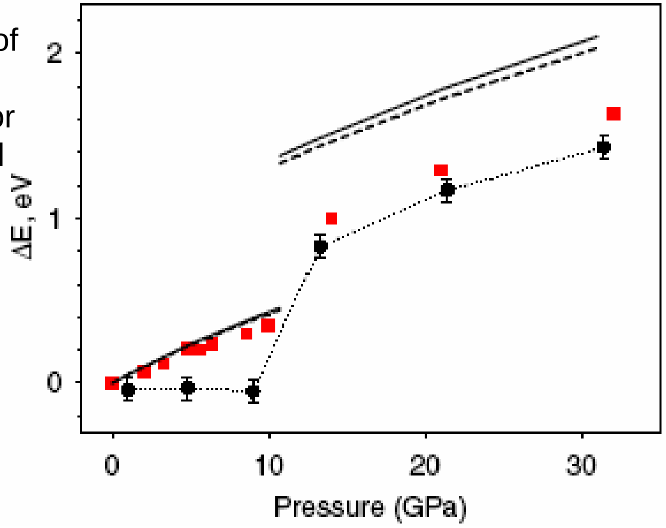
Valence Band Width Near Pressure-Induced Metallization of Ge

Pressure causes drastic changes in electronic bandwidth and band gap, and leads to metallization of solids. New high-pressure x-ray emission spectroscopy technique developed at HPCAT has been used to probe the valence-band emission line in Ge from 4*p* states to 1*s* core state (Kβ2 at 11100.8 eV). The band widths for the semiconductor phase below 10 GPa and the metallic phase at higher pressures are determined, and the results are used to guide theoretical models beyond the current density-functional theory and the *GW* approximation.

Valence-band emission spectra of Ge at high pressures



Valence-band width of Ge under pressure. Solid circles with error bars—experiment; all other symbols and curves -- theories



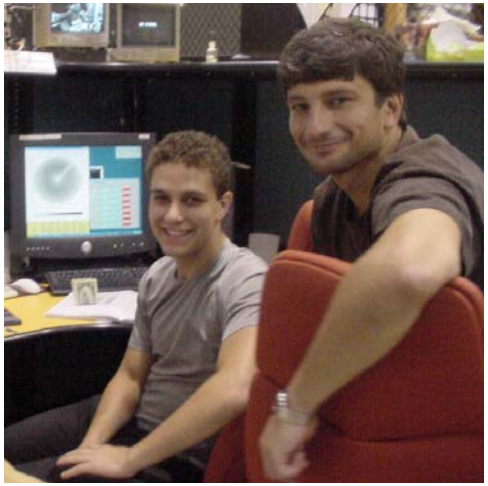
V. V. Struzhkin *et al.,* Valence band x-ray emission spectroscopy of compressed germanium, *Phys. Rev. Lett. 96,* 137402 (2006)

Novel Osmium and Iridium Nitrides

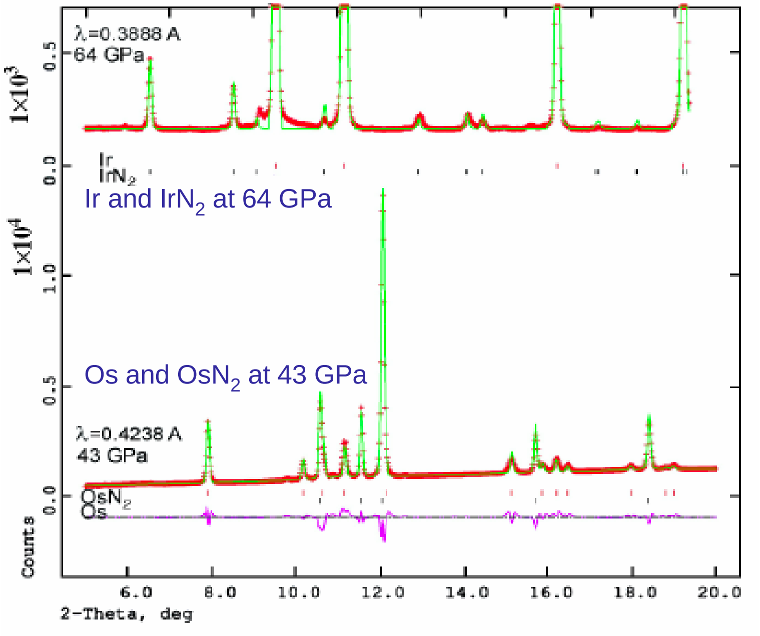
Carnegie summer undergraduate intern Andrea F. Young discovered two novel transition metal nitrides, IrN_2 and OsN_2. Synchrotron x-ray diffraction at HPCAT was used to determine the structures of nitrides and the equations of state for both the parent metals as well as the newly synthesized materials. These new compounds have bulk moduli comparable with those of traditional superhard materials. *Ab initio* calculations indicate that both compounds have a metal:nitrogen stoichiometry of 1:2 and that nitrogen intercalates in the lattice of the parent metal in the form of singly bonded N-N units.

A. F. Young *et. al.*, Synthesis of novel transition element nitrides IrN_2 and OsN_2, *Phys. Rev. Lett. 96,* 155501 (2006)

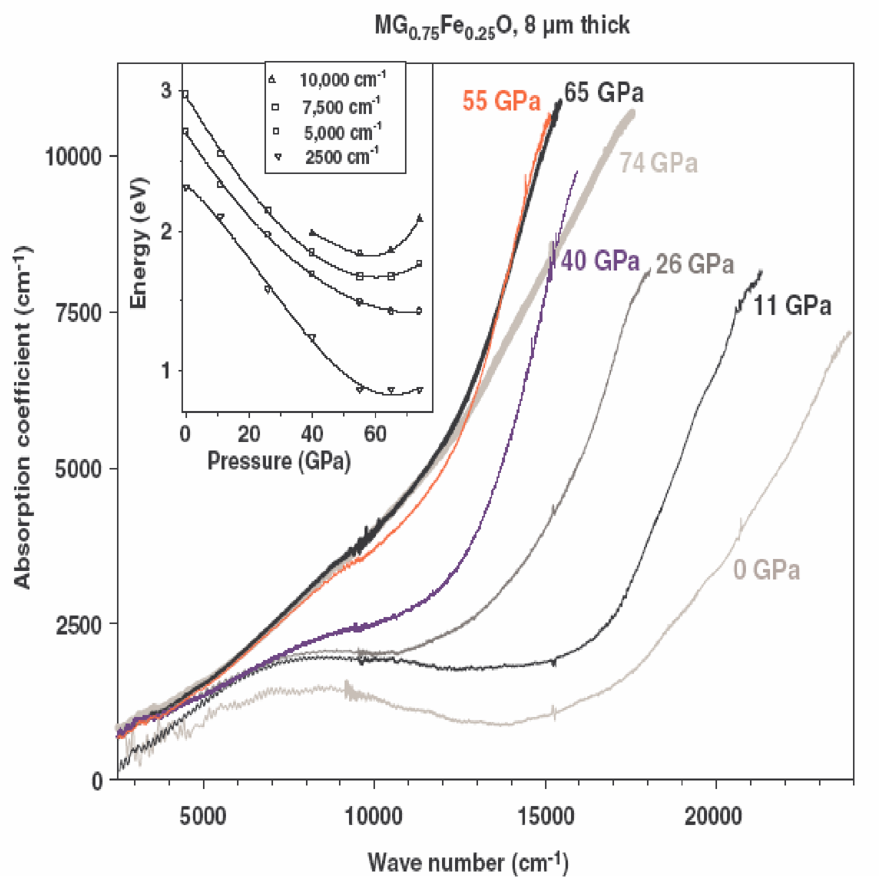
Intern Andrea Young (left) and mentor Eugene Gregoryanz (right) at HPCAT

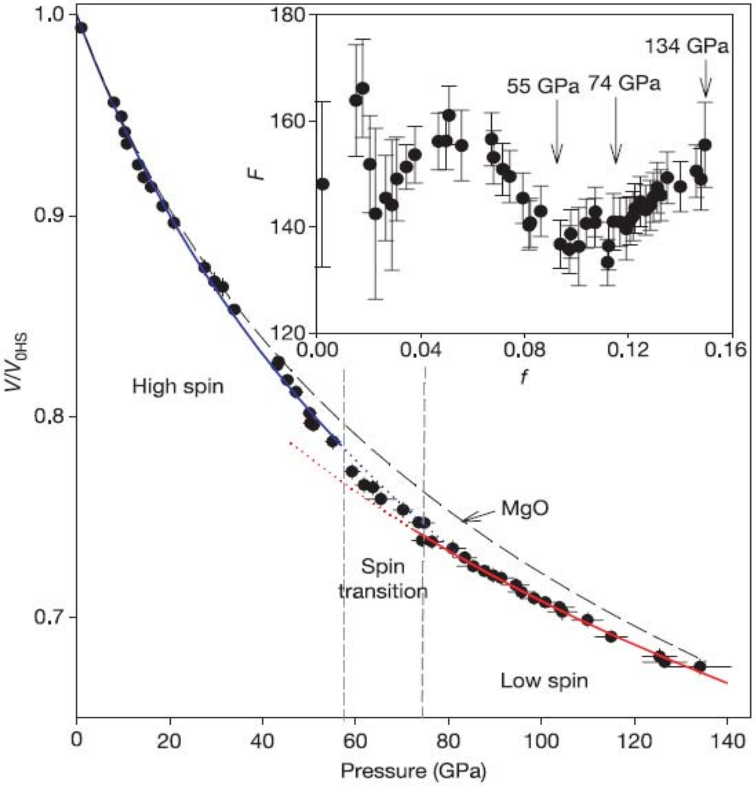


X-ray diffraction patterns



Low-spin (Mg,Fe)O magnesiowüstite in the lower mantle



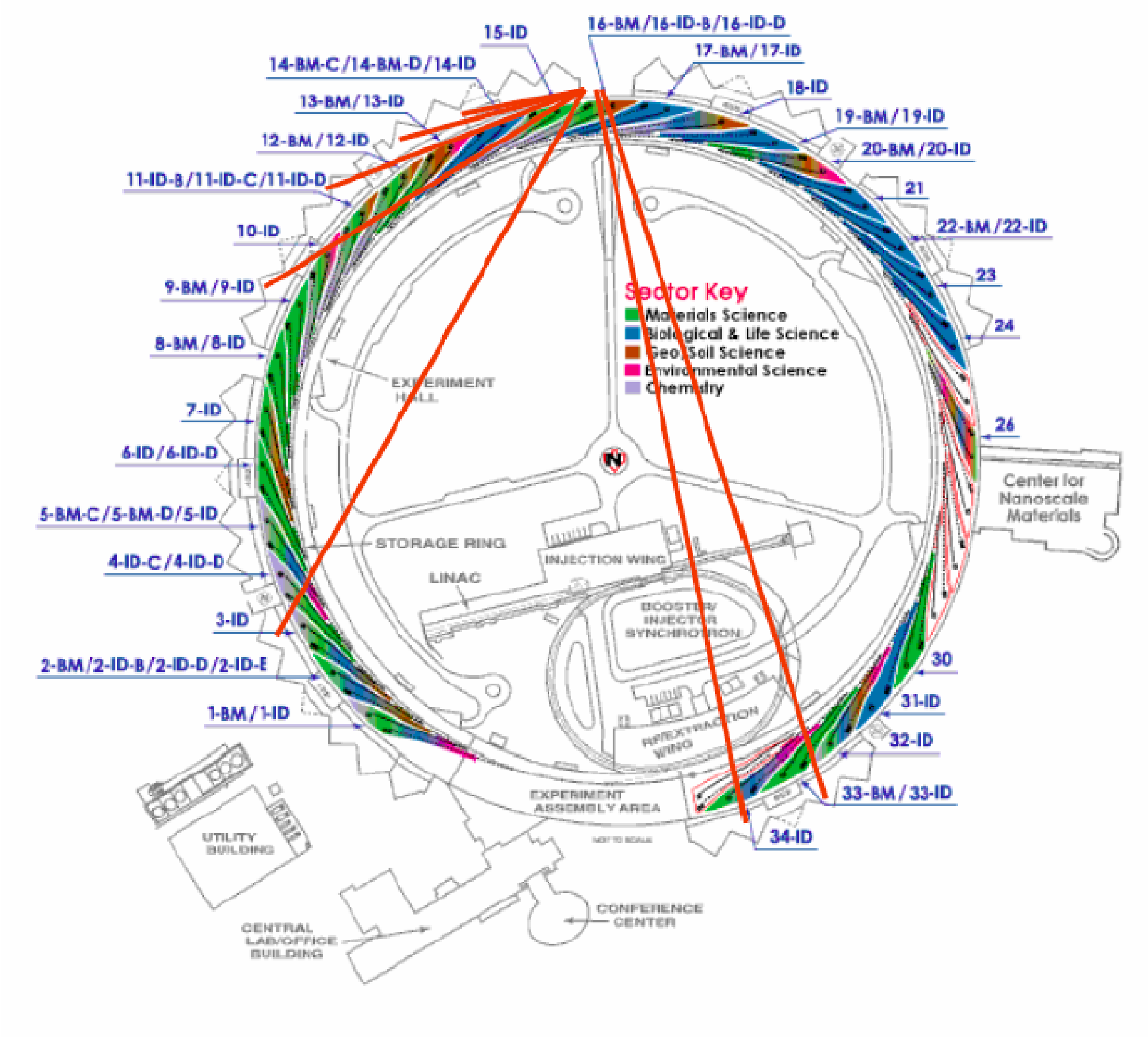


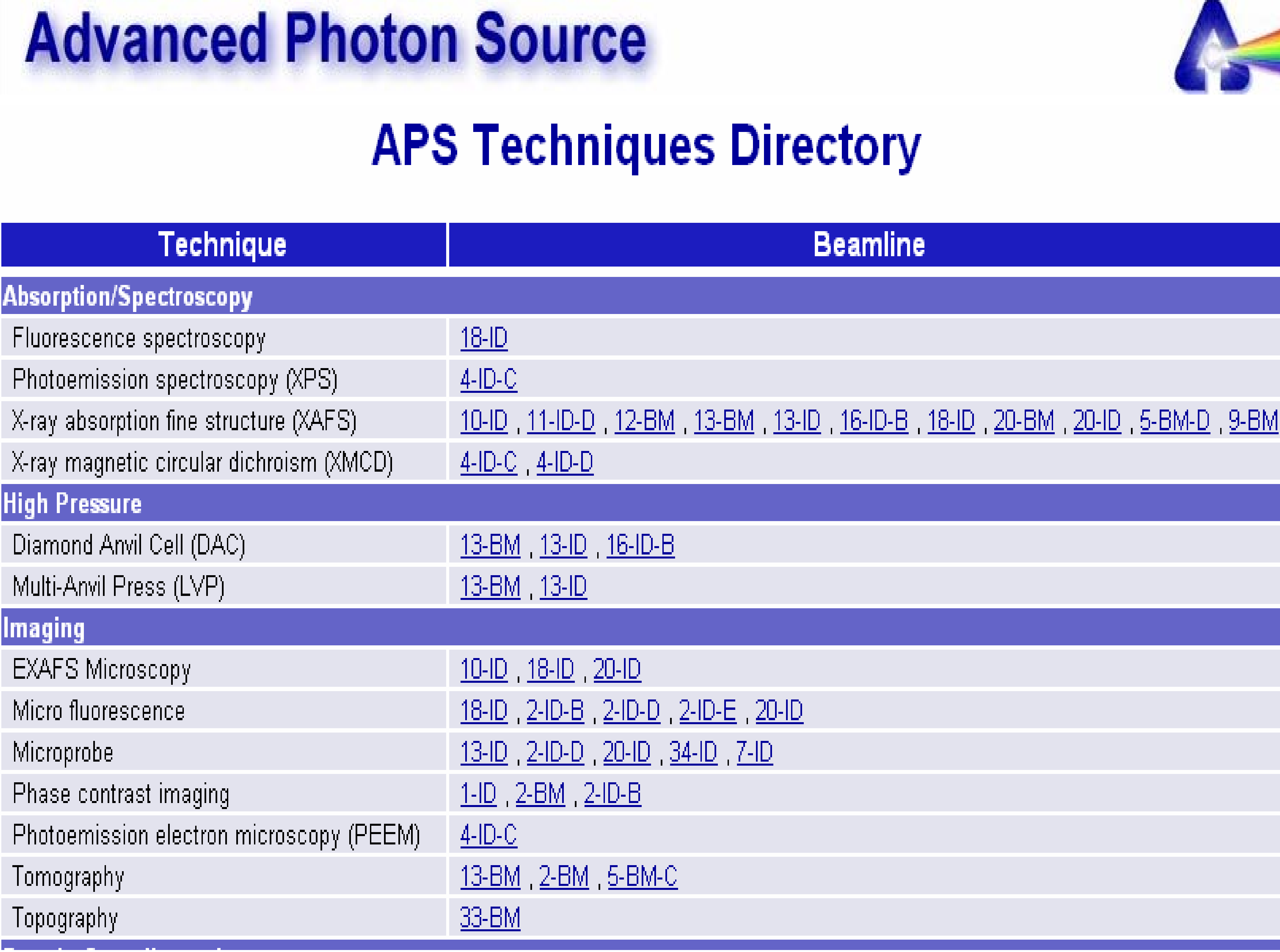
A. Goncharov, V. Struzhkin, S. Jacobsen, Reduced low spin (Mg,Fe)O in the lower mantle, *Science. 312,* 1205 (2006)

J-F Lin, Spin transition of iron in magnesio-wüstite in the Earth's lower mantle" *Nature* **436**, 377 (2005)

HPCAT 2006 highlight **X-ray diffraction, x-ray emission spectroscopy, optical absorption**

HP-CAT collaboration with other APS sectors has been very successful but enormous capabilities at APS has yet been tapped….





Advanced Photon Source

APS Techniques Directory

Technique	Beamline
Absorption/Spectroscopy	
Fluorescence spectroscopy	18-ID
Photoemission spectroscopy (XPS)	4-ID-C
X-ray absorption fine structure (XAFS)	10-ID , 11-ID-D , 12-BM , 13-BM , 13-ID , 16-ID-B , 18-ID , 20-BM , 20-ID , 5-BM-D , 9-BM
X-ray magnetic circular dichroism (XMCD)	4-ID-C , 4-ID-D
High Pressure	
Diamond Anvil Cell (DAC)	13-BM , 13-ID , 16-ID-B
Multi-Anvil Press (LVP)	13-BM , 13-ID
Imaging	
EXAFS Microscopy	10-ID , 18-ID , 20-ID
Micro fluorescence	18-ID , 2-ID-B , 2-ID-D , 2-ID-E , 20-ID
Microprobe	13-ID , 2-ID-D , 20-ID , 34-ID , 7-ID
Phase contrast imaging	1-ID , 2-BM , 2-ID-B
Photoemission electron microscopy (PEEM)	4-ID-C
Tomography	13-BM , 2-BM , 5-BM-C
Topography	33-BM

Protein Crystallography	
Macromolecular crystallography	14-BM-C , 14-BM-D , 14-ID , 17-ID , 19-BM , 19-ID , 22-ID , 31-ID , 5-ID , 8-BM
Multi wavelength anomalous dispersion (MAD)	14-BM-D , 14-ID , 17-ID , 19-BM , 19-ID , 22-ID , 8-BM
Scattering	
Anomalous and Resonant Scattering	15-ID , 33-ID , 4-ID-D
Coherent x-ray scattering	2-ID-B , 34-ID , 8-ID
Compton scattering	16-ID-B
Diffraction anomalous fine structure (DAFS)	10-ID , 20-BM , 20-ID
Fiber Diffraction	18-ID
General Diffraction	11-ID-D , 12-BM , 2-BM , 20-BM , 20-ID , 33-BM , 33-ID
High energy x-ray scattering	1-ID , 11-ID-B , 11-ID-C , 5-BM-D , 6-ID-D
Inelastic scattering	13-ID , 16-ID-B , 3-ID , 33-ID
Liquid scattering	15-ID , 6-ID , 9-ID
Magnetic x-ray scattering	4-ID-C , 4-ID-D , 6-ID , 6-ID-D
Micro - diffraction	13-ID , 16-ID-B , 18-ID , 2-BM , 2-ID-D , 34-ID
Nuclear Resonant Scattering	16-ID-B , 3-ID
Polymer	5-BM-D , 5-ID
Powder diffraction	1-BM , 12-BM , 16-ID-B , 33-BM , 5-BM-C , 5-ID , 6-ID , 6-ID-D
Reflectivity	1-BM
Single crystal diffraction	33-BM
Small angle x-ray scattering (SAXS)	12-ID , 15-ID , 18-ID , 33-ID , 5-ID , 8-ID , 9-ID
Surface diffraction	33-ID , 6-ID
Time-resolved x-ray scattering	14-ID , 15-ID , 18-ID , 6-ID-D , 7-ID , 8-ID
Ultra-small Angle X-ray Scattering	33-ID
Wide angle x-ray scattering (WAXS)	15-ID , 8-ID

… but beamline scientists’ main obligations are to build, maintain, operate, and support users. Little time for HP-SR development at other beamlines

Arunkumar Bommannavar
Paul Chow
Yang Ding
Daniel Hausermann
Michael Hu
Peter Liermann
Haozhe Liu
Yue Meng
Veronica O’Connor
Eric Rod
Guoyin Shen

Former staff:
Daniel Errandonea
Maddury Somayazulu

GL/CDAC
Russell Hemley
Ho-kwang Mao
Stephen Gramsch

Malcolm Nicol – UNLV
Choong-Shik Yoo – LLNL
Murli Manghnani – U Hawaii

Users attempting to develop novel experiments at other beamlines are facing the same challenge as in the first decade.

HPSynC

A team similar to a beamline, not working as beamline scientists but bridging…

- **Scientific disciplines and community**
- **High *P-T* vessels**
- **Analytic probes and facilities**

Retrospective facility-wide coordination is difficult at existing facility.

It should be built in at the planning stage, such as ERL and NSLS-II