International Workshop on Synchrotron High-Pressure Mineral Physics and Material Science
The Advanced Photon Source, ANL - Dec. 6-7, 2007

X-ray Microtomography at High Pressure

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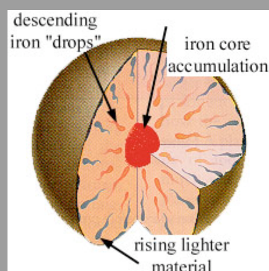


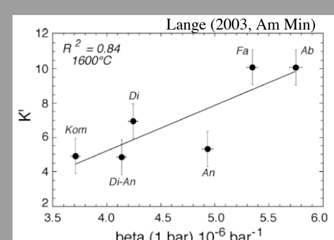
THE DEPARTMENT OF GEOLOGY UNIVERSITY OF CALIFORNIA, DAVIS

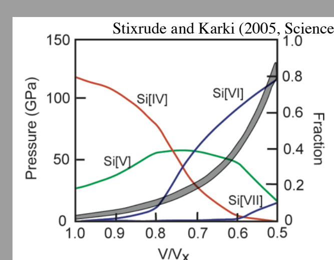
GeoSoilEnviroCARS-APS

Compressibility & thermal expansion of silicate melts - f(P,T)

- Fusion curve analysis
- Sink-float experiments
- Shock-compression
- Sound speed
- MD simulations

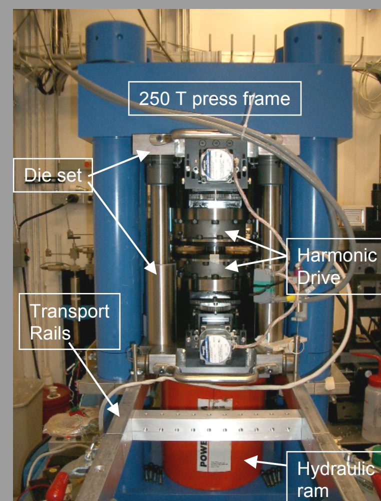
- X-ray absorption
- Direct volume determination (X-ray CT)





Instrument Requirements

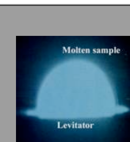
- Panoramic X-ray access under pressure
- At least 180° sample rotation, under pressure
- Ability to use various pressure cells
- Bulk characterization (at least mm sized samples)
- High temperature capability
- Deformation capability
- Mono- and polychromatic X-rays

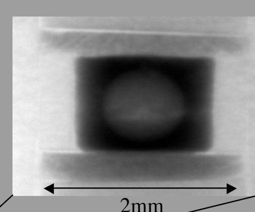


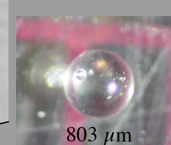
Rotating Anvil Apparatus (RAA)
13-BM-D, GSECARS-APS

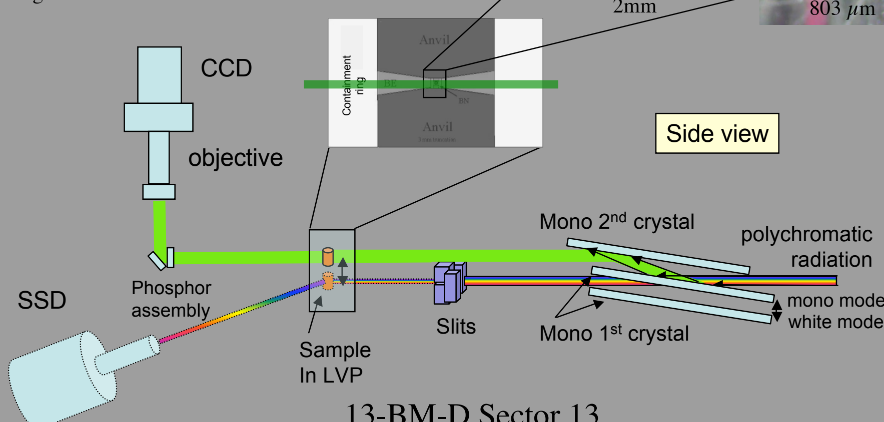
Wang, Y., T. Uchida, F. Westferro, M.L. Rivers, N. Nishiyama, J. Gebhardt, C.E. Lesher, S.R. Sutton (2005) High-Pressure X-ray Tomography Microscope: Synchrotron Computed Microtomography at High Pressure and Temperature, Rev. Sci. Instrum., **76**, 073709, doi: 10.1063/1.1979477.

- •Mono energy: 35-50 keV
- •Pixel size: ~1.5-2 microns unbinned; 3-4 microns 2x2 binned
- •Exposure times: 30 s unbinned; ~10 s 2x2 binned
- •Rotation range: 0°-179.5° (0.5°;steps)
- •Dark current/white field images collected before and after data collection, horizontally 3 – 4 mm from the sample center through borated epoxy and containment ring



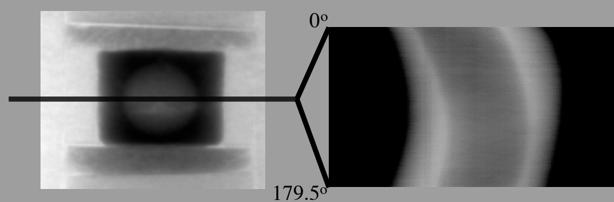






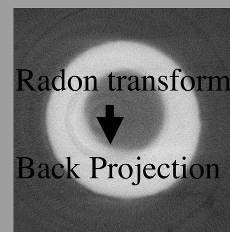
13-BM-D Sector 13

Computed Tomography



Radiograph

Signogram

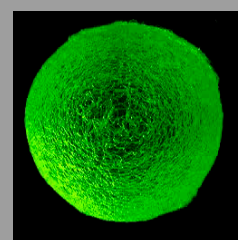


2-D map of μ_L

Beer-Lambert law: $dI/I = -\mu_L dx$

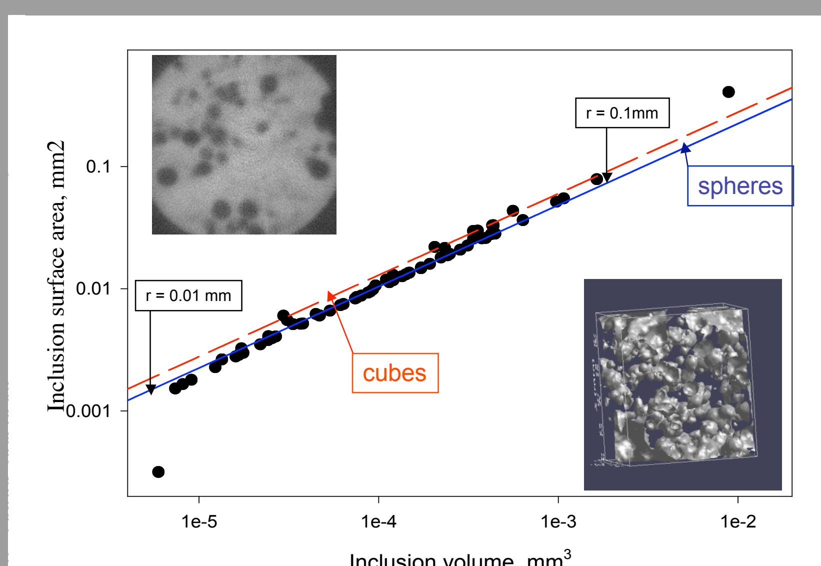
$$I = I_o \exp(-\mu_L x)$$

I_o - intensity of unattenuated radiation
I - intensity of transmitted flux after traversing thickness x
μ_L - linear attenuation coef. = mass absorption coef (μ_m)* mass density(ρ)



Rendered volume

Spatial resolution test – Blob3d extraction



Wang et al. (2005)

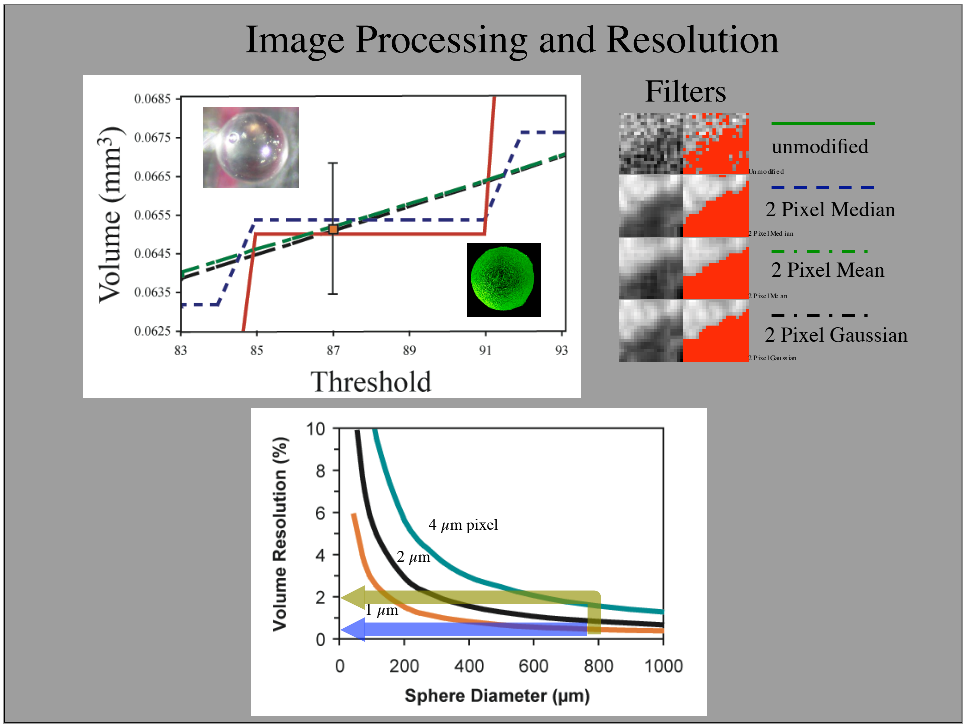
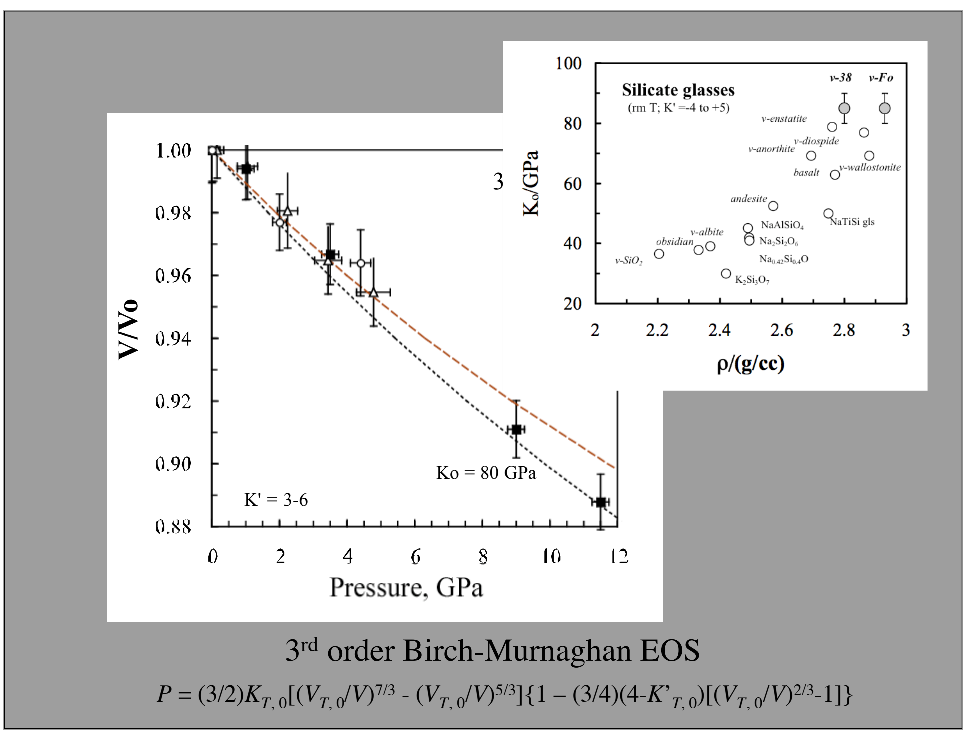
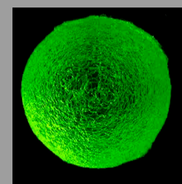


Image Processing and Resolution
Filters
Volume (mm³)
Threshold
unmodified
2 Pixel Median
2 Pixel Mean
2 Pixel Gaussian
Volume Resolution (%)
Sphere Diameter (µm)
4 µm pixel
2 µm
1 µm


Silicate glasses
(rm T; K' =-4 to +5)
v-38
v-Fo
v-enstatite
v-diopside
v-anorthite
basalt
v-wallostonite
andesite
NaTiSi gls
NaAlSiO₄
Na₂Si₂O₅
v-albite
obsidian
v-SiO₂
K₂Si₃O₇
K₀/GPa
ρ/(g/cc)
V/Vo
Pressure, GPa
Ko = 80 GPa
K' = 3-6
3rd order Birch-Murnaghan EOS
P = (3/2)K_{T,0}[(V_{T,0}/V)^{7/3} - (V_{T,0}/V)^{5/3}]{1 – (3/4)(4-K'_{T,0})[(V_{T,0}/V)^{2/3}-1]}

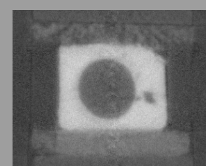
Direct volume determination:



But, there is another way:

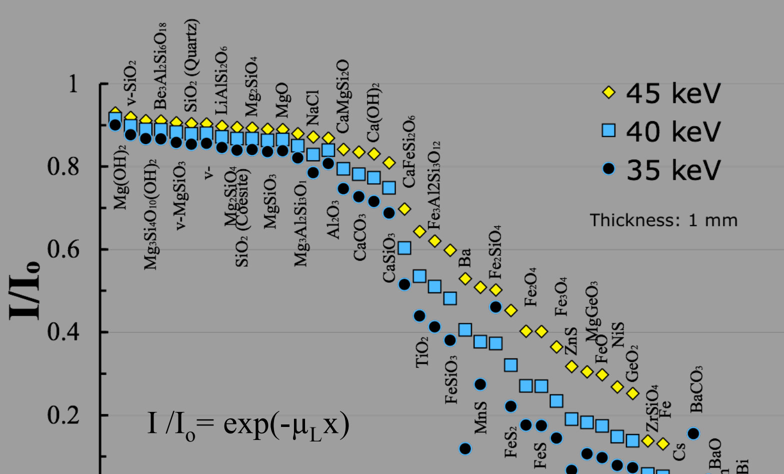
Absorption:

$\mu_L = \mu_M * \rho$

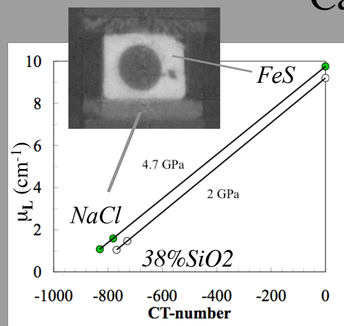


Requires *in situ* calibration:

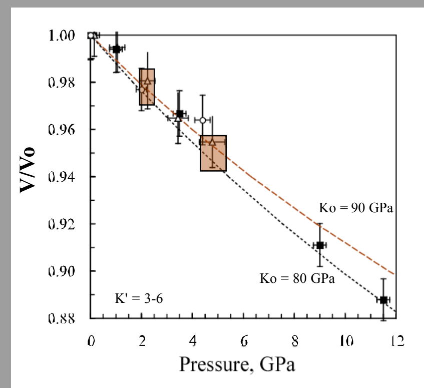
CT-number = $1000*[(\mu'_{L,i} - \mu'_{L,std})/\mu'_{L,std})]$



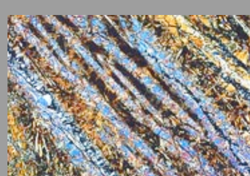
Calibration

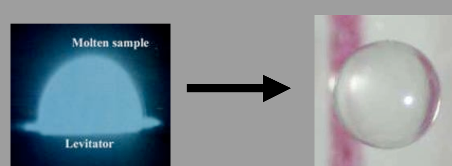


4.7 GPa (15 ton)	μ_L (cm^{-1})	μ_M (cm^{2}g^{-1})	ρ (g cm^{-3})
FeS	9.756	1.891	5.16
NaCl	1.584	0.624	2.54
38%SiO2	1.074	0.354	**3.03** (3.04)



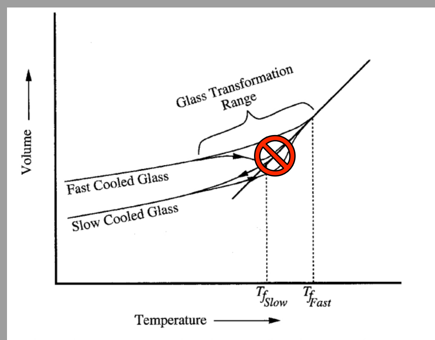
Melt properties by HPXMT

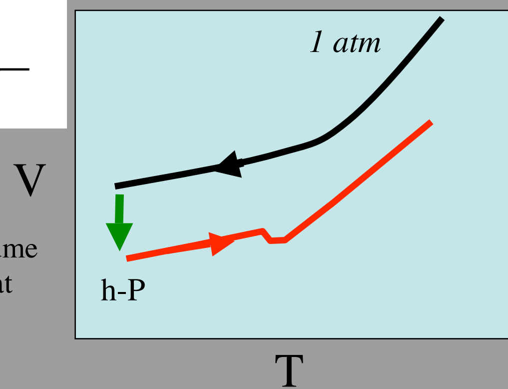




spinifex in komatiite

A possible strategy for refractory melts

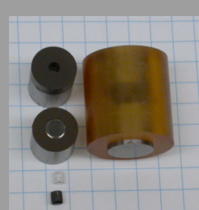


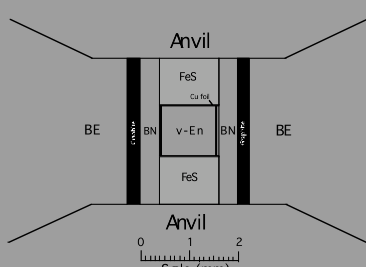


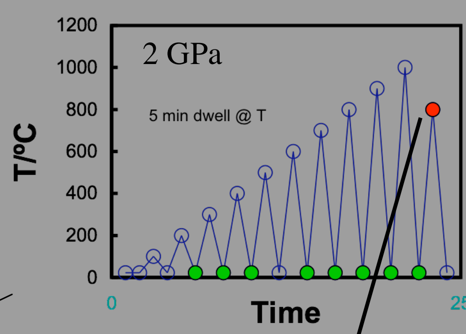
Can we detect changes in volume (density) passing through Tg at pressure by HPXCT?

Experimental Design

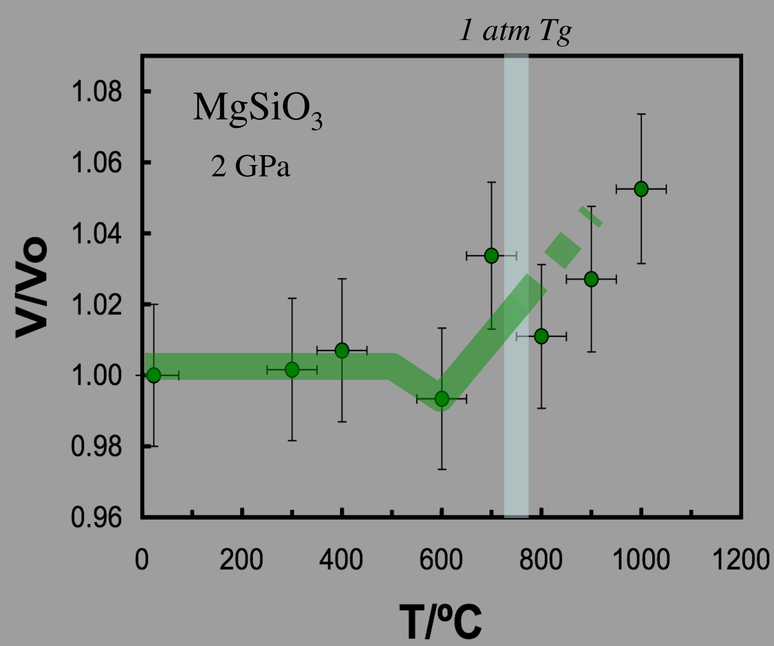
High T Drickamer Cell





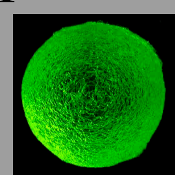


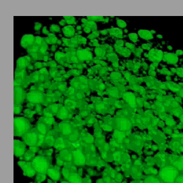
800º C/2 GPa

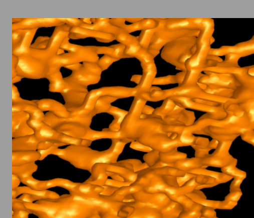


Present and Future for HPXMT

- P-V-T relations glasses/melts
  - Direct volume determination
  - Density from absorption
- Other applications
  - Textural/phase relations
  - Partial melting
  - Deformation
  - among others
- Diffraction/scattering
  - Monochromatic mode
  - CAESAR mode
- Ultrasonics
- Neutrons







Dunite + FeS melt