X-Ray Diamond Anvil Cell Facility at NSLS: 2010 Progress Report

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Beamline Scientists: Zhiqiang Chen, Xinguo Hong

2010 COMPRES Annual Meeting – Stevenson, WA

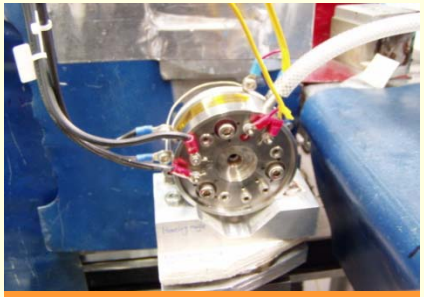
X17 DAC Facilities Overview

- Includes X17B3, X17C beamlines and Support Laboratory (serves for HPDAC research at X17B3, X17C, X14A, X27A, X11A and U2A)
- Powder X-ray Diffraction, Total Scattering Pair-Distribution Function (PDF) under high P and high T (Resistive Heating is available and Laser Heating System is being built up)
- ~60 user groups (over 150 users) from 50 international institutions annually, 5 new users in Sept-Dec 2010
- ~ 45 publications per year

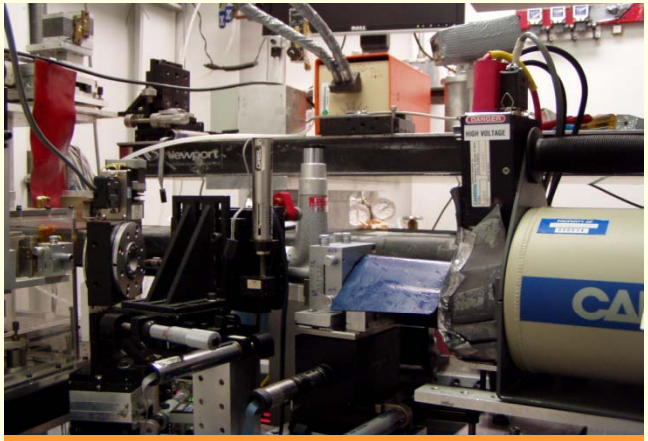
X17C Beamline

Main techniques: EDXD and ADXD on polycrystals under high P and high T
Studies of: EOS, phase transitions, structure refinements, yield strength, amorphization, texturing, compressibility

Angle Dispersive X-ray Diffraction by MarCCD


Hydrothermal DAC (Bassett)

R-Heating (upto ~900K)
Protected by H_2(4%)/Ar

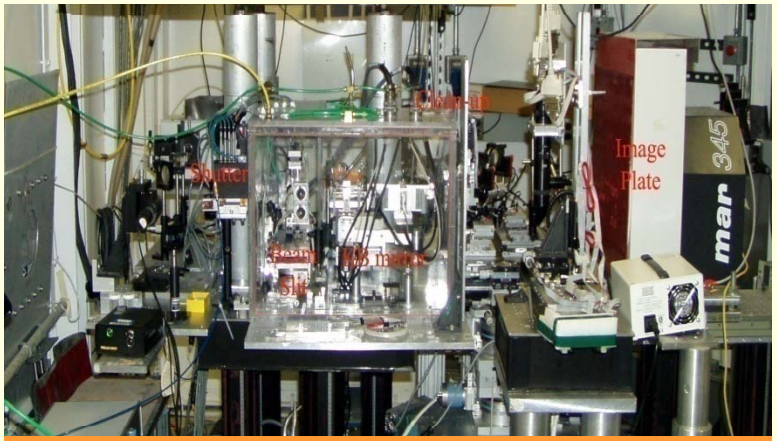

Energy Dispersive X-ray Diffraction by Ge detector

Monochromator	Sagittally bent Si Laue crystals
Beam energy	tunable 20 keV to 40 keV
Focusing mirror	K-B mirror
Primary beam size	0.90 mm x 0.90 mm
Focused beam size	0.025 mm x 0.020 mm
Detector	Rayonix SX-165 CCD detector

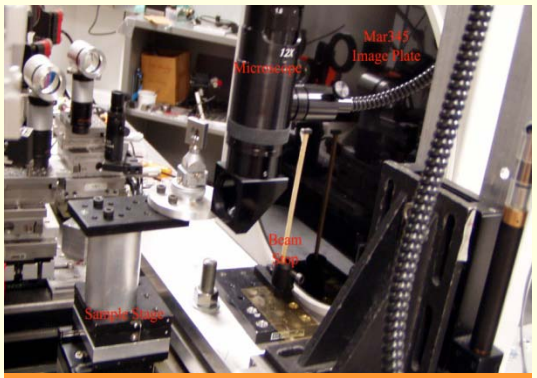
White beam E range	20 keV to 100 keV
Focusing mirror	K-B mirror
Primary beam size	0.070 mm x 0.070 mm
Focus beam size	0.025 mm x 0.020 mm
Detector	Ge solid state detector

X17B3 Beamline

Main techniques: EDXD and ADXD on polycrystals under high P and high T; High energy total x-ray scattering (PDF analysis),



ADXD Setup of Mar345 Image Plate



PDF Scattering

Angle-dispersive x-ray diffraction

Monochromator	Sagittally bent Si Laue crystals
Beam energy	30 keV/ 80 keV
Focusing mirror	K-B mirror (30 keV only)
Primary beam size	0.10 mm x 0.08 mm
Focused beam size	0.015mm x 0.010 mm (30 keV only)
Detector	MAR345 Image Plate/Perkin Elmer flat panel detector

Energy-dispersive x-ray diffraction

White beam E range	20 keV to 100 keV
Focusing mirror	K-B mirror
Primary beam size	0.10 mm x 0.080 mm
Focus beam size	0.010 mm x 0.010 mm
Detector	Ge solid state detector

Summary of Operation Status

- User agreement with NSLS: 50% general user time and 50% contributing user
- X-17C operates 100% of time:
- X-17B3 operates 33% time dedicated, 33% parasitic with X-17B2

	2009		2010	
	X17C	X17B3	X17C	X17B3
# proposals	48	18	55	24
# days requested	296	187	304	103
#days beamtime available *	210.73	120.66	195	100
Oversubscription	1.40	1.55	1.56	1.03
Funding **				
NSF	30	13	26	14
DOE	10	1	14	3
DOD	3	0	2	0
Foreign	9	4	16	7

- Available beamtime includes all beam setup, maintenance and development. 2010 beam time is reduced due to X17 wiggler cooling system leakage.
- ** Some proposals are supported by more than one funding.

Staffing

Sanjit K. Ghose (09/01/2008-12/31/2009)
current position: Assoc. Scientist at the XPD project beam line NSLS II
with 10% time at NSLS and member management Team

Zhiqiang Chen (joined 11/01/2008)
zchen@bnl.gov

Xinguo Hong (joined 06/01/2010)
xhong@bnl.gov

New Beamline Scientists joined and delivered responsibly and smoothly running of X17 DAC beamlines and support laboratory.

Beamline Developments in 2009-2010

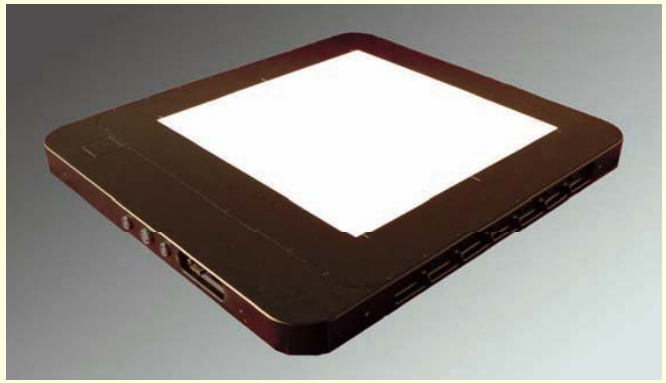
X17C: New area detector: Rayonix SX-165 CCD has been used since Mar 2009

Type	Single CCD; single fiber-optic taper
X-ray Sensitive Surface	Round, 165mm diameter
Number of Pixel	2048 x 2048
Pixel Size	80 x 80 μm
Read Noise	9 e-/pixel @ 3.5 sec. readout
Dark Current	<0.01 e-/pixel/sec. @ 2048 × 2048

Rayonix SX-165 CCD

X17B3: New COMs Perkin Elmer Detector is installed and being tested

Pixel number	2048 x 2048
Pitch	200 μm
Total area	409.6 x 409.6 mm^2
Frame rate (max)	15 Hz @ 200 μm
	30 Hz @ 400 μm
Radiation energy	20 keV – 15 MeV

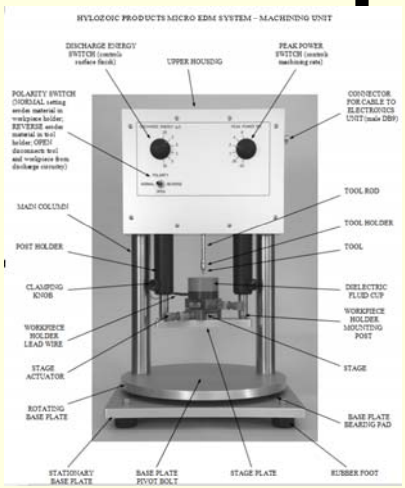


Perkin Elmer Flat Digital X-ray detector (XRD 1620 CN ES)

Sample Stages: All Kinematic Bases have been replaced by new magnetic model (BK-3A)

Support Laboratory Developments

- Sample Prepare Lab:
  - New Leica Microscopy (MZ165)
  - New Micro EDM system from Hylozoic Product
  - Several new types of DAC: Panoramic DAC, Short-piston Symmetric DAC, Almax Plate DAC



Micro EDM system

- Ruby Fluorescence System:
  - New Solid state Laser (Ventus, 250mW, 532nm)

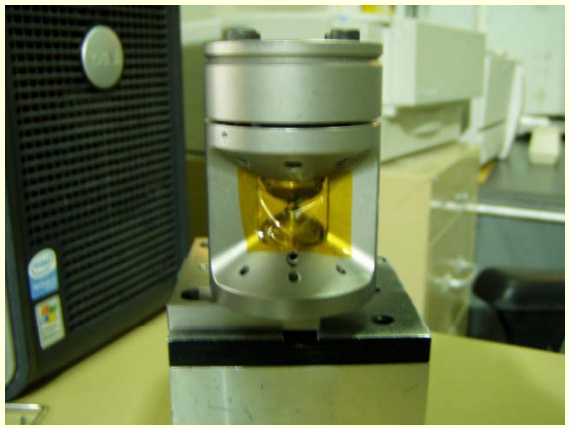
Working distance
59-97mm
Max Magnification
x 300
LED Ring Light
Analog Camera

Leica Microscopy (MZ165)

Diamond Anvil Cell Capabilities

- Symmetric DAC (Princeton)
- Short-piston DAC (Princeton)
- Panoramic DAC (Princeton)
- Plate DAC (Almax), on order

Short-Piston DAC (left) and Symmetric DAC (right)



Panoramic DAC

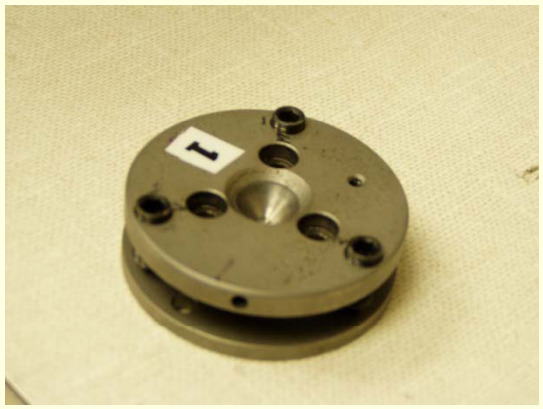
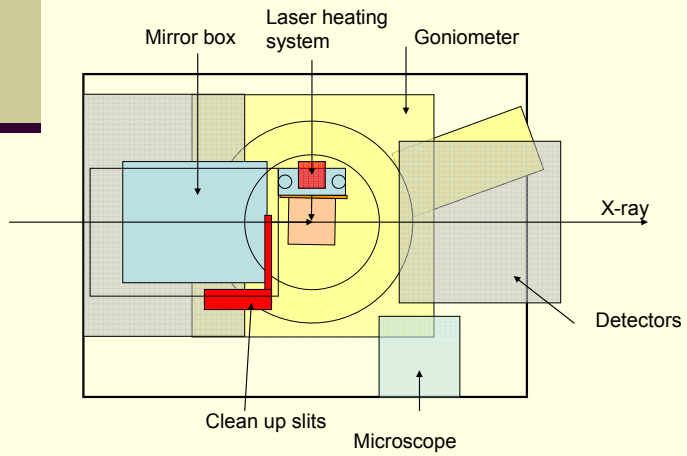


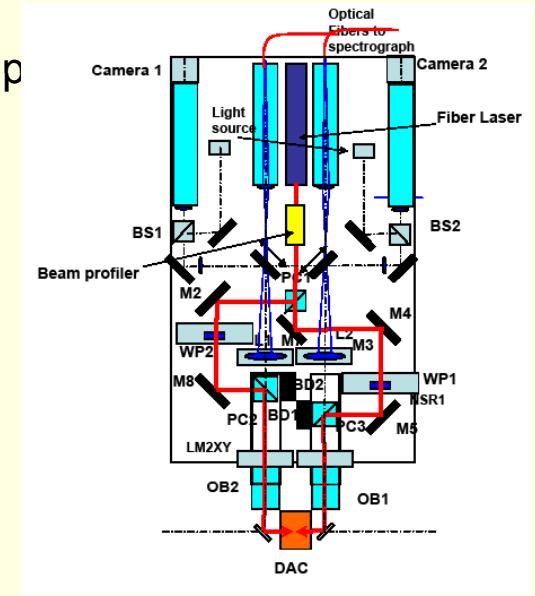
Plate DAC (Almax)

Laser Heating System Upgrade

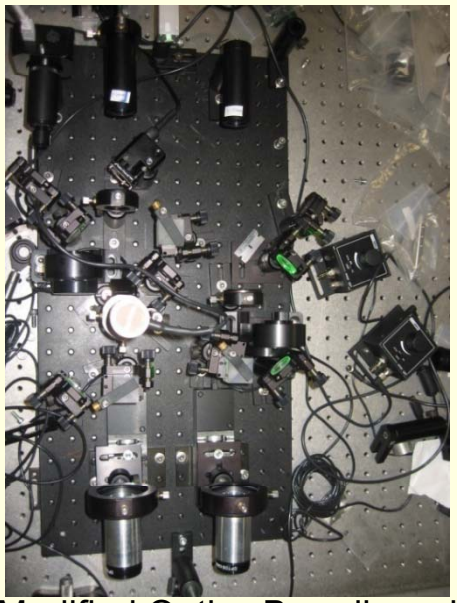
- Interlock system has been installed and Temp SOP for Laser Setup has been approved
- Fiber Laser (100W, 532nm) and shutter have been mounted and tested
- Portable optical breadboard (A. Gonchrov) has been assembled and now is being modified
- Spectrometer and CCD detector (Princeton Instrument) for T measurement is being tested
- New KB Mirror Enclosure is being built up



Layout of beamline setup



Schematic Optical Layout



Modified Optics Breadboard

2009 Stimulus Funds (ARRA)

COMPRES received ~ $850,000 in stimulus funds for the 4 facilities (X17B2, X17B3, X17C and U2A) of NSLS (average of $212,500 per facility)

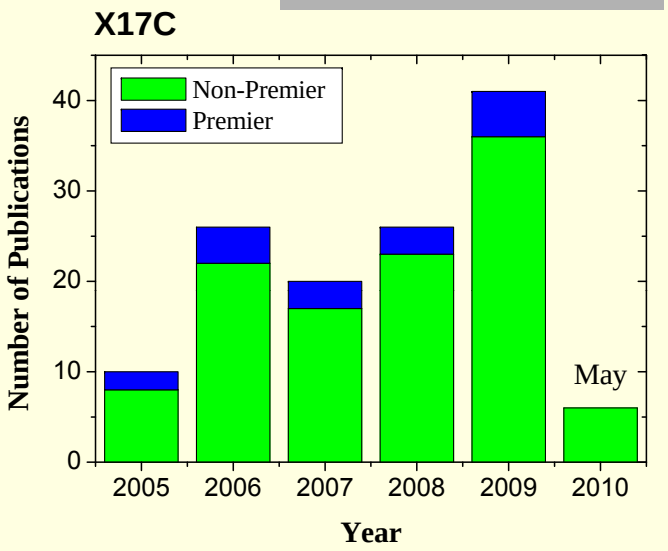
Flat Panel Detector (Perkin Elmer)	$119,000
Portable Ruby/Raman System	$25,000
Laser Heating System Upgrade	$29,200
Membrane Diamond Anvil Cell System, *on quoting*	$44,300
Networking upgrade, *on quoting*	$ 2,000
Total Request	**$219,500**
Already Spent	**~ $173,000**

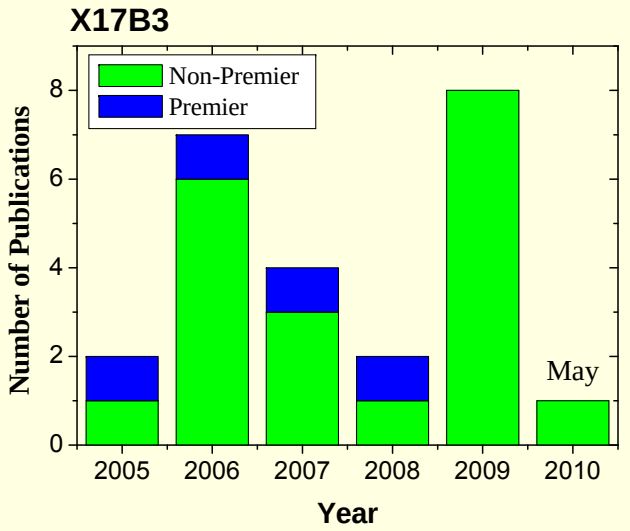
Development Plan (2010-2011)

- Network: improve network speed by a 24 port Gigabyte Switch and new CAT 5e or CAT 6 Ethernet cables
- X-ray beam transportation:
  - X-95 rails (New Port)
  - Lead Shielding
- Membrane Diamond Avail Cell:
  - Membrane capillary 1/32’ with micro-valve (Screw)
  - Pneumatic Drive system (200 bars)
  - External Heating system
- Cryostat: high pressure and low T (down to L He or L N_2)

X17 DAC – Publications

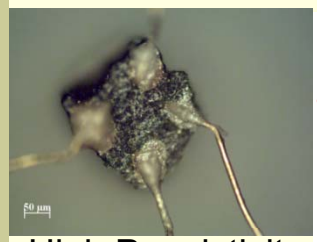
- **Since 2007 (COMPRES II era) :**
- **--12 PhD theses (+1 MS thesis)**
- **-- more than 90 Publications in the Peer-reviewed literature**
- -- **High-Profile Journals:** Science (1), Nature (2), Nature Materials (1), PNAS (2), Phys Rev. Lett. (3), Europhysics Letters (1)
- --**Earth Science**: American Mineralogist, Earth Planet. Sci. Letters, J. Geophys. Res., Phys. Earth Planet Int., Phys. Chem. Minerals
- --**Physics**: Physical Review B, Journal Physics Condensed Matter, J. Appl. Phys., Appl. Phys. Lett.
- --**Chemistry**: Chem. Phys. Lett., J. Solid State Chem., J. Am Chem. Soc., J. Chem. Phys.

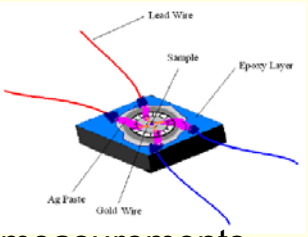




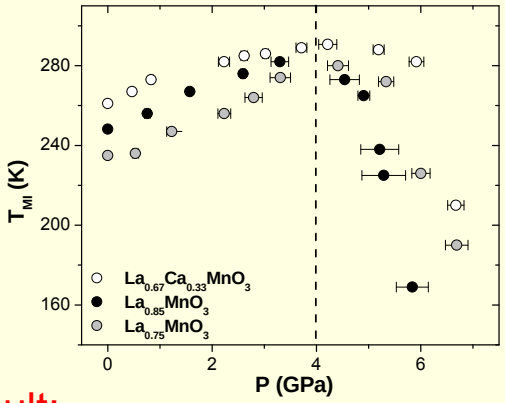
Non-linear Pressure Effects on CMR materials ($La_{1-x}Ca_xMnO_3$)

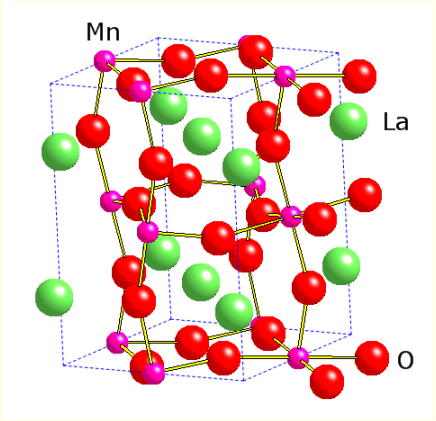
Purpose: study pressure effects on structure, magnetic and electronic properties of LCMO



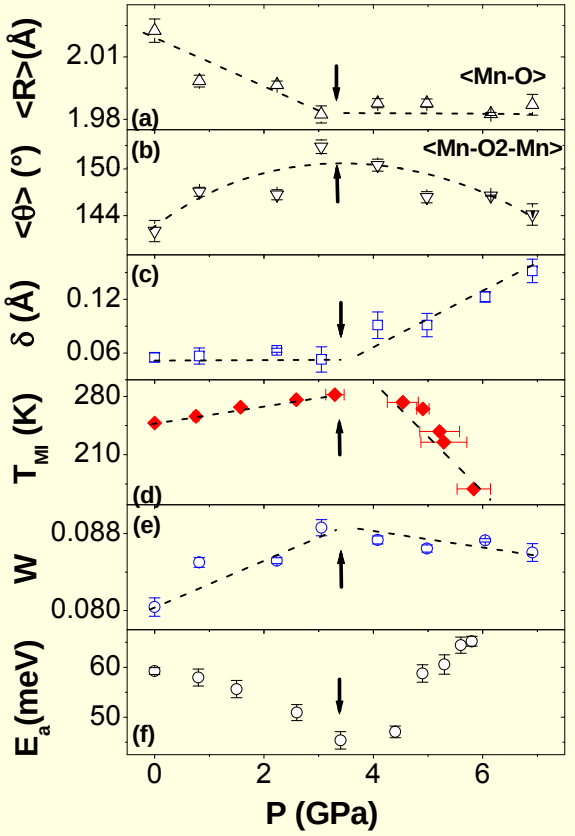


High P resistivity measurements





Monoclinic *I 2/a*



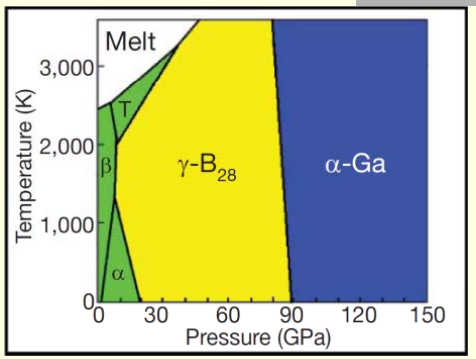
Result:

- T_{MI} of $La_{1-x}Ca_xMnO3$ is optimized near 3-4 GPa.
- At low-pressure range the bandwidth increases T_{MI}
- Above P^* the octahedral distortion decreases T_{MI}.

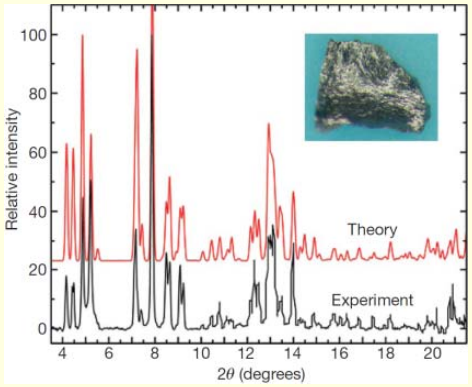
Z. Chen, T. A. Tyson et. al., to be published in Journal of Magnetism and Magnetic Materials

Ionic high-pressure form of elemental boron

- **Purpose**
  - To study the phase of Boron
- **Experiment**
  - In-situ high-pressure energy-dispersive XRD was performed at X17C of NSLS
- **Results**
  - High-pressure experiments and *ab* initio evolutionary crystal structure predictions reveal a partially ionic high pressure Boron phase. This new phase is stable between 19 and 89 GPa, and can be quenched to ambient conditions. It has a hitherto unknown structure (space group Pnnm, 28 atoms in the unit cell) consisting of icosahedral B12 clusters and B2 pairs in a NaCl-type arrangement.



Phase Diagram of Boron.

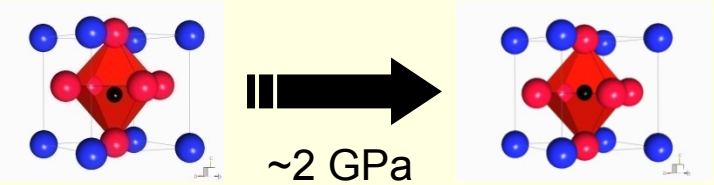


Calculated and measured XRD of γ-B28.

Artem R. Oganov and Yanzhang Ma, et. al., Nature,457,863(2009)

X-ray total scattering

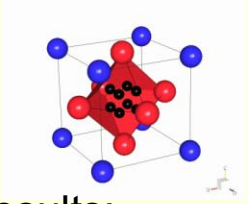
Pressure induced phase transition in $BaTiO_3$



tetragonal cubic

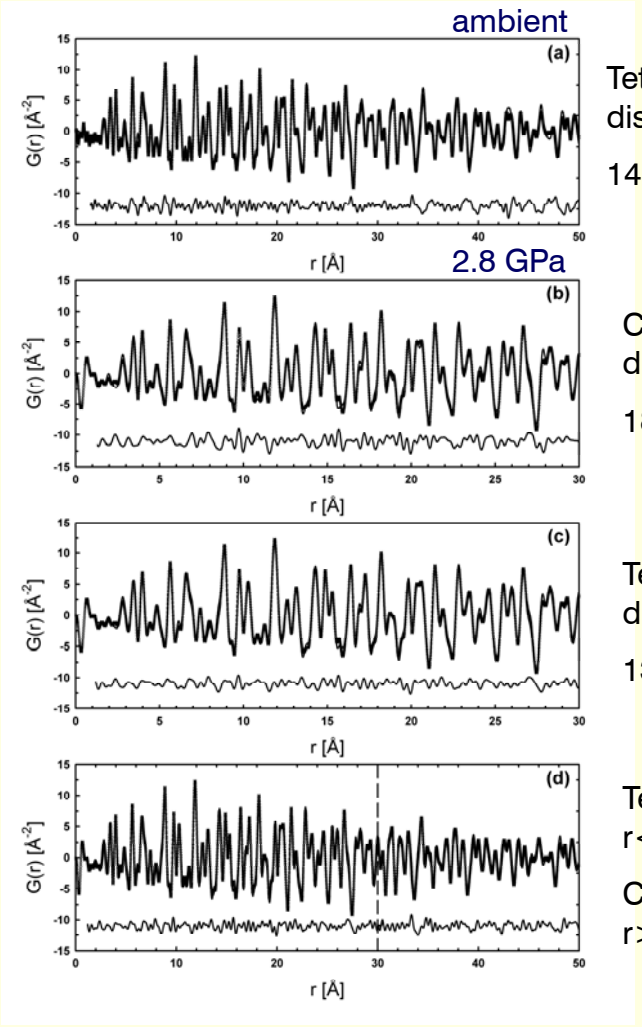
Differences of local and average structure

XAS shows Ti disorder in tetragonal and cubic phase → Ti displaced along {111}



HP XAS shows Ti displaces from center ~ 10 GPa

Diffuse scattering up to 11 GPa



Tetragonal disordered

14.7 %

Cubic disordered

18.1 %

Tetragonal disordered

13.4 %

Tetragonal r< 30 Å

Cubic r> 30 Å

Results:

Local high pressure structure is tetragonal r < 30Å

r < 30Å suggests existence of tetragonal (nano)-domains → domain size decreases with pressure

→ Domains disappear at 9.6 GPa

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