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LLNL-CONF-703240

# Time resolved x-ray diffraction: dynamics of pressure induced structural phase transition in bismuth.

Z. Jenei, W. J. Evans, Z. Konopkova, H. P. Liermann, Y. Kono

September 19, 2016

5th Workshop on High Pressure Planetary and Plasma Physics
Hamburg, Germany
September 14, 2016 through September 16, 2016

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# Time resolved x-ray diffraction: dynamics of pressure induced structural phase transition in bismuth.

5th High Pressure Planetary and Plasma Physics Workshop
Hamburg, Germany

Zsolt Jenei, William Evans
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Zuzana Konopkova, H-P. Liermann
*Extreme Conditions Beamline, DESY*
Y. Kono,
*HPCAT, Advanced Photon Source*

September 16, 2016

LLNL-PRES-703240
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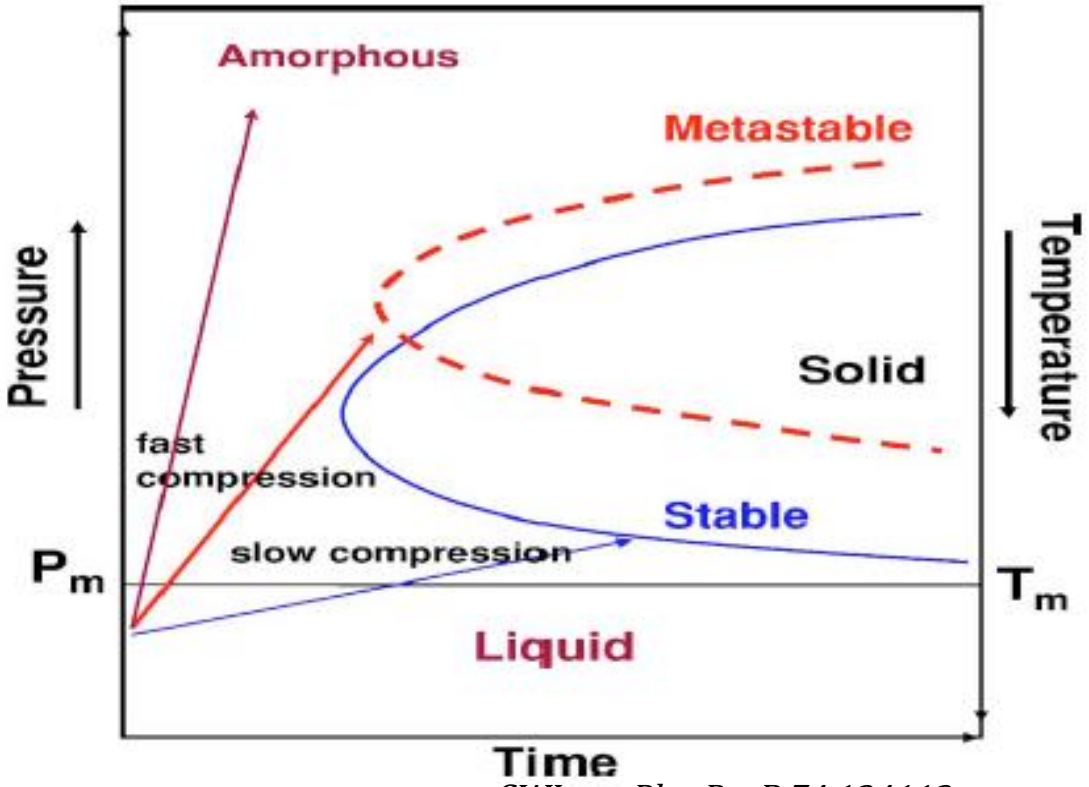
Lawrence Livermore National Laboratory

# Kinetic studies with the diamond anvil cell (DAC)

## Methods:

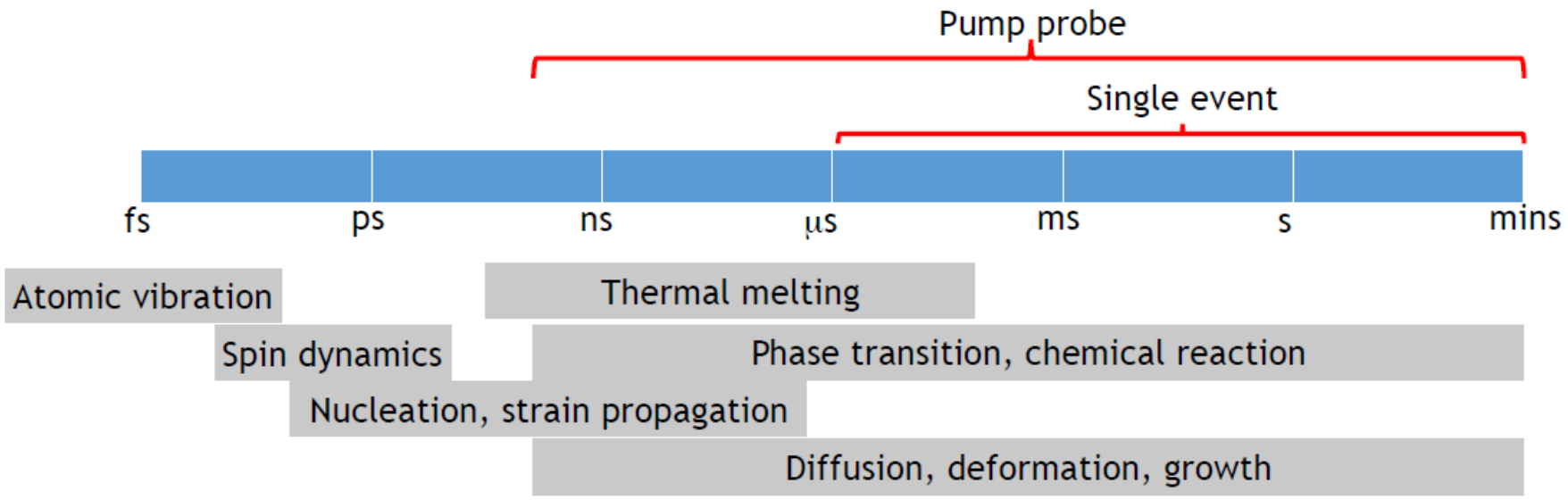
- Fast compression with gas membrane
- Piezo electric actuator driven diamond anvil cell (dDAC)




*GWLee - PhysRevB.74.134112*

d-DAC is capable of precisely controlling pressures and compression rates, which enables kinetic studies nearby the phase boundaries

# Time scale of dynamic phenomena



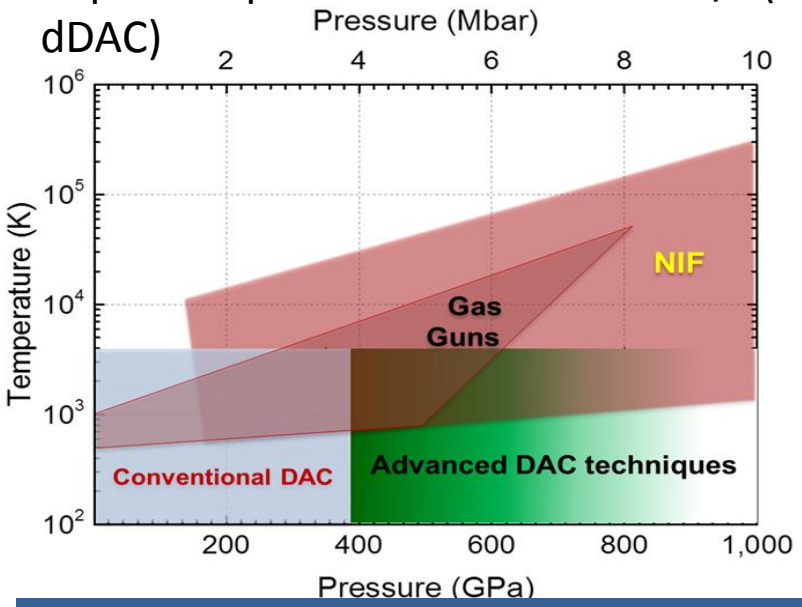

*GShen – ESRF dynamic workshop 2013*

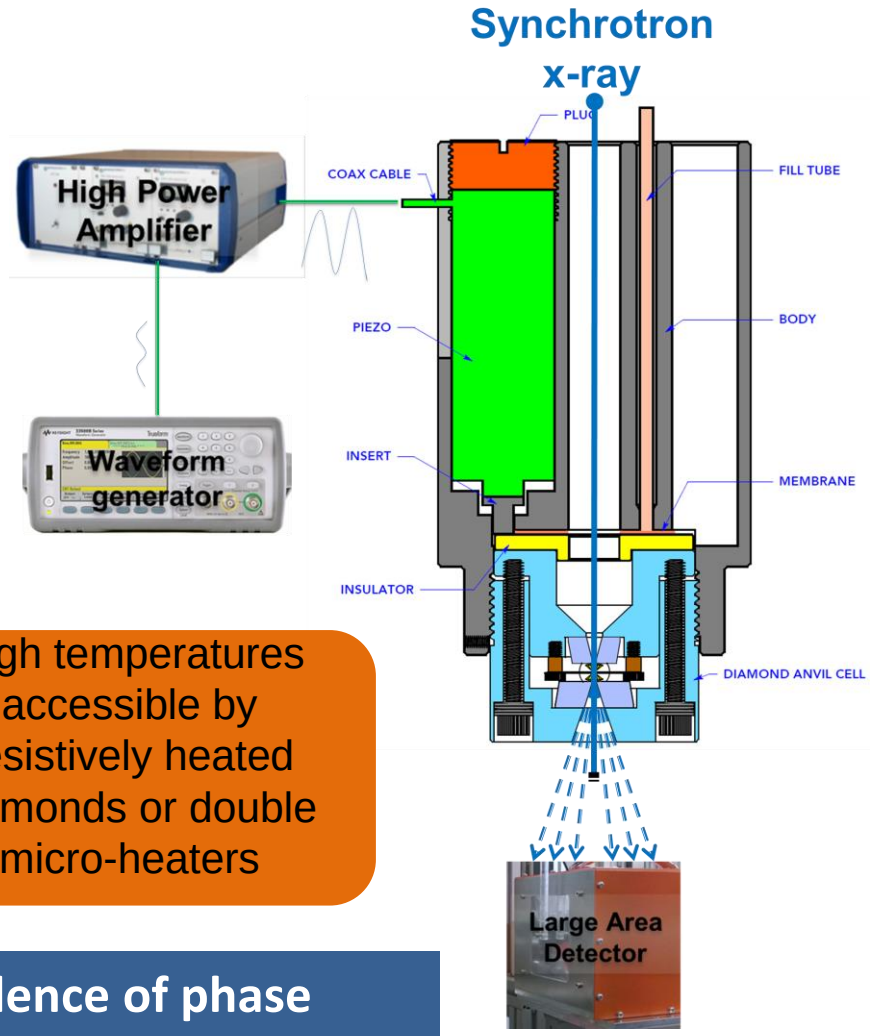
dDAC with 3rd gen x-ray light source

# Dynamic diamond anvil cell (d-DAC)

## Dynamic DAC

- conventional DAC + piezo actuators
- Accessible pressure range: **0-350 GPa**
- Precise control over pressure and compression rates
- Rapid compression rates 1000 GPa/s (DESY/ECB dDAC)




High temperatures accessible by resistively heated diamonds or double micro-heaters

**dDAC probes compression rate dependence of phase transitions along isotherms**

# Dynamic diamond anvil cell (d-DAC)

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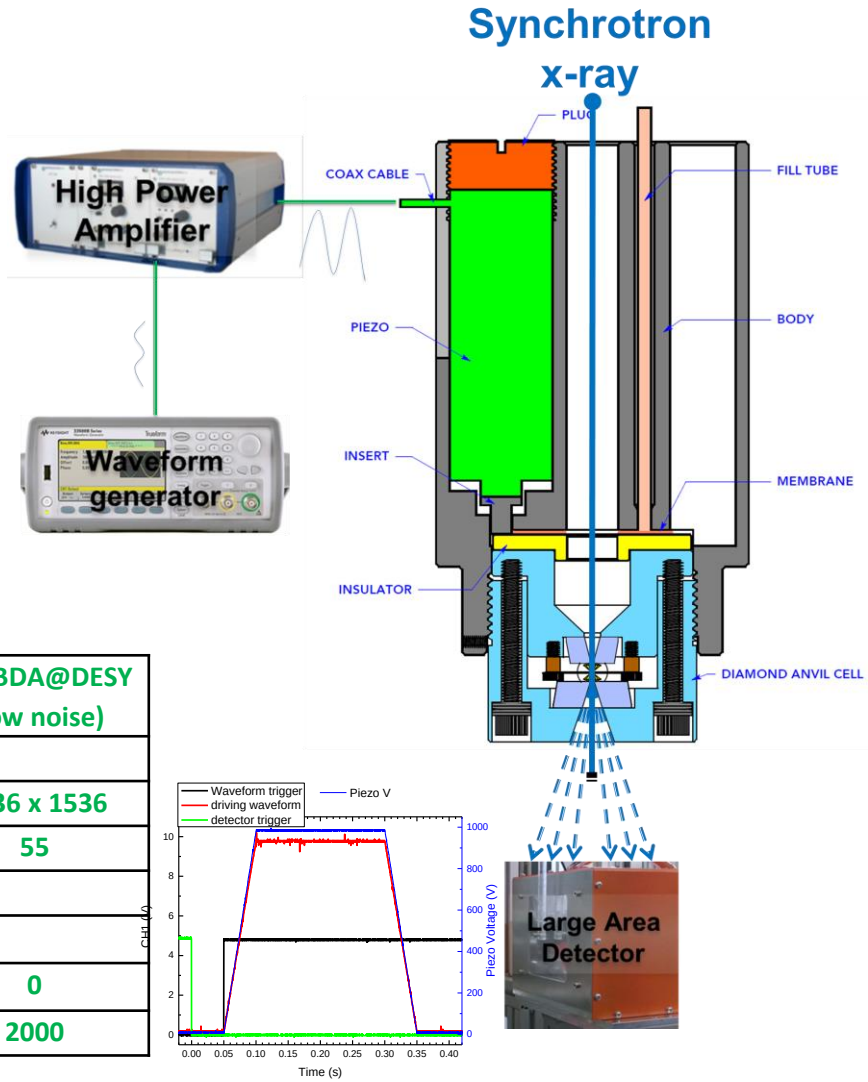
## Time resolved x-ray diffraction

- 3rd generation synchrotron (APS, DESY)
- XFEL (>20 keV)
- Fast Detectors




| DETECTOR | Pilatus (Low Background) | | | Perkin Elmer (HB) | LAMBDA@DESY (Low noise) |
|---|---|---|---|---|---|
| model | 100K | 300K | 1M | XRD 1621 | |
| Active size(pixels) | 487 x 195 | 487 x 619 | 981 x 1043 | 2048 x 2048 | 1536 x 1536 |
| Pixel size (μm) | 172 | | | 200 | 55 |
| Energy Range (keV) | 3-30 | | | 20-450 | |
| Quantum efficiency | 80%-99%(8keV)-12% | | | | |
| Readout time (ms) | 2.7 | 3.6 | 3.6 | | 0 |
| Frame rate (Hz) | >200 | 200 | 30 | 15-30 | 2000 |

# Dynamic diamond anvil cell (d-DAC)

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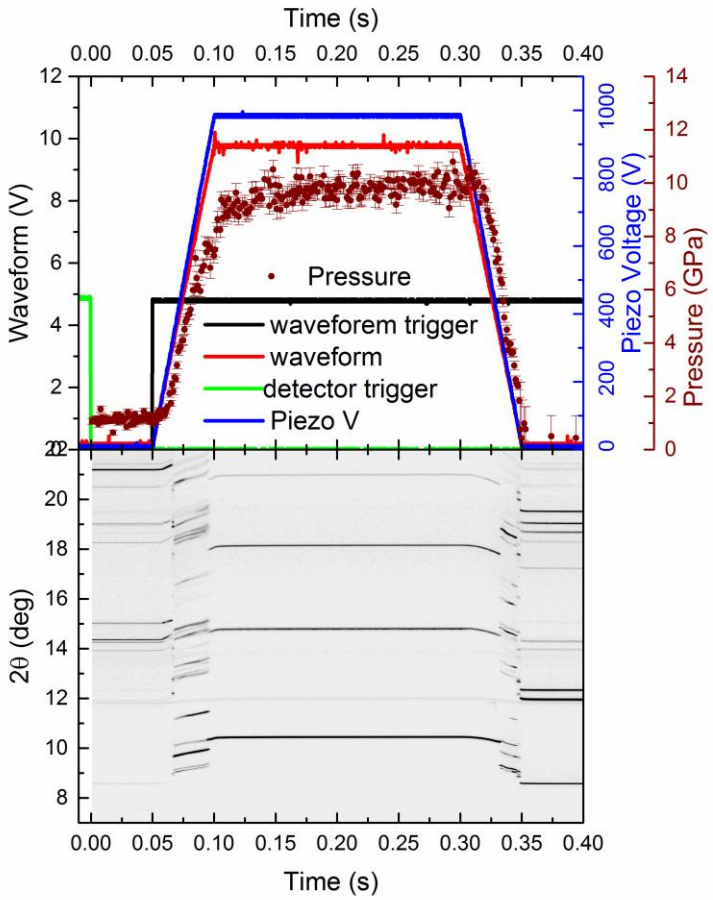
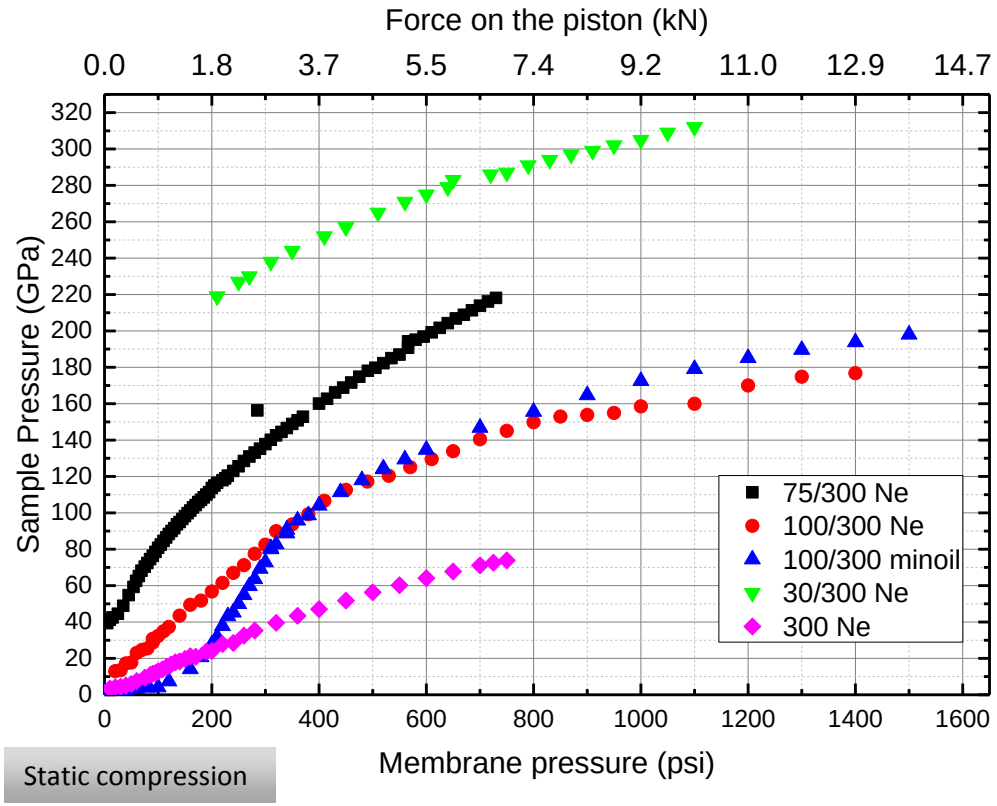


# Force requirements for multi-megabar pressures




Static compression

**Force on the piston :**

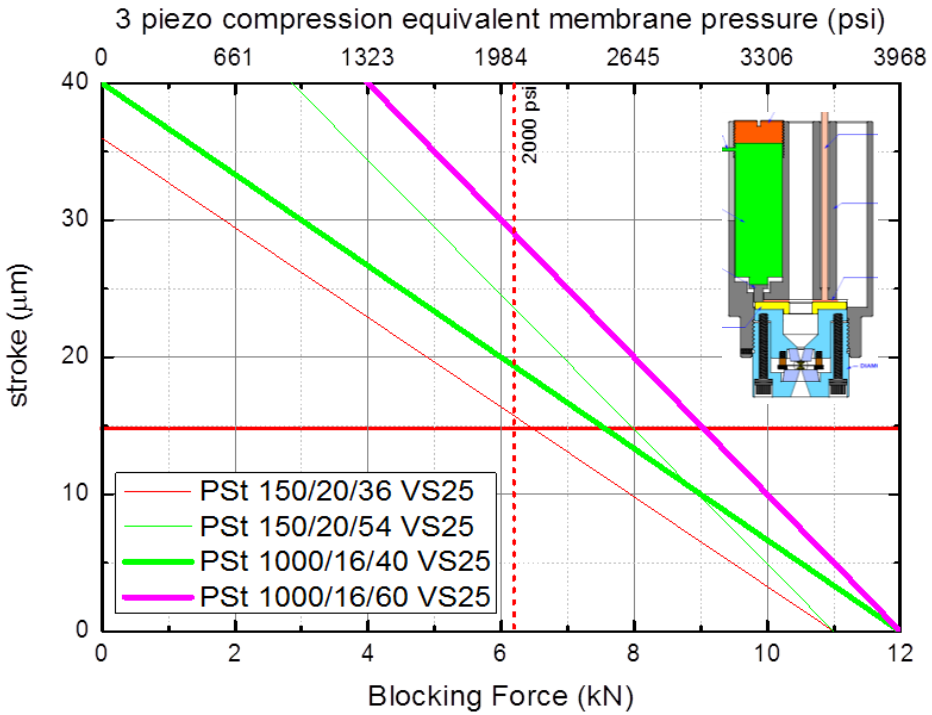
- 10 kN on the piston generates 320 GPa with a 30/300 culet and only 160 GPa with a 100/300 culet

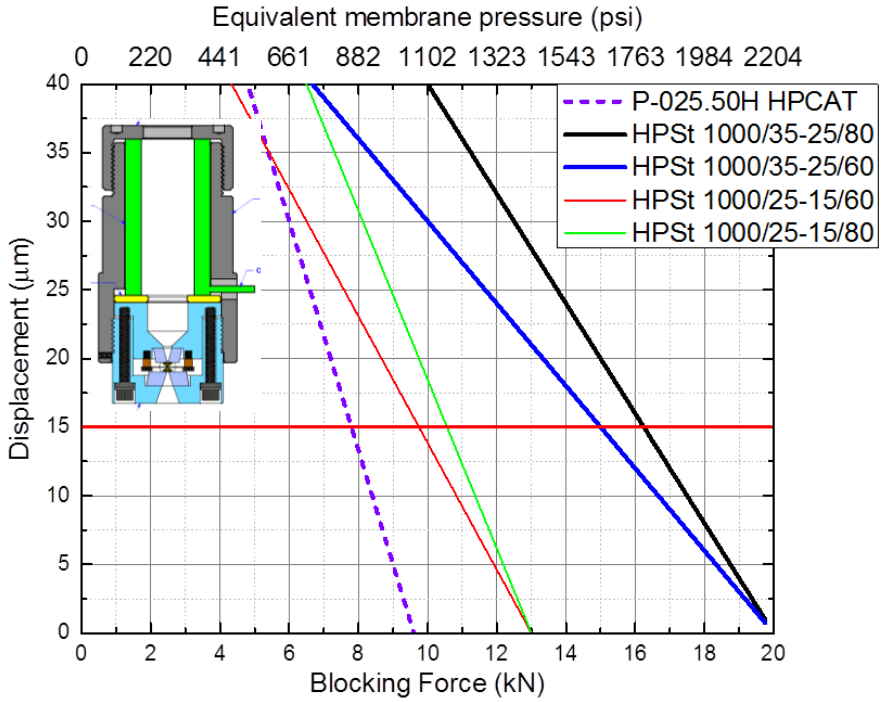
**Assumptions:**

- all the compression will be performed by the piezo actuators [no-pre pressurization with membrane]
- Start with a 20-23 µm thick gasket, sample at ~10 GPa ➔ expect at least 15 µm expansion of the piezo actuator

For multi-megabar pressures piezoactuators need to deliver more than 10 kN force (when expanded at max pressure in the dDAC assembly)

# Aim to deliver more than 10 kN for single piezo and close to 20 kN with 3 piezo configuration



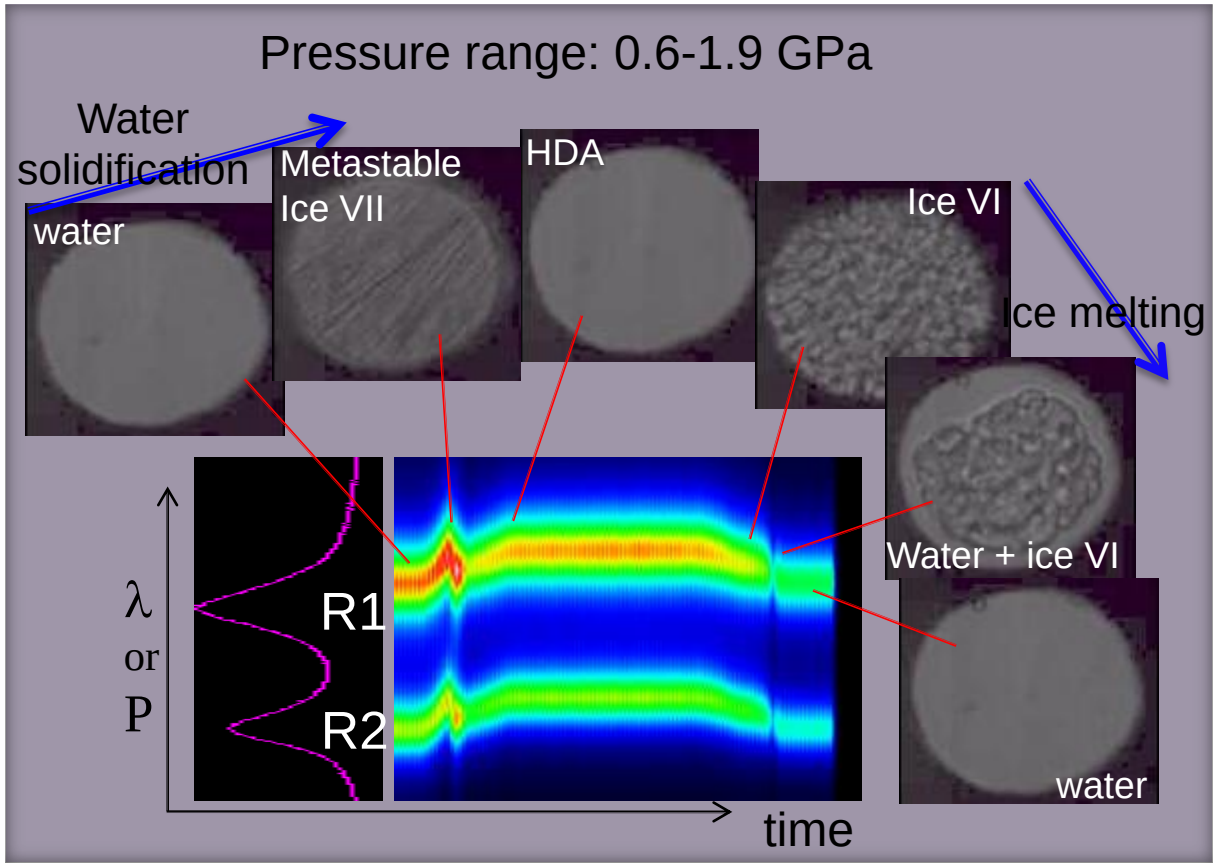



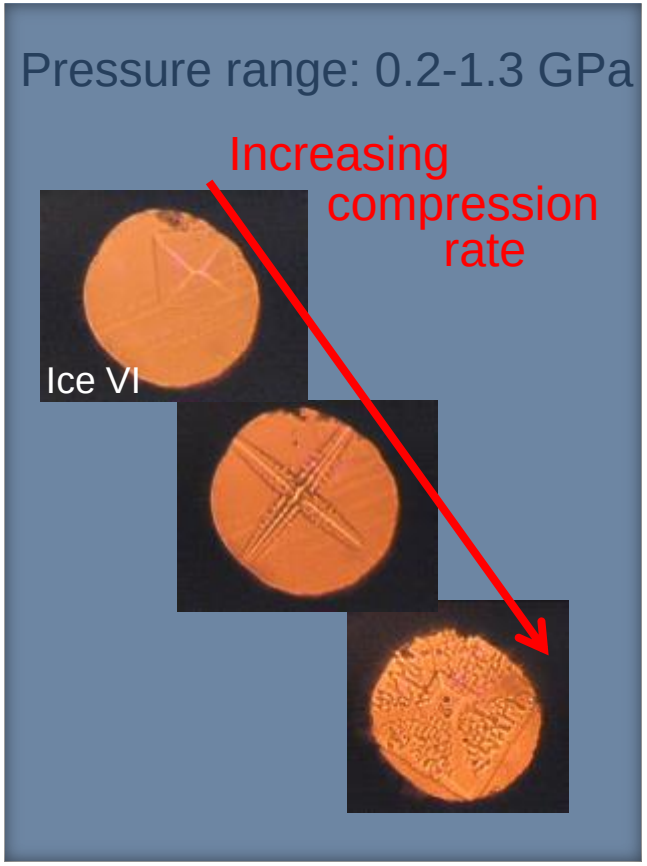

PSt 1000/16/60, 1kV 7A:
Blocking force: approx. 5…6 kN [1500-1900 psi eq.]
Electrical rise time for 3 actuators: **450 µs**

1x HPSt 1000/35-25/80, 1kV 7A:
Blocking force: approx. 10…11 kN [~1200 psi eq.]
Electrical rise time for 3 actuators: **370 µs**

Compression rates of $1–3 \cdot 10^2$ GPa/s can be achieved

# Metastable high-pressure phases at low pressure are observed under rapid compression in $H_2O$



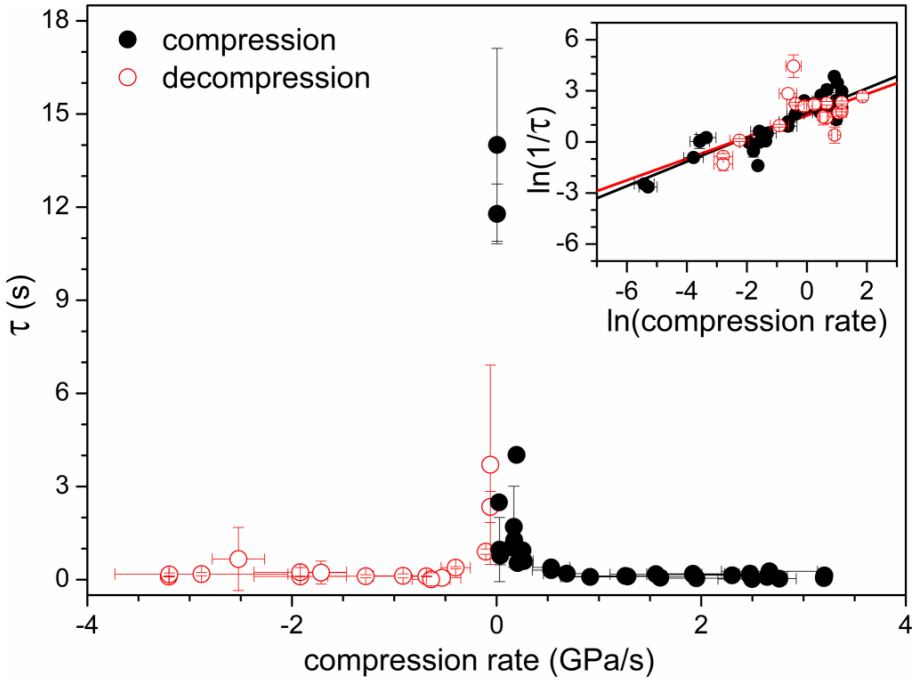




*Geun Woo Lee, William J. Evans, and Choong-Shik Yoo, Phys. Rev. B* ***74****, 134112, (2006).*
*Geun Woo Lee, William J. Evans, and Choong-Shik Yoo, PNAS* ***104****, 9178, (2007).*
*Jing-Yin Chen and Choong-Shik Yoo, PNAS* ***108****, 7685, (2011).*

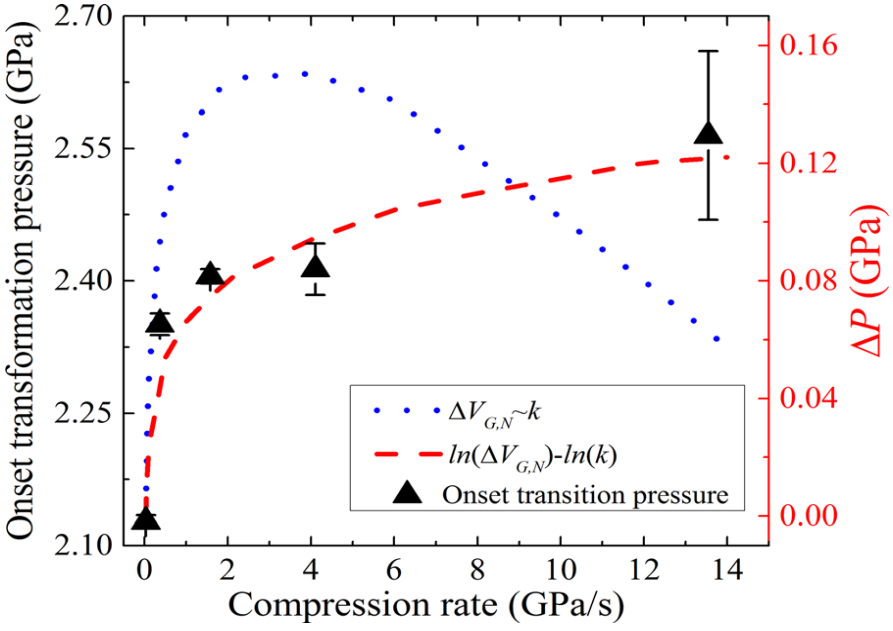
Crystal texture and growth direction is dependent on compression rates

Slide from J.Y Chen

# Compression rate dependence on phase transition pressure in Kr and KCl




*J.Y. Chen et al – PHYS. REV. B 90, 144104 (2014)*

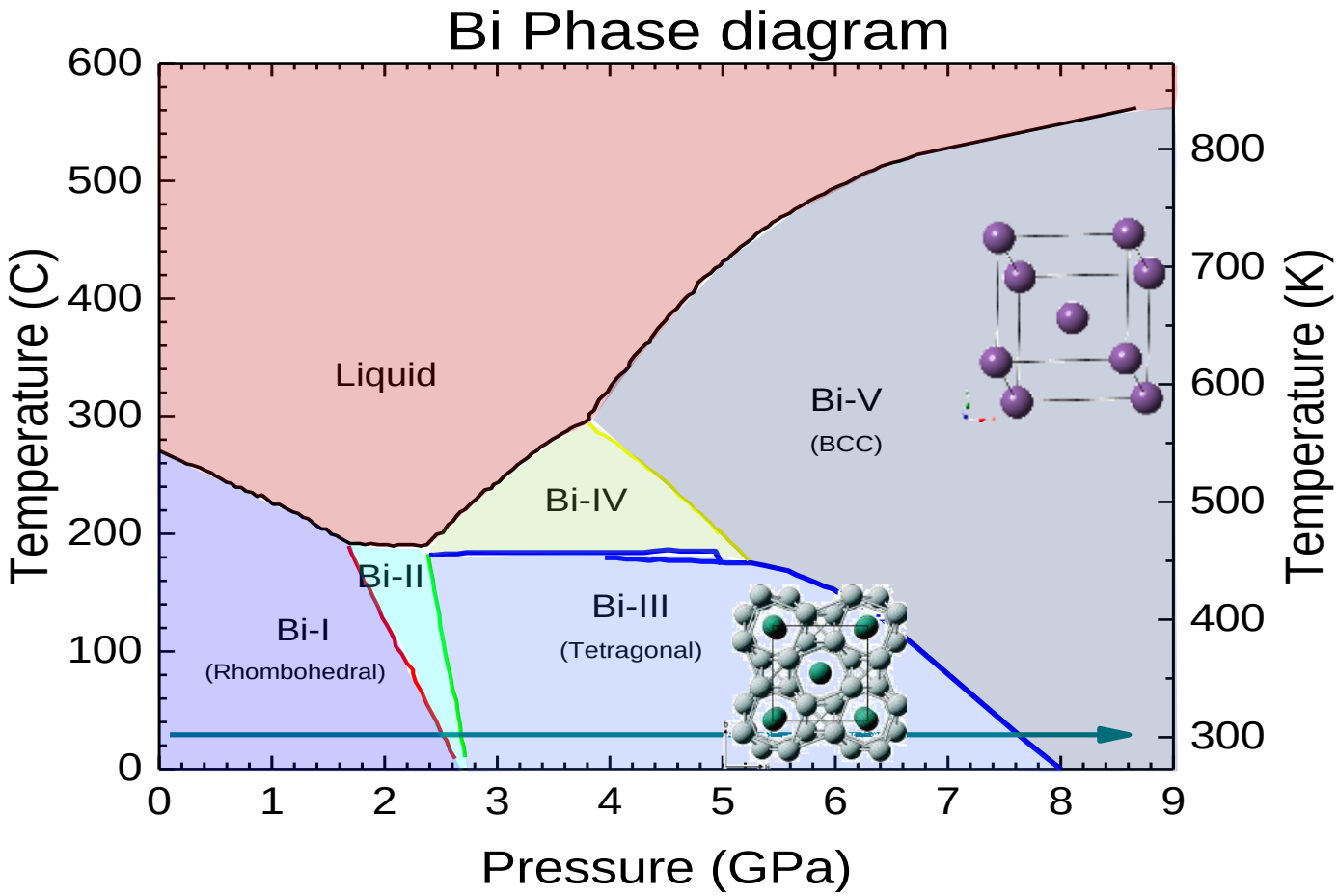



*Journal of Applied Physics 119, 045902 (2016);*

Strong compression-rate dependence of the solidification/melting process in liquid Kr.

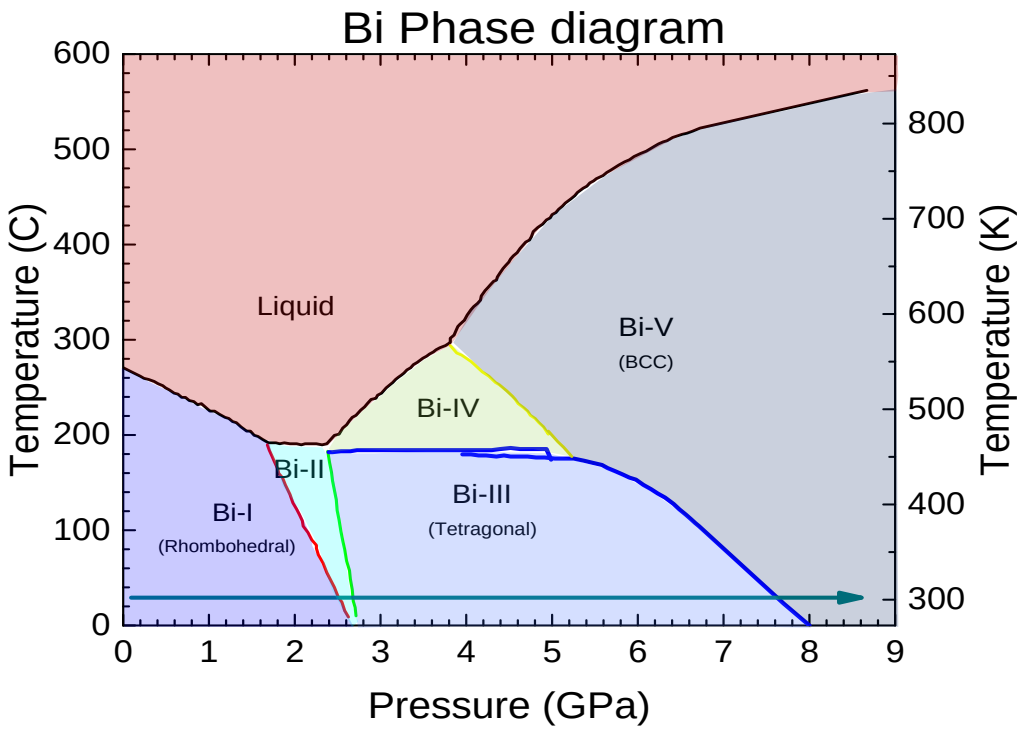
FIG. 10. The over-pressurization effect reflected by the onset phase transformation pressures at various compression rates ($k$). Black triangles are experimental results from this study. Lines represent the transition pressure

# Bismuth phase diagram – compression path




Phase transition pressures static:
I→II 2.5 GPa; II→III: 2.7 GPa; III→V 7.7 GPa

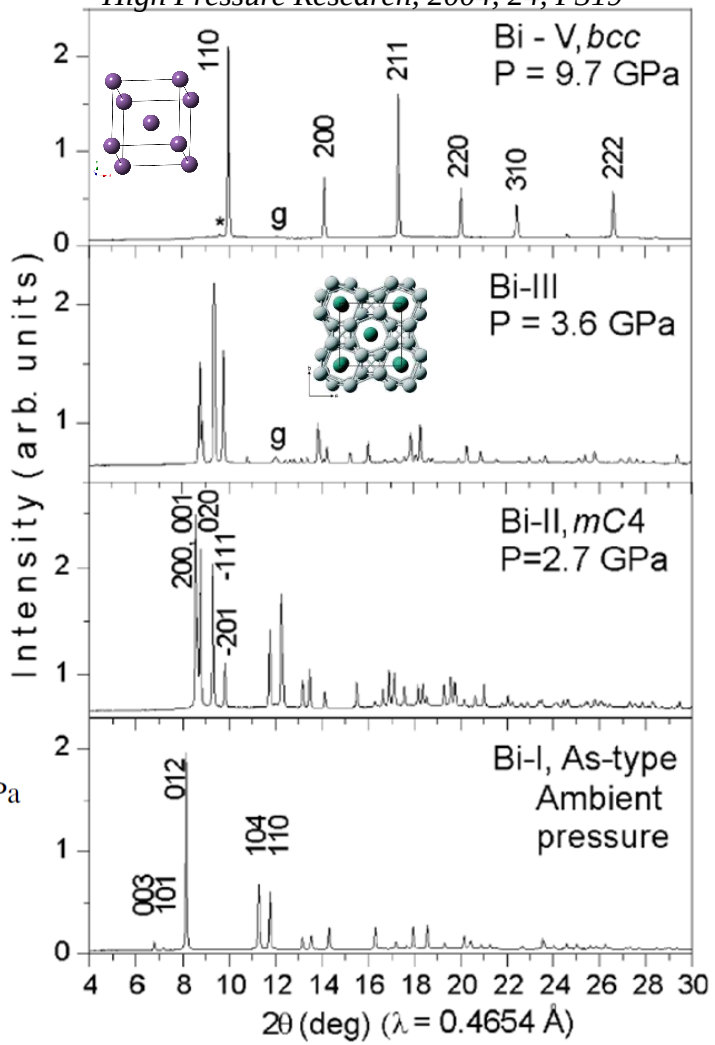
# Bismuth phase diagram – compression path




Bi: Bi-I (As-type, $hR2$) —2.55 GPa→ Bi-II ($mC4$) —2.7 GPa→ Bi-III (tetr. host–guest) —7.7 GPa→ Bi-V (bcc, $cI2$) < 220 GPa

Phase transition pressures static:
I→II 2.5 GPa; II→III: 2.7 GPa; III→V 7.7 GPa

*High Pressure Research, 2004, 24, P319*



# Under dynamic compression phase lines shift

PRL 101, 065701 (2008) PHYSICAL REVIEW LETTERS week ending 8 AUGUST 2008

## Ultrafast Dynamic Compression Technique to Study the Kinetics of Phase Transformations in Bismuth

R. F. Smith, J. H. Eggert, M. D. Saculla, A. F. Jankowski, M. Bastea, D. G. Hicks, and G. W. Collins

*Lawrence Livermore National Laboratory, P.O. Box 808, Livermore, California 94550, USA*

(Received 29 February 2008; revised manuscript received 25 June 2008; published 8 August 2008)

Preheated Bi (296–532 K) was ramp compressed with 15–35 ns rise times to a peak stress of ~11 GPa to explore structural phase-transformation kinetics under dynamic loading conditions. At high strain rates, $\dot{\epsilon} > 5 \times 10^6$ $s^{-1}$, deviation from equilibrium phase boundaries suggests that compression time scales are comparable to the new phase incubation period. The dependence of $\Delta P/kT$ on $\dot{\epsilon}$ is consistent with a thermally activated transformation.

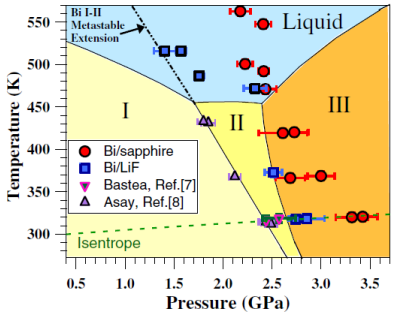



FIG. 4 (color online). *P-T* plot of Bi shown with I, II, and liquid equilibrium phase boundaries. Points from a series of preheated Bi samples are shown for the two different window materials used (LiF, sapphire) along with the points taken by Bastea [7] and Asay [8].

*High Pressure Research*, 1990, Vol. 4, pp. 336–338
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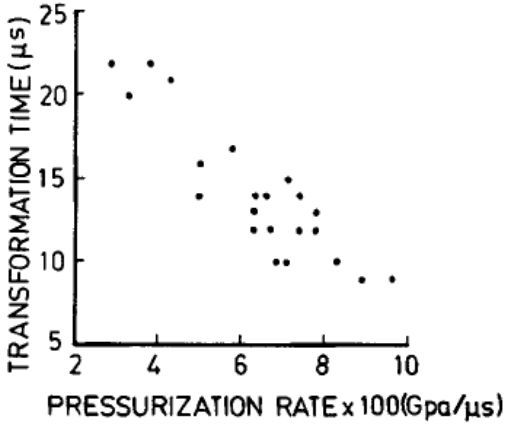
## KINETICS OF Bi(I) → Bi(II) TRANSITION UNDER VERY HIGH PRESSURIZATION RATES

ANIL K. SINGH

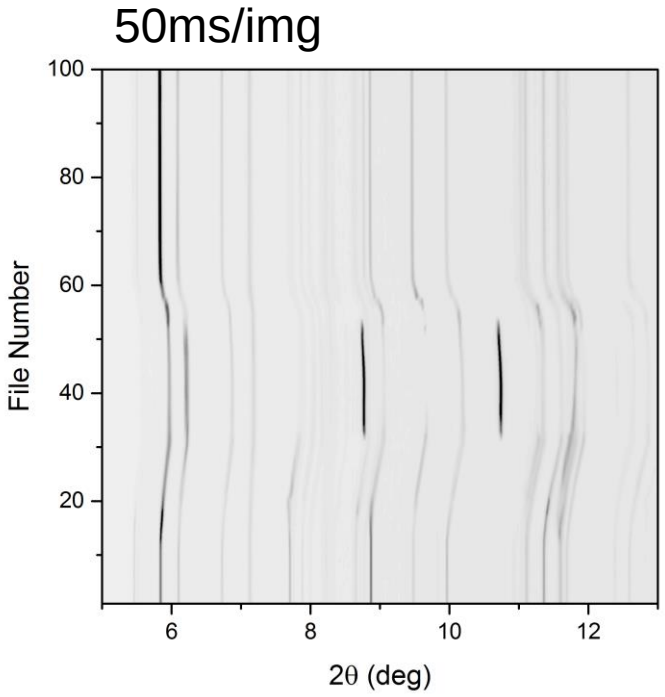
*Materials Science Division, National Aeronautical Laboratory, Bangalore 560017, INDIA.*

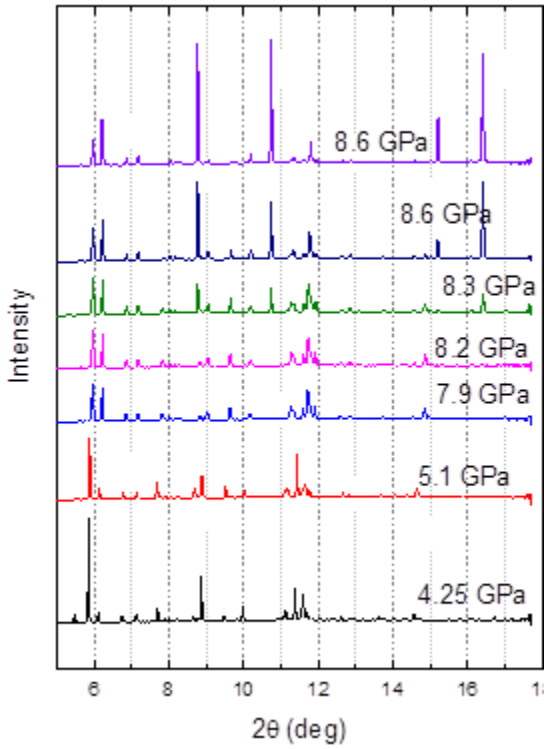
The occurrence of Bi(I) → Bi(II) transition has been examined under constant pressurization rates in the range 0.03 to 0.1GPa/(μs) by monitoring the electrical resistance of the specimen as a function of time. Such high pressurization rates have been obtained by impacting a tungsten carbide opposed anvil setup with a low velocity projectile [Rev. Sci. Instum. **60**, 253 (1989)]. The time taken for the completion of the transition decreases with the increasing pressurization rate.

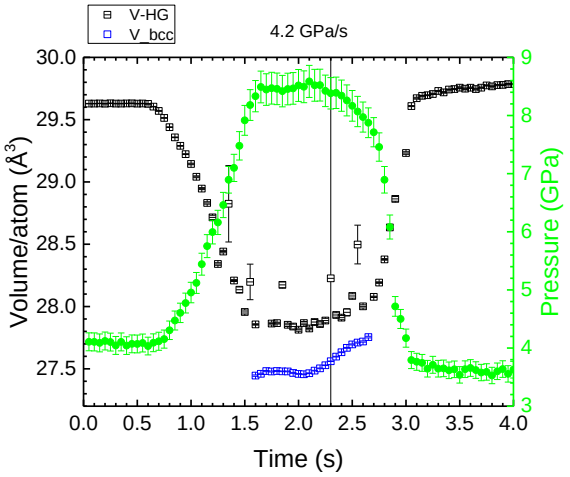


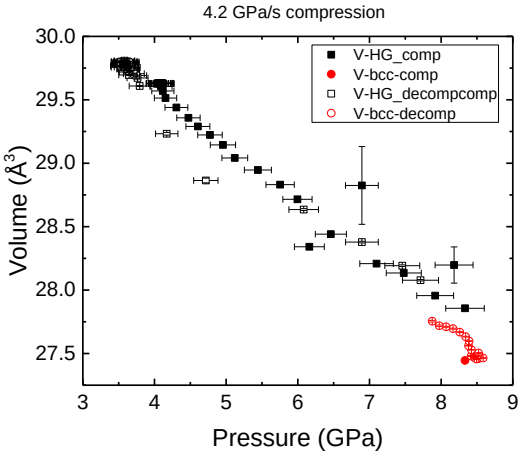
# Kinetic studies of Bismuth ~~I-II~~-III-V phase transitions

## 4 GPa/s



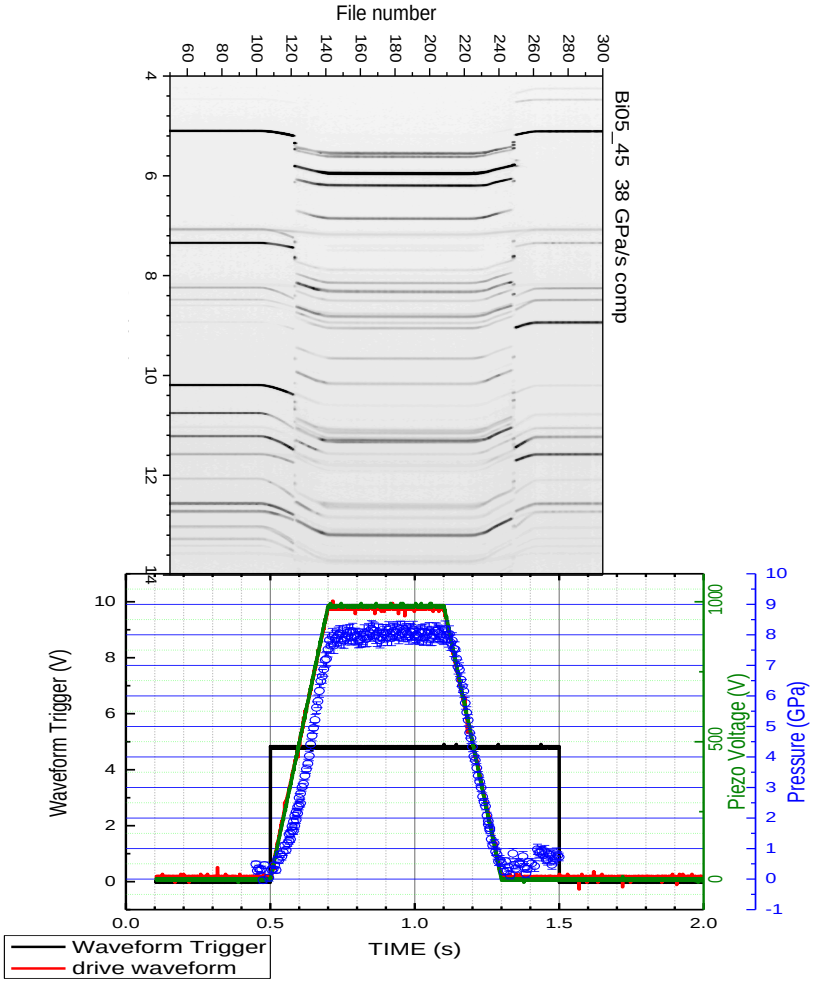



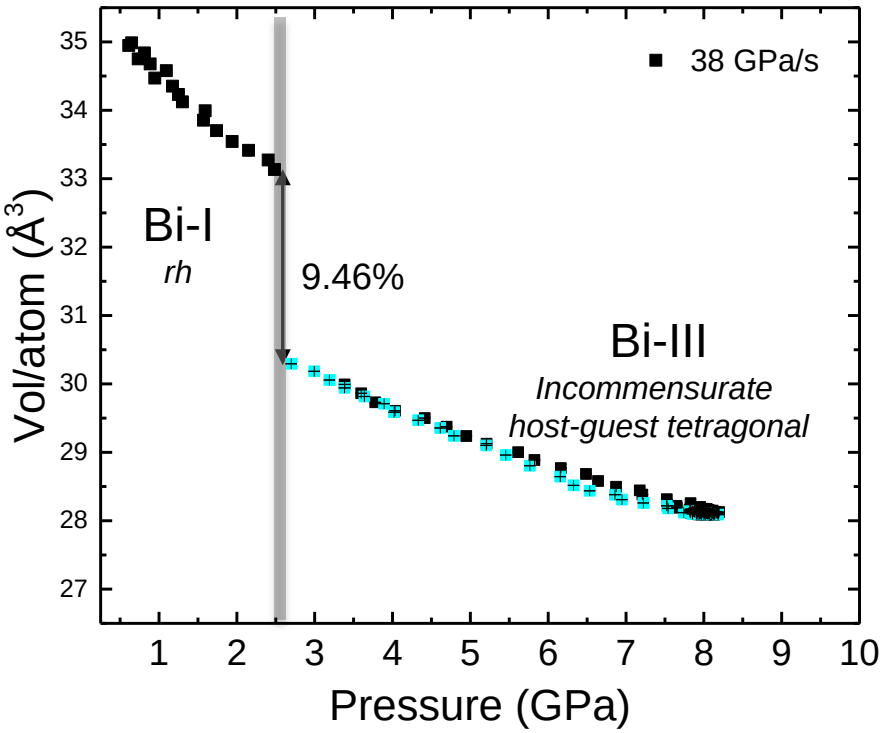




Bi-III→V phase transition pressures shifted higher to 8.2 GPa at 4.2 GPa/s compression rate

# Kinetic studies of Bismuth I-II-III-~~V~~ phase transitions

## 38 GPa/s compression



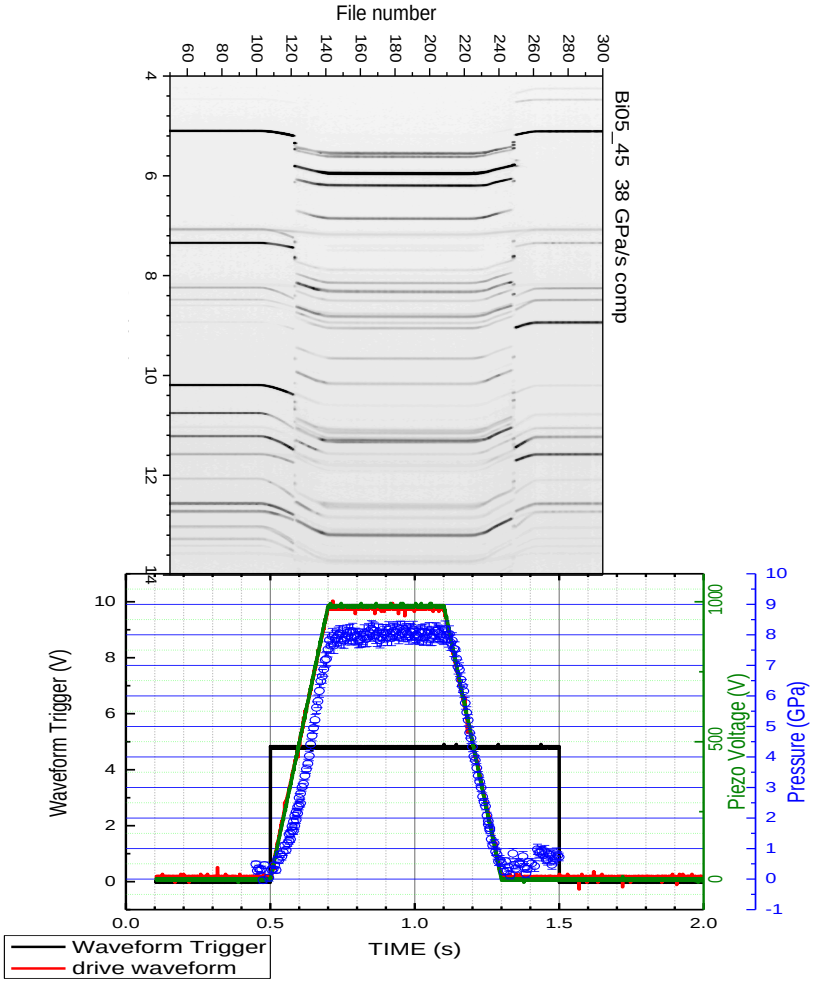



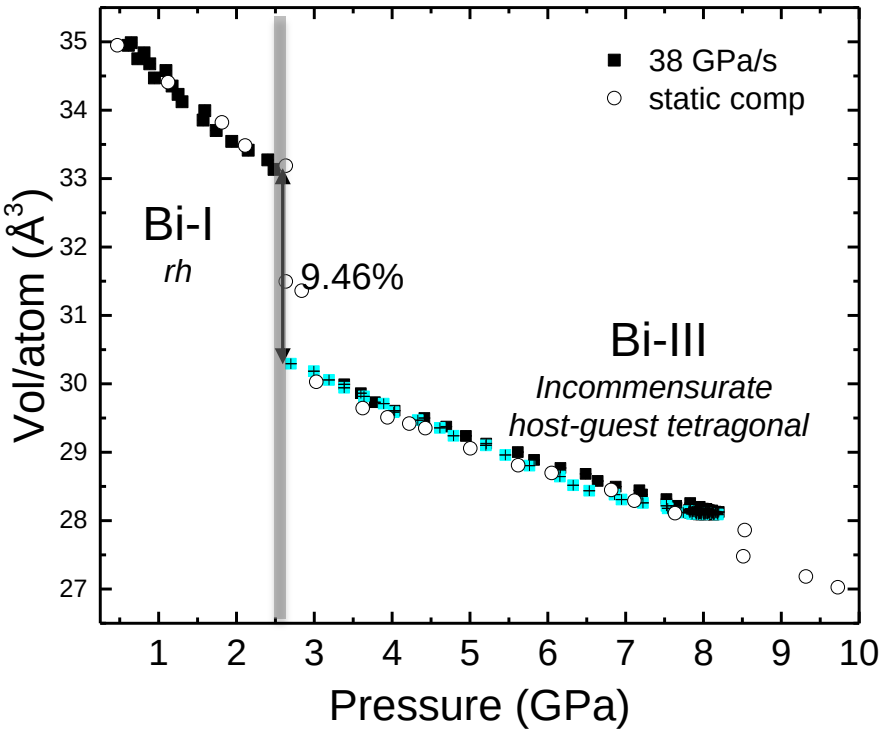

Bi-V not observed up to 8.1 GPa

Bi-III→V phase transition pressures higher than 8.1 GPa at 38 GPa/s compression rate

# Kinetic studies of Bismuth I-II-III-~~V~~ phase transitions

## 38 GPa/s compression



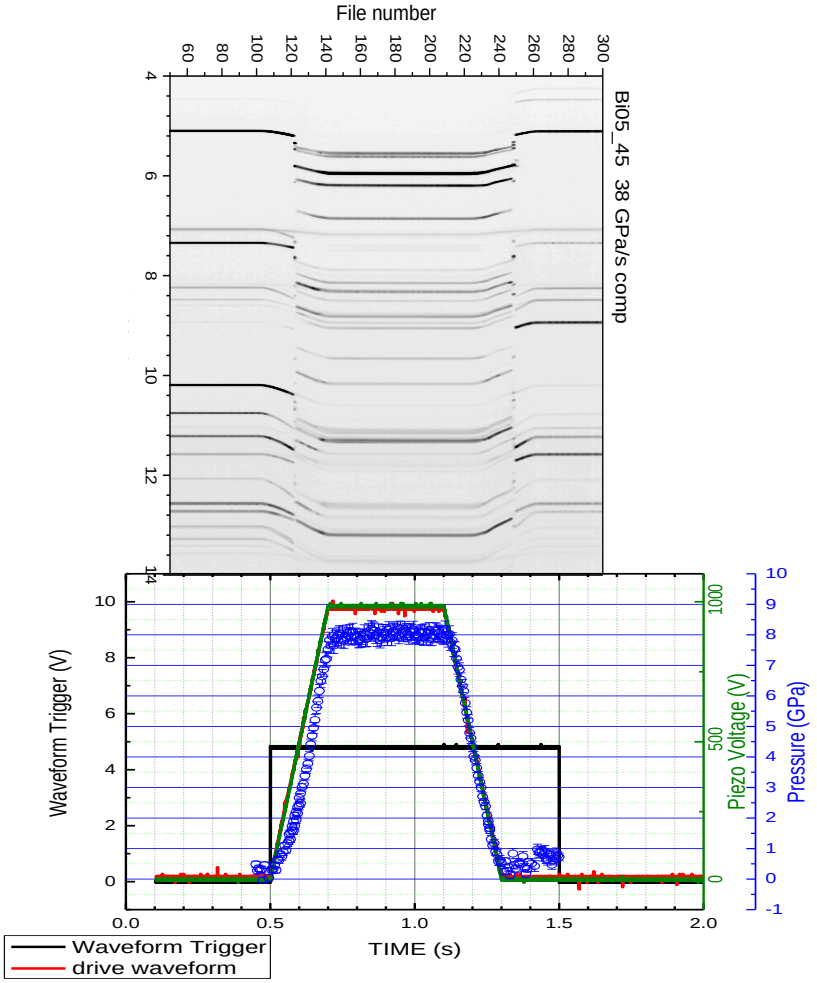



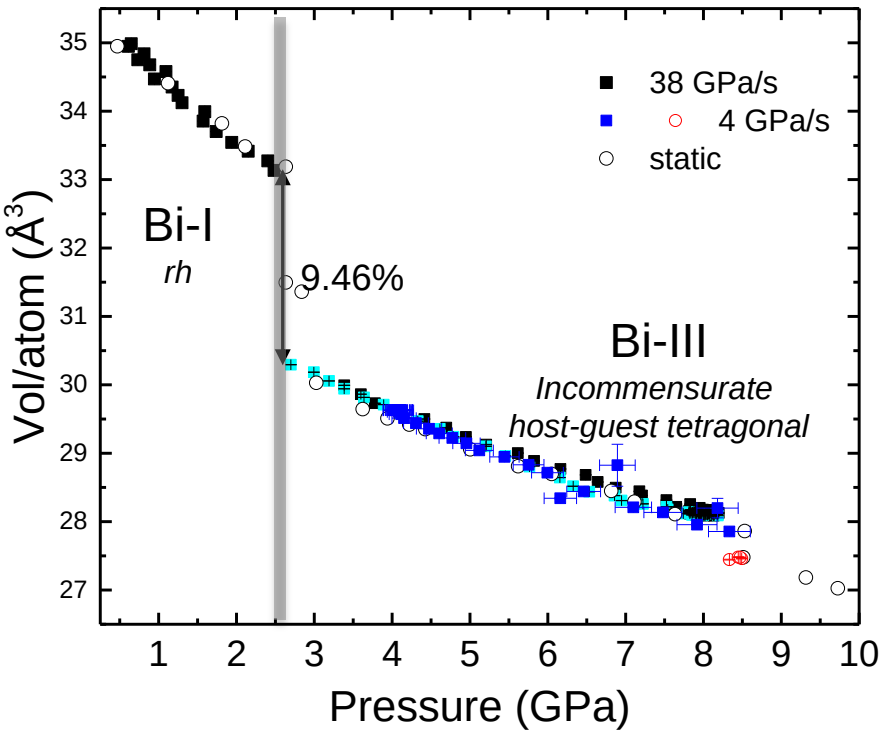

Bi-V not observed up to 8.1 GPa

Bi-III→V phase transition pressures higher than 8.1 GPa at 38 GPa/s compression rate

# Kinetic studies of Bismuth I-II-III-~~V~~ phase transitions

## 38 GPa/s compression



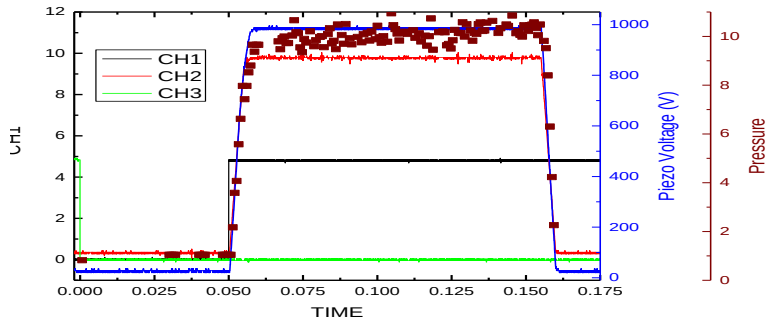



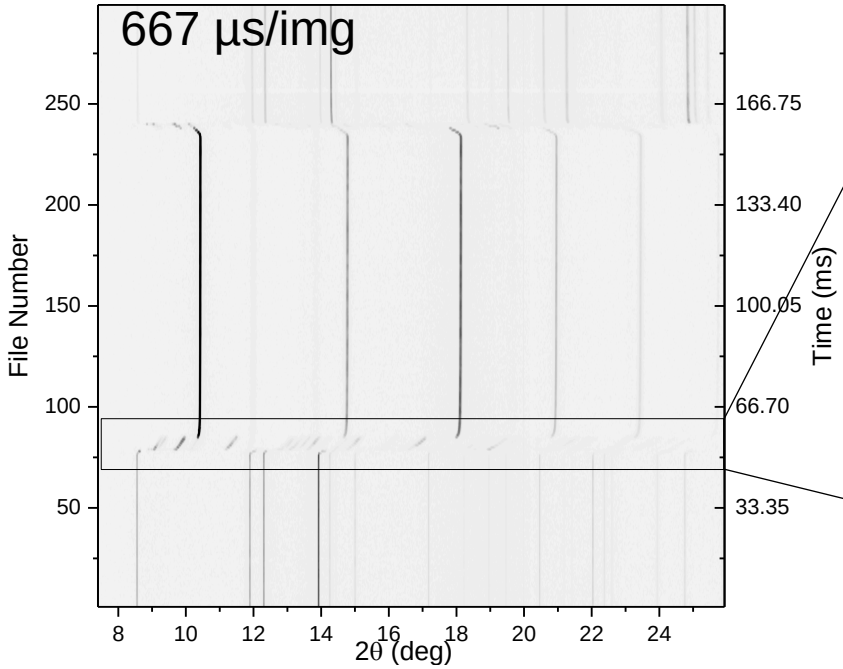

Bi-V not observed up to 8.1 GPa

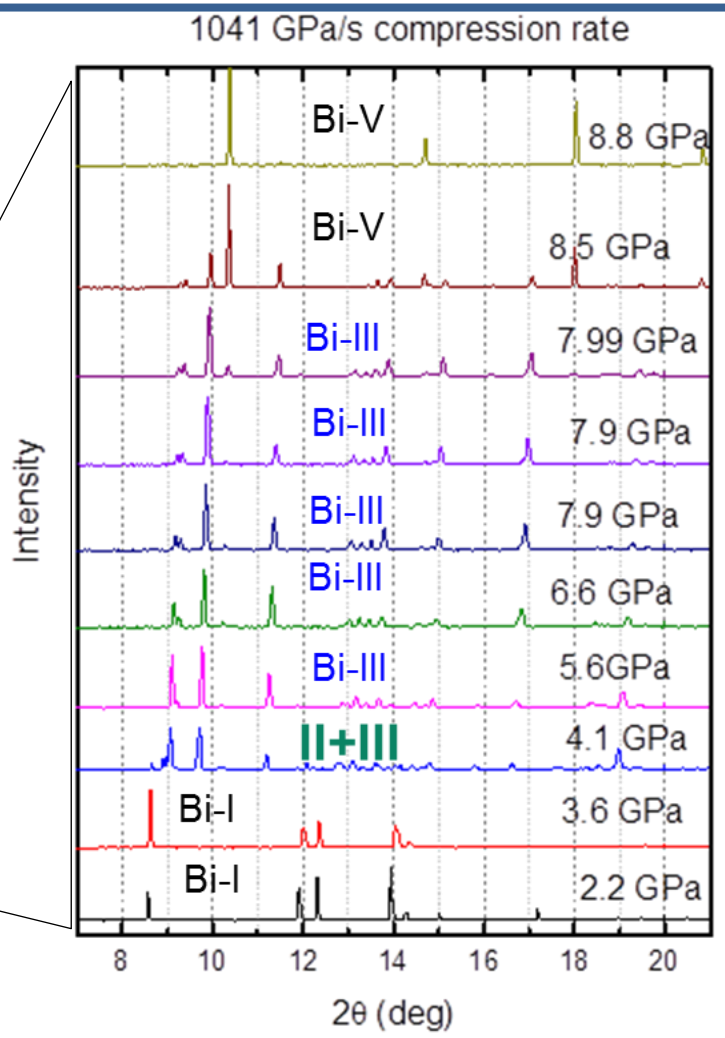
Bi-III→V phase transition pressures higher than 8.1 GPa at 38 GPa/s compression rate

# Kinetic studies of Bismuth I-II-III-V phase transitions

## 1041 GPa/s average compression rate

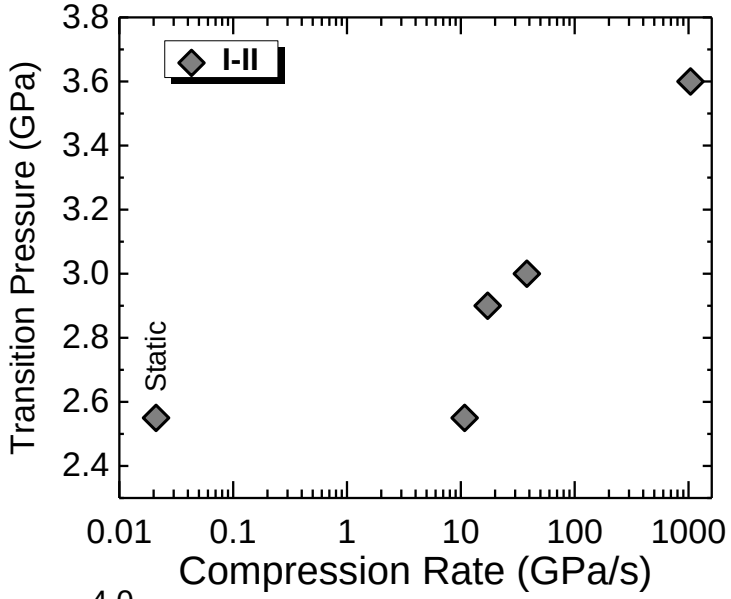


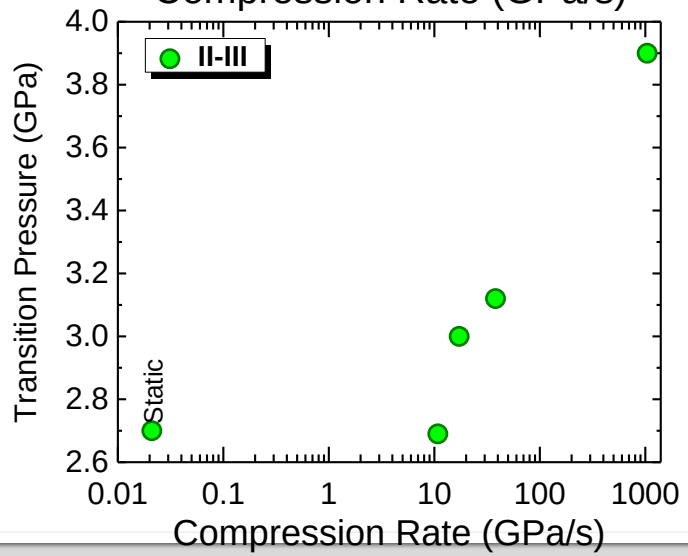



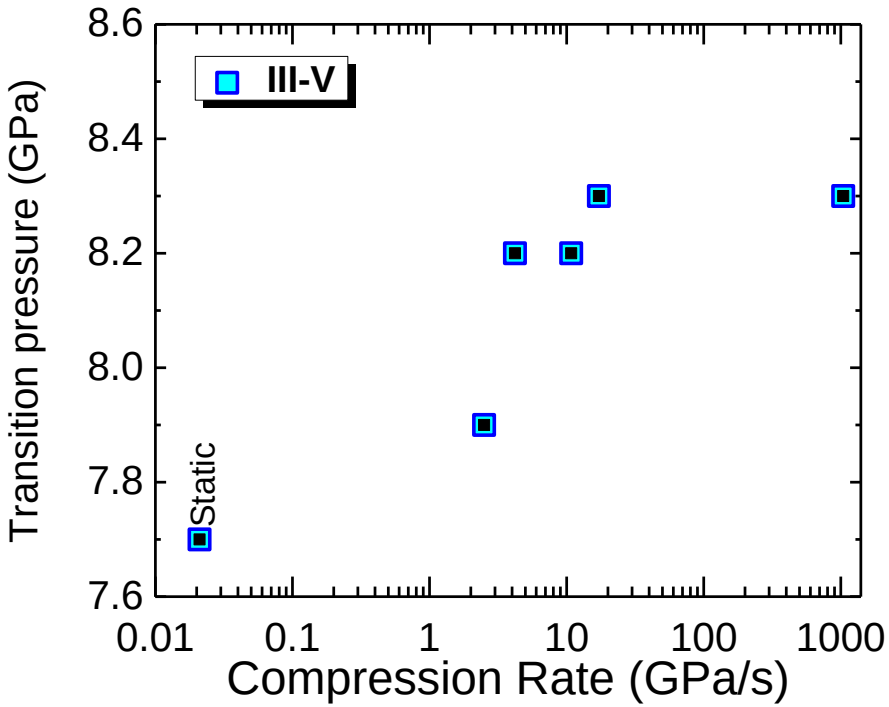




Phase transition pressures shifted higher at 1041 GPa/s compression rate

# Bismuth summary

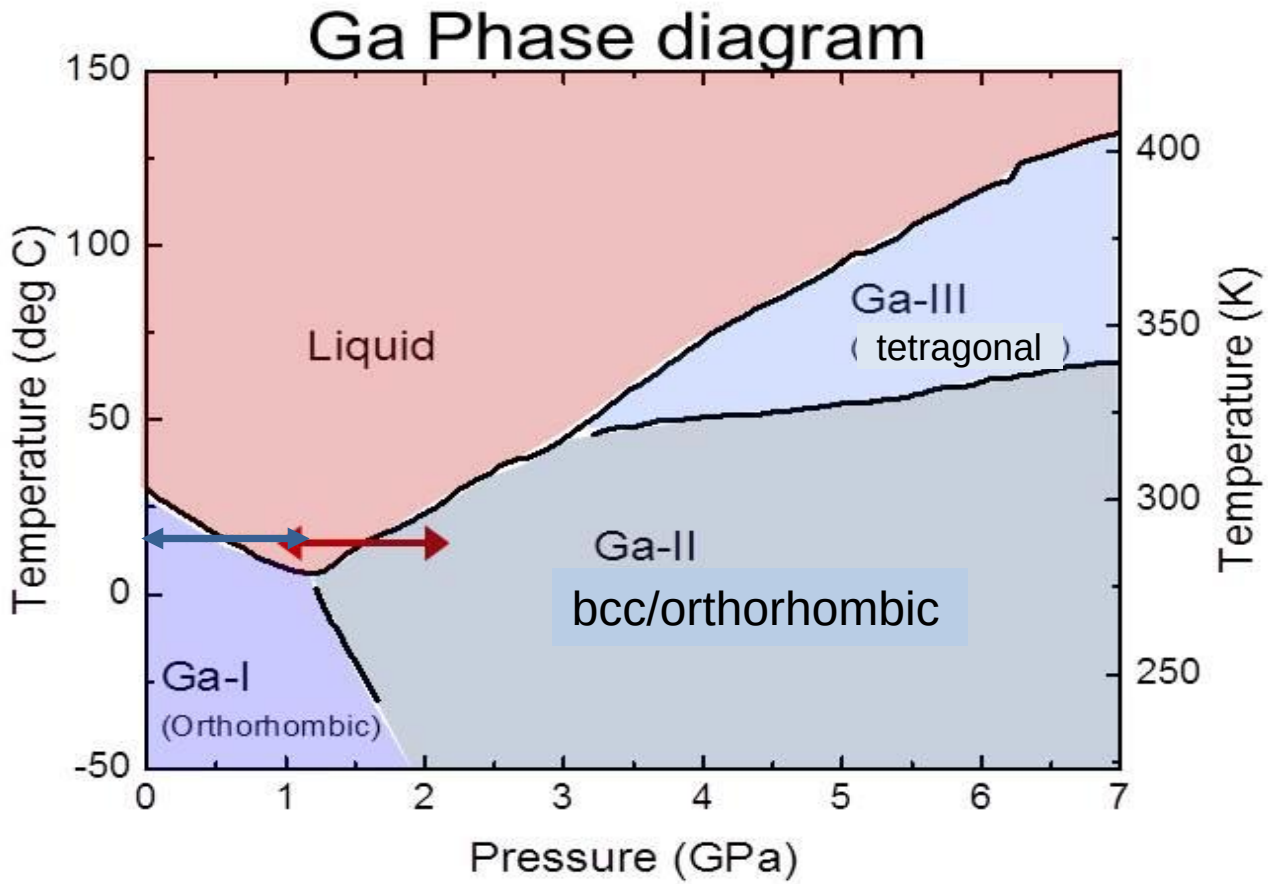



Phase transition pressures shifted higher at compression rates higher than 10 GPa/s

# Complex phase diagram of Ga

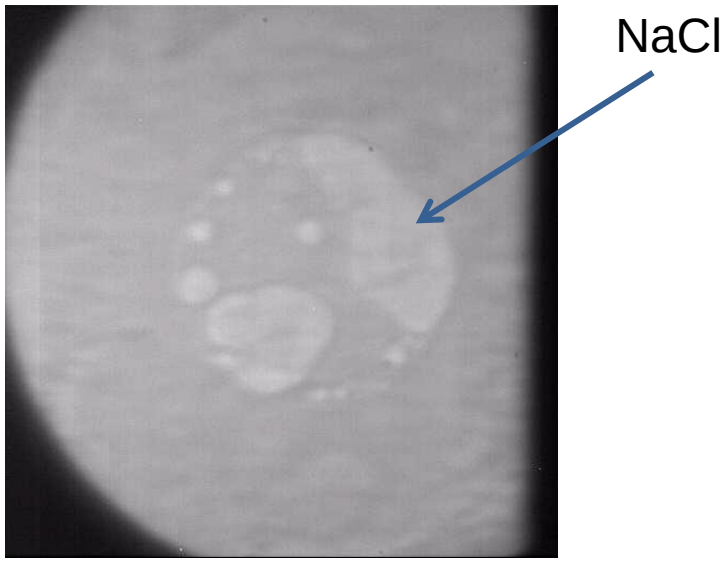



Ga-II cannot be accessed in nDAC by isothermal compression

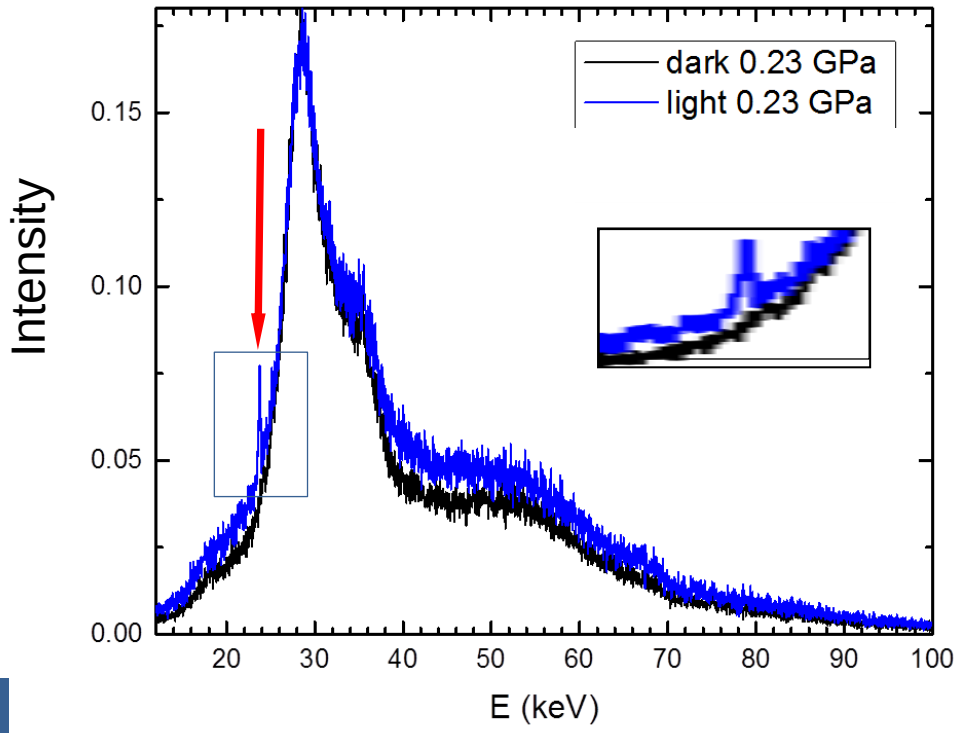
# Liquid solid phase transition in Ga

*phase contrast imaging and x-ray diffraction*

**Ga-I ←→ liquid**




Energy dispersive x-ray diffraction patterns taken at light (blue) and dark (black) regions of the Ga




X-ray phase contrast imaging of Ga-I ←→ liquid phase transition
(500 ms sinusoidal modulation 0.05-1 GPa)

# Acknowledgements

Portions of this work were performed under the auspices of the U.S. DOE by LNL under Contract DE-AC52-07NA27344.
Portions of this research were carried out at Petra-III at DESY, a member of the Helmholtz Association (HGF).

Lawrence Livermore National Laboratory

DESY

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