Tailoring high-pressure experiments for the energy, flux, and resolution of IXS at NSLS-II

Hokwang (David) Mao
Carnegie Institution for Science

NSLS-II Early Experiment Workshop –
IXS Focused Session
September 30, 2013

Count Rate Estimates (9.1 vs 21.7 keV)

- Given the same q resolution, I/I_0 is proportional to analyzer solid angle $(\delta\theta)^2$ and sample absorption length l_{abs} (volume):

$$\frac{I}{I_0} \propto \left(\frac{\delta q}{E_i}\right)^2 l_{abs} = \left(\frac{2}{\hbar}\delta\vartheta\right)^2 l_{abs}$$

H. Sinn (2001)

Count rate obtained on ID28 at ESRF (Data courtesy of M. Krisch)

Sample	Thickness used (μm)	E_{ino} (keV)	ΔE_{ino} (meV)	Intensity (phs/sec)	Acceptance (mrad²)	Q (nm⁻¹)	ΔE_{total} (meV)	Count Rate (cnts/sec)
v-SiO_2	~2100	21.7	1.0	4×10^9	27	1 – 4	1.6	~0.2
H_2O (HP)	~1000	21.7	1.0	4×10^9	27	3 – 13	1.6	~1.0
La_2CuO_4	~75.3	17.8	2.0	2.7×10^{10}	27	44.4	3.3	~0.5
Solid O_2	~10	17.8	2.0	1.0×10^{10}	27	37.5	3.3	~1.5

Estimated count rate for NSLS-II instrument at 1 meV resolution

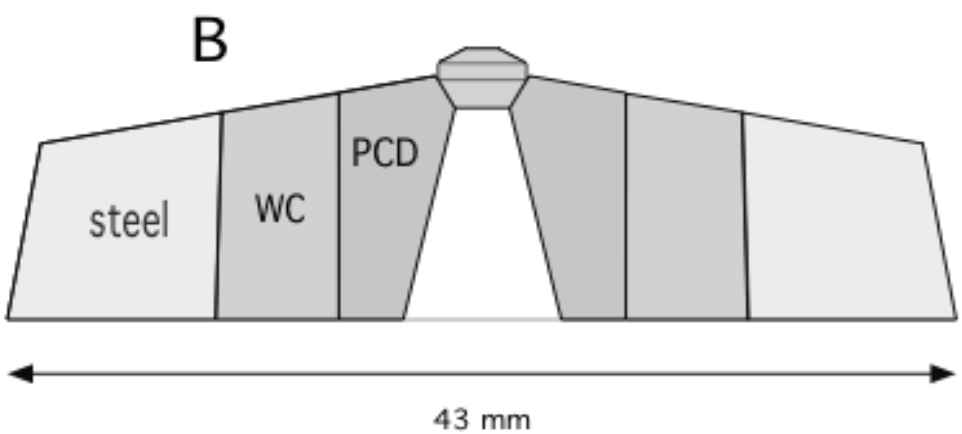
Sample	I_0 Gain	I_{abs} Gain	$(\delta\theta)^2$ Gain	Efficiency Gain	Abs. Correction	Count Rate (cnts/sec)
v-SiO_2	1.6/0.4	190/2100	25/27	2.5	1	0.2
H_2O (HP)	1.6/0.4	1000/1000	25/27	2.5	0.72/0.92	~7
La_2CuO_4	1.6/2.7	7.13/75.3	25/27	1.0	1	~0.03
Solid O_2	1.6/1.0	10/10	25/27	1.0	0.72/0.9	~2.0

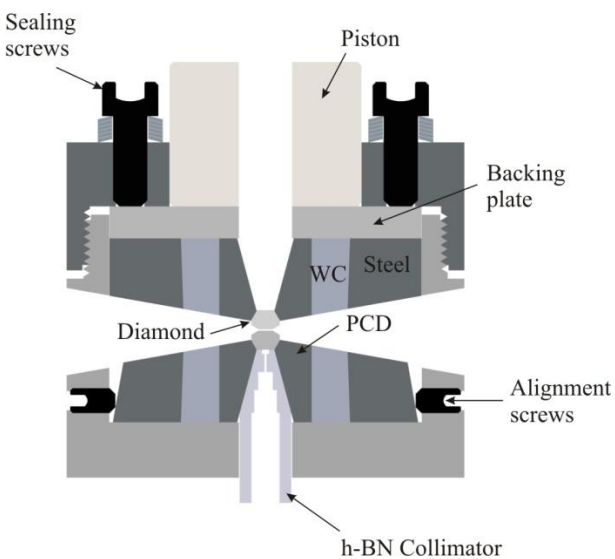
Better performance for low-Z materials!

Tailoring high-pressure IXS experiments for the energy, flux, and resolution of NSLS-II

Using 9.1 keV, NSLS-II is particularly advantageous for low-Z materials which will benefit from thicker samples. We shall modify the DAC design to optimize the thickness without sacrificing the pressure range. The goals is four times the present limit to reach:

- 500 μm thickness at 100 GPa
- 2000 μm at 10 GPa





Key high-pressure experiments to utilize the IXS capabilities of NSLS-II

- Vibrational spectroscopy with dispersion—hydrogen vibron; diamond and graphite Raman.
- Low-Z liquid and amorphous materials—amorphous and nano carbon; Li and Na high-*P* melts.
- Phonon and elasticity –
  high *P*-*T* Fe; single crystal graphite-diamond transition.

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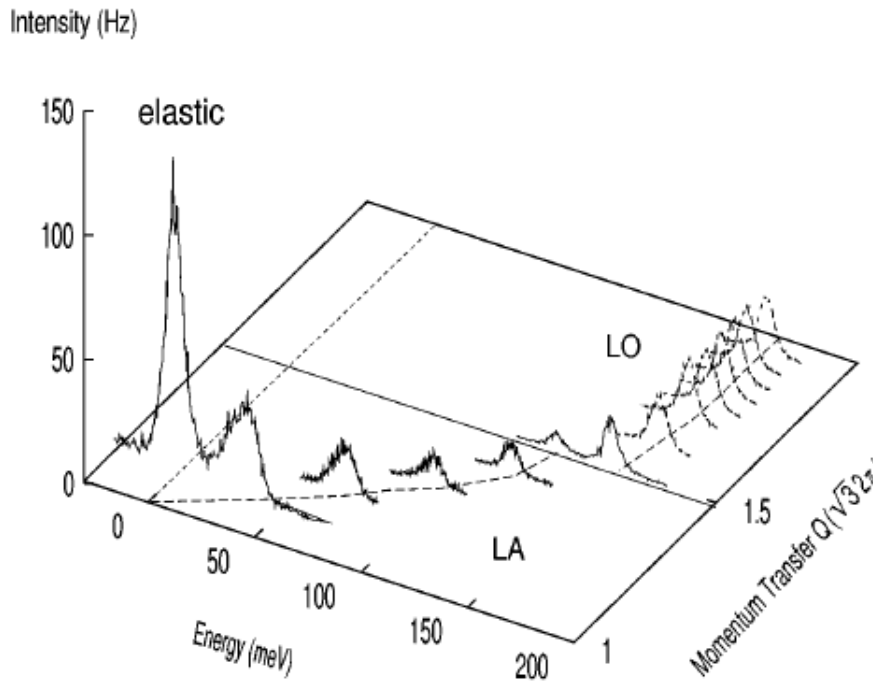
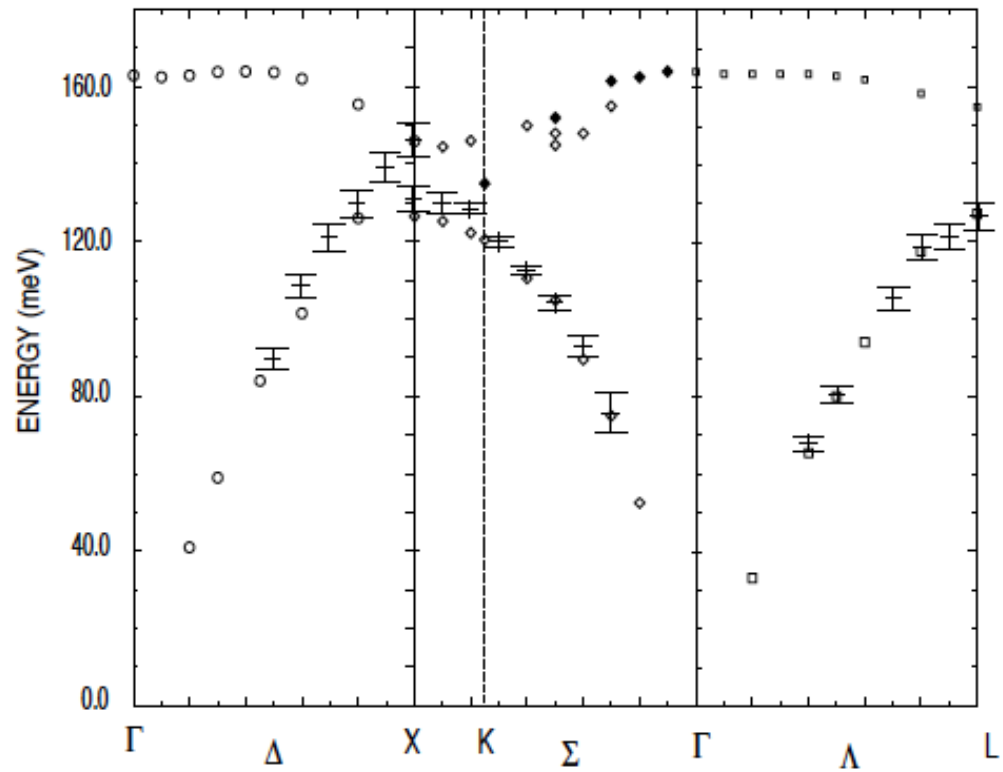
VOLUME 80, NUMBER 25 PHYSICAL REVIEW LETTERS 22 JUNE 1998

Phonon Dispersion of Diamond Measured by Inelastic X-Ray Scattering

M. Schwoerer-Böhning* and A. T. Macrander

6 meV resolution





Kuida et al, PRB (2002)

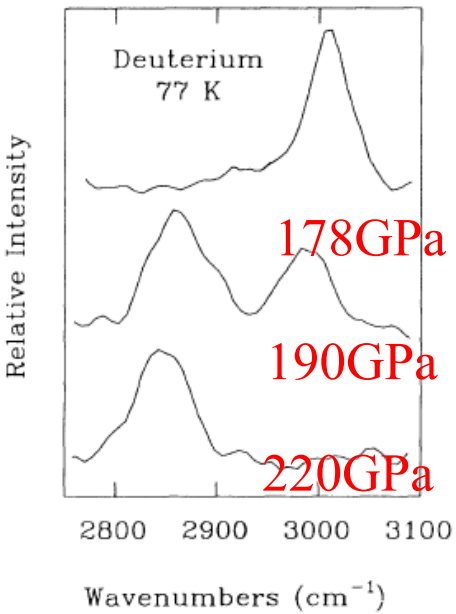
Hydrogen Raman vibron

VOLUME 63, NUMBER 13 PHYSICAL REVIEW LETTERS 25 SEPTEMBER 1989

Isotope Effects in Dense Solid Hydrogen: Phase Transition in Deuterium at 190 ± 20 GPa

1989

R. J. Hemley and H. K. Mao
Geophysical Laboratory, Carnegie Institution of Washington, 2801 Upton Street, N.W., Washington, D.C. 20008-3898
(Received 30 June 1989)

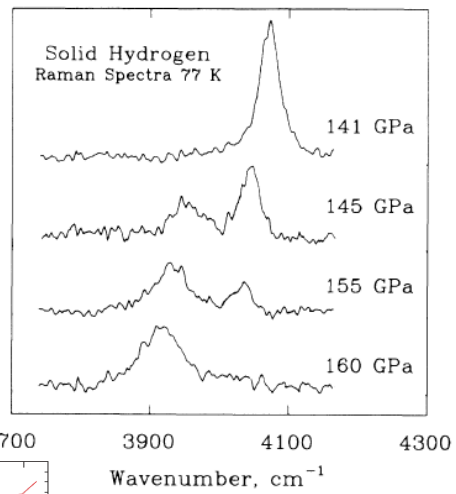


VOLUME 61, NUMBER 7 PHYSICAL REVIEW LETTERS 15 AUGUST 1988

Phase Transition in Solid Molecular Hydrogen at Ultrahigh Pressures

1988

R. J. Hemley and H. K. Mao
Geophysical Laboratory, Carnegie Institution of Washington, Washington, D.C. 20008-3898
(Received 31 May 1988)

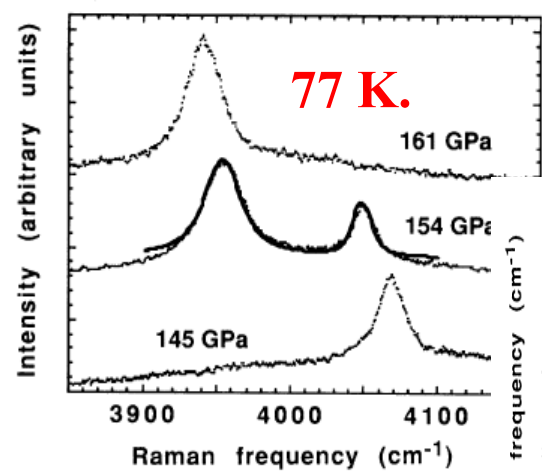


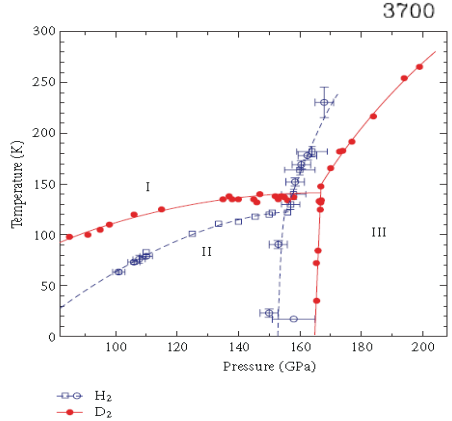
LUME 63, NUMBER 19 PHYSICAL REVIEW LETTERS 6 NOVEMBER 1989

Evidence for a Structural Phase Transition in Solid Hydrogen at Megabar Pressures

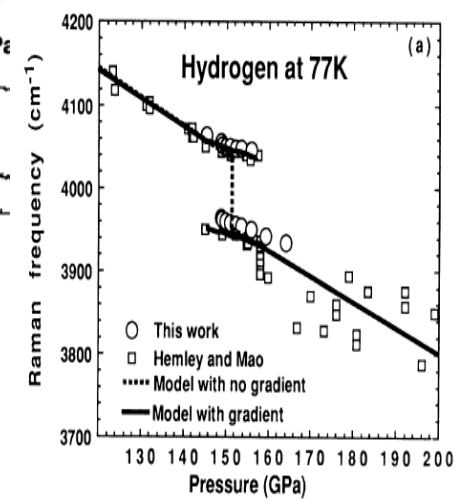
1989

Hector E. Lorenzana, Isaac F. Silvera, and Kenneth A. Goettel
Lyman Laboratory of Physics, Harvard University, Cambridge, Massachusetts 02138
(Received 22 June 1989)

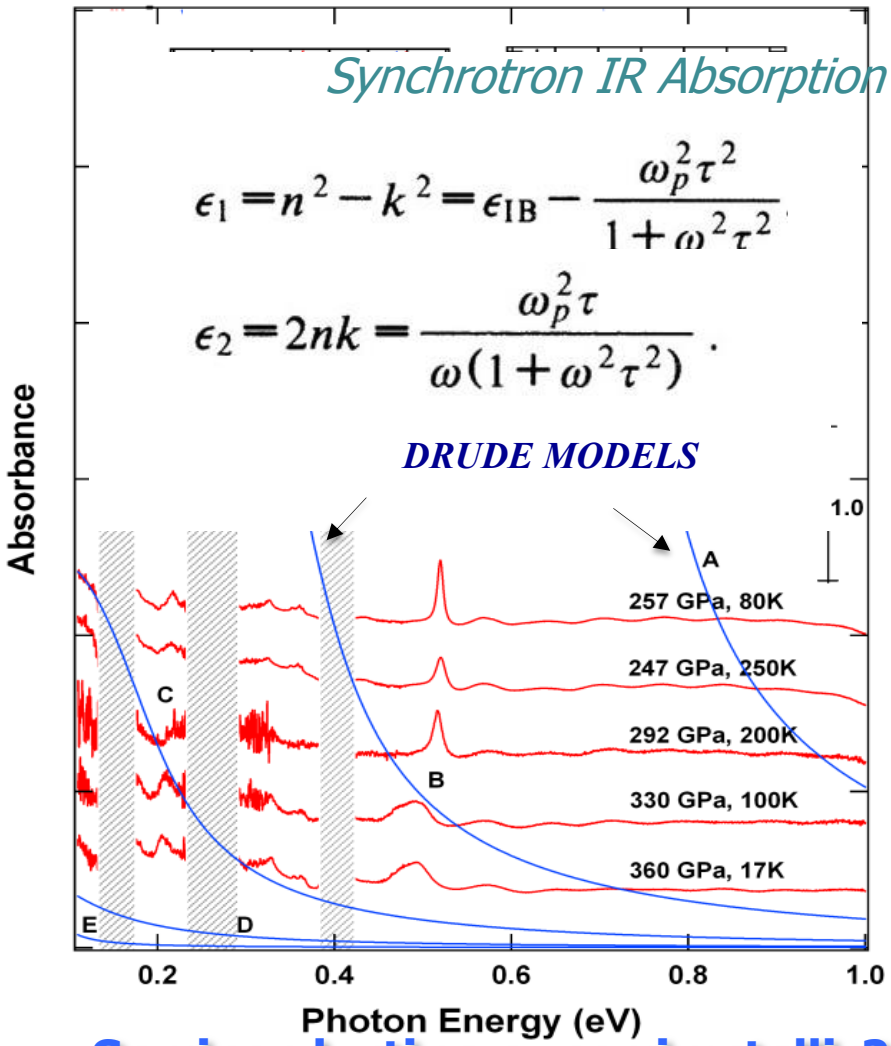




1 meV = 8 cm^{-1}

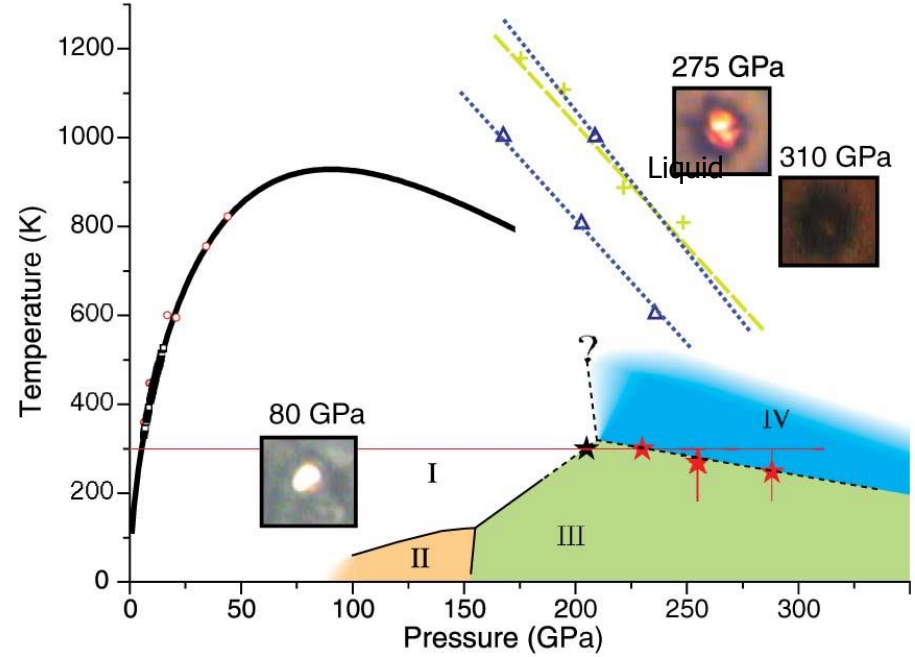


Hydrogen IR vibron



Semiconducting or semimetallic?

Zha *et al., Phys. Rev. Lett.* ***108,*** 125501 (2012); *Phys. Rev. Lett.* ***110,*** 217402 (2013)
Carnegie Institution for Science



Evidence for a new phase

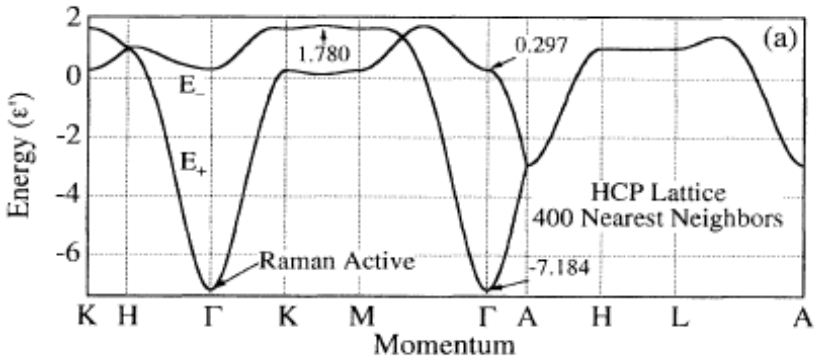
Howie *et al., Phys. Rev. Lett.* **108,** 125501 (2012)
University of Edinburgh

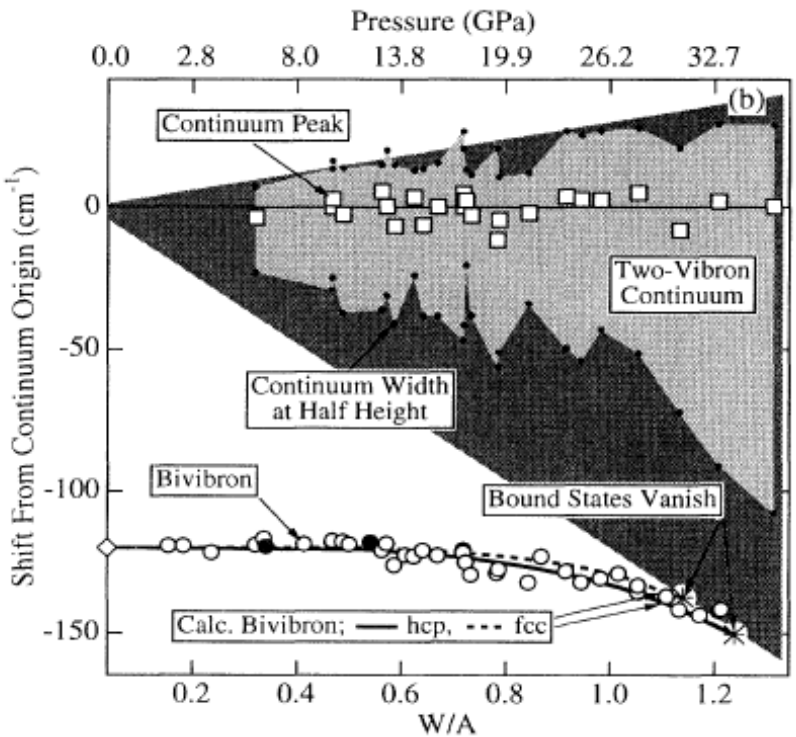
- **Continued challenge**
- **EFree experiment/theory synergy**

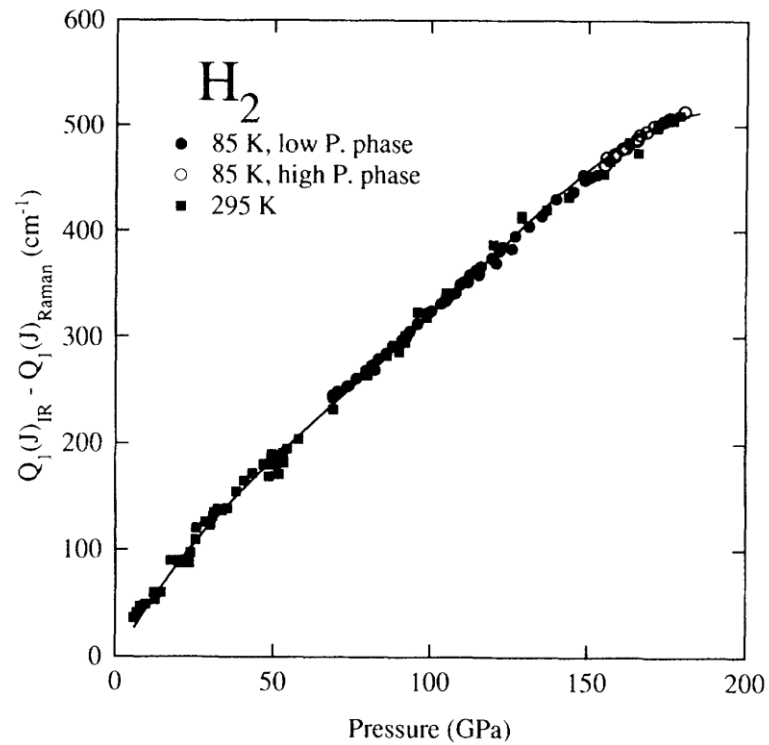
VOLUME 70, NUMBER 15 PHYSICAL REVIEW LETTERS 12 APRIL 1993

Observation of a Two-Vibron Bound-to-Unbound Transition in Solid Deuterium at High Pressure

Jon H. Eggert, Ho-kwang Mao, and Russell J. Hemley



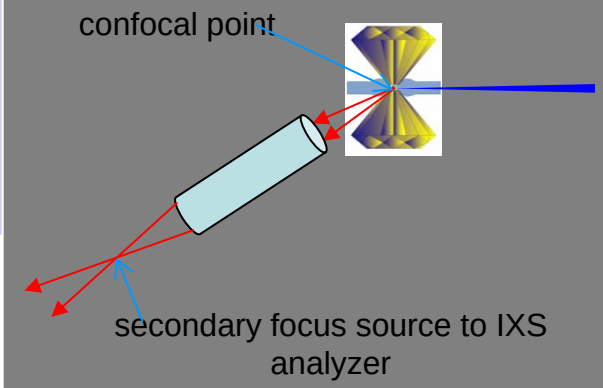


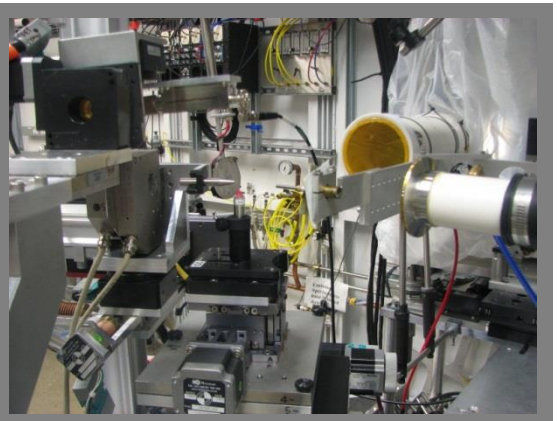


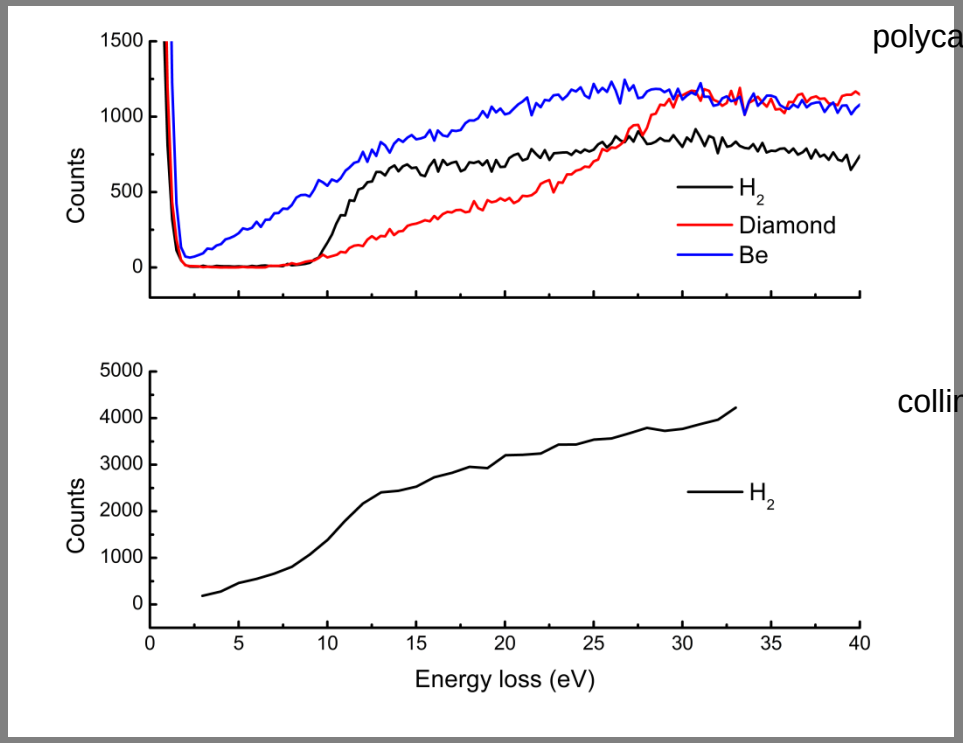
Hanfland, Hemley, Mao, PRL, 1992; 1993

Inelastic X-ray Scattering with Polycapillary Lens

- Improved the collimation and suppressed the background signal by an order of magnitude
- Enabled new measurements of band structures of H_2 which was not feasible with the conventional slit collimation







Yuming Xiao, Paul Chow, Yang Ding, Junyue Wang, Dave Mao, to be published

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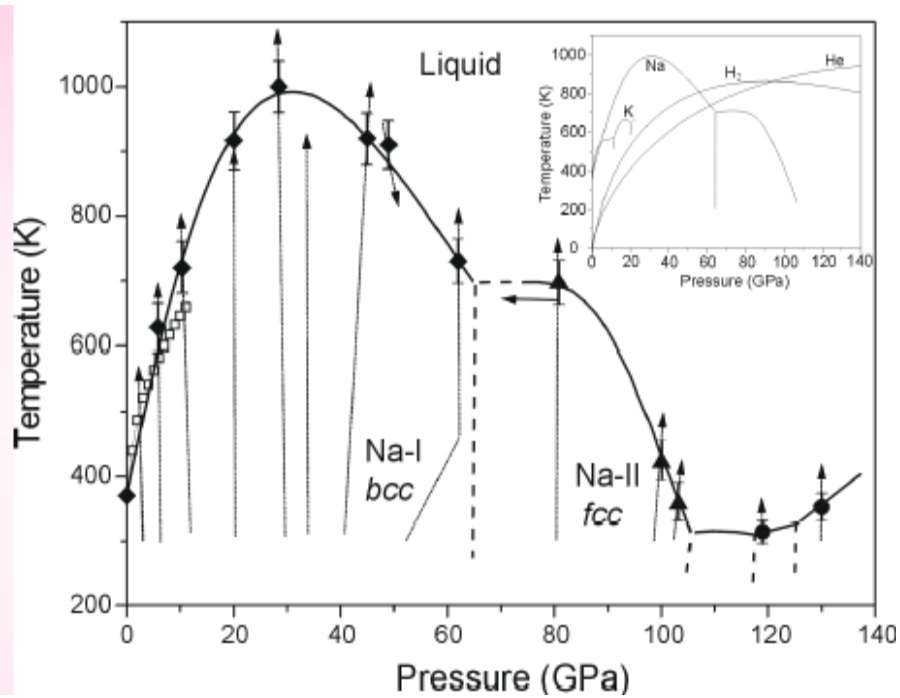
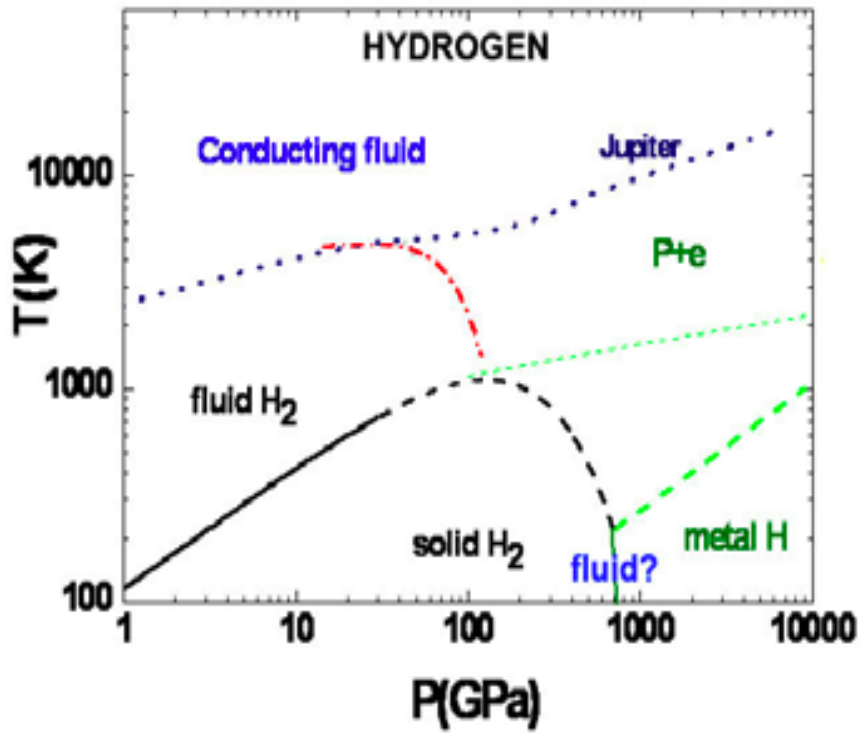
- **Low-Z liquid and amorphous materials—amorphous and nano carbon; Li and Na high-*P* melts.**
- Phonon and elasticity – high *P-T* Fe; single crystal graphite-diamond transition.

Alkali metal has a meltdown



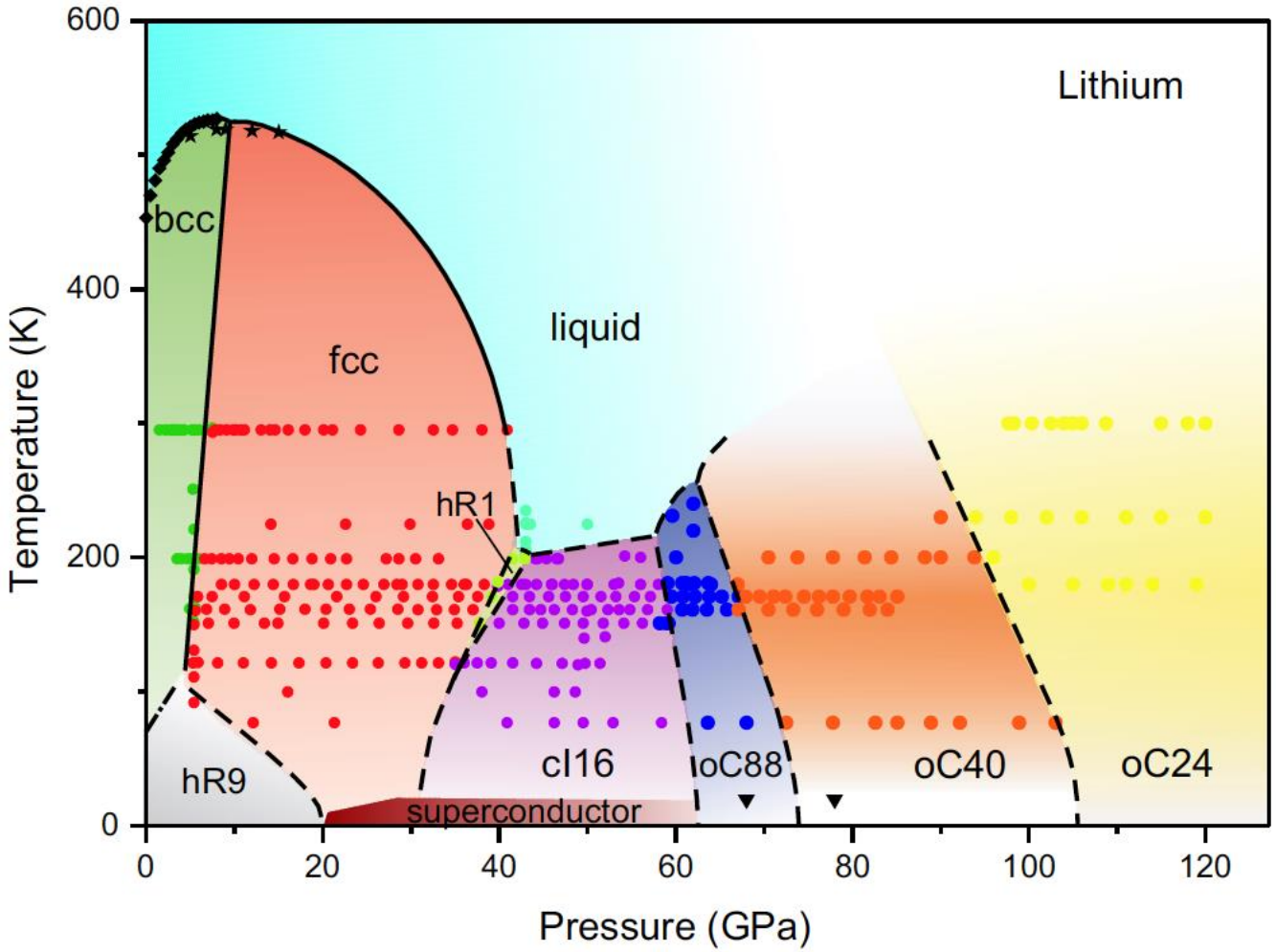


Gregoryanz, *et al*., *PRL 94*, 185502 (2005)
Gregoryanz, *et al*., *Science 320*, 1054 (2008)
Ashcroft, *Nature 458*, 158 (2009)
Ma, *et al.*, *Nature 458*, 182 (2009)
Matuoka, *et al.*, *Nature 458*, 186 (2009)

Babaev *et al*, *Nature 431,* 666 (2004)
Bonev *et al*, *Nature 431,* 669 (2004)
Pickard and Needs, *Nature Phys. 2,* 473 (2007)
Babaev *et al*, *Nature Phys. 3,* 530 (2007)
Attaccalite & Sorella, *PRL 100,* 114501 (2008)

Cold melting and solid structures of dense lithium

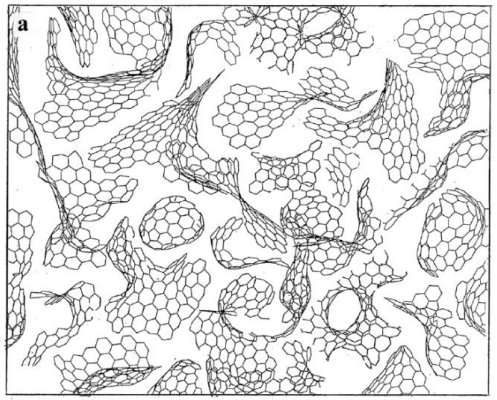
Christophe L. Guillaume, Eugene Gregoryanz, Olga Degtyareva, Malcolm I. McMahon, Shaun Evans, Michael Hanfland, Malcolm Guthrie, Stas V. Sinogeikin, & H-K. Mao, *Nature Physics 7, 211 (*2011)



PRL 107, 175504 (2011) PHYSICAL REVIEW LETTERS week ending 21 OCTOBER 2011

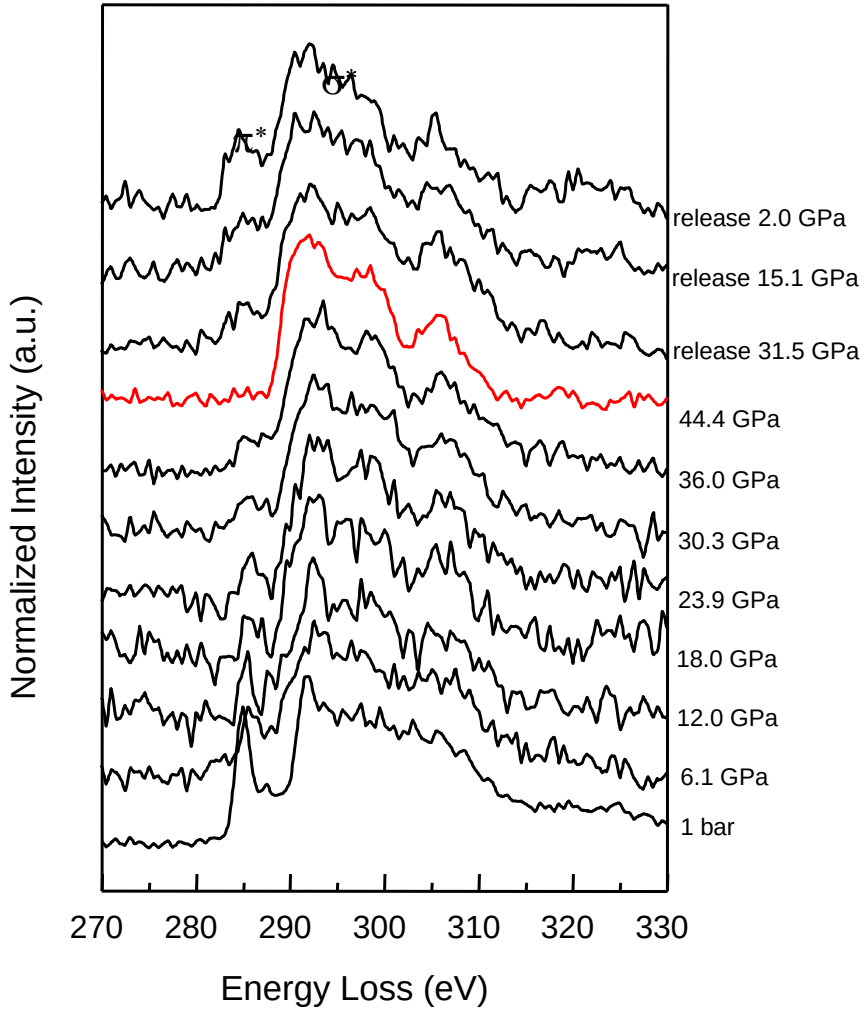
Amorphous Diamond: A High-Pressure Superhard Carbon Allotrope

Yu Lin,[1,*] Li Zhang,[2] Ho-kwang Mao,[2] Paul Chow,[3] Yuming Xiao,[3] Maria Baldini,[4] Jinfu Shu,[2] and Wendy L. Mao[1,5]

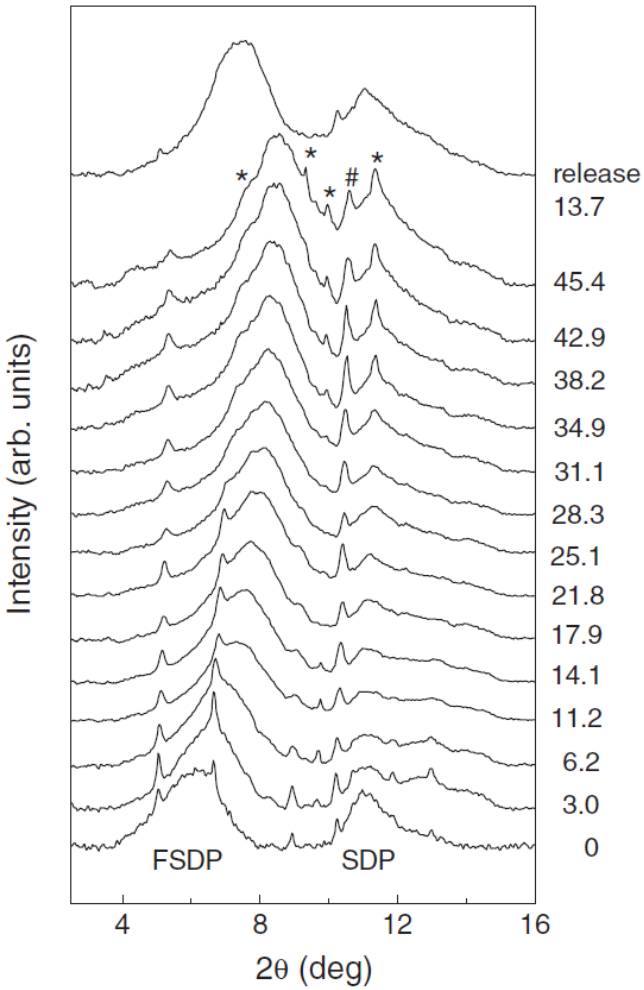


Harris, *Philos. Mag. 2004*

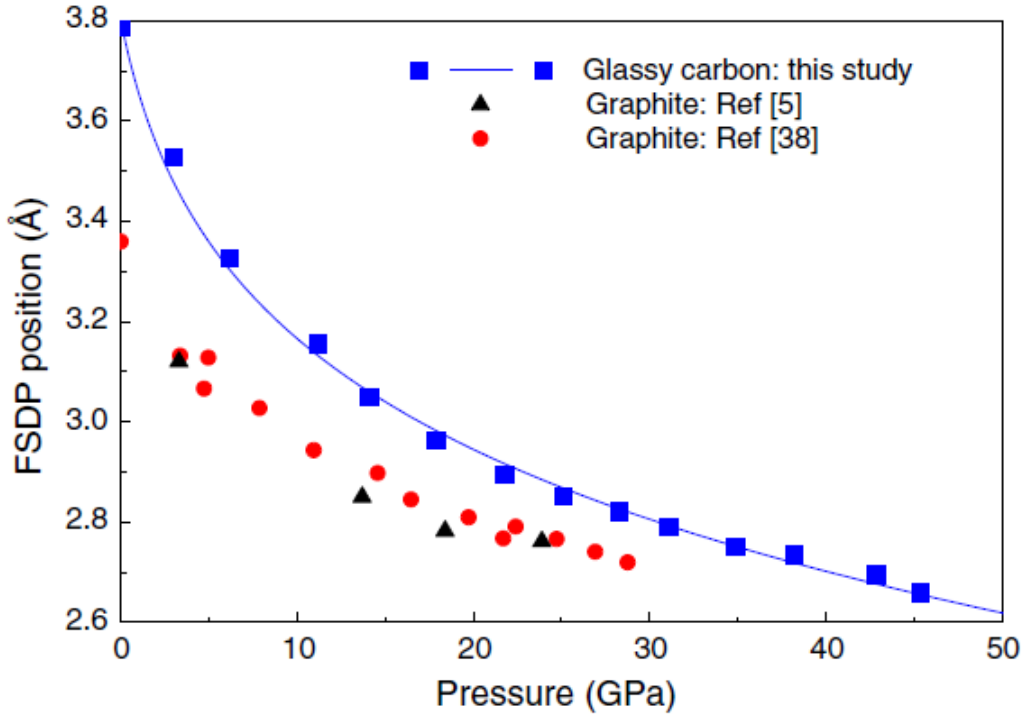
Xray Raman spectroscopy of the carbon K-edge showing the transition of sp^2-^{sp3} bonding



Glassy carbon

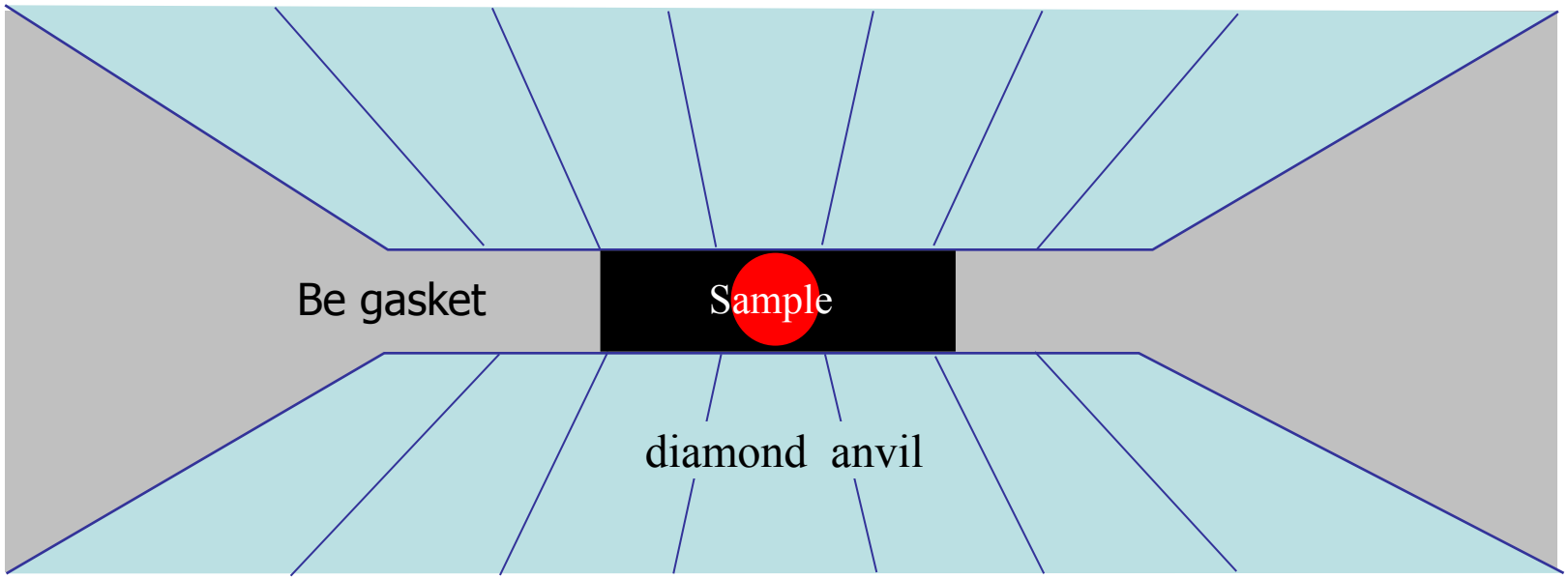


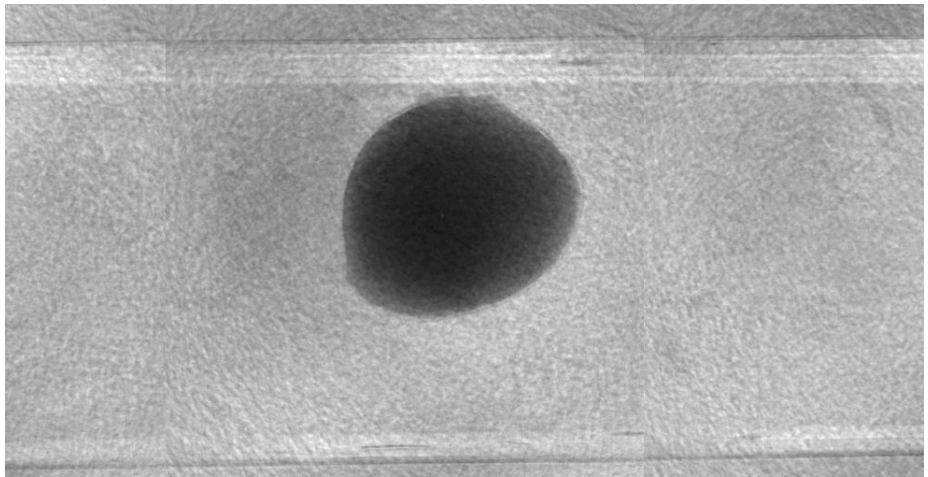
X-ray diffraction showing the compression of amorphous peaks



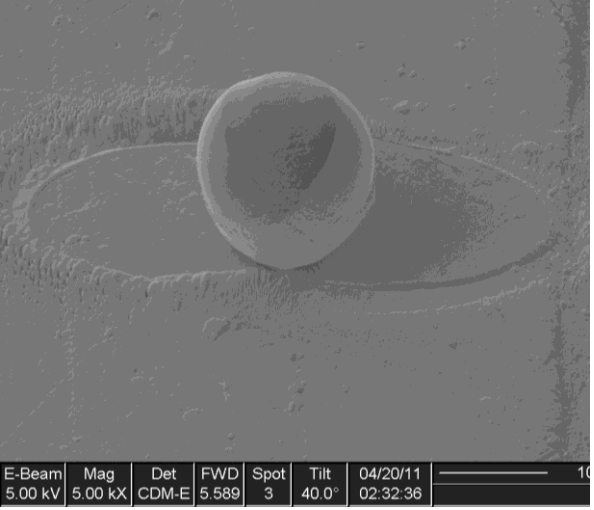
Y. Lin et al, *PRL* 2011

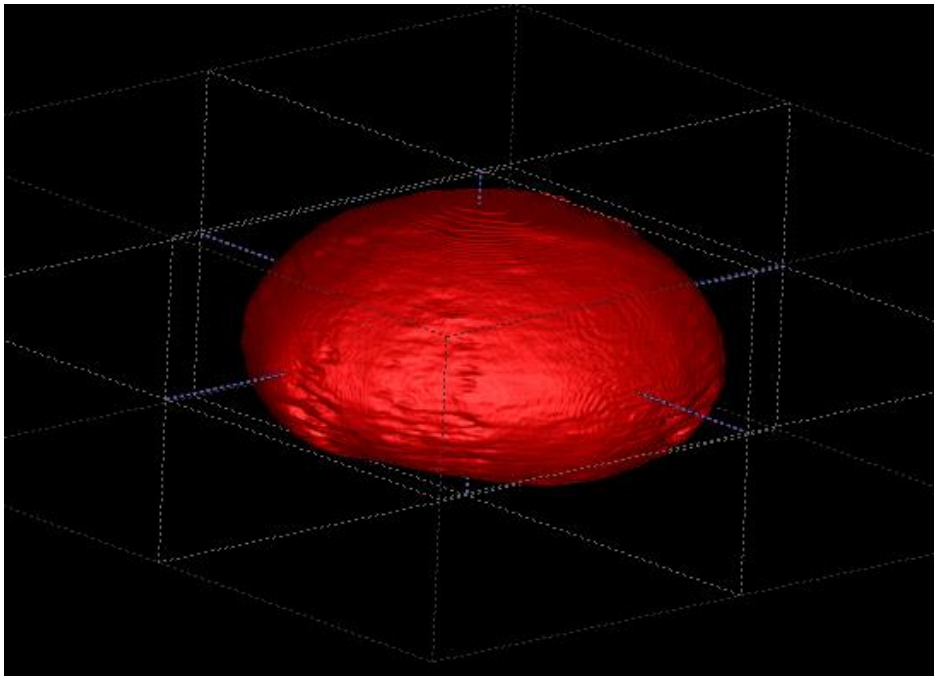
Transmission X-ray microscopy (TXM) showing the change of shape and volume



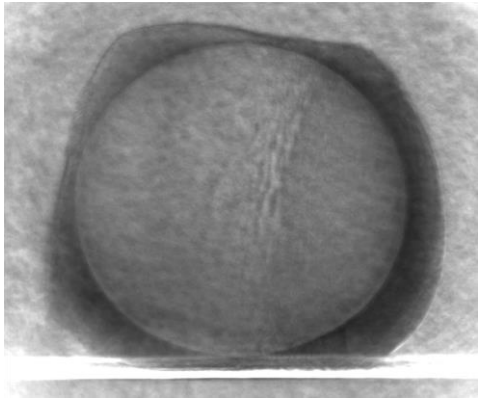


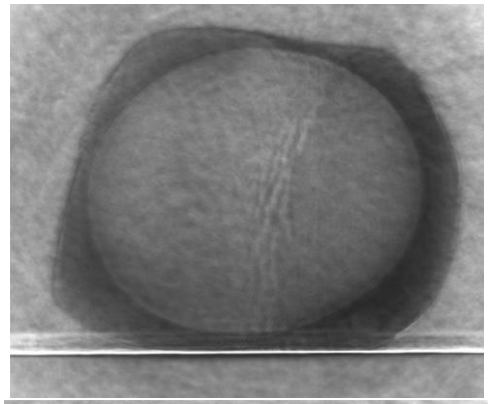
Glassy carbon



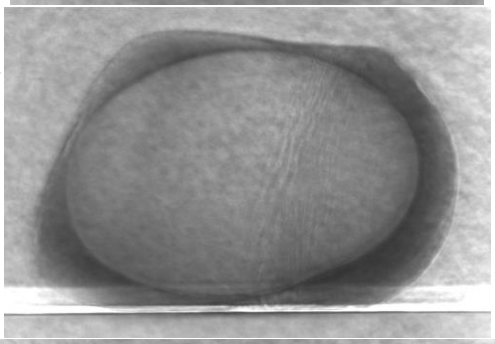


4.7 GPa

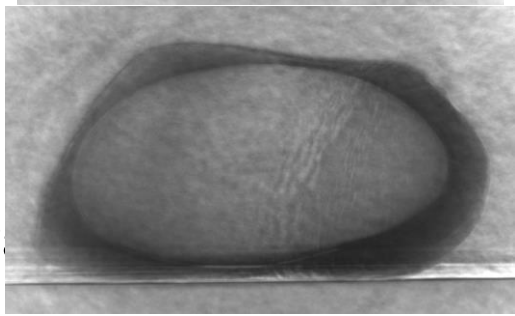




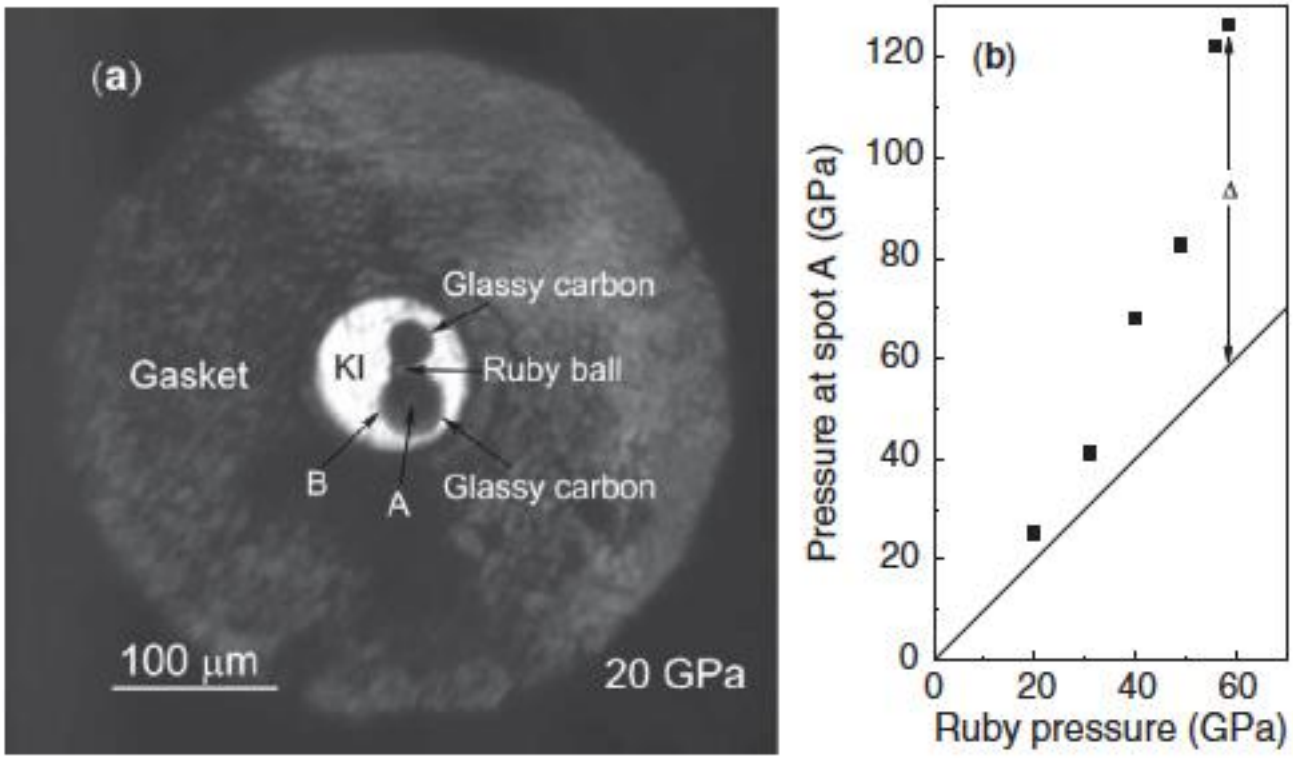
7.1 GPa



11.3 GP



Glassy carbon became superhard and act as the second-stage anvils to reach 120 GPa

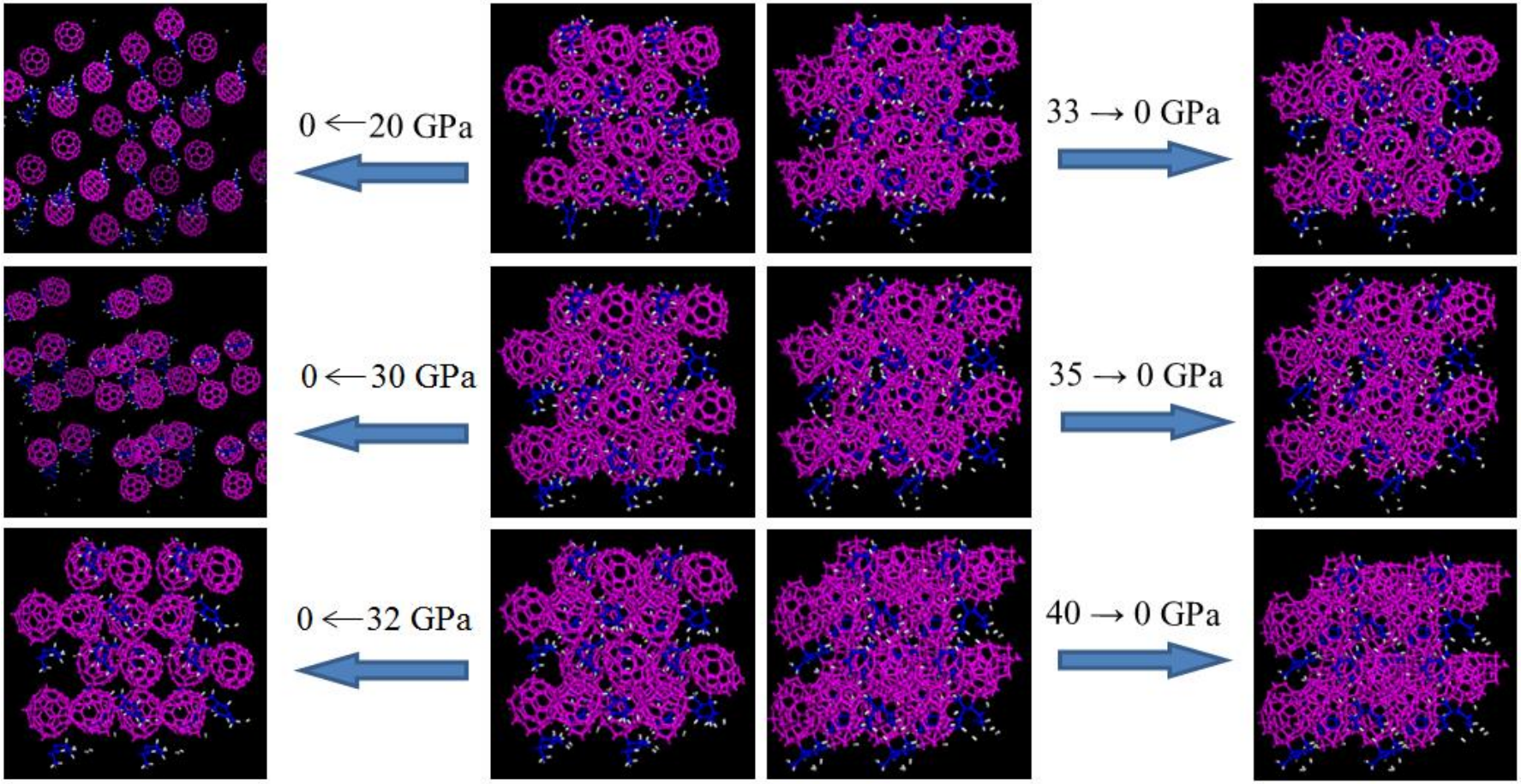


Long-Range Ordered Carbon Clusters: A Crystalline Material with Amorphous Building Blocks

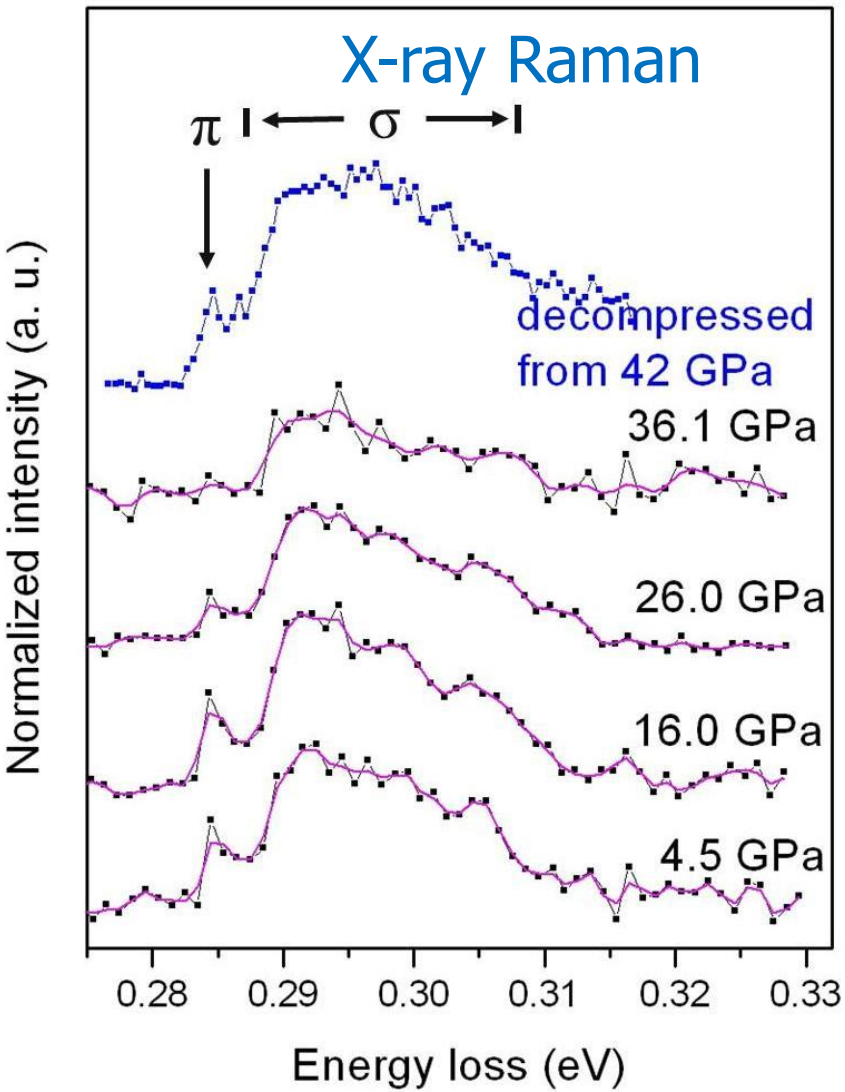
Lin Wang *et al.*

Science **337**, 825 (2012);

C_{60} & m-xylene solvent: Quantum Molecular Dynamics Simulation

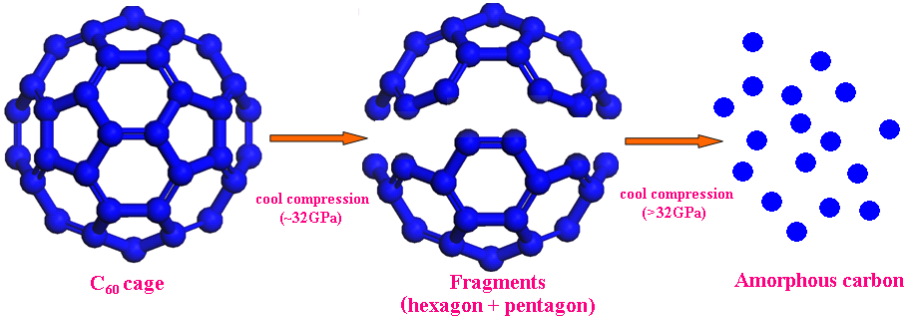


Pressure crushed individual C_{60} but preserved the arrangement



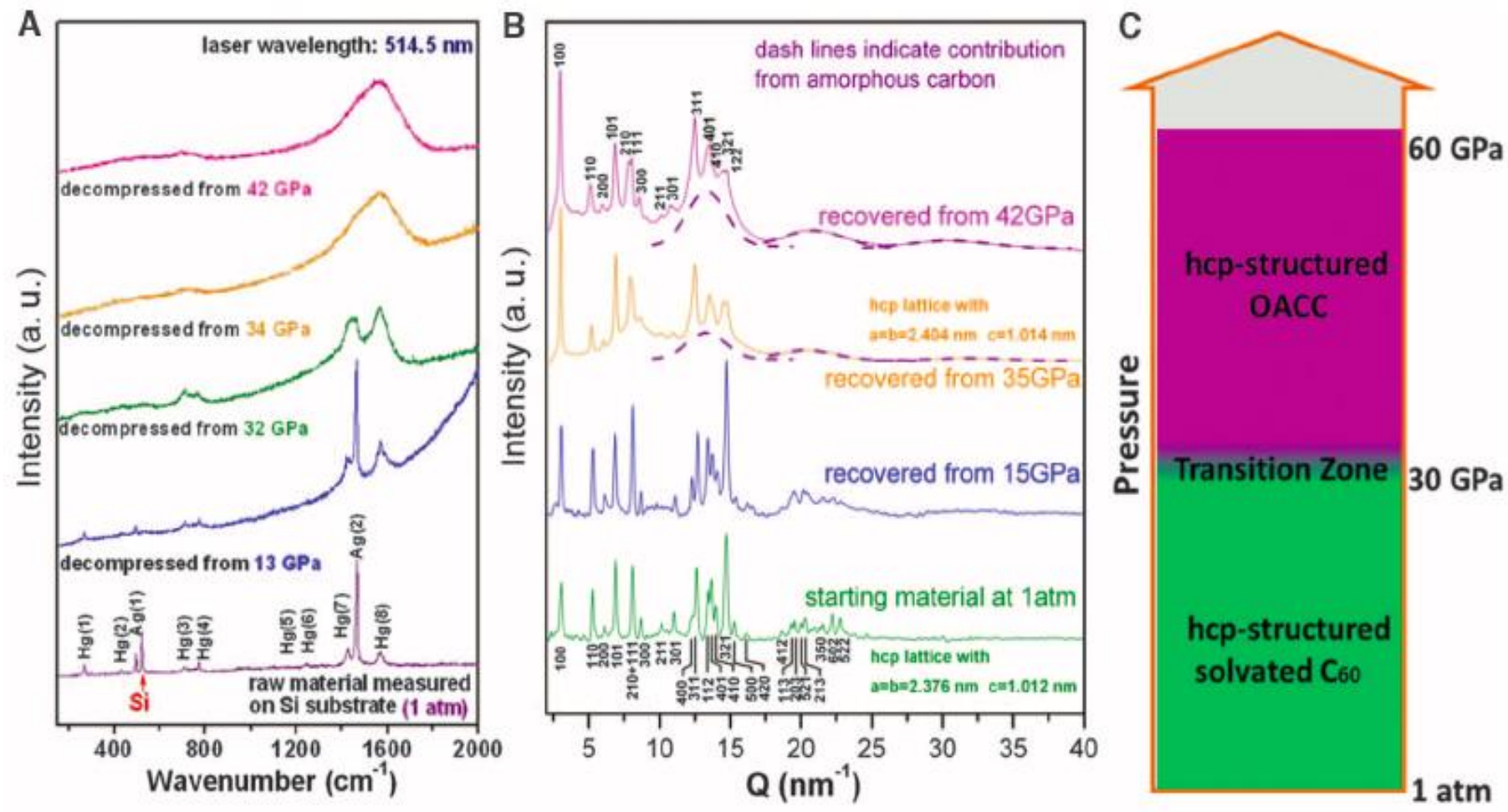
Xray Raman spectroscopy
of the carbon K-edge showing
the transition of sp^2-^{sp3} bonding

Optical Raman spectroscopy
showing the crushed C60



Wang et al., *Science* ***337***, 825 2012

C_{60} solvate transforms into new carbon form at high pressure

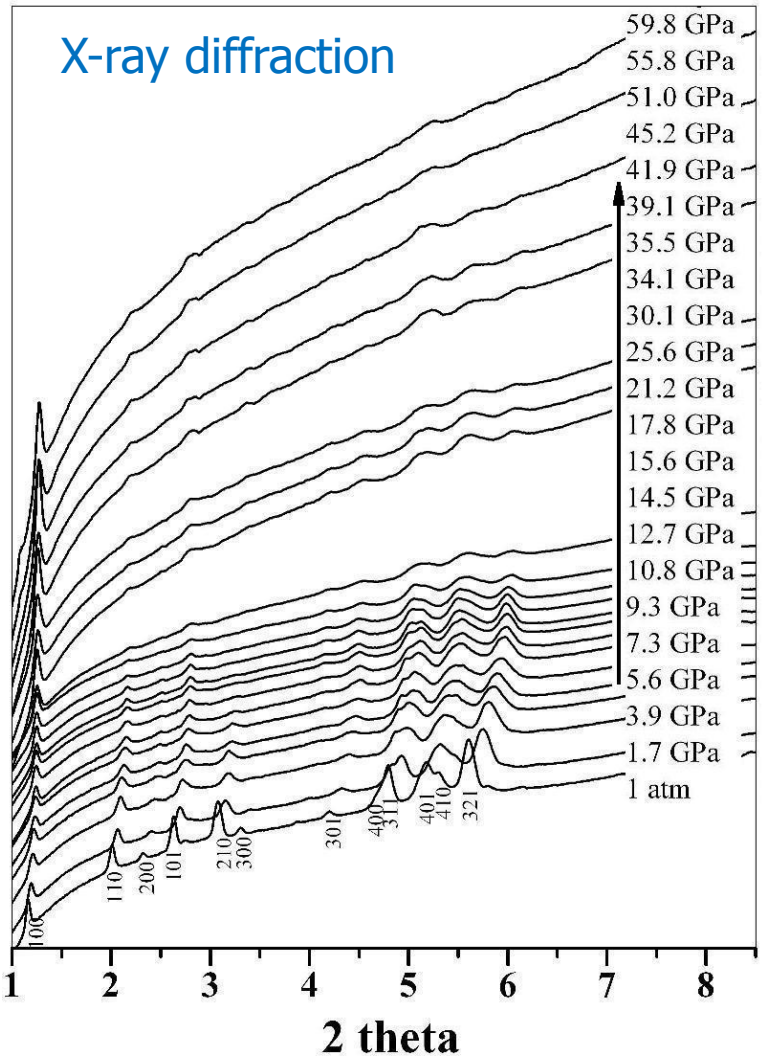


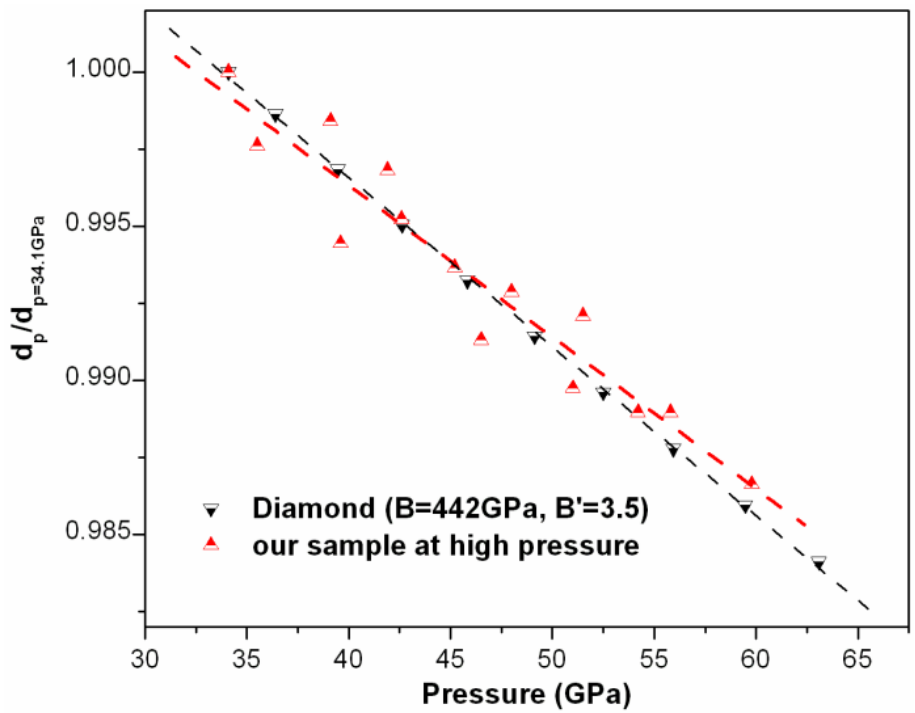
Optical Raman

X-Ray Diffraction

Wang et al., *Science* 2012

The new carbon form is more incompressible than diamond





Wang et al., *Science* 2012

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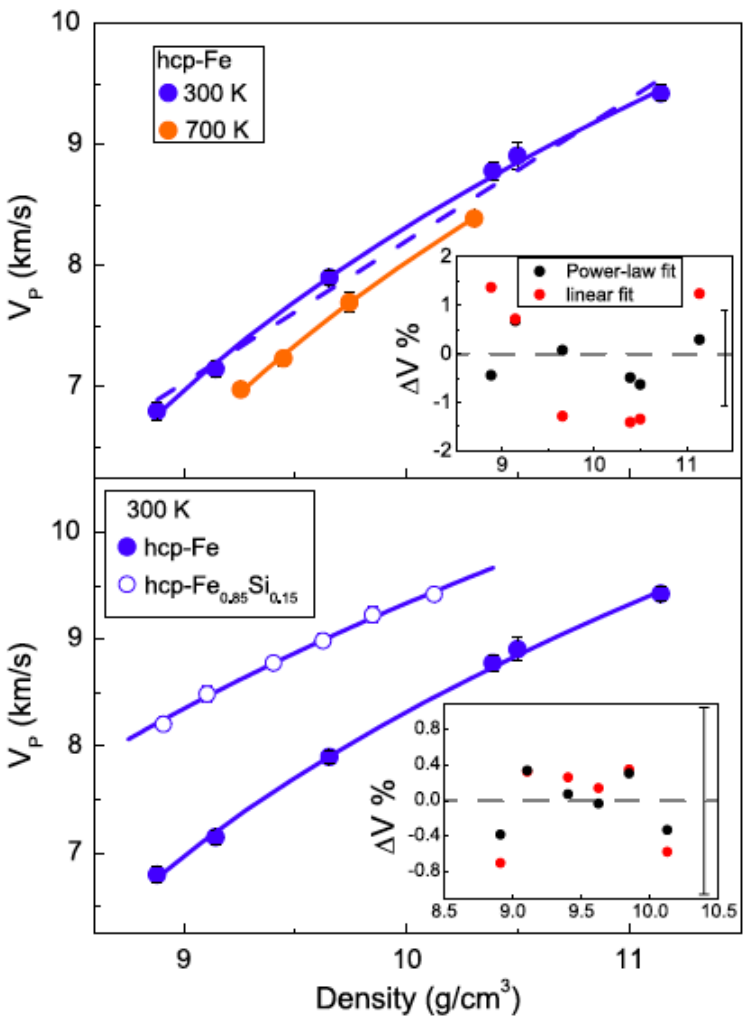
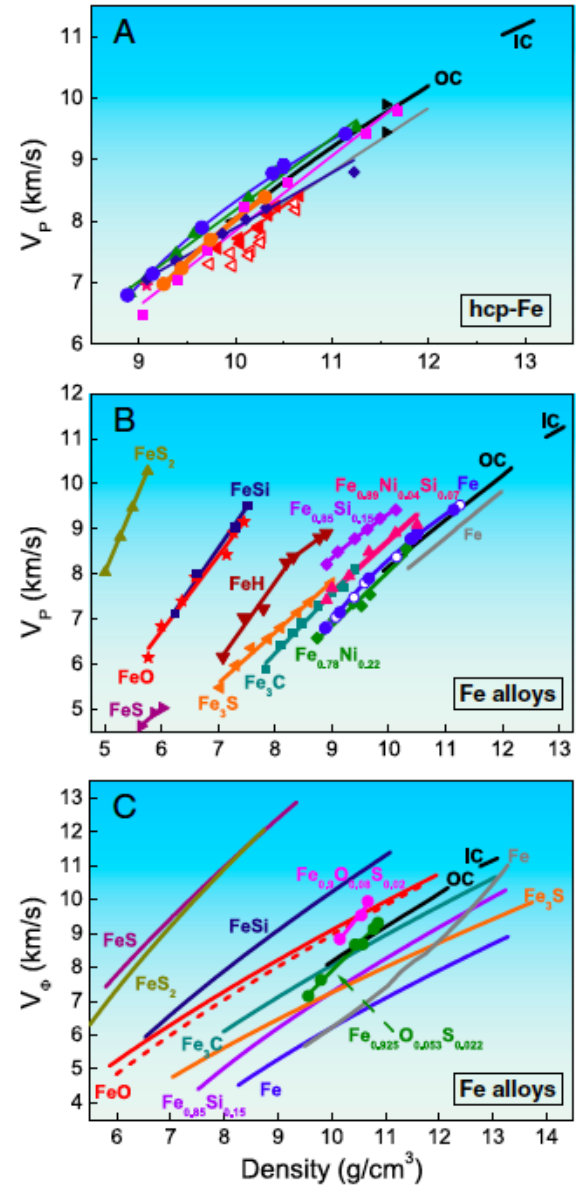
Sound velocities of Fe and Fe-Si alloy in the Earth's core

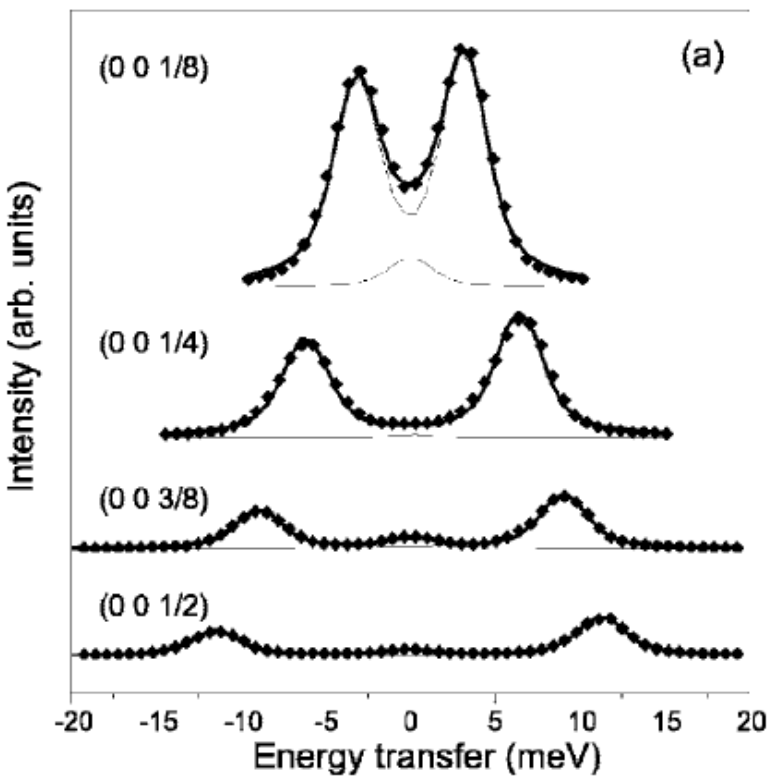
Zhu Mao[a,1], Jung-Fu Lin[a], Jin Liu[a], Ahmet Alatas[b], Lili Gao[b], Jiyong Zhao[b], and Ho-Kwang Mao[a]

PNAS | June 26, 2012 | vol. 109 | no. 26 | 10239–10244

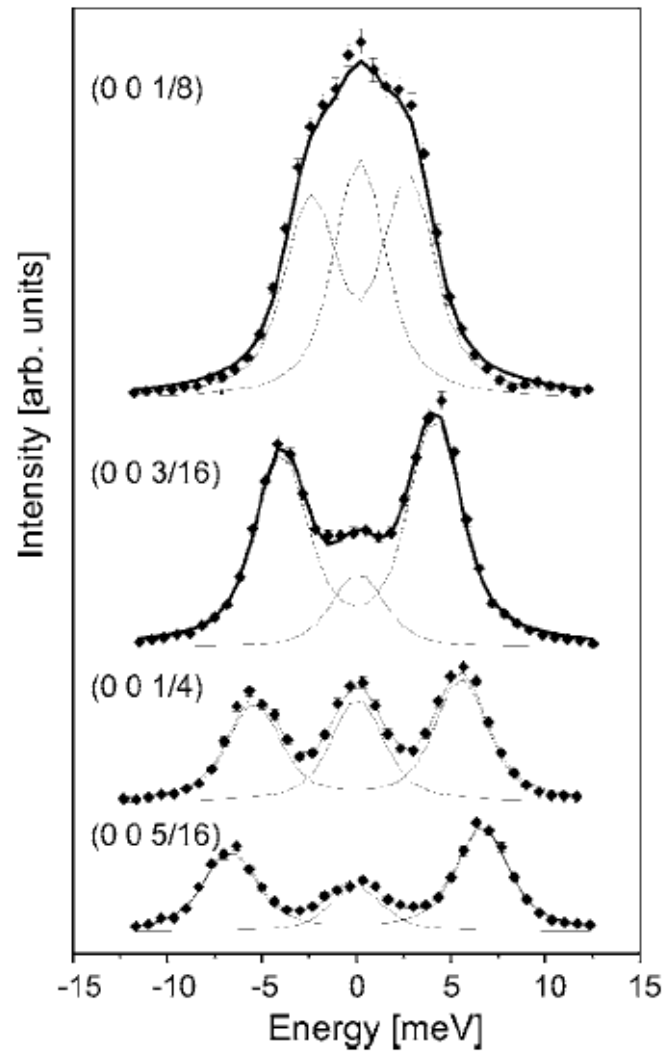




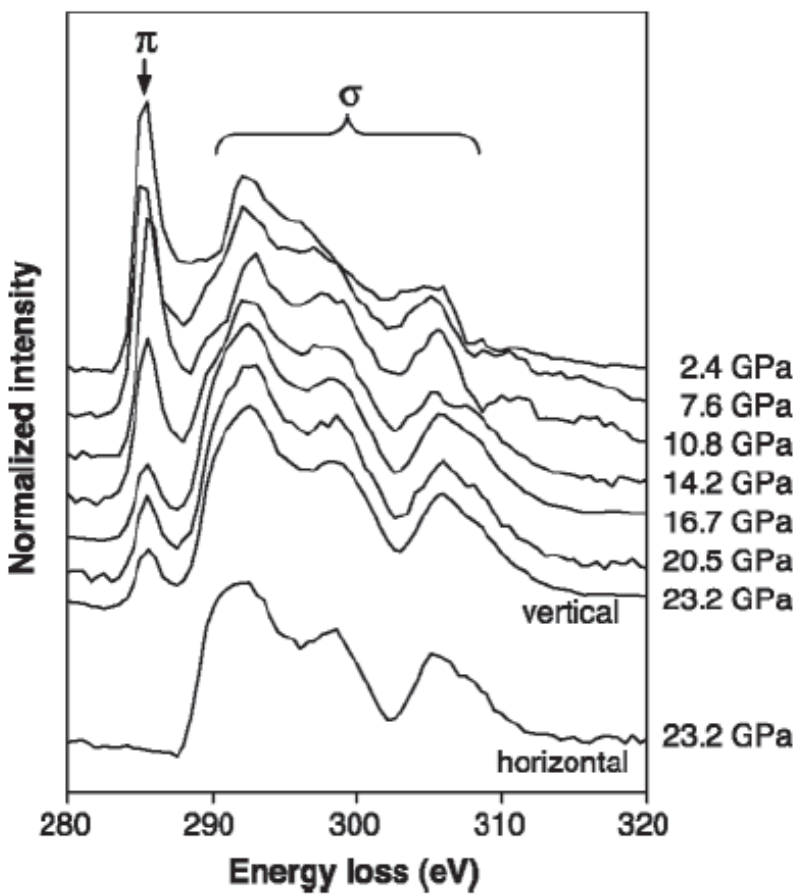
Elasticity of single-crystal graphite
Bosak & Krisch, PRB (2007)



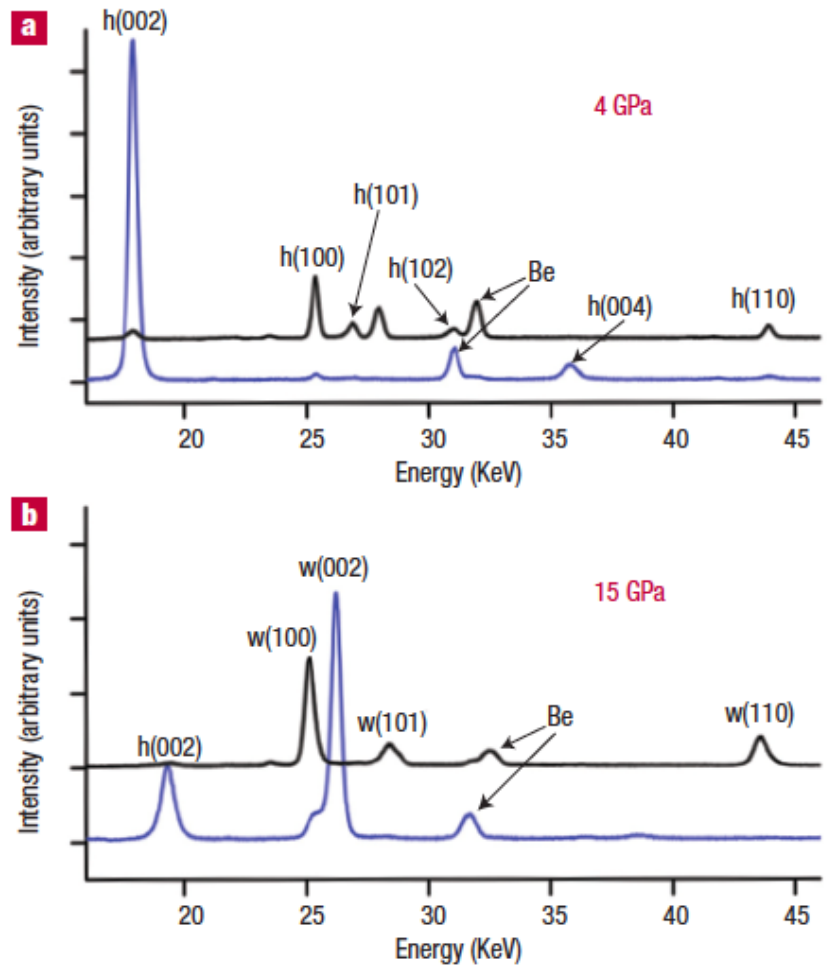
Elasticity of single-crystal hBN
Bosak & Krisch, PRB (2006)



Pressure-induced sp2-sp3 transition in graphite, Mao et al, Science (2003)



Pressure-induced sp2-sp3 transition in hBN, Meng et al, Nature Mat. (2004)



Early high-pressure experiments

1. Dispersion of hydrogen vibron.
2. Single crystal graphite-diamond transition.
3. Amorphous and nano carbon
4. high-*P* melts of Li and Na .
5. Phonon and elasticity of high *P*-*T* Fe.