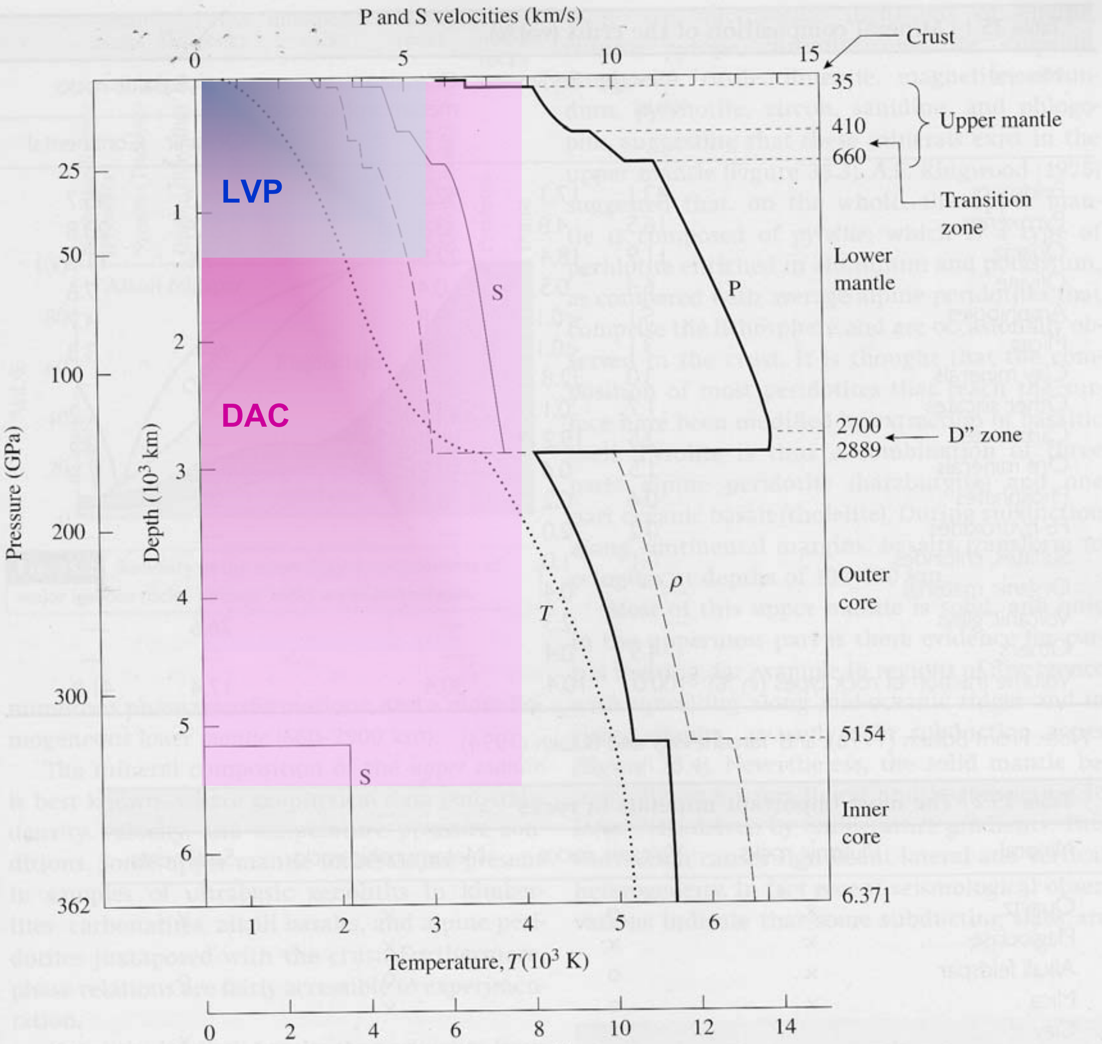
New developments in diamond anvil cell facilities at GSECARS

Vitali Prakapenka, Mark Rivers and Steve Sutton

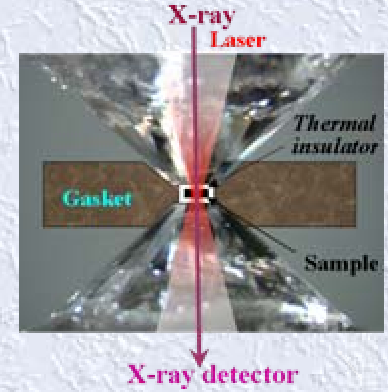
CARS, University of Chicago, Chicago, Illinois 60637, USA

http://cars9.uchicago.edu/gsecars/index.html

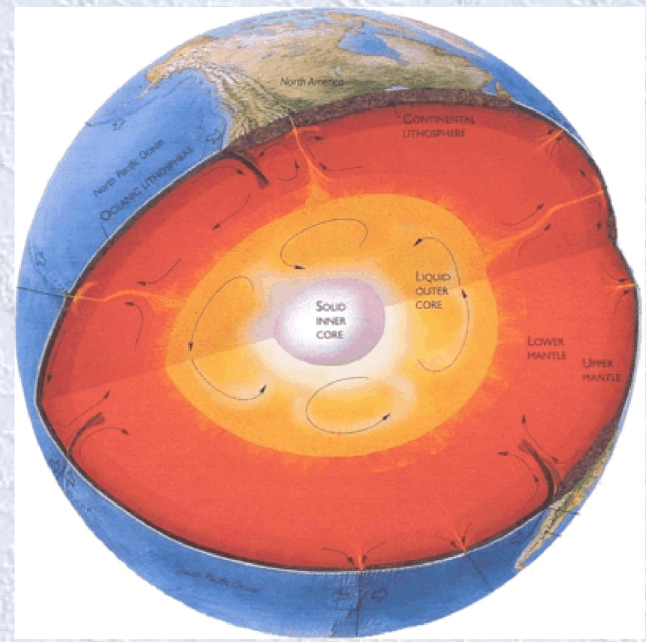
Significance

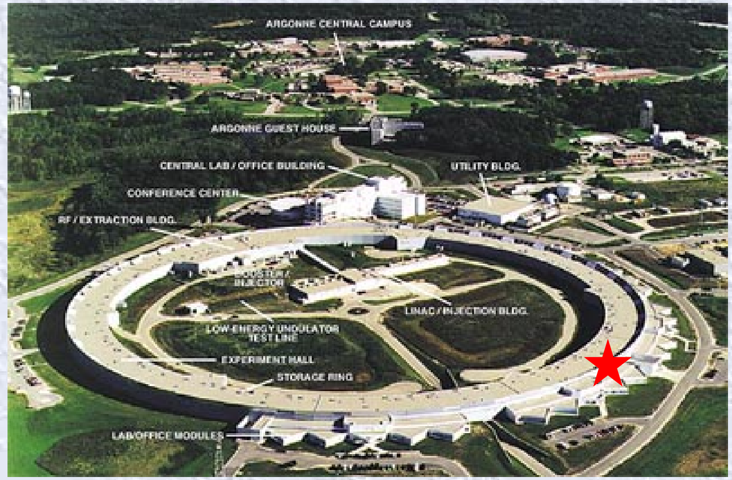


R.Wenk, 2005



P=F/S





GeoSoilEnviroCARS Sector 13

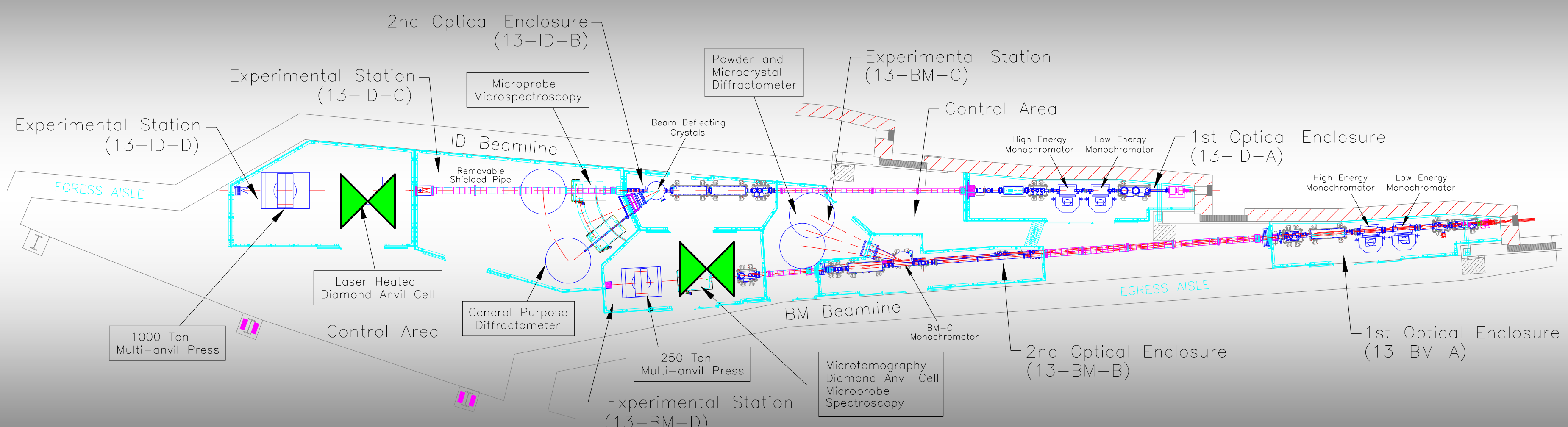
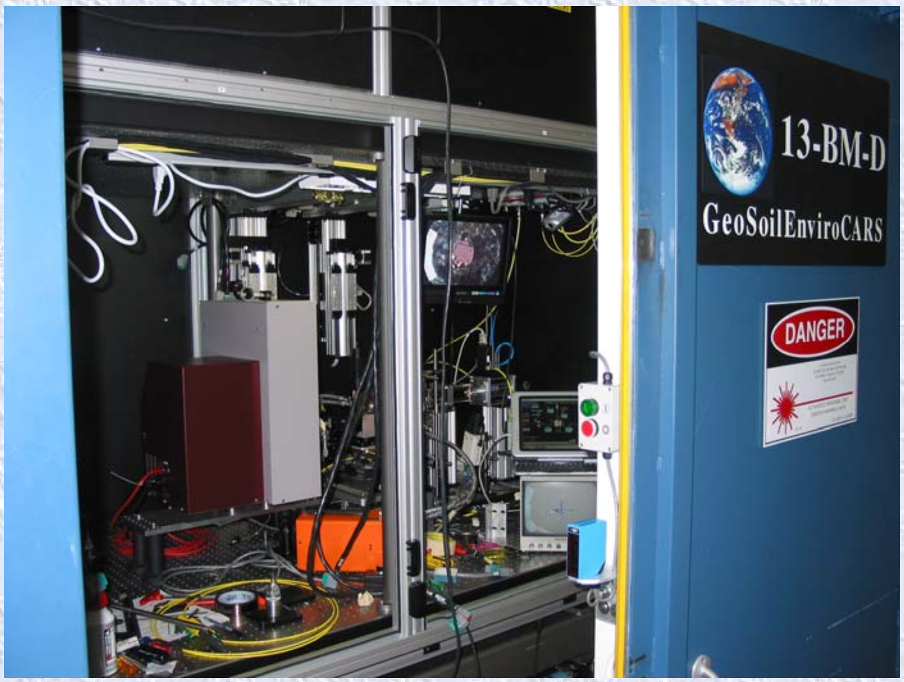
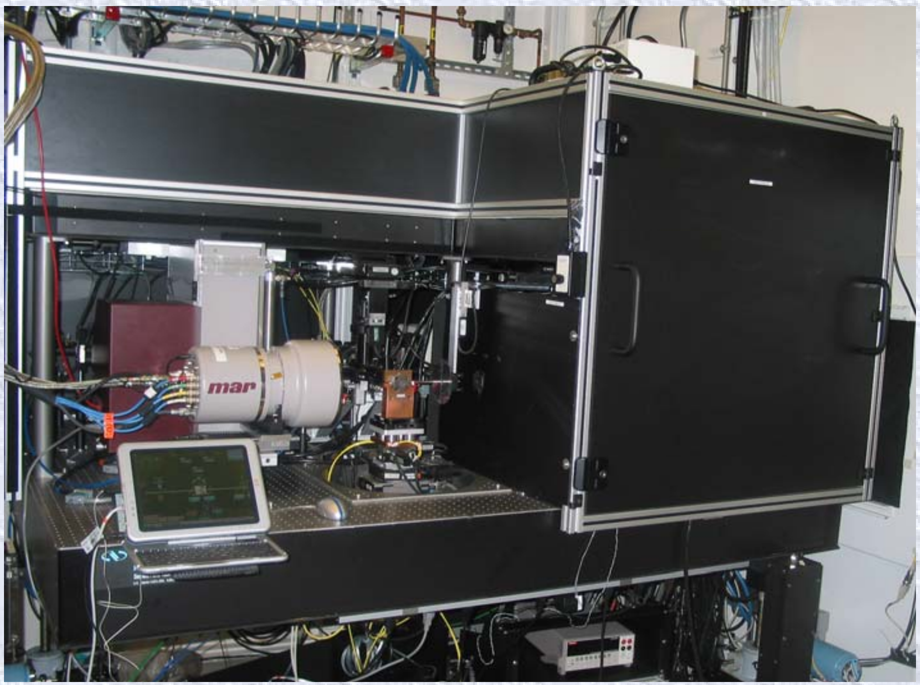


Figure C.1-1

13-BMD, GSECARS



13-IDD, GSECARS



	BM-D Specs:	ID-D Specs:
Beam size (μm at FWHM)	6 (H) x 15(V)	5 (H) x 5(V)
Energy range	5-75 keV	5-45keV
Flux (photon/s) at the sample position at 37 keV	5×10^8	5×10^{11}
X-ray detectors	MAR-IP, MAR-CCD	

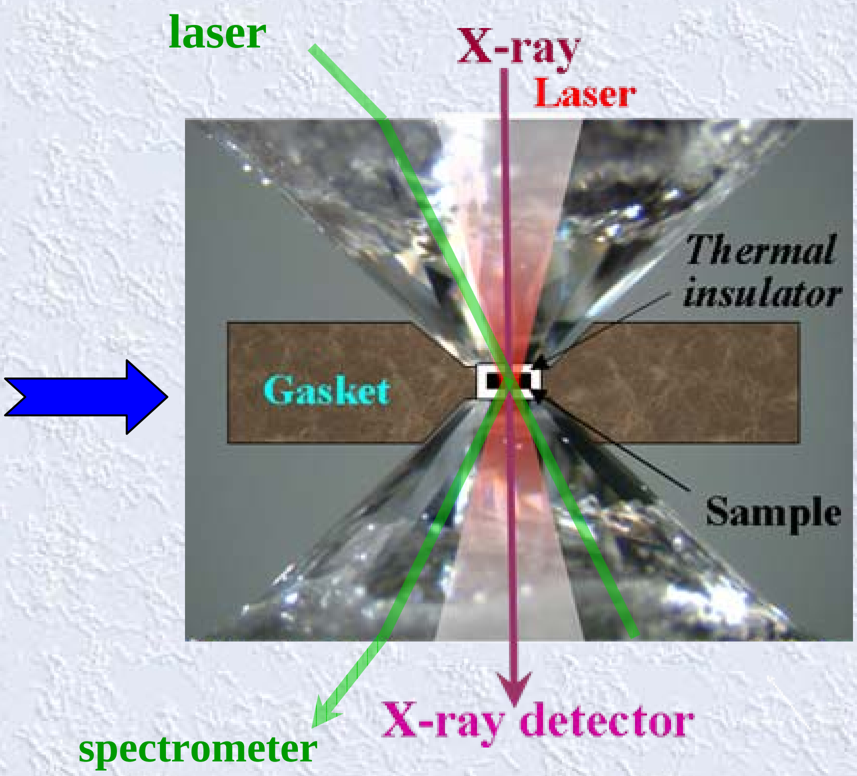
Diamond Anvil Cell Techniques at GSECARS

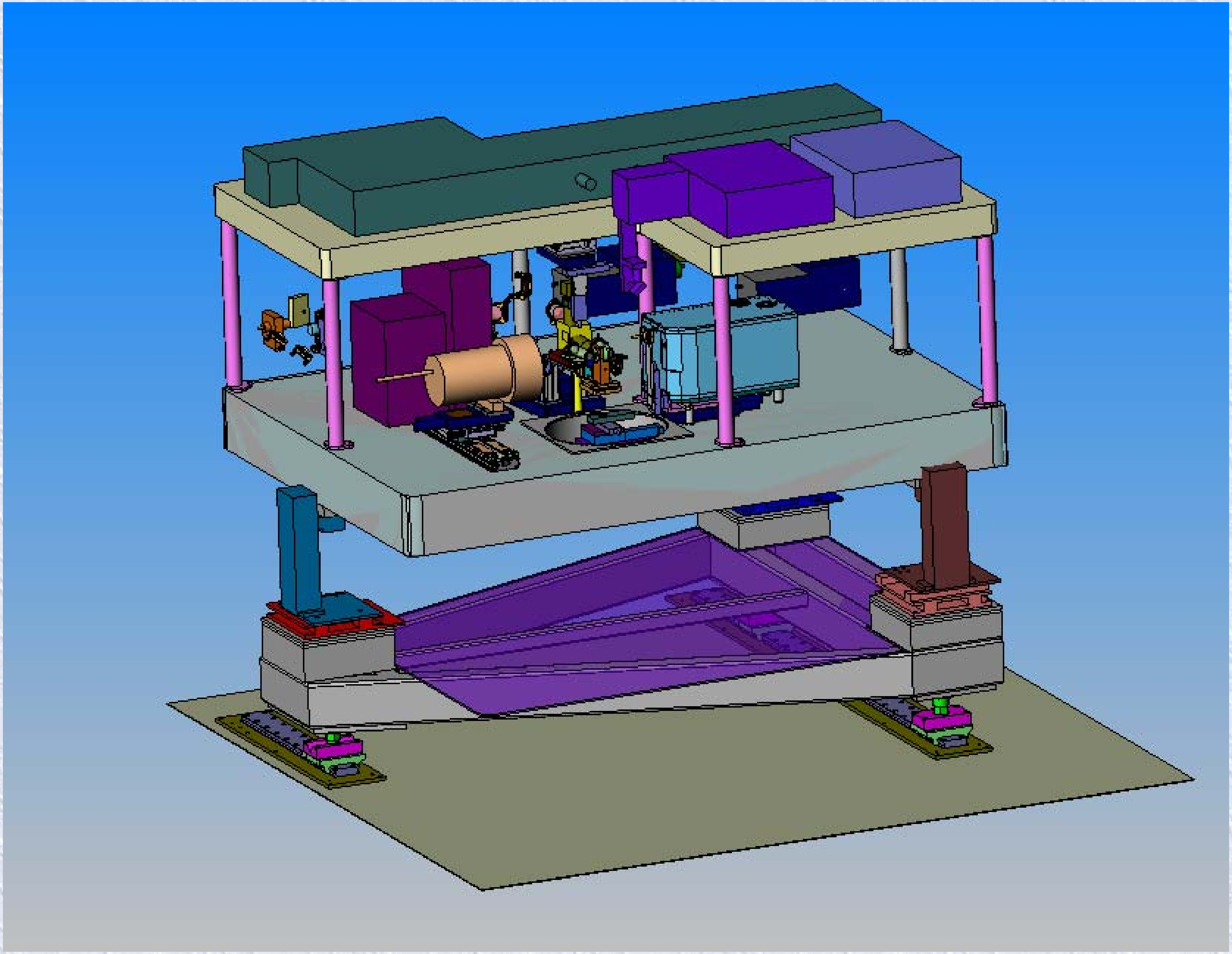
X-ray techniques

- *X-ray diffraction*
- *X-ray transmission scattering*
- *X-ray absorption spectroscopy*
- *X-ray emission spectroscopy*
- *X-ray Raman spectroscopy*
- *X-ray radiography*
- *EXAFS*

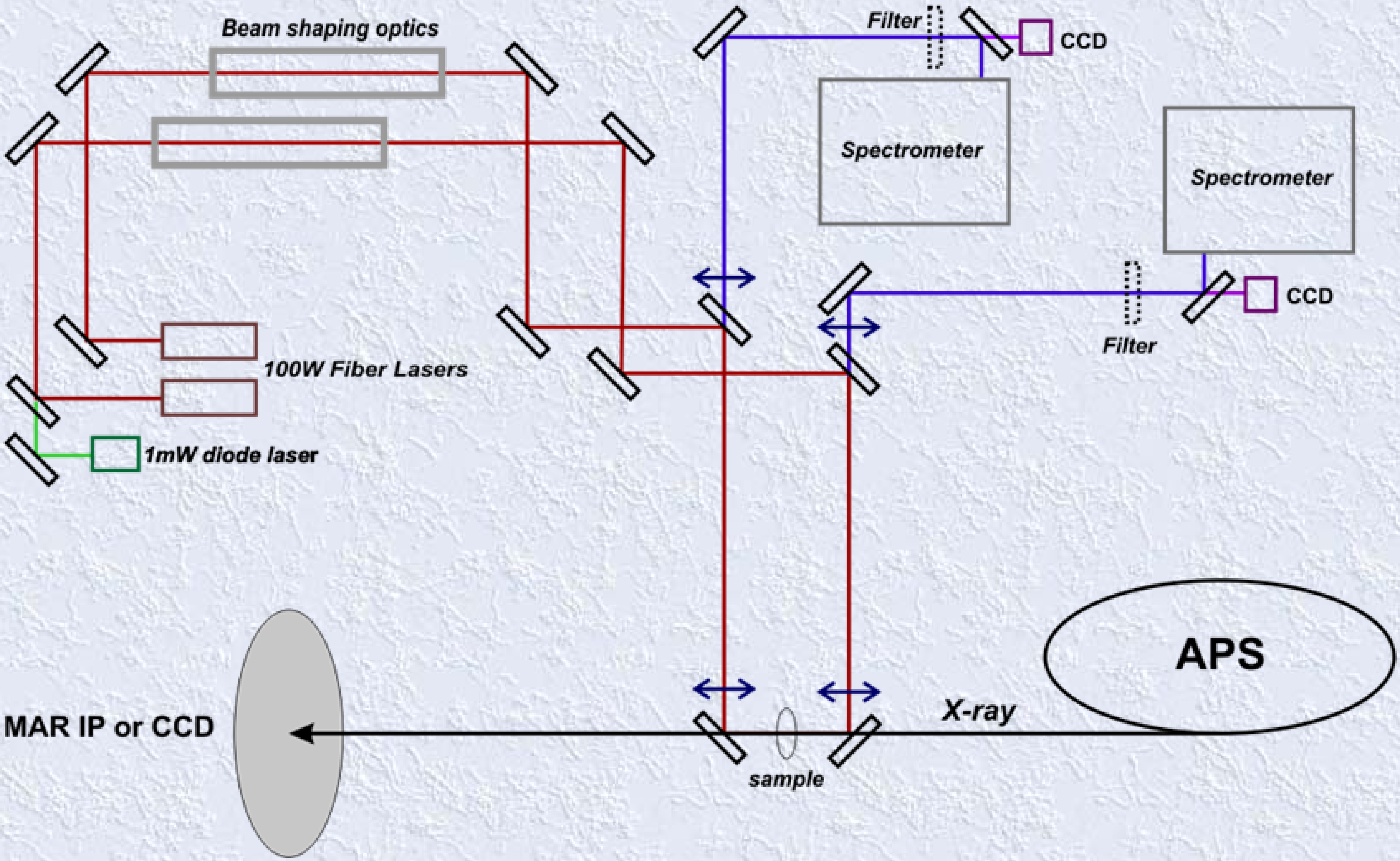
VIS and IR techniques

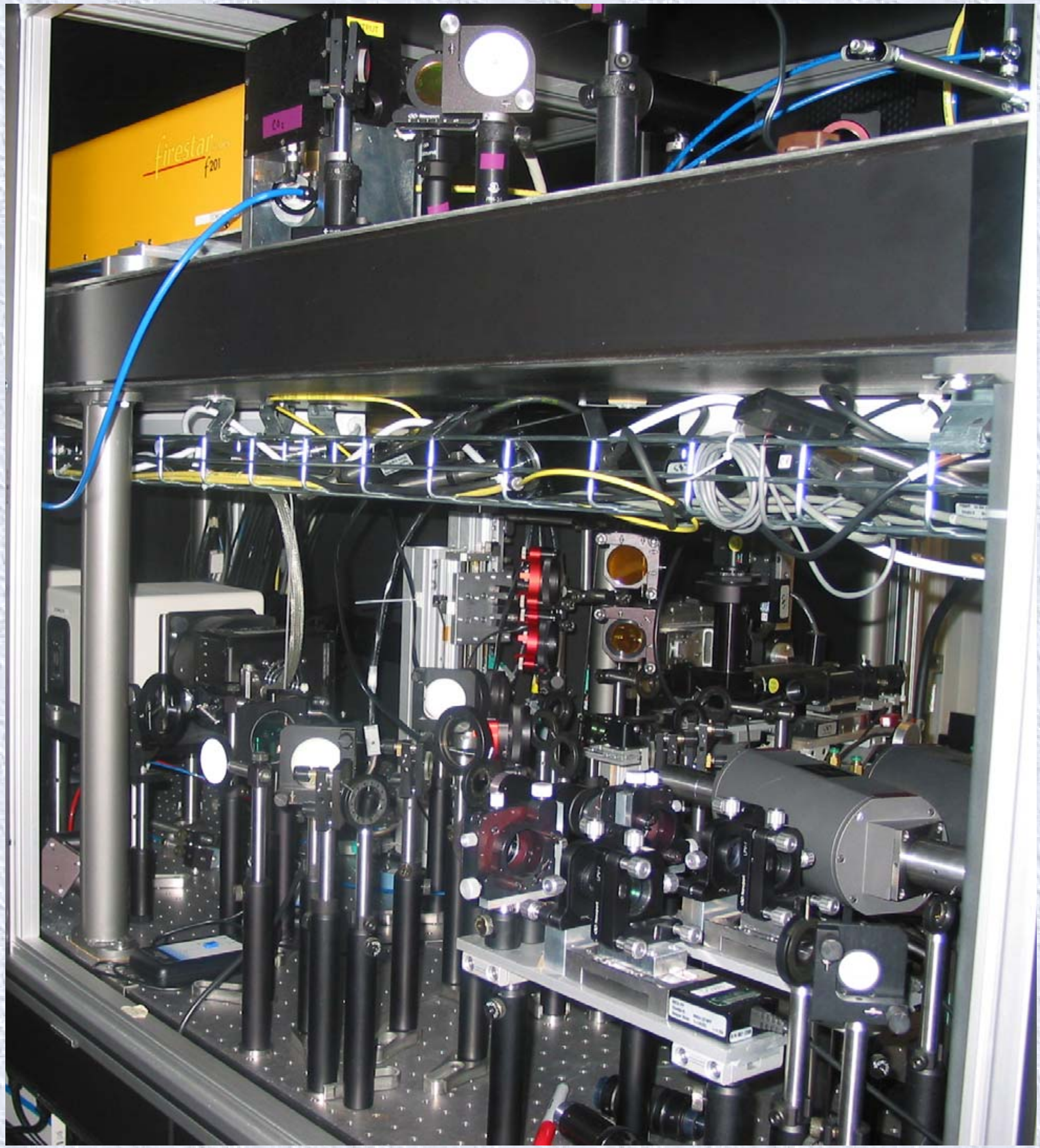
- *Laser heating*
- *Brillouin spectroscopy*
- *Raman spectroscopy*
- *Fluorescence spectroscopy*
- *Temperature measurements*

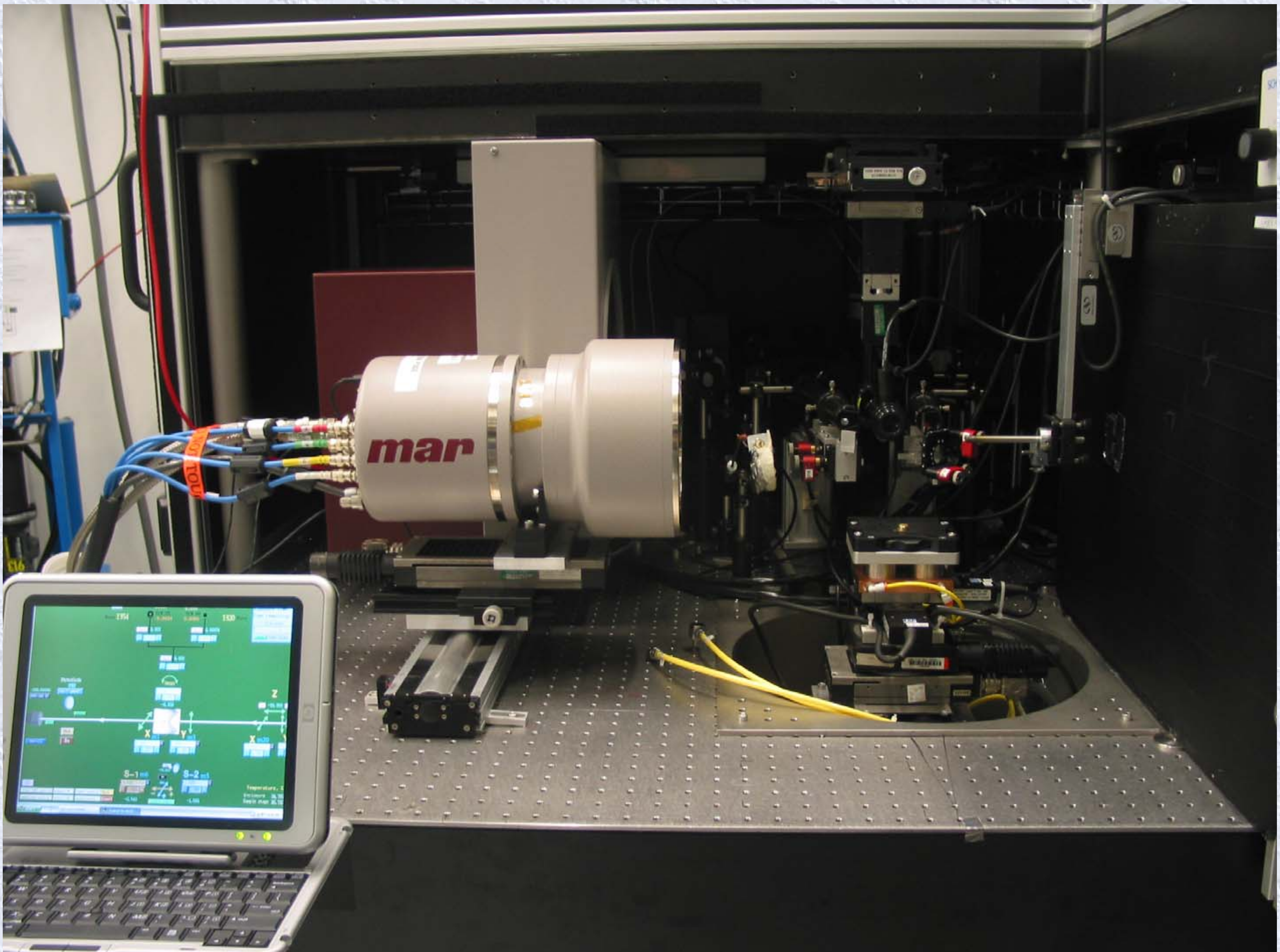


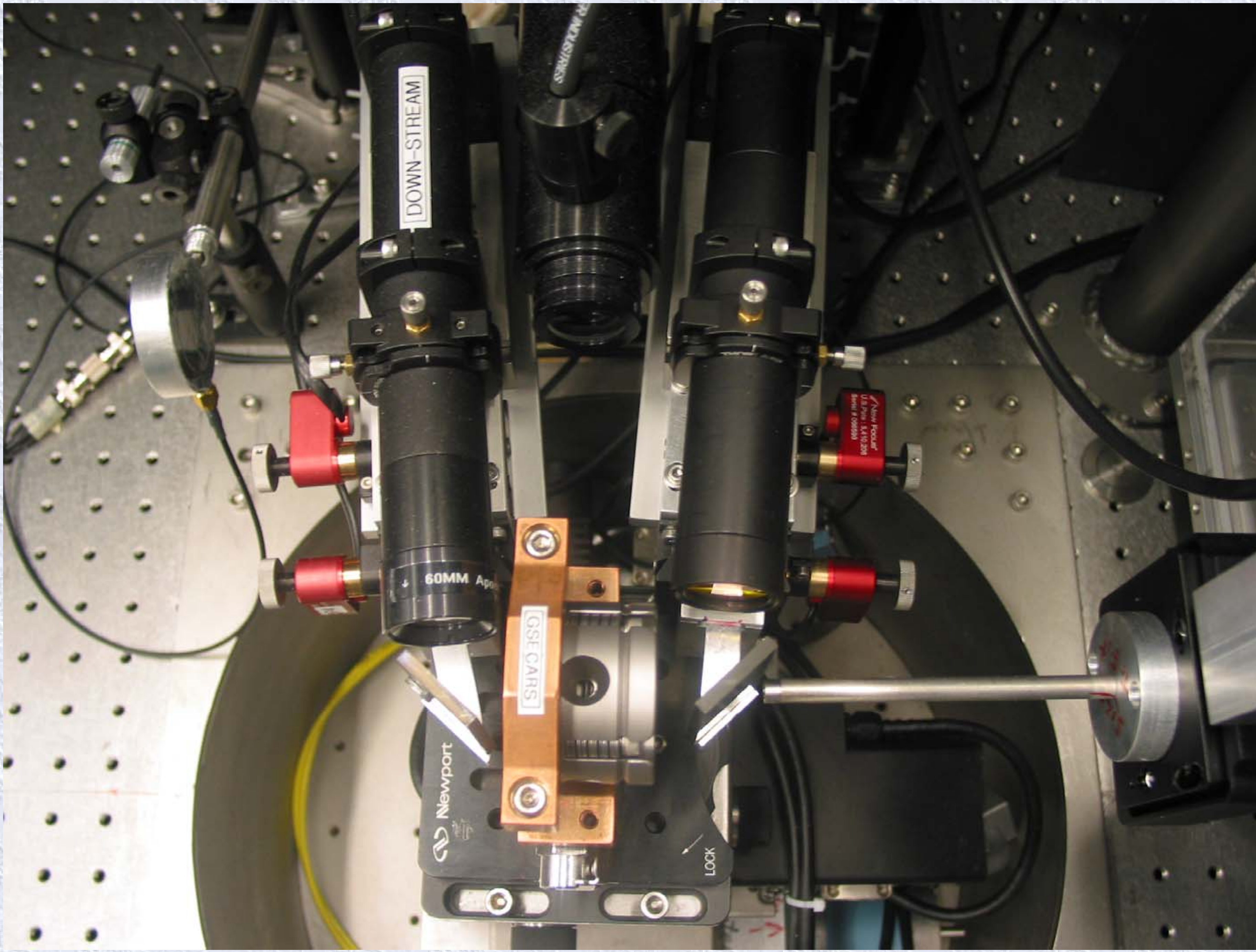


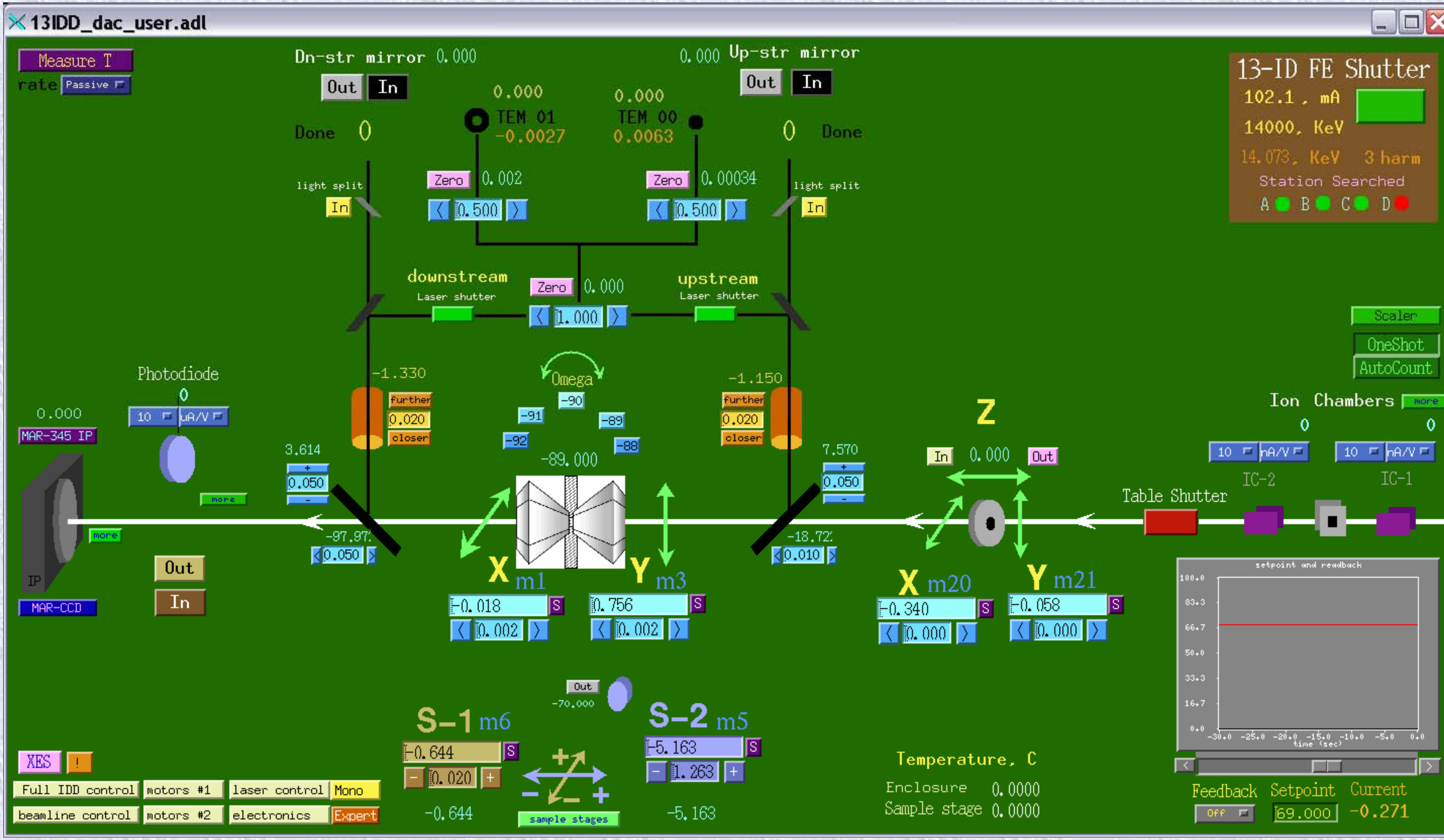
Double side laser heating at 13-IDD



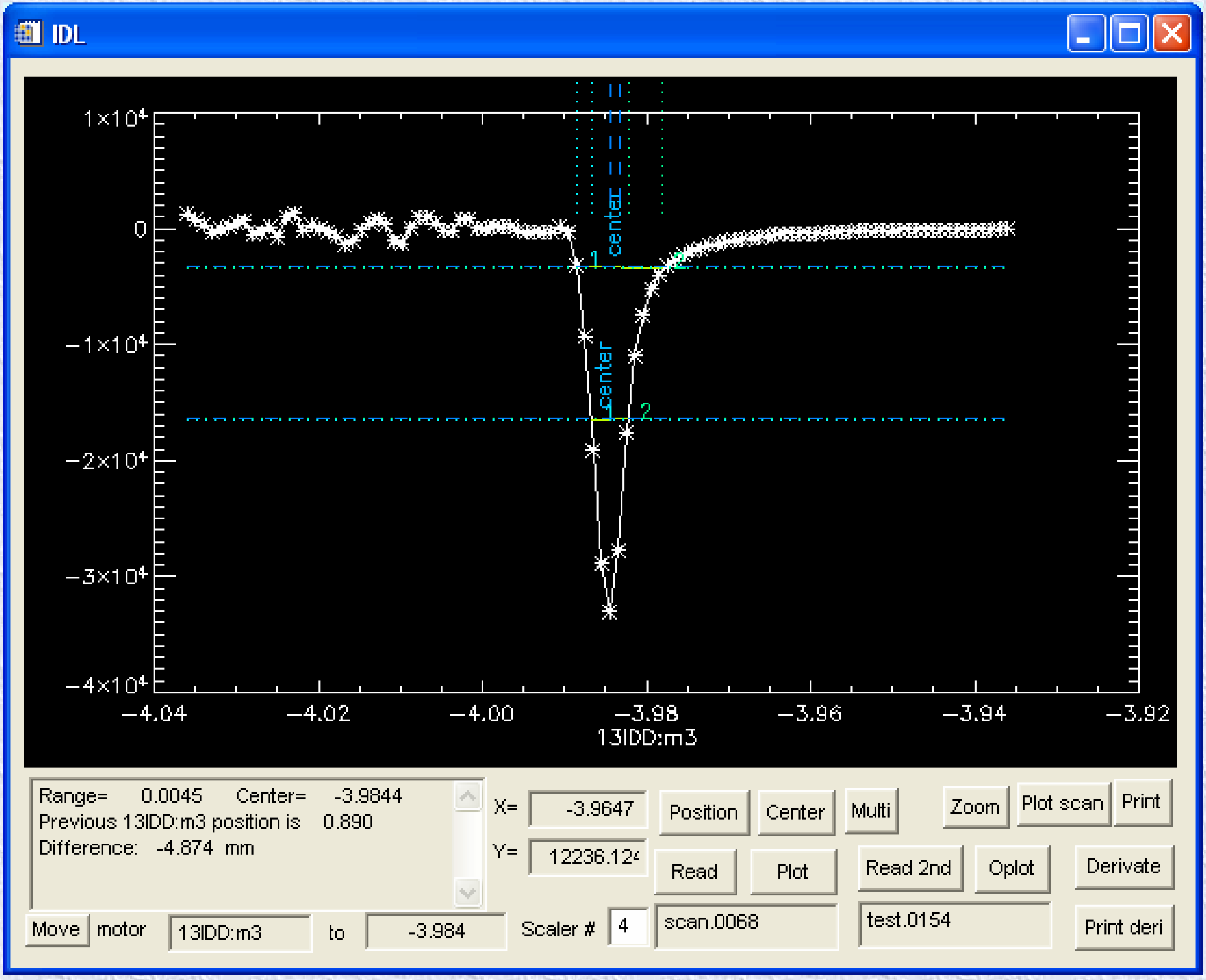

firestar
f201


mar


DOWN-STREAM
60MM
GSE CARS
Newport
LOCK



13IDD_dac_user.adl
Measure T
rate Passive
Dn-str mirror 0.000
Out In
Done 0
0.000 TEM 01 -0.0027
0.000 TEM 00 0.0063
0.000 Up-str mirror
Out In
0 Done
light split In
Zero 0.002
0.500
Zero 0.00034
0.500
light split In
downstream Laser shutter
Zero 0.000
1.000
upstream Laser shutter
13-ID FE Shutter
102.1 , mA
14000. KeV
14.073, KeV 3 harm
Station Searched
A B C D
Scaler
OneShot
AutoCount
Ion Chambers more
Photodiode
0
10 uA/V
0.000
MAR-345 IP
more
-1.330
further 0.020 closer
Omega
-90 -91 -89 -92 -88
-89.000
-1.150
further 0.020 closer
3.614
0.050
-97.97
0.050
7.570
0.050
-18.72
0.010
Z
In 0.000 Out
0 0
10 nA/V 10 nA/V
IC-2 IC-1
Table Shutter
IP
MAR-CCD
Out
In
X m1
-0.018 S
0.002
Y m3
0.756 S
0.002
X m20
-0.340 S
0.000
Y m21
-0.058 S
0.000
setpoint and readback
Out
-70.000
S-1 m6
-0.644 S
0.020
-0.644
S-2 m5
-5.163 S
1.263
-5.163
sample stages
Temperature, C
Enclosure 0.0000
Sample stage 0.0000
Feedback Setpoint Current
Off 69.000 -0.271
XES !
Full IDD control motors #1 laser control Mono
beamline control motors #2 electronics Expert


IDL
1×10⁴
0
−1×10⁴
−2×10⁴
−3×10⁴
−4×10⁴
−4.04
−4.02
−4.00
−3.98
−3.96
−3.94
−3.92
13IDD:m3
center
center
Range= 0.0045 Center= -3.9844
Previous 13IDD:m3 position is 0.890
Difference: -4.874 mm
X= -3.9647
Y= 12236.124
Position
Center
Multi
Zoom
Plot scan
Print
Read
Plot
Read 2nd
Oplot
Derivate
Move
motor
13IDD:m3
to
-3.984
Scaler #
4
scan.0068
test.0154
Print deri

IPG's YLR-SM Series represents a break-through generation of diode pumped single mode CW Ytterbium fiber lasers of near infrared spectral range (~1070nm) with a unique combination of high power, ideal beam quality, fiber delivery, high wall-plug efficiency, compactness and reliability.

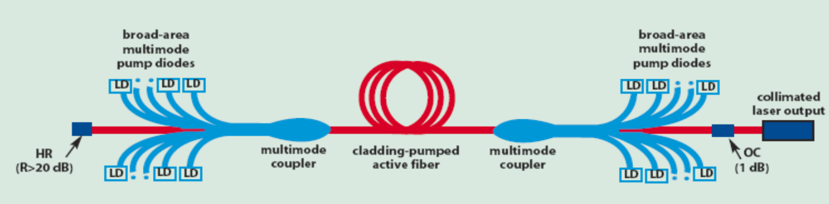
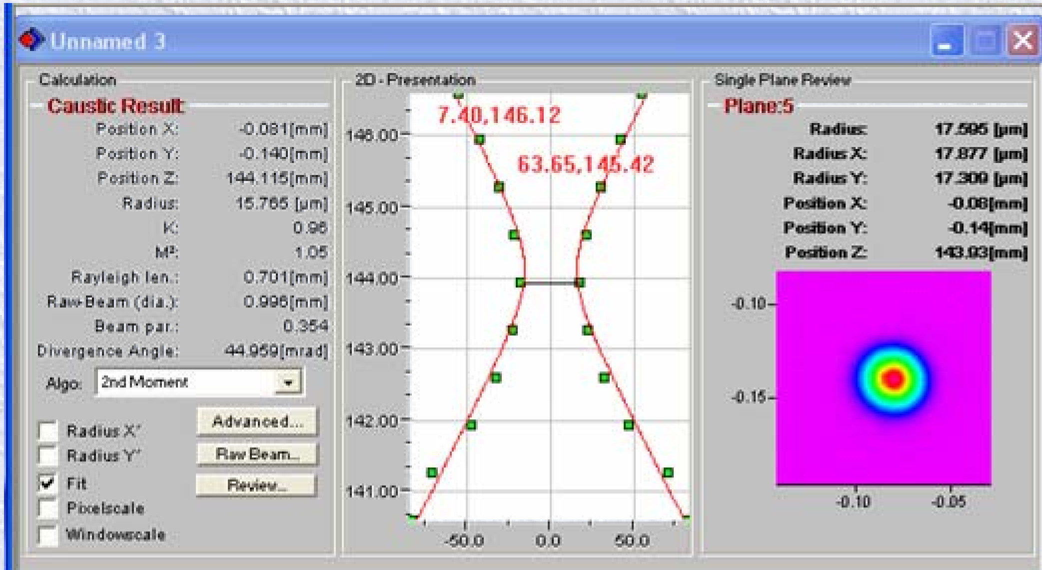
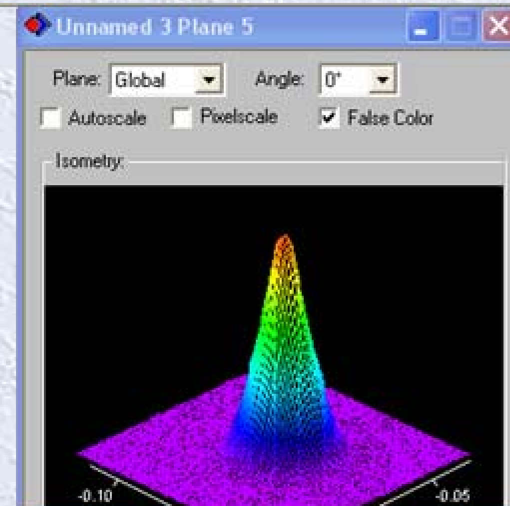
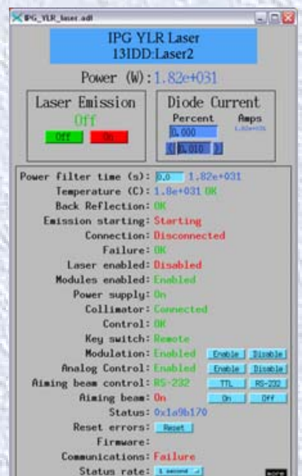
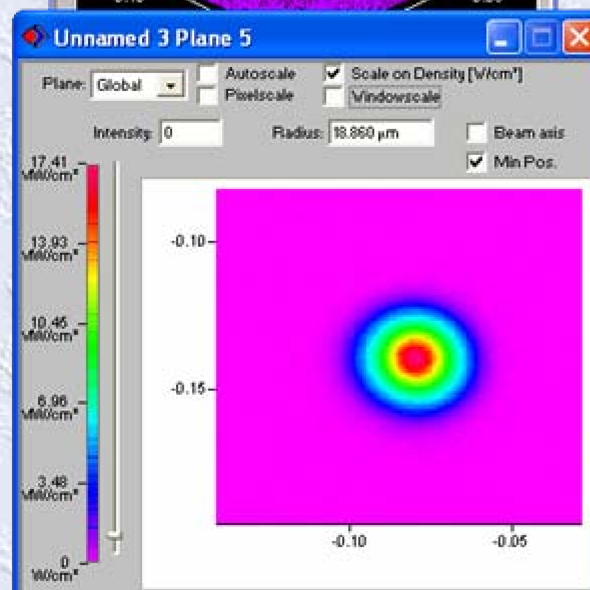


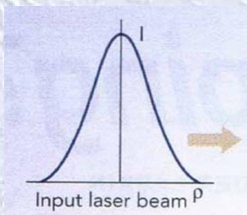
Figure 1. Schematic of a typical single-mode fiber laser utilizing single-emitter diodes.



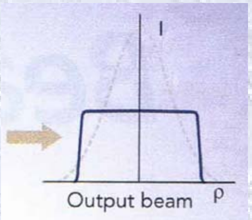






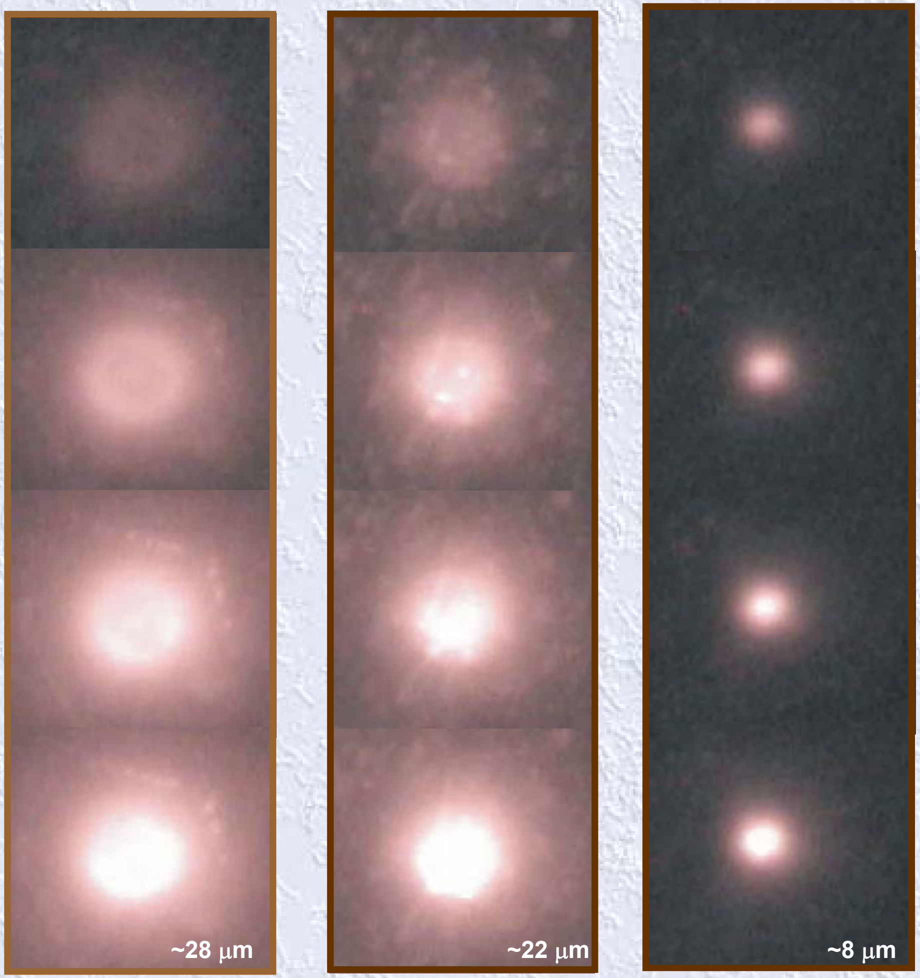


Beam shaping in focal plane

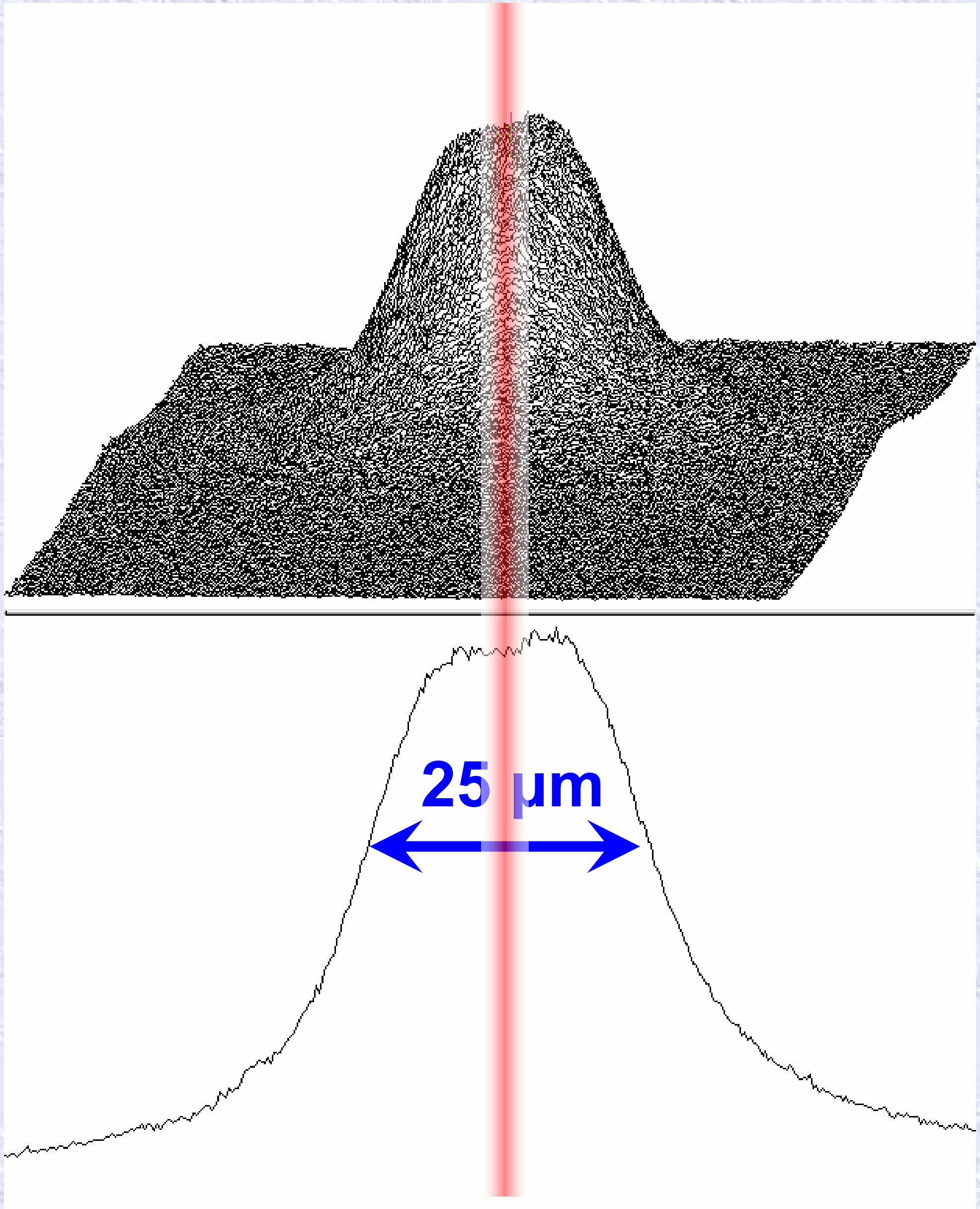


TEM_{01}

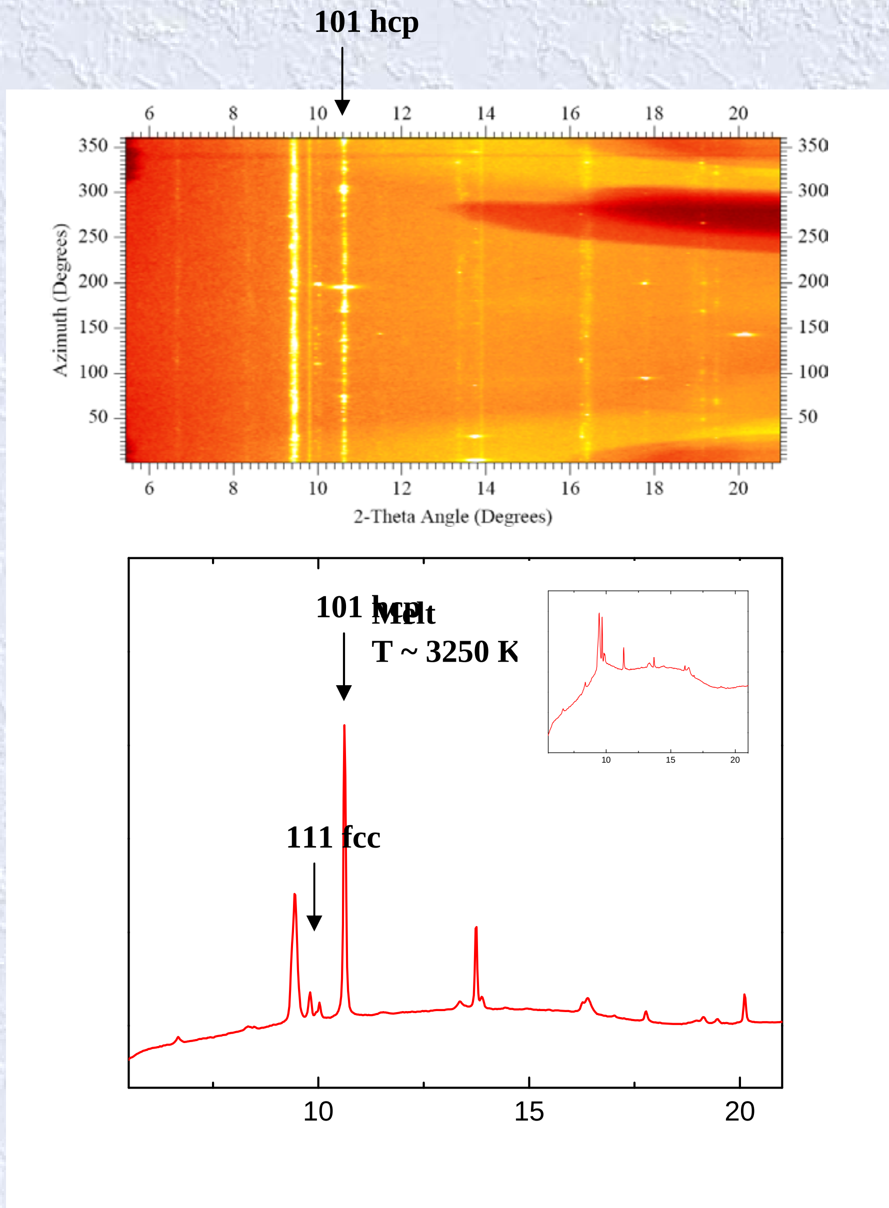
TEM_{00} + π-Shaper



X-ray



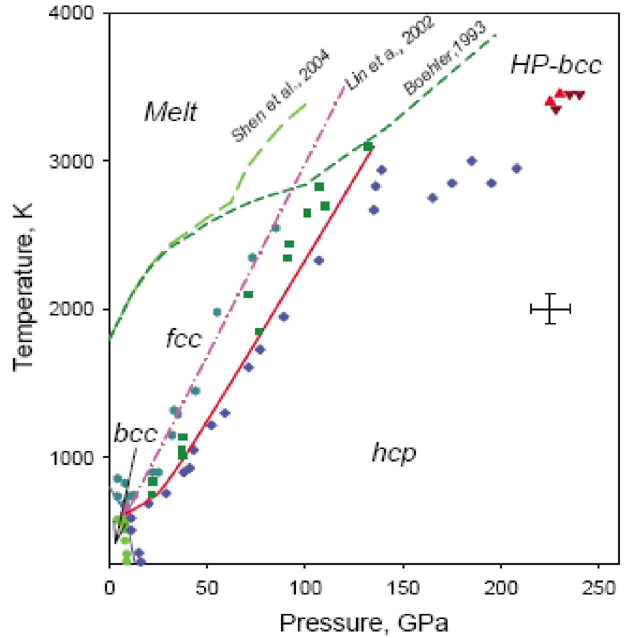
Melting of Fe-Ni alloy at 60 GPa

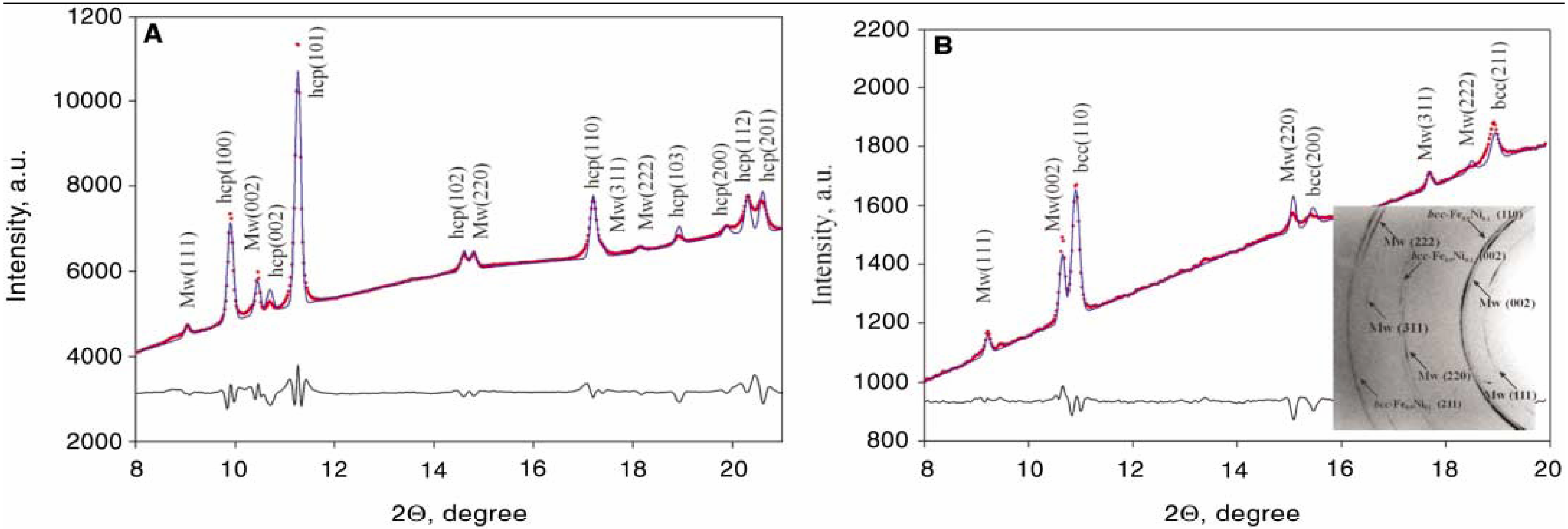


Body-Centered Cubic Iron-Nickel Alloy in Earth's Core

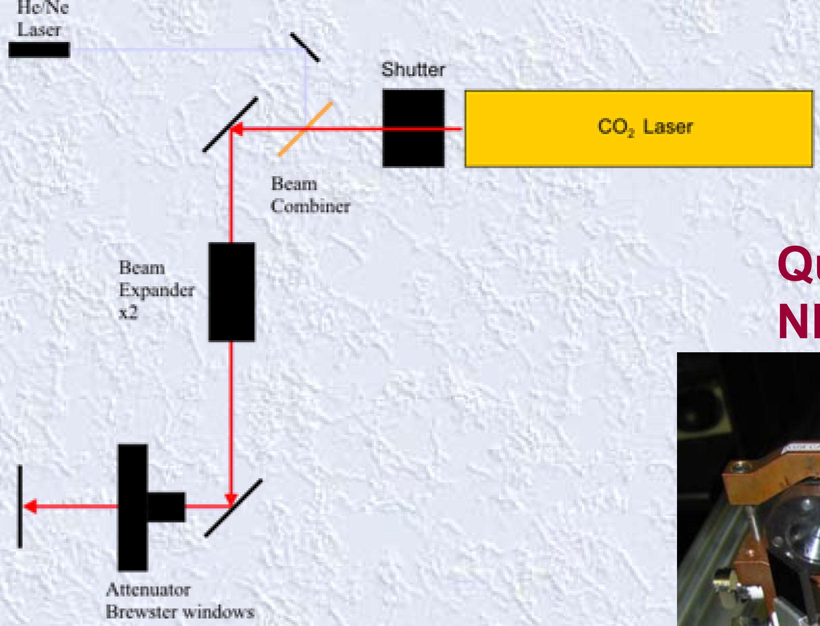
L. Dubrovinsky,[1] N. Dubrovinskaia,[2] O. Narygina,[1] I. Kantor,[1] A. Kuznetzov,[3] V. B. Prakapenka,[3] L. Vitos,[4,5,6] B. Johansson,[4,5] A. S. Mikhaylushkin,[6,7] S. I. Simak,[7] I. A. Abrikosov[7]

At pressures above 225 gigapascals and temperatures over 3400 kelvin, Fe0.9Ni0.1 adopts a bodycentered cubic structure

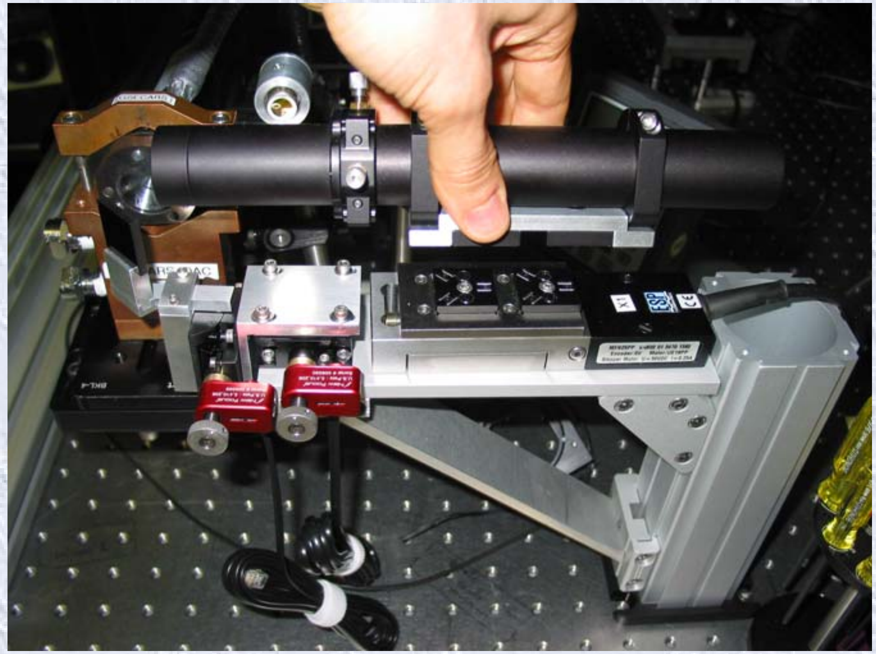


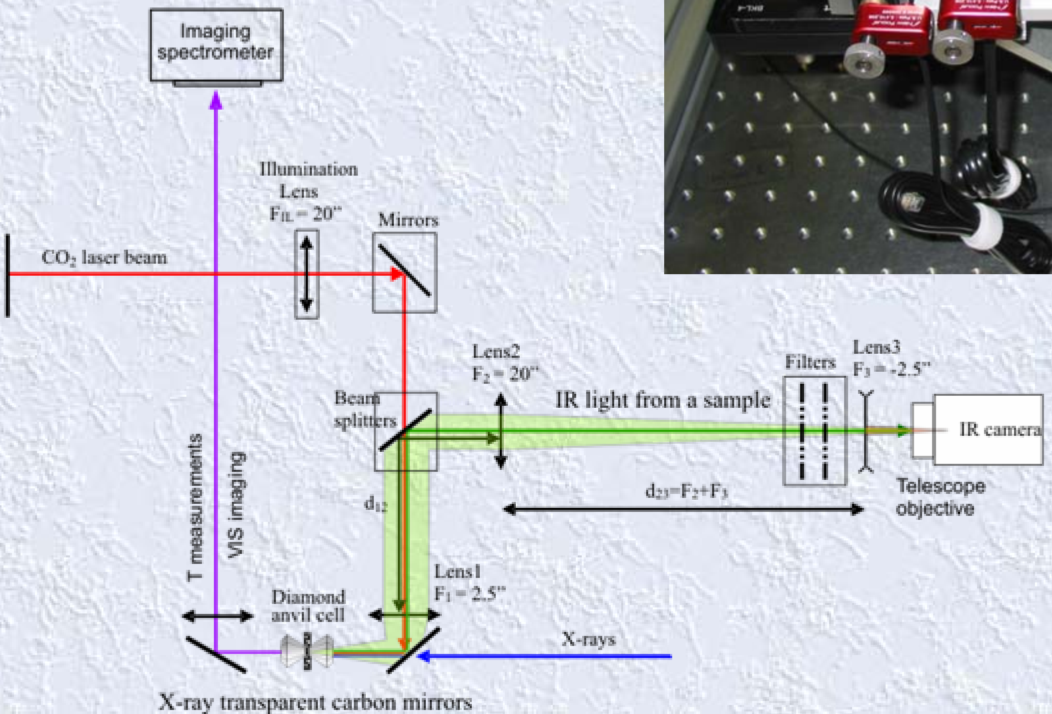


L. Dubrovinsky, *et al. Science* 316, 1880 (2007)

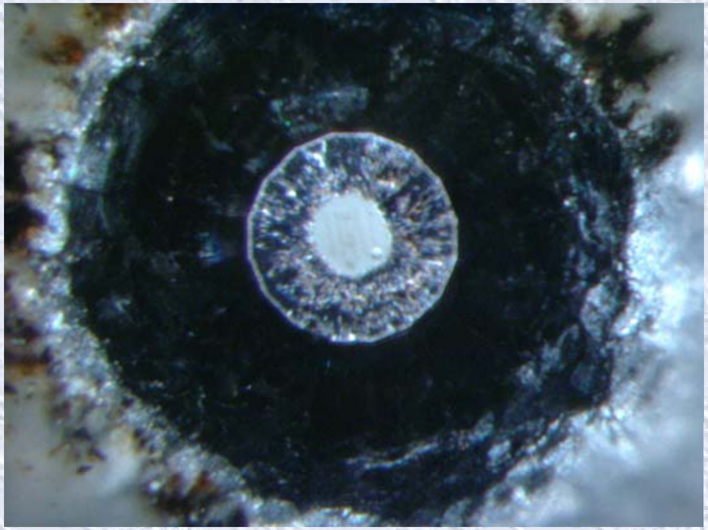


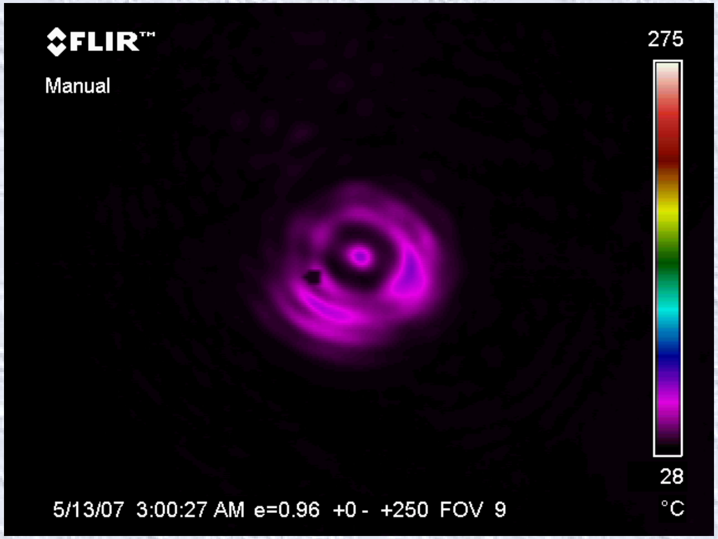
Quick switch between the NIR and CO2 laser heating



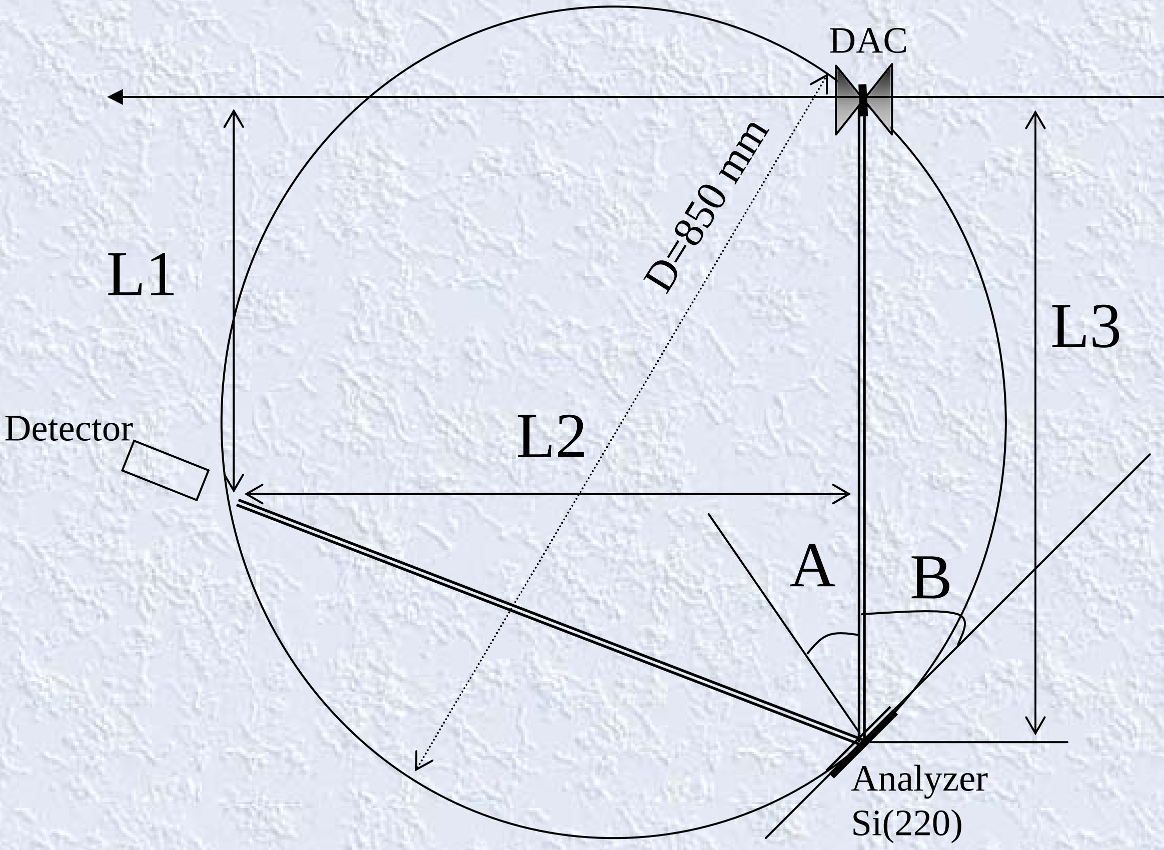


Simultaneous on-line sample monitoring in IR (7.5-13.5 μm) and visible wavelength range



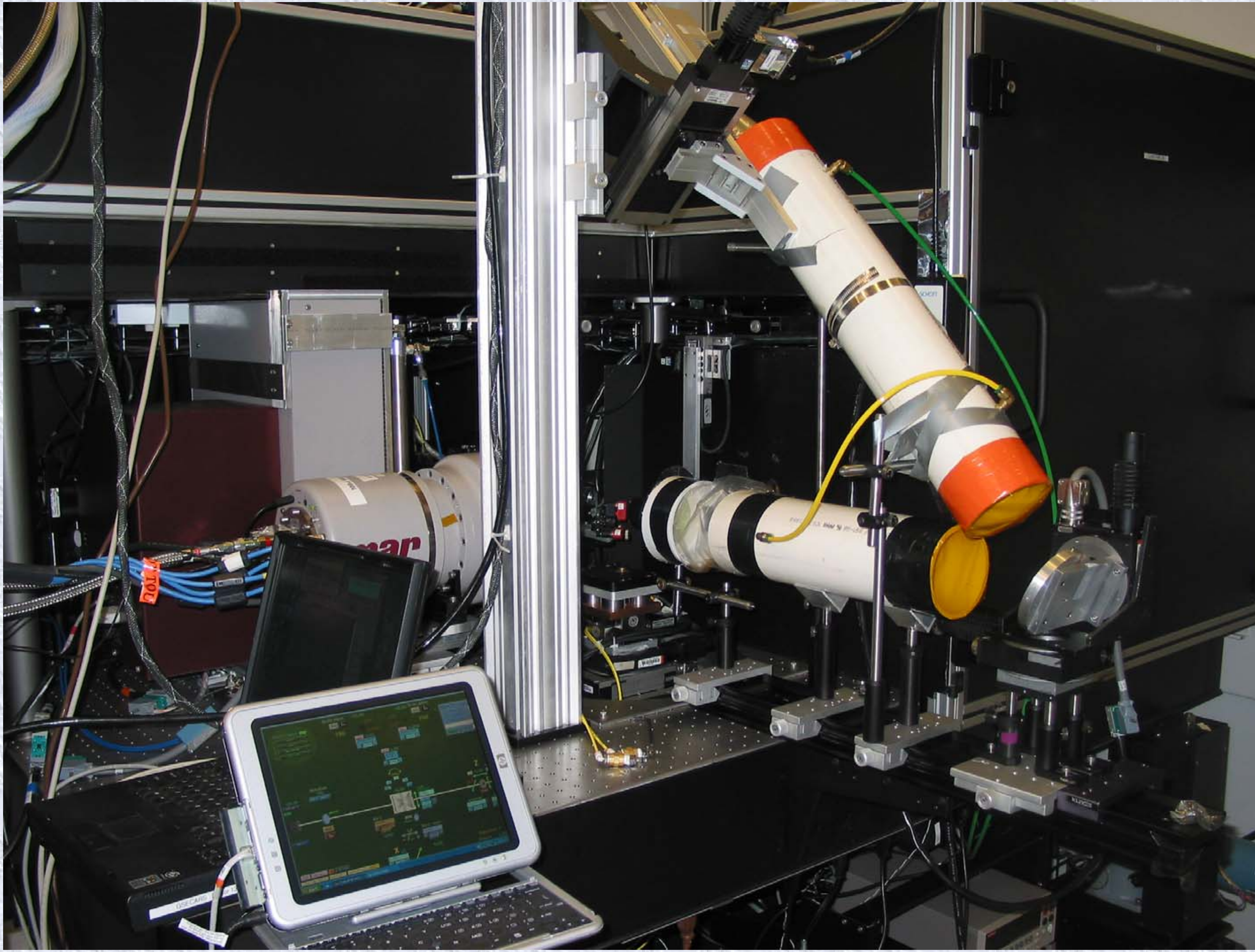


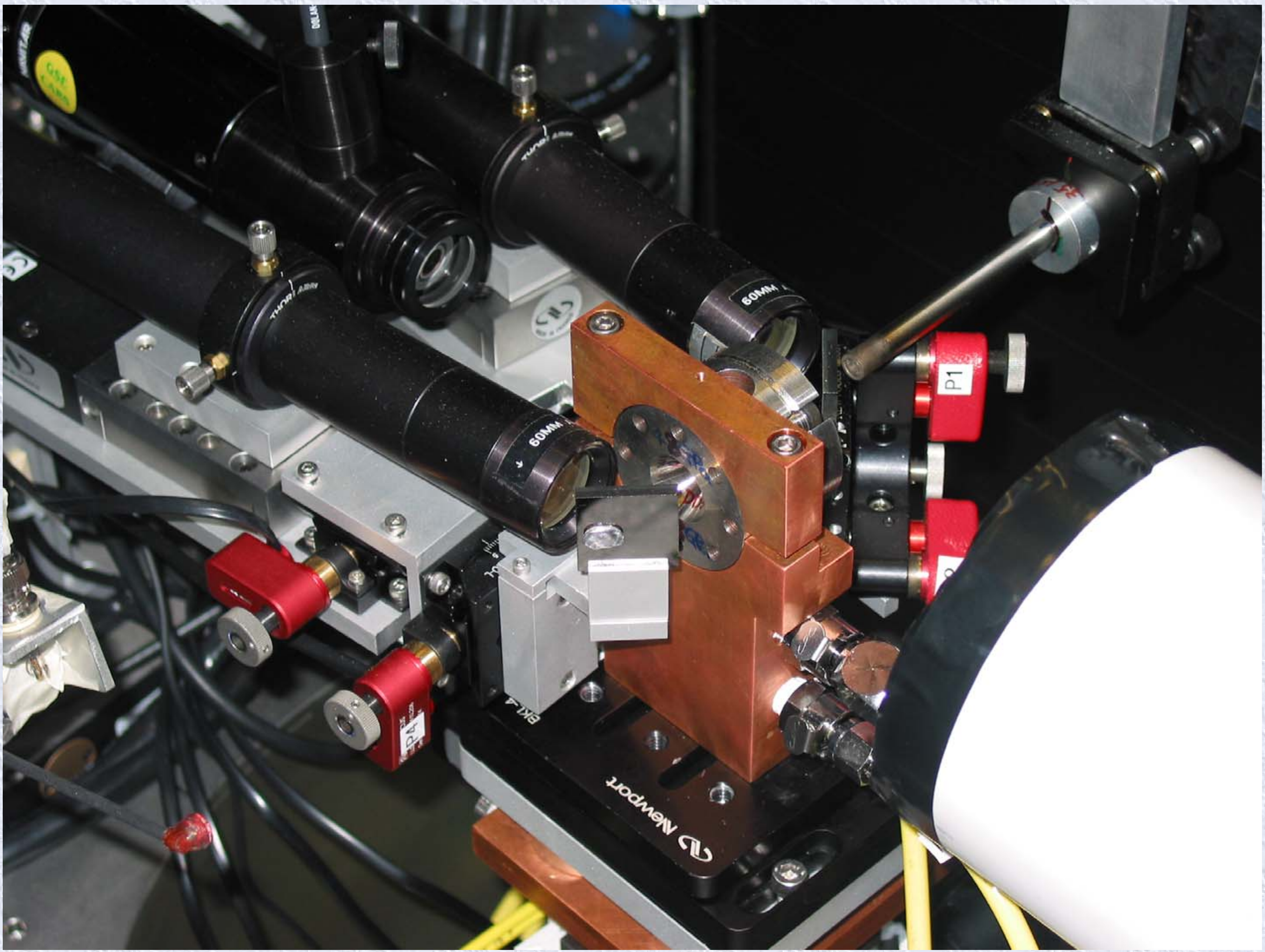
Emission spectroscopy setup



d [Si(220)]=1.9201
$B=\mathrm{asin}(6.19921/(d*E_{kb1}))$
$A=90-B$
$L3=D*\cos(A)$
$L1=L3*(1-\cos(2A))$
$L2=L3*\sin(2A)$

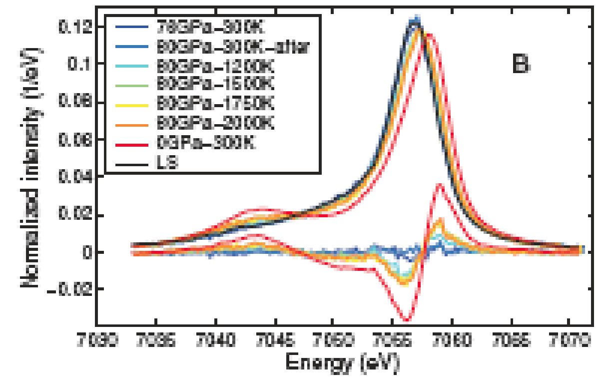
	Fe
K-beta	**7057.98**
A (degree)	**62.78**
B (degree)	**27.22**
L1 (mm)	**614.91**
L2 (mm)	**316.3**
L3 (mm)	**388.8**



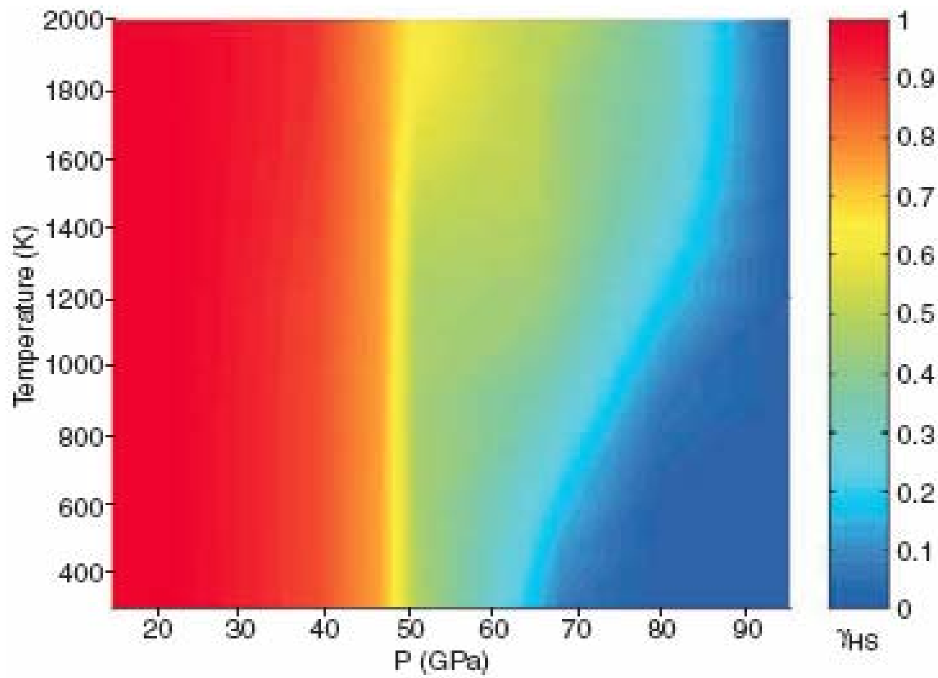

60MM
60MM
P1
Newport

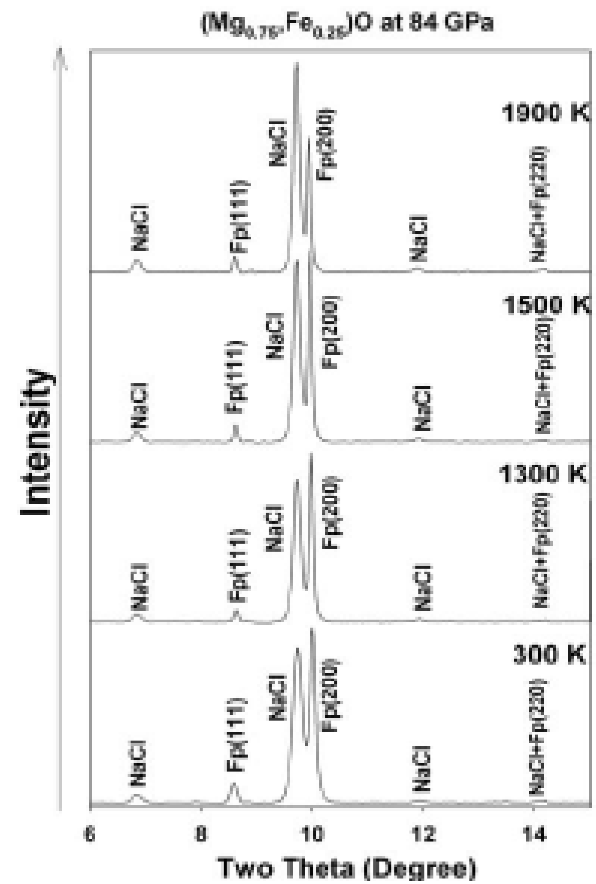
Spin Transition Zone in Earth's Lower Mantle

Jung-Fu Lin,[1] György Vankó,[2,3] Steven D. Jacobsen,[4] Valentin Iota,[1] Viktor V. Struzhkin,[5] Vitali B. Prakapenka,[6] Alexei Kuznetsov,[6] Choong-Shik Yoo[1]*



A gradual spin transition of iron occurs over a pressure-temperature range extending from about 1000 kilometers in depth and 1900 kelvin to 2200 kilometers and 2300 kelvin in the lower mantle



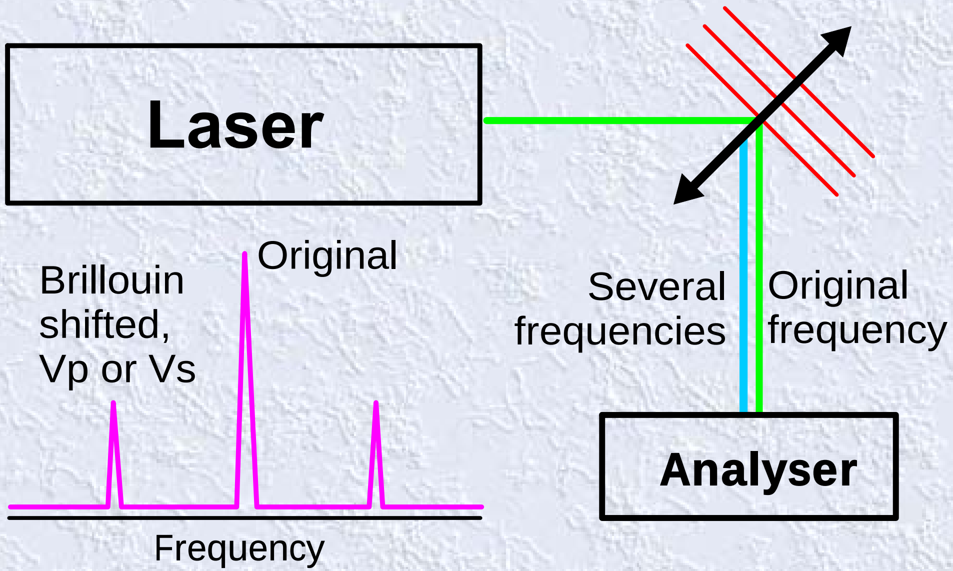


Jung-Fu Lin, *et al.*, *Science* 317, 1740 (2007)

Brillouin scattering

- *Acoustic waves present in a solid due to thermal motion of atoms*
- *Laser light interacts with phonons (or density / refractive index fluctuations) and is scattered with Doppler shifted frequency $\Delta\omega$*
- *In symmetric platelet geometry the Brillouin shift is directly proportional to acoustic velocity*

$$V_i = \Delta\omega\lambda / 2\sin(\theta/2)$$



S. V. Sinogeikin, J. D. Bass, V.B. Prakapenka et al, Rev. Sci. Instrum. 77, 103905 2006

Brillouin spectroscopy

Advantages:

- direct measurements of acoustic velocities
- nondestructive optical spectroscopy
- very small samples (down to 10-20 μm)
- from single crystal to non-crystalline materials
- measurements at P-T conditions of the Lower Mantle

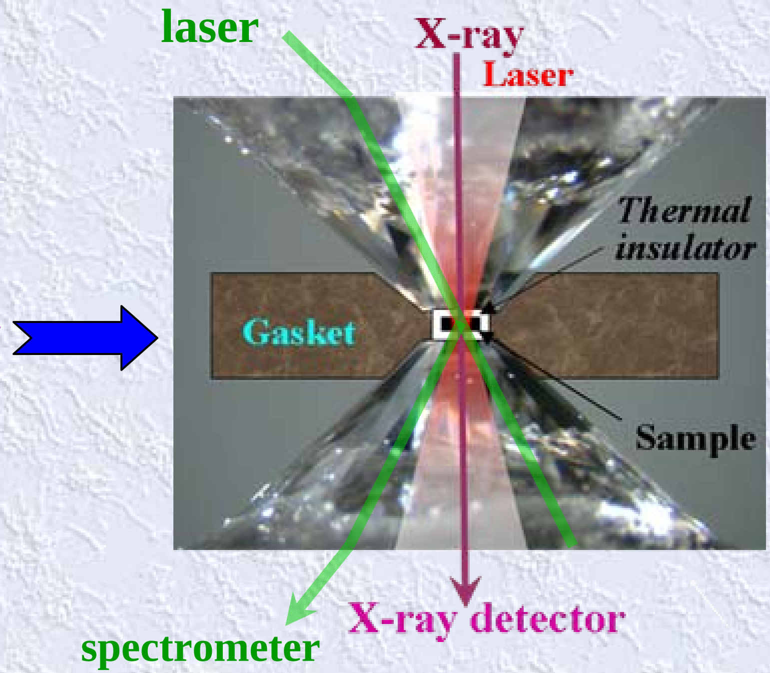
Disadvantages:

- complicated optical system
- transparent and translucent samples
- elastic moduli only for known density

Shear: $\mu=\rho V^2_S$

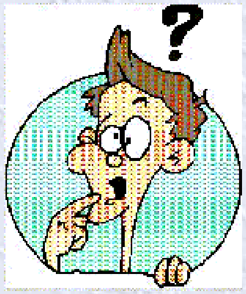
Bulk: $K_S=\rho V^2_P - 4/3\,\mu$

Single-crystal: $C_{11} = \rho V_p{}^2{}_{(100)}$



S. V. Sinogeikin, J. D. Bass, V.B. Prakapenka, Rev. Sci. Instrum. 77, 103905 2006

Why do we need Brillouin spectroscopy on-line?

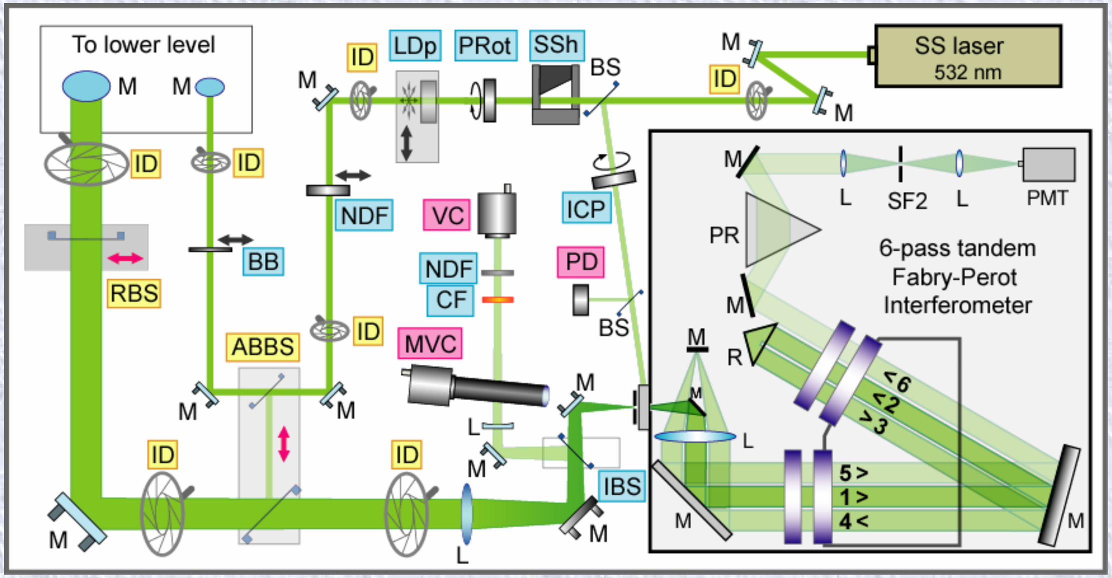


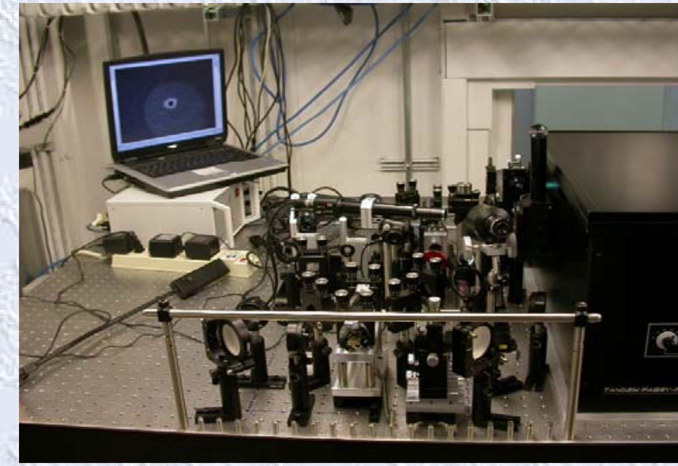
*-measuring sound velocities and densities of materials simultaneously results in a **pressure scale** without reference marker and important materials properties (equations of state, elasticity, etc) as a function of pressure and temperature*

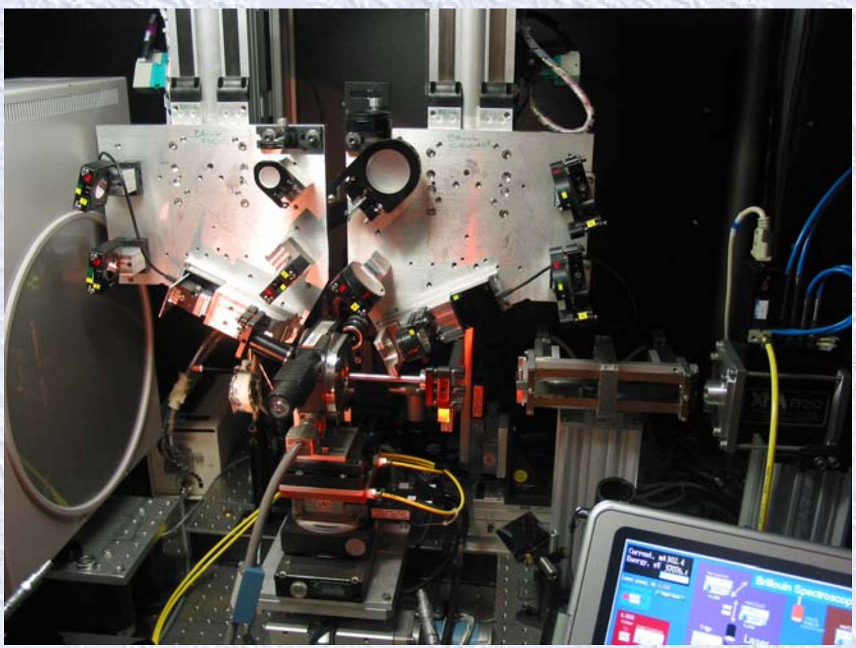
*-the experimental data collected with XRD and BS at the same pressure-temperature conditions provide information essential for comparing with seismic observations and modelling the **composition and evolution of the Earth***

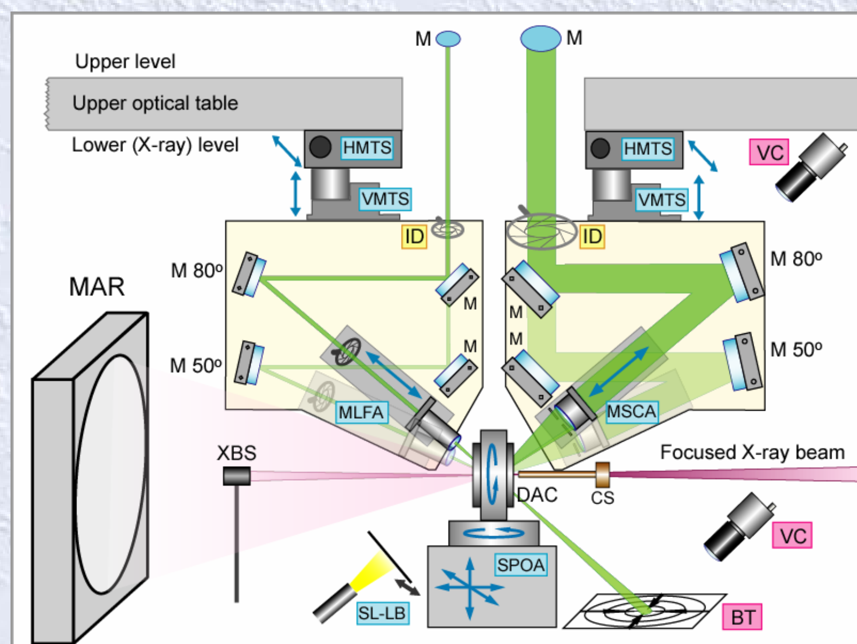
*-Brillouin spectroscopy combined with XRD **open a new area** of in-situ studies of materials at extreme conditions*

S. V. Sinogeikin, J. D. Bass, V.B. Prakapenka, Rev. Sci. Instrum. 77, 103905 2006



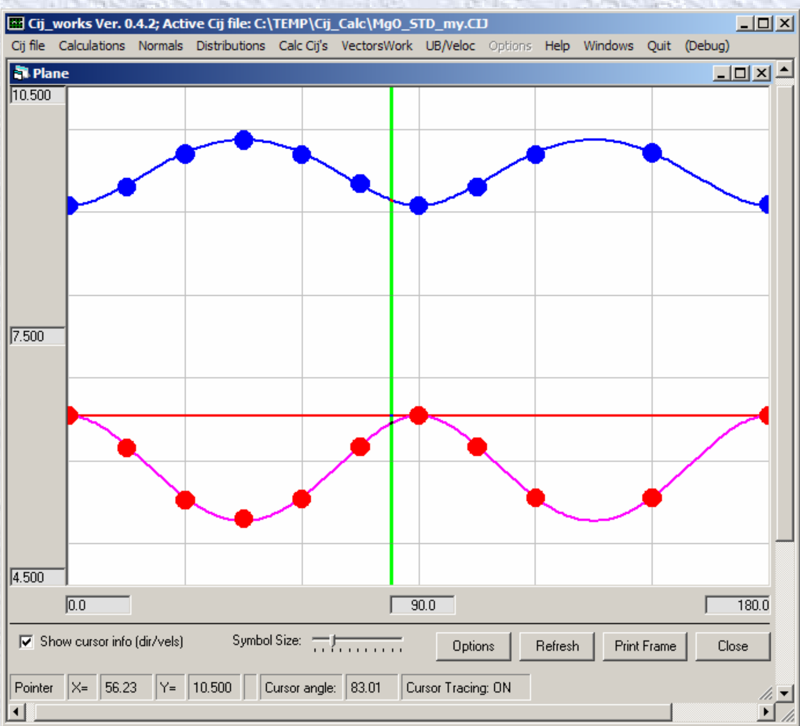


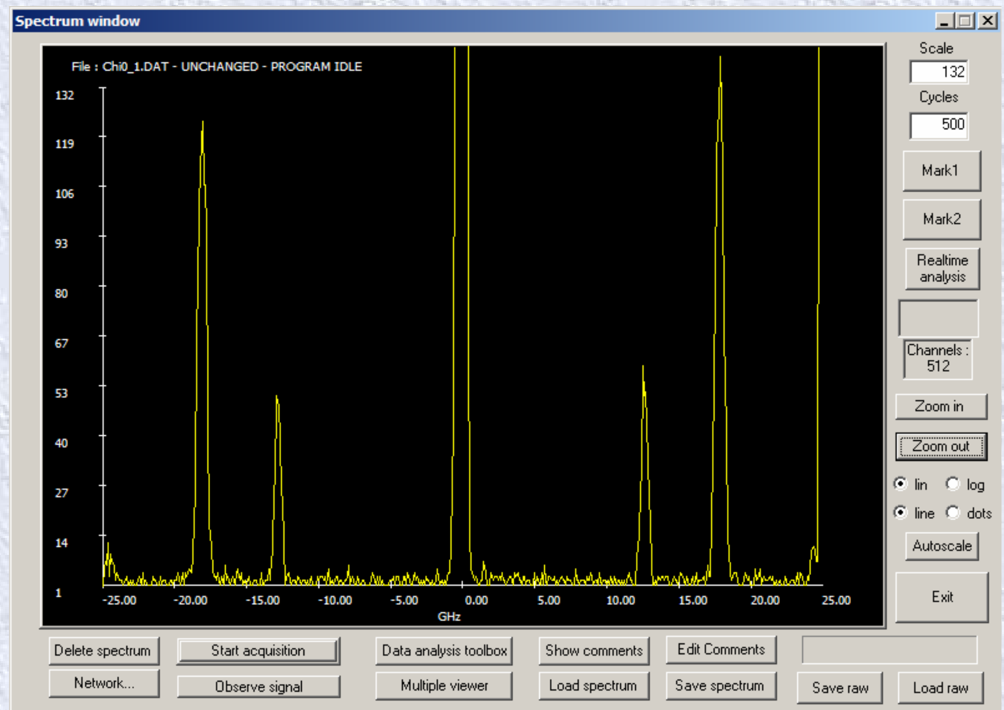




S. V. Sinogeikin, J. D. Bass, V.B. Prakapenka, Rev. Sci. Instrum. 77, 103905 2006

Calibration Brillouin system with MgO single crystal starting with [100] direction at ambient conditions





collection time is 5 minutes

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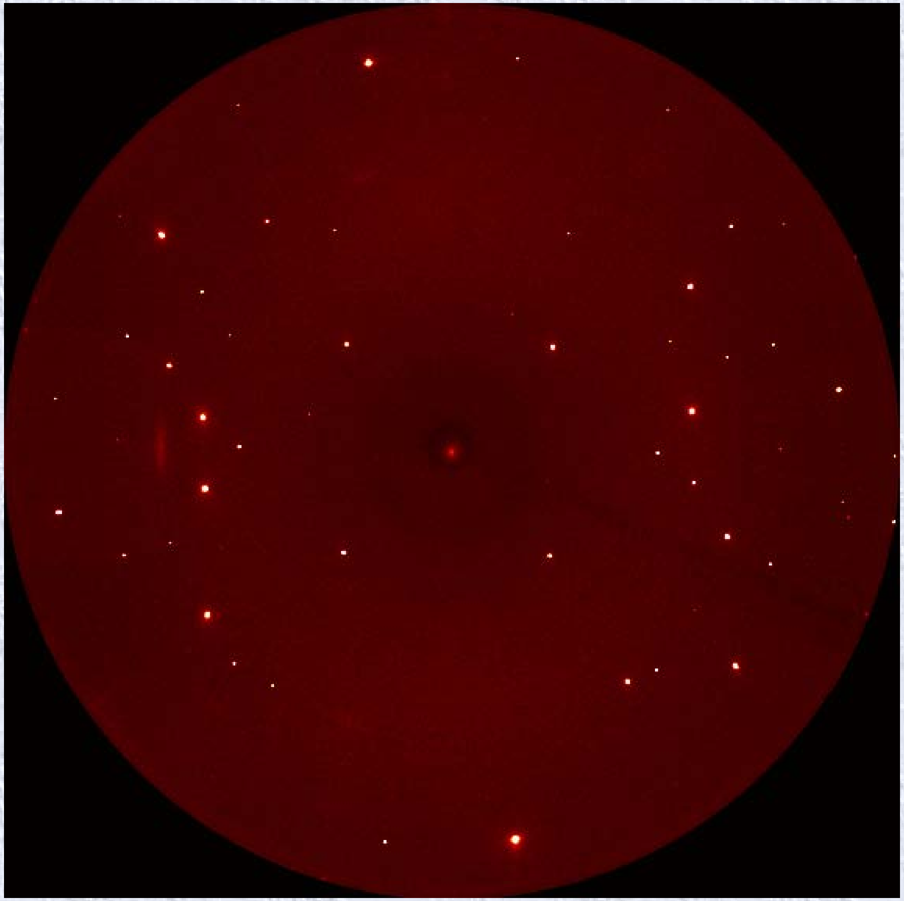
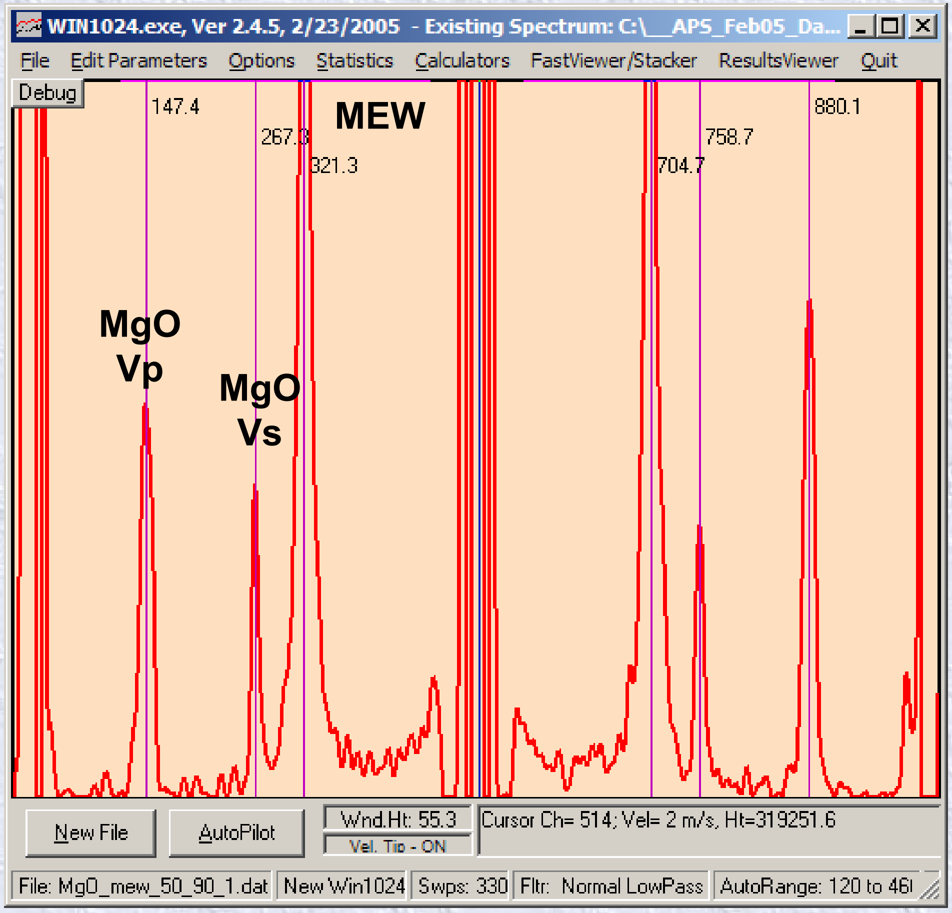
XRD and BS of single crystal MgO in [100] direction in the DAC at 4 GPa





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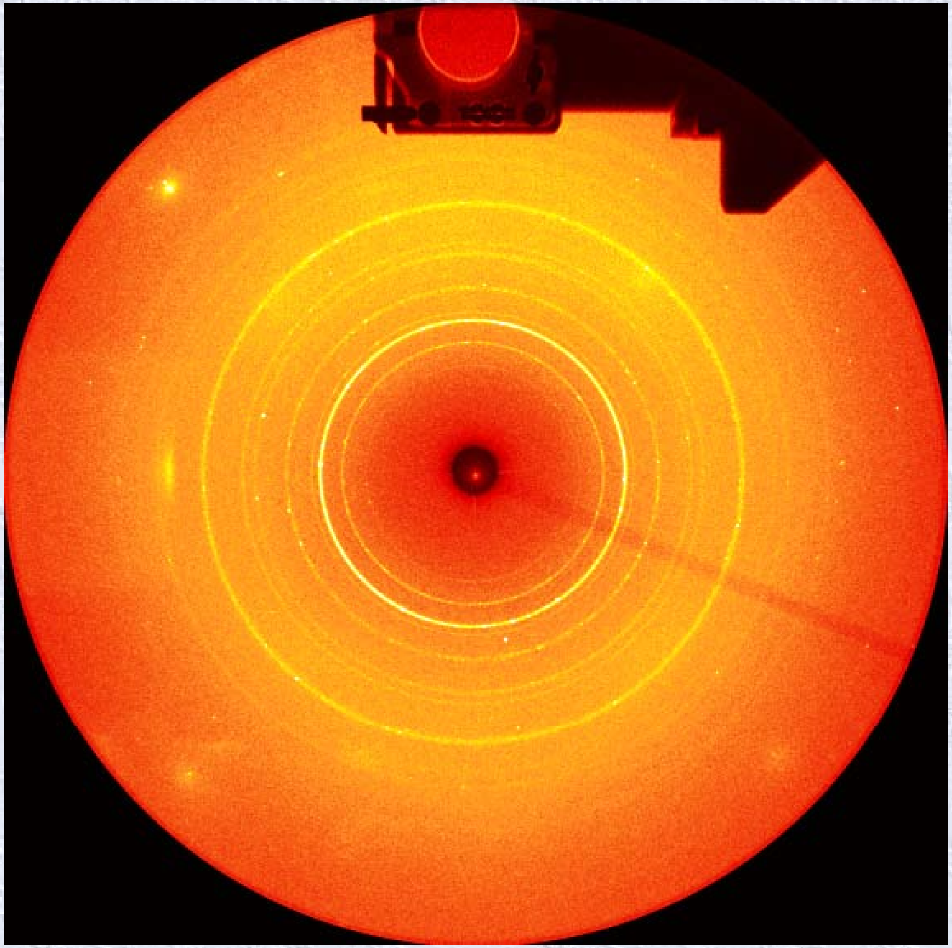
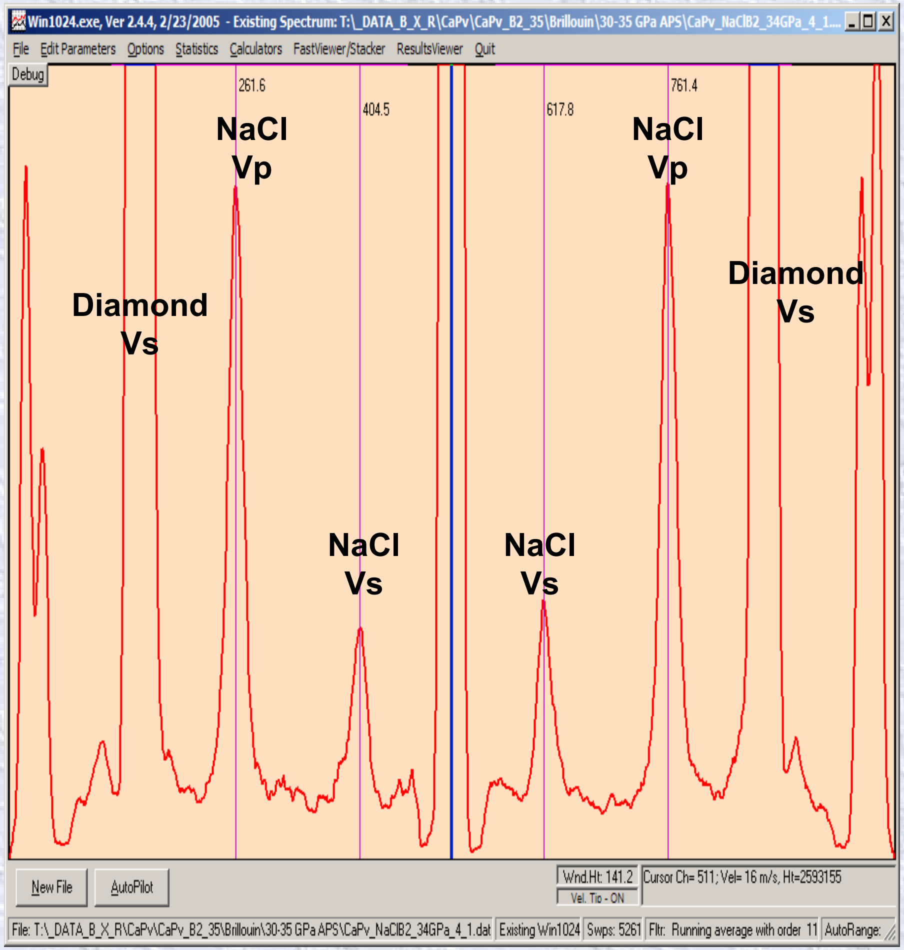
XRD and BS of NaCl at 35 GPa collected simultaneously





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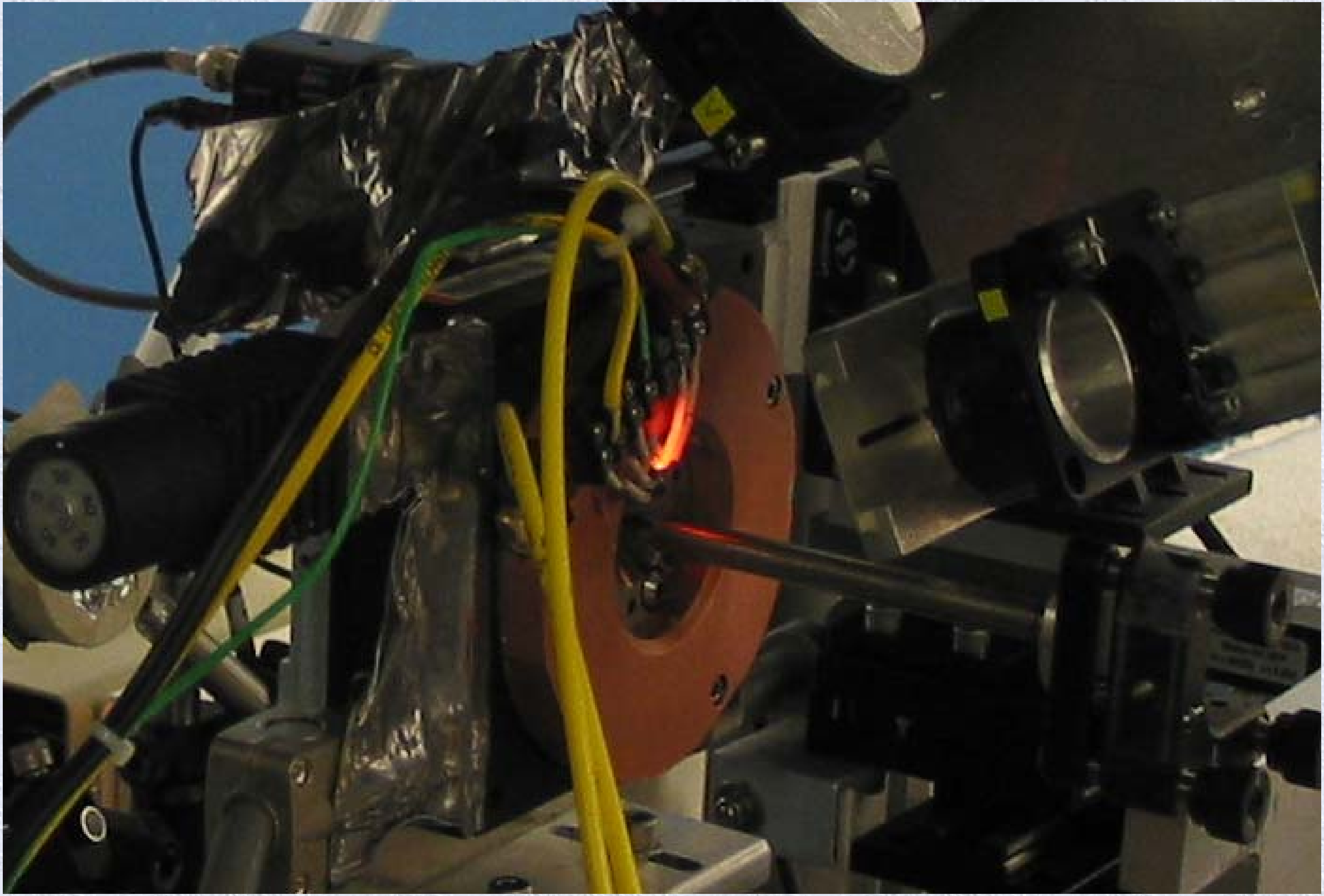

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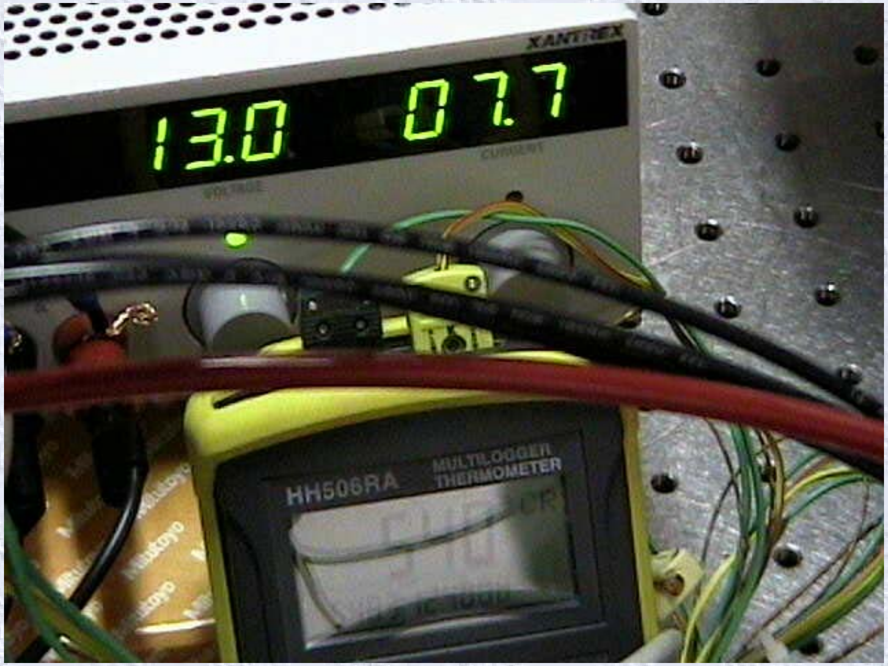
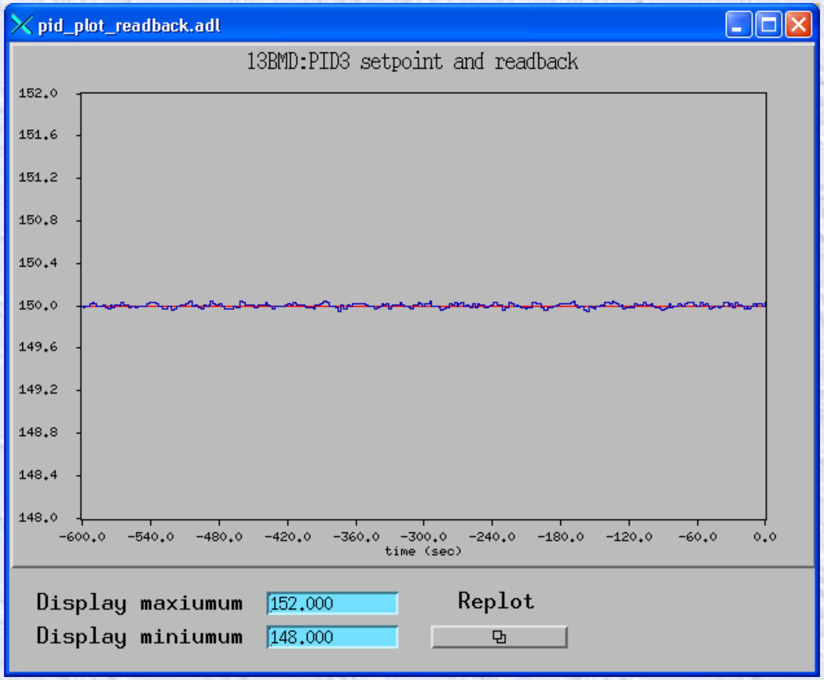
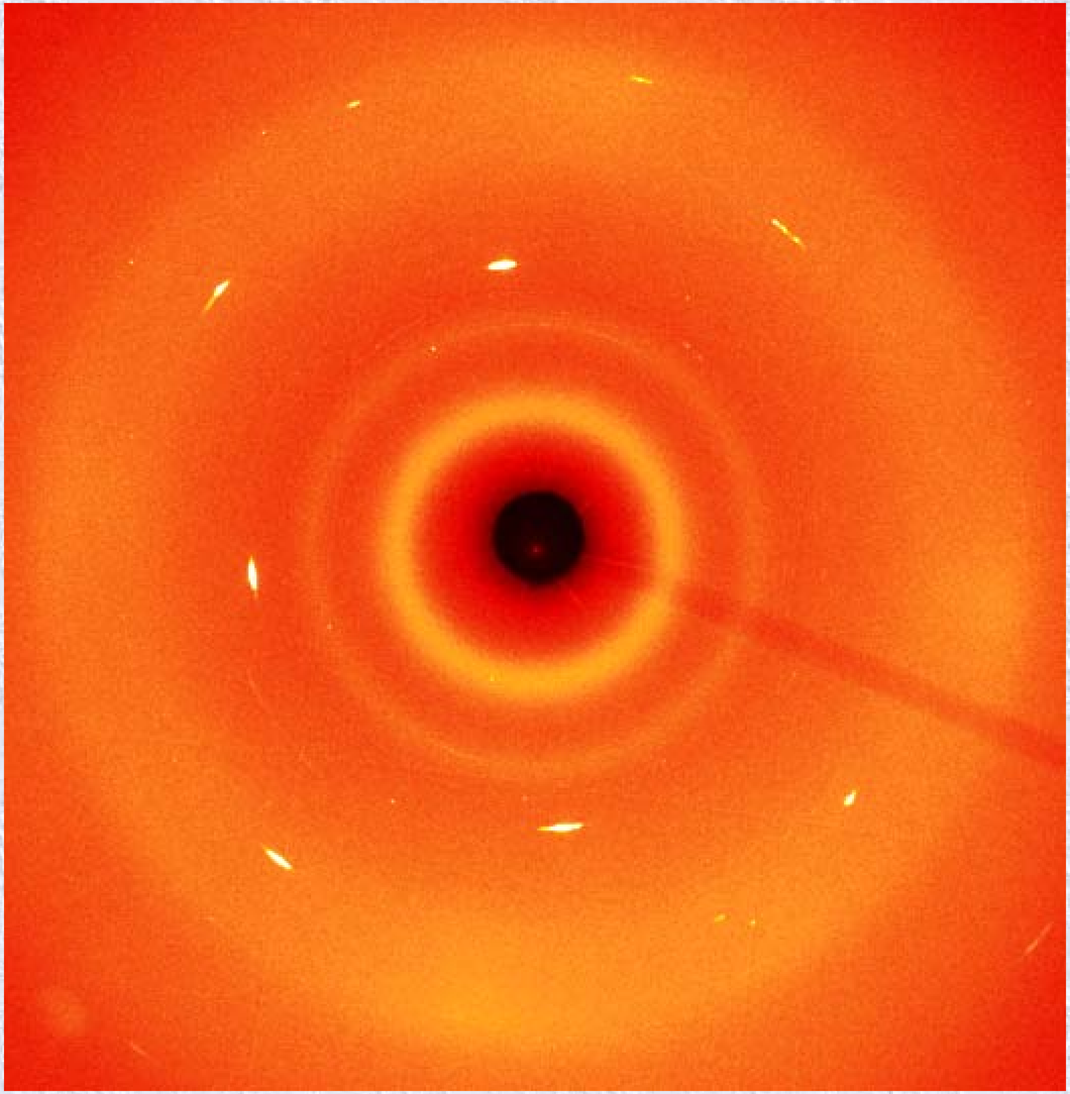
Temperature control
(slow feedback)



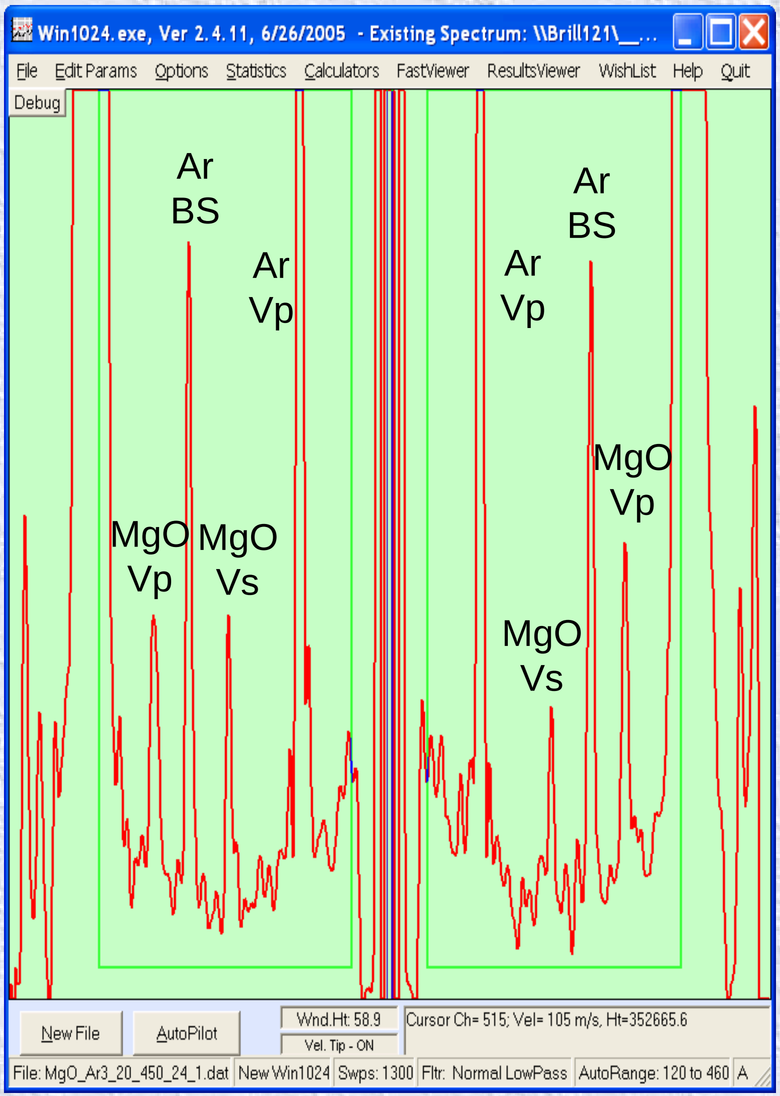


S. V. Sinogeikin, J. D. Bass, V.B. Prakapenka, Rev. Sci. Instrum. 77, 103905 2006

XRD and BS of MgO in [100] direction in the DAC at 8 GPa and ~800 K

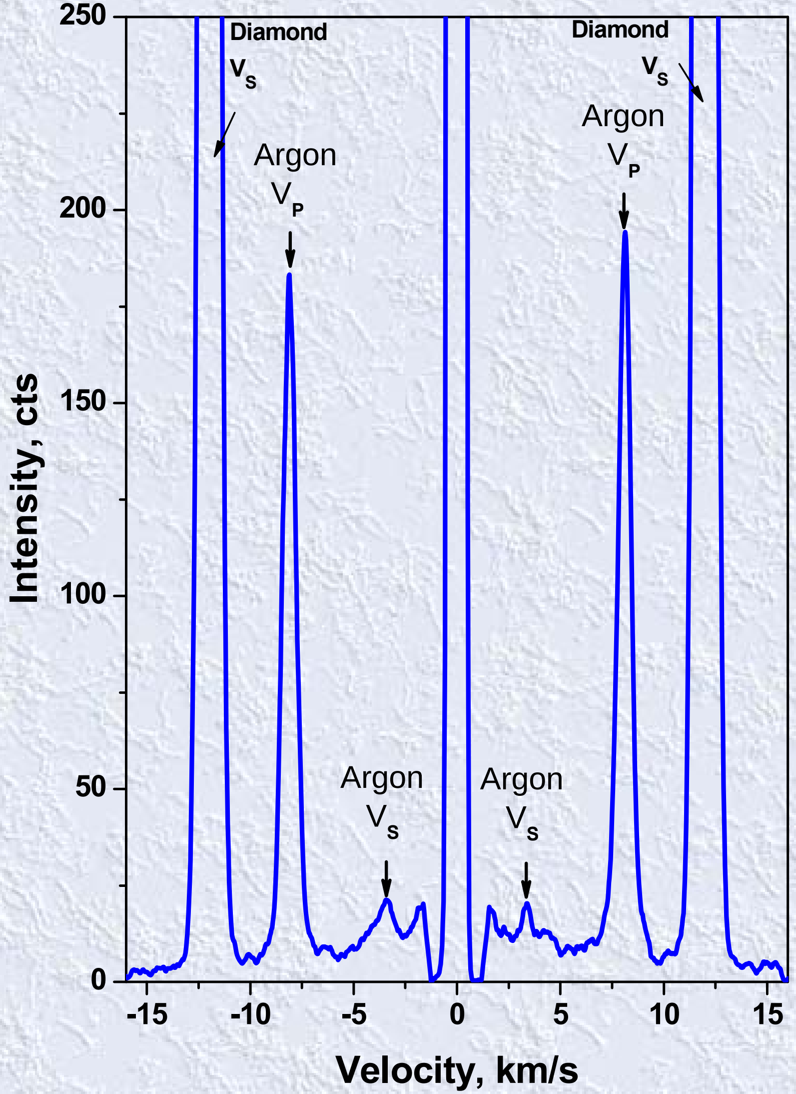


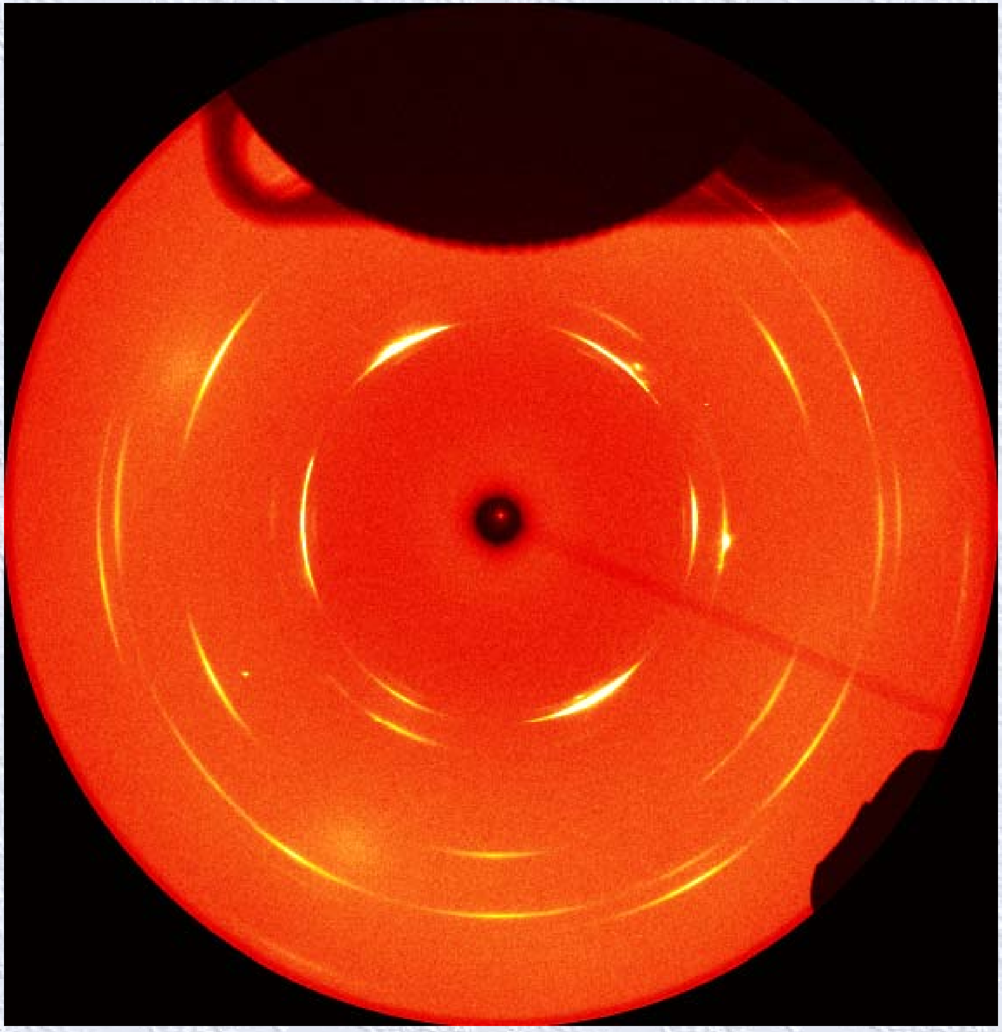
Ar pressure medium is melted



collection time ~10 minutes

In situ XRD and BS of argon at 60 GPa and ~700 K



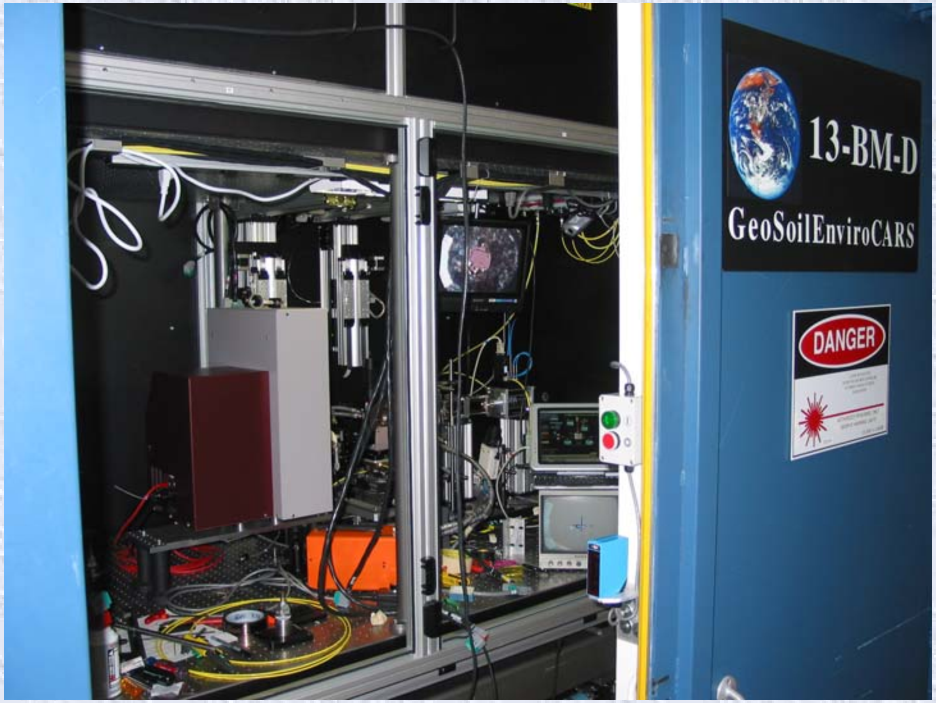


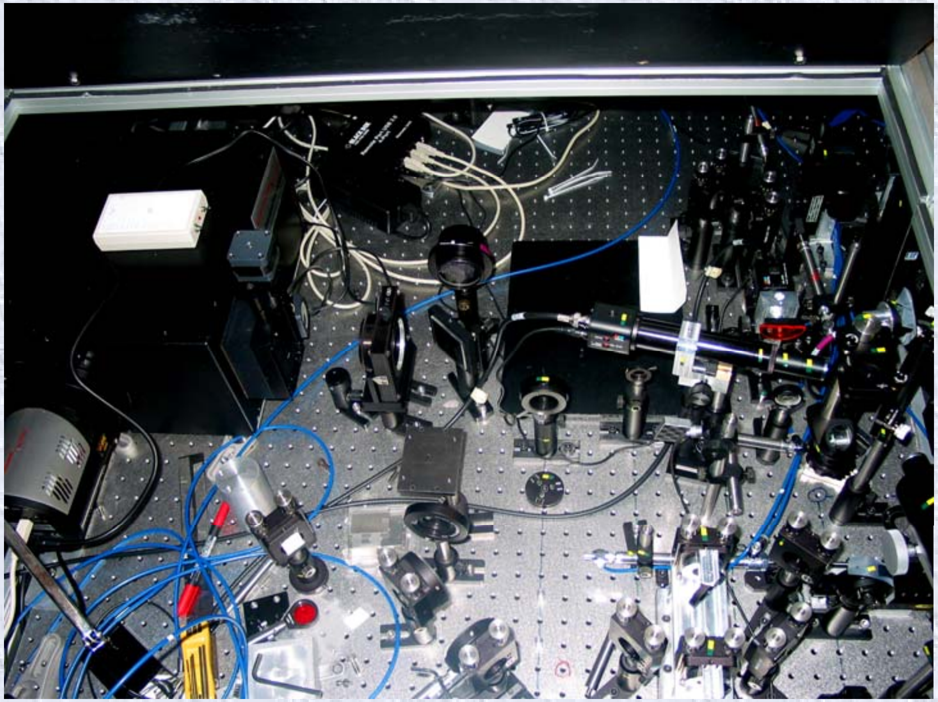
S. V. Sinogeikin, J. D. Bass, V.B. Prakapenka, Rev. Sci. Instrum. 77, 103905 2006

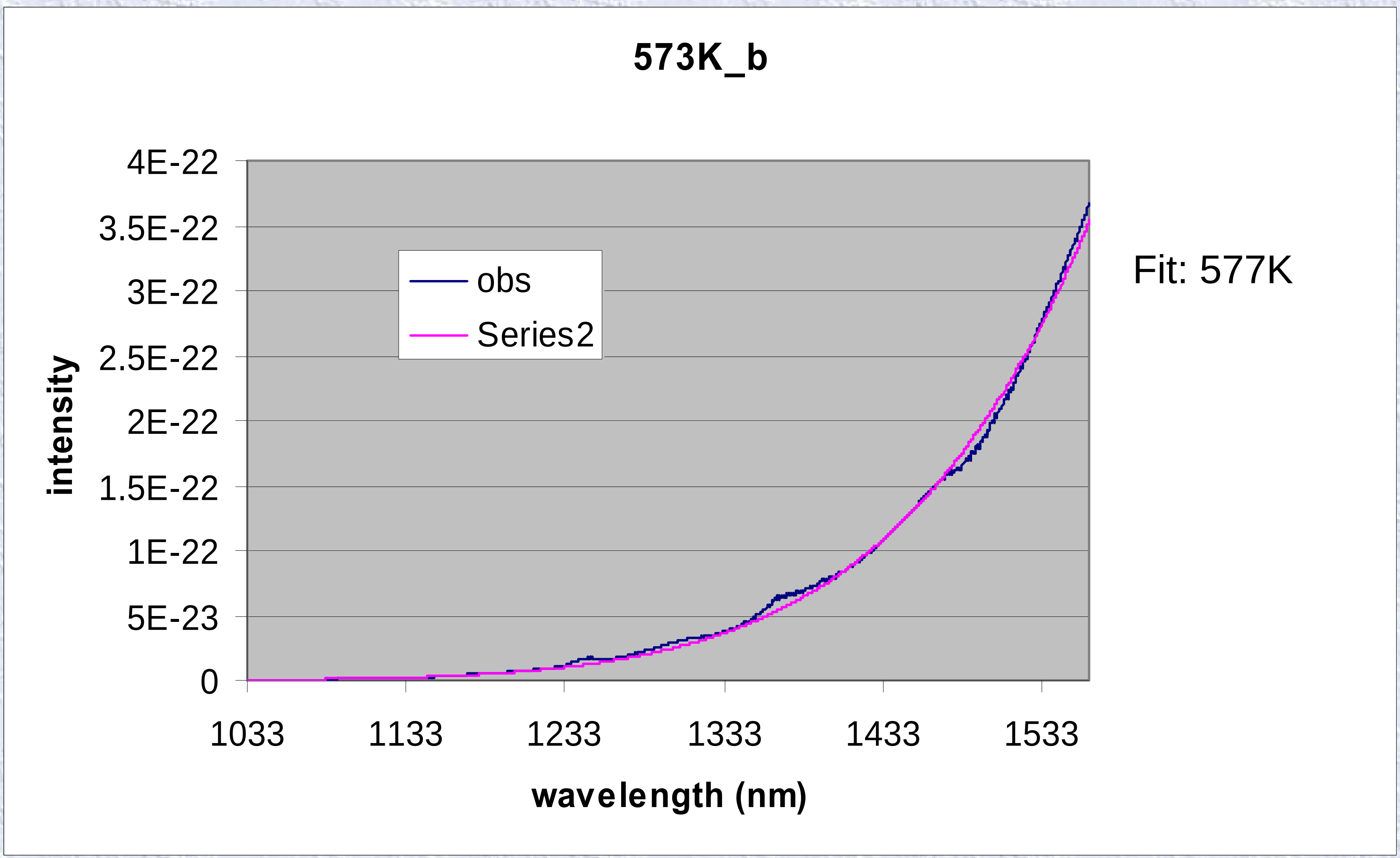
Remarkable combination of in-situ high resolution microdiffraction at HP/HT in DAC with optical spectroscopy:

- *Brillouin spectroscopy*
- *Raman spectroscopy*
- *Fluorescent spectroscopy (ruby)*
- *Optical spectroscopy (500-1600 nm)*

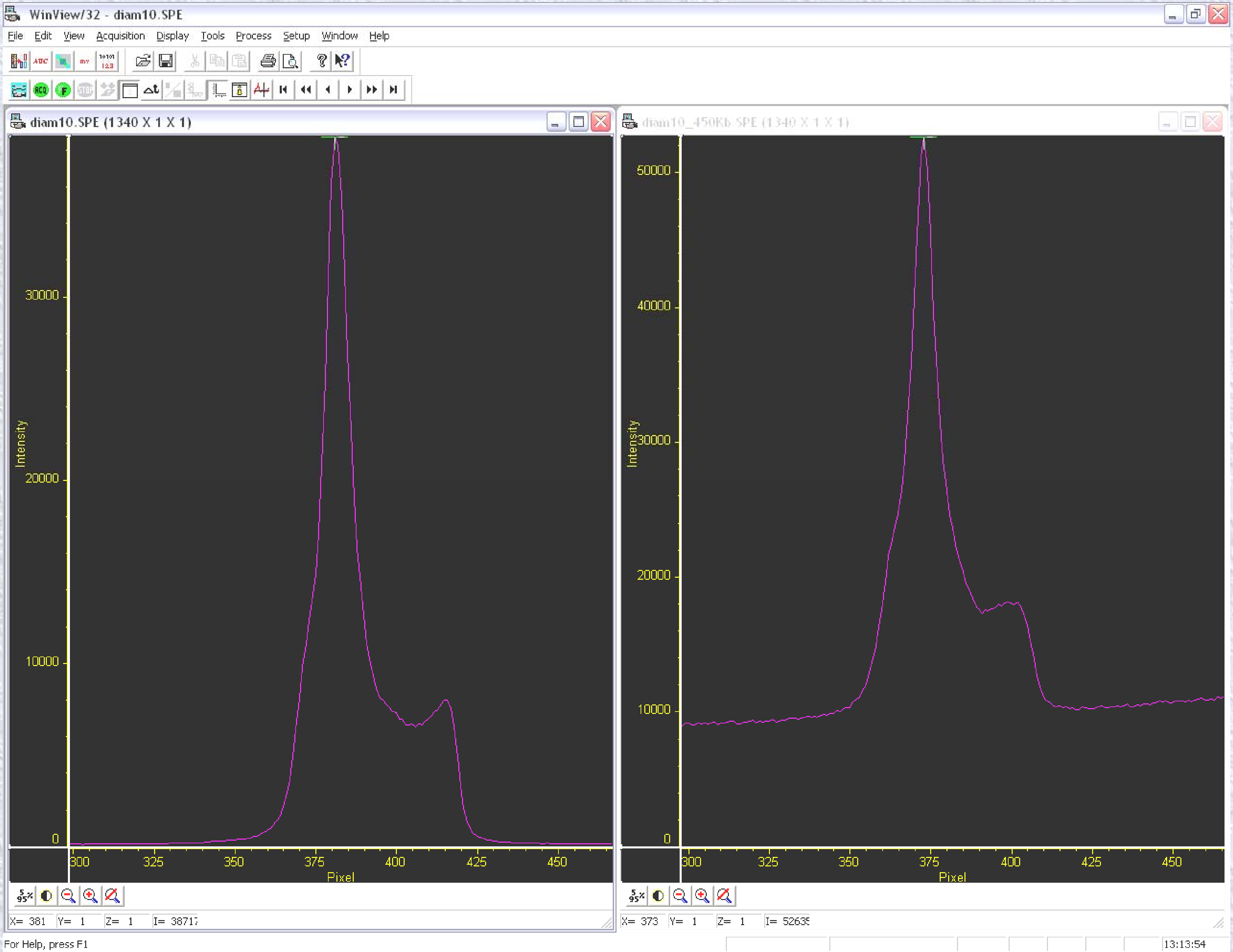
13-BM-D (GSECARS)



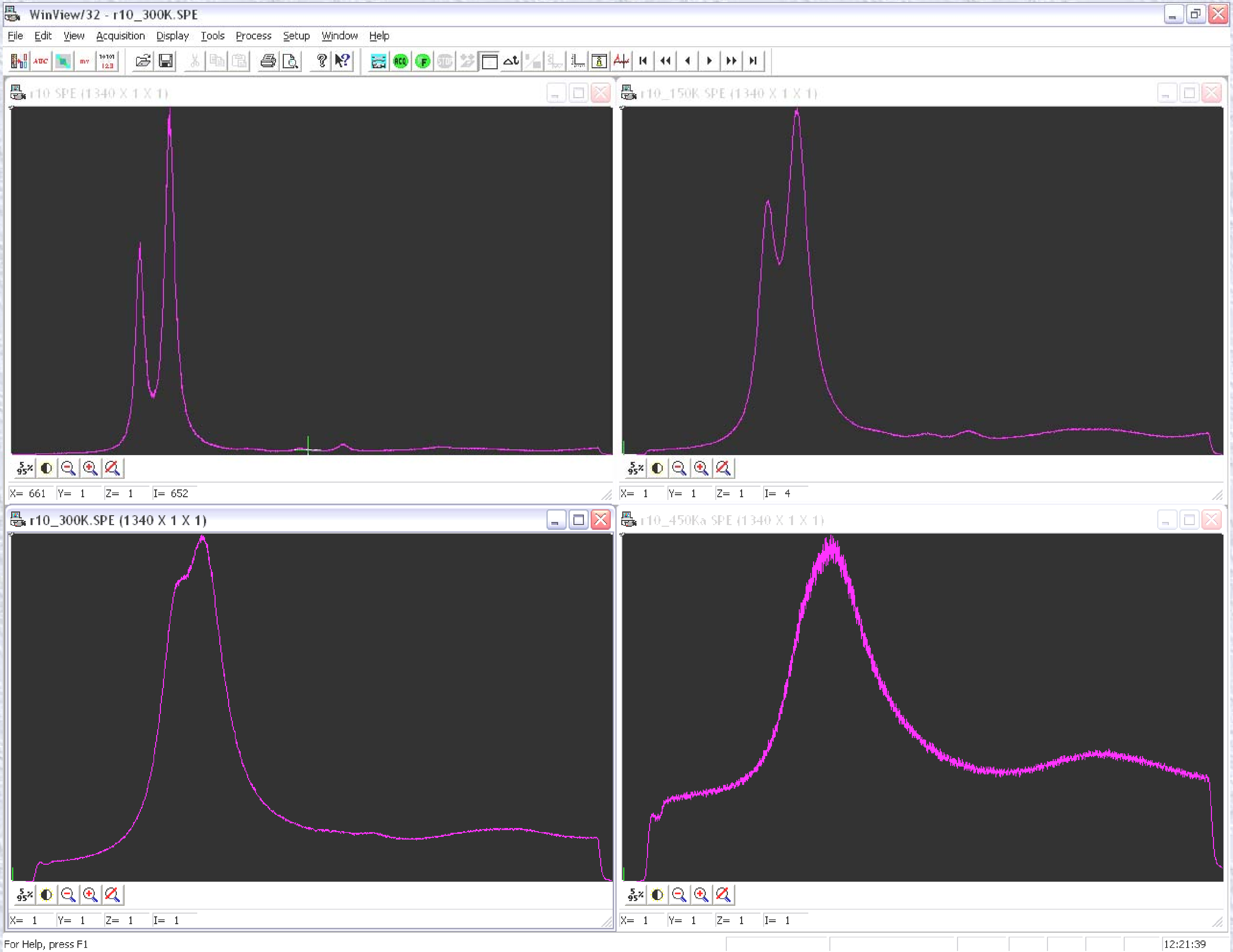




573K_b
4E-22
3.5E-22
3E-22
2.5E-22
2E-22
1.5E-22
1E-22
5E-23
0
intensity
obs
Series2
1033
1133
1233
1333
1433
1533
wavelength (nm)
Fit: 577K

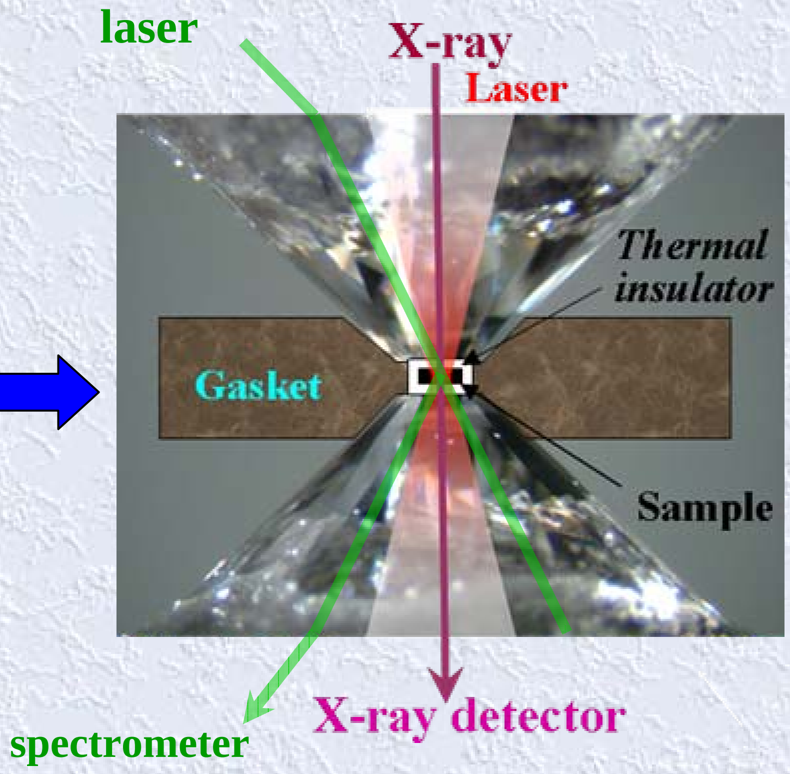


WinView/32 - diam10.SPE
File Edit View Acquisition Display Tools Process Setup Window Help
diam10.SPE (1340 X 1 X 1)
Intensity
30000
20000
10000
0
300 325 350 375 400 425 450
Pixel
X= 381 Y= 1 Z= 1 I= 38717
diam10_450Kb.SPE (1340 X 1 X 1)
Intensity
50000
40000
30000
20000
10000
0
300 325 350 375 400 425 450
Pixel
X= 373 Y= 1 Z= 1 I= 52635
For Help, press F1
13:13:54

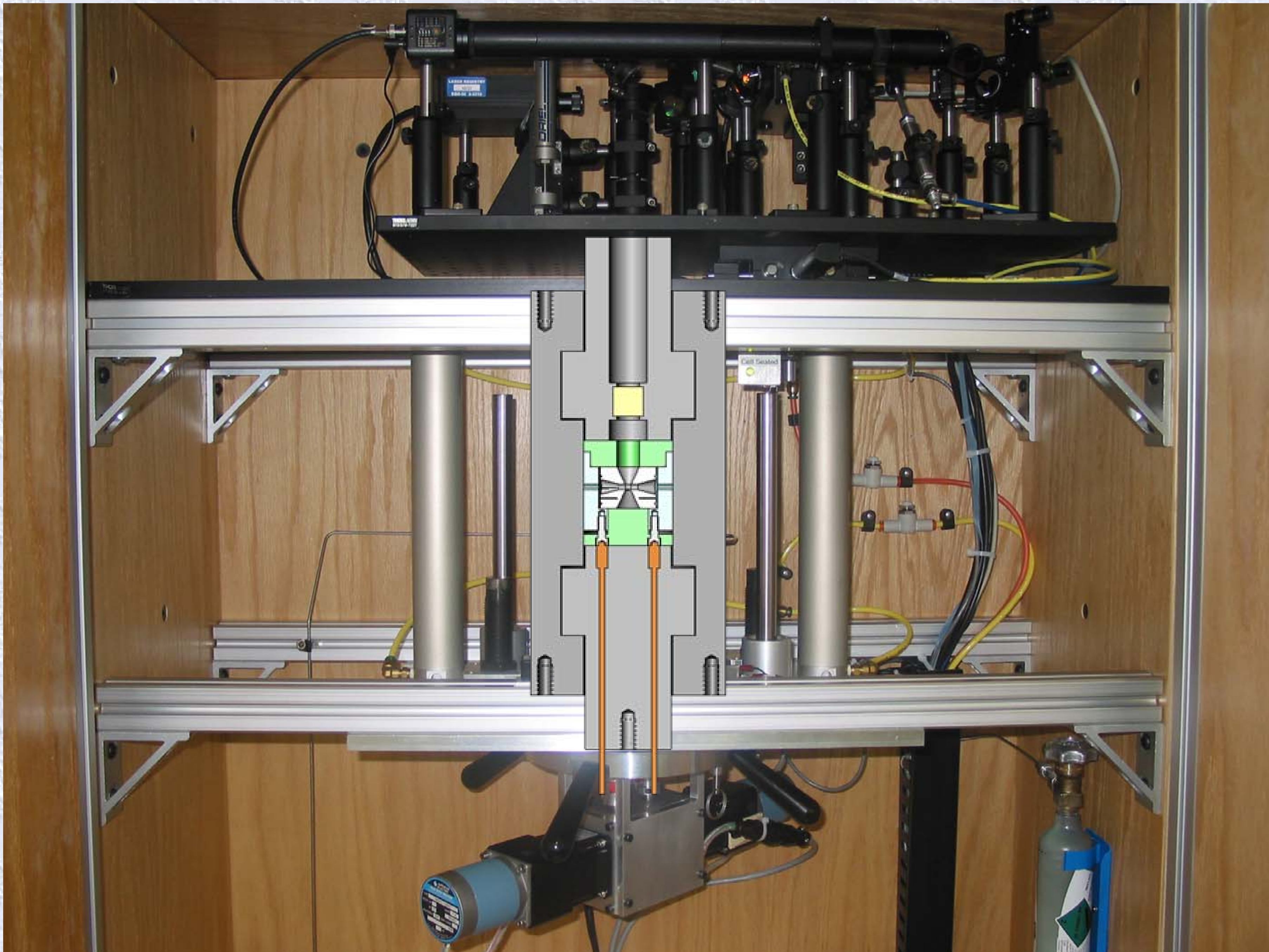

WinView/32 - r10_300K.SPE
File Edit View Acquisition Display Tools Process Setup Window Help
r10.SPE (1340 X 1 X 1)
X= 661 Y= 1 Z= 1 I= 652
r10_150K.SPE (1340 X 1 X 1)
X= 1 Y= 1 Z= 1 I= 4
r10_300K.SPE (1340 X 1 X 1)
X= 1 Y= 1 Z= 1 I= 1
r10_450Ka.SPE (1340 X 1 X 1)
X= 1 Y= 1 Z= 1 I= 1
For Help, press F1
12:21:39

13-BMD, GSECARS

The full spectroscopic characterization of a sample is possible in-situ at extreme high pressure/temperature conditions with powerful combination of x-ray diffraction, Brillouin, Raman and fluorescence spectroscopy applied to a single point on the sample in a diamond anvil cell

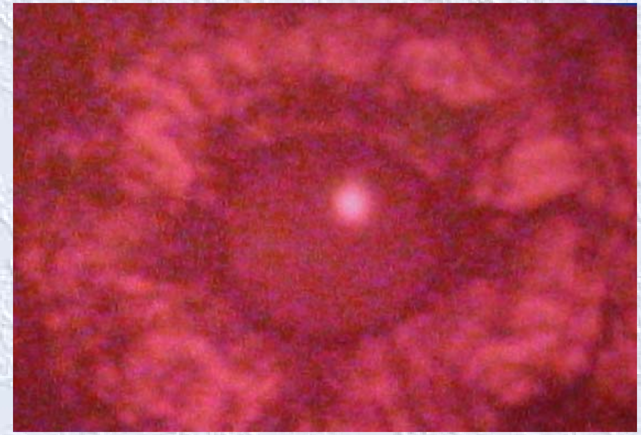


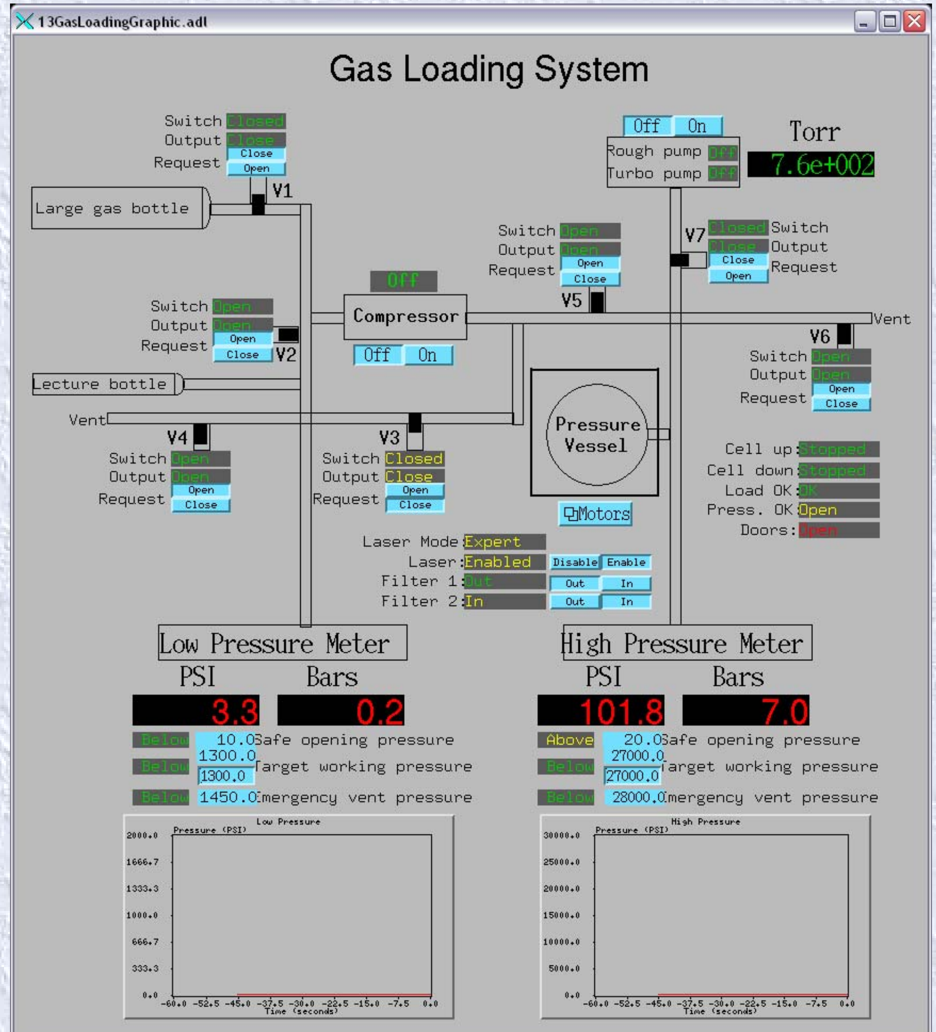

QUAD MOTOR DRIVER
GeoSoilEnviroCARS
The University of Chicago

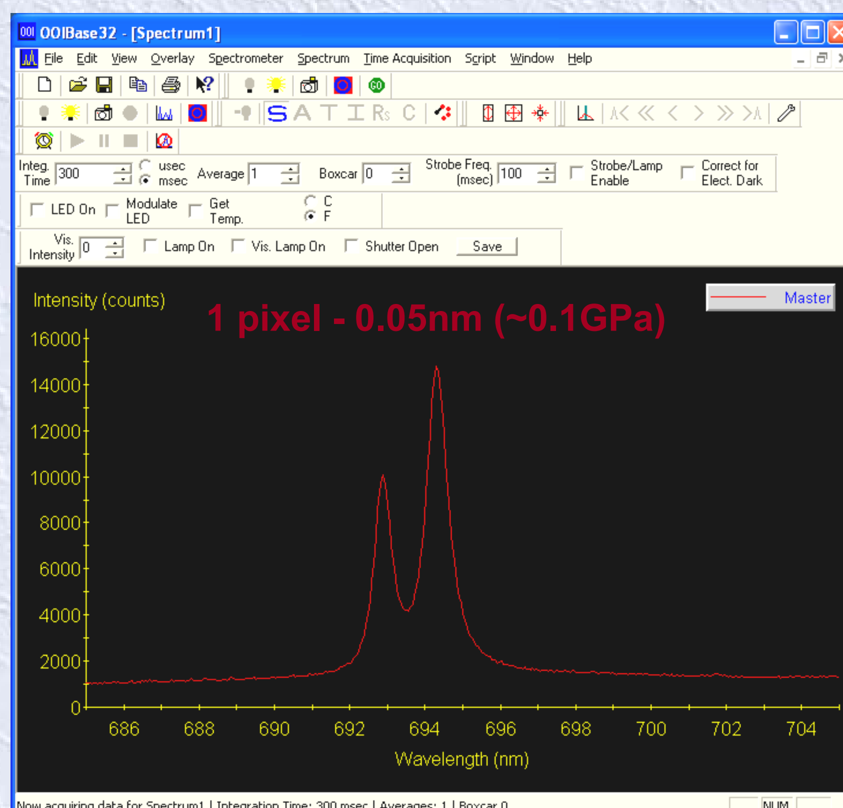

Cell Seated

The gases can be loaded at pressures up to 29000 PSI (~0.2 GPa)

- ➤*Able to load many kinds of cells*
- ➤*Closure mechanism with motor driven screws*
- ➤*Optical access for in-situ pressure measurements*
- ➤*Vacuum pump to clean system before loading*







Acknowledgements

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- Charlie Smith

Heavy fog, GSECARS, 8 a.m., October 2007