



*From quasi static to dynamic compression: A unique opportunity to study the influence of kinetics on various physical phenomena*

A. Sollier, F. Occelli, E. Lescoute, C. Pepin, A. Dewaele, S. Brygoo, L. Videau, V. Recoules, J. Bouchet, J. Bontaz, L. Soulard, and P. Loubeyre



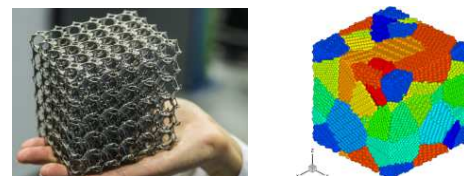
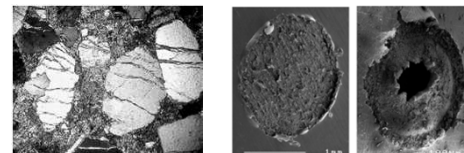
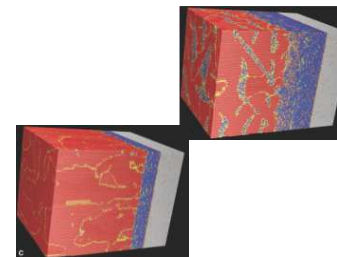
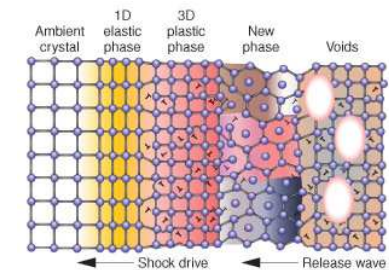
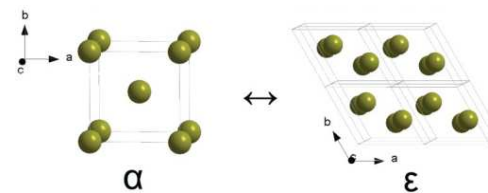
- ❑ Why do we want to perform dynamic compressions experiments in front of synchrotrons?
- ❑ What is our experimental strategy to do it ?
- ❑ First results and examples of possible future experiments

# Relevance of synchrotron X-Ray measurements for dynamic compression studies

Understanding the dynamic behavior of materials require experiments that provide real-time, in-situ, spatially resolved measurements on relevant length and timescales

## Topics of interest:

- Phase transitions, chemical reactions
  - Kinetics
  - Nucleation & growth, diffusion
  - Atomic rearrangement, microstruture evolution
- Metastability, intermediate phases
  - Metastable phases
  - Over heating / Over compression
- Plasticity
  - Dislocations dynamics
- Damage & Failure
  - Nucleation & growth
- Real, Complex Materials
  - Role of grains interfaces
  - Void compaction
- ...



Diagnostics such as X-ray diffraction (XRD), X-ray absorption spectroscopy (XAS) and Phase contrast imaging (PCI) available at ESRF offer unique opportunities for such ultrafast spatially resolved measurements

# A strategy for Studies of Dynamic Behavior of Materials at ESRF

Apply dynamic loadings at different time scales on the same material and characterize it with different X-ray techniques

Samples + Loading platforms + Synchrotron

High purity (>99.9%)  
Which impurities ?  
Grain size ?  
Oxydation layer ?

Compatible with X-rays !

Different loading paths  
and time scales



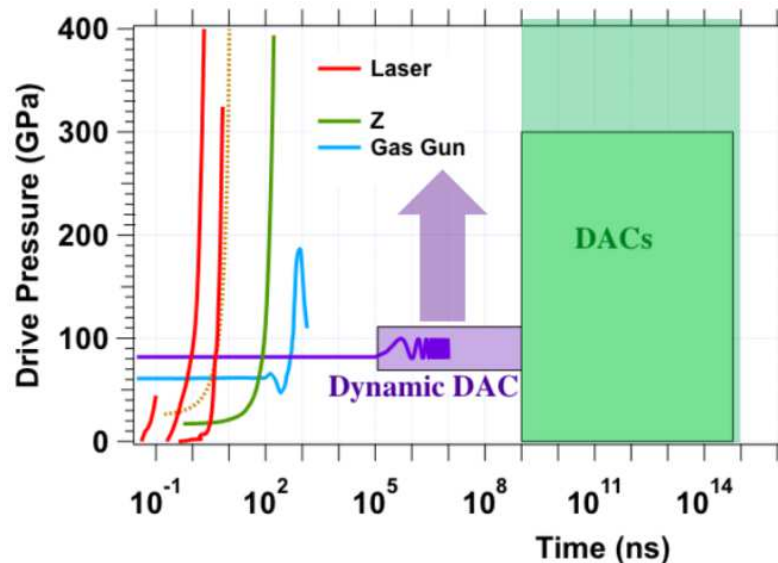
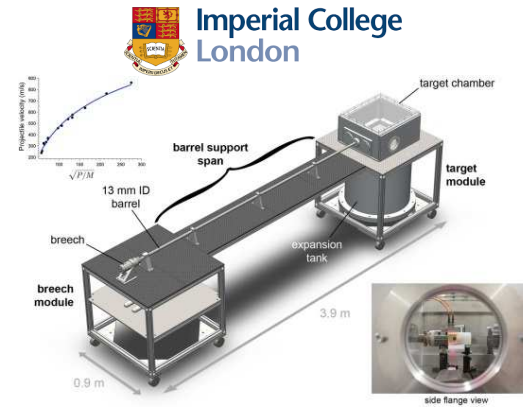
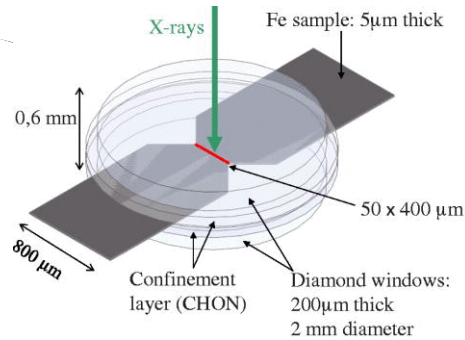
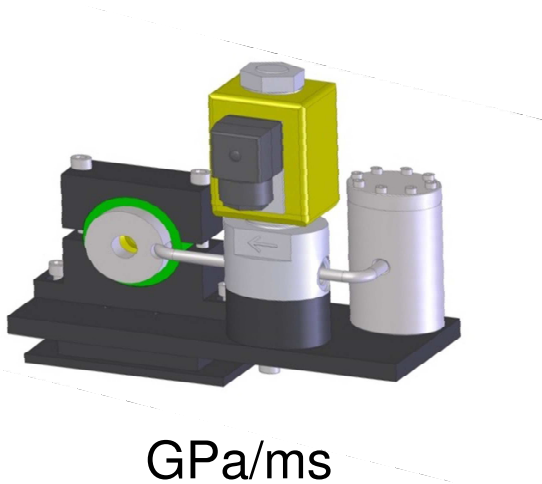
P/T measurements with  
relevant time resolution

XRD, XAS, PCI  
XMCD and CDI ?

- Need to adapt the sample and the loading device to the beamline environment
- Need to adapt the beamline to the time-resolved conditions
- Need to implement P/T measurements

# A strategy for Studies of Dynamic Behavior of Materials at ESRF

- Various dynamic loading facilities available or in development at ESRF
  - Different loading paths & strain rates
  - Large P/T range



A unique opportunity to investigate a phenomena at various time scales with the same microscopic information from X-ray measurements (XRD, XAS, PCI, ...)



# ms dynamic membrane DAC

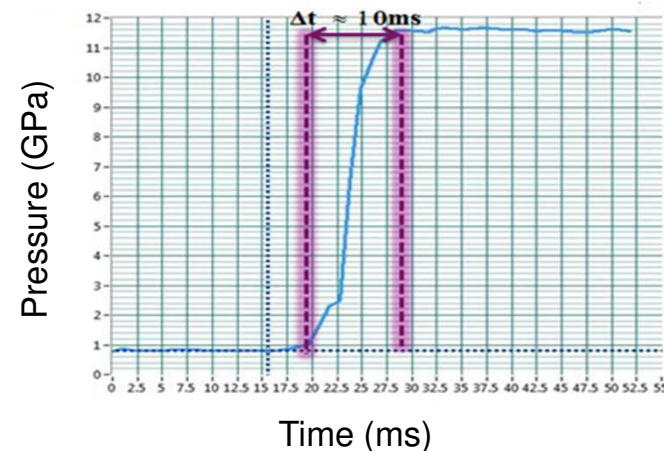
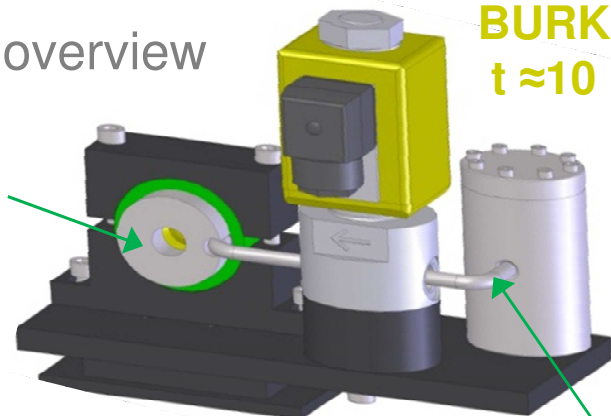
Setup overview

Electrovalve  
BURKERT  
 $t \approx 10$  ms

Gas tank

Membrane DAC

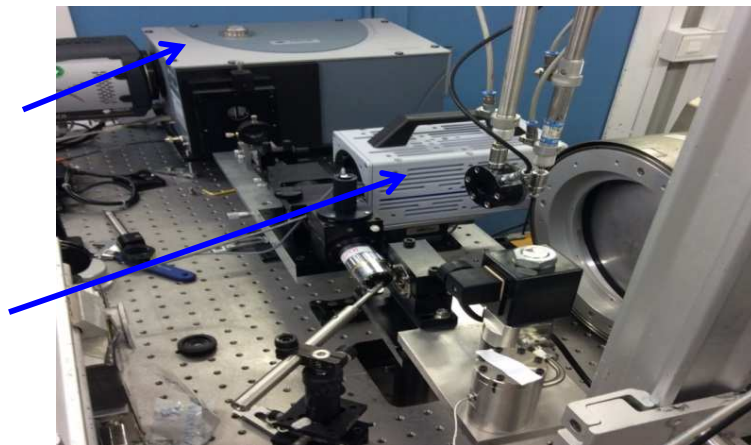
1/4" tubing: large gas flow rate



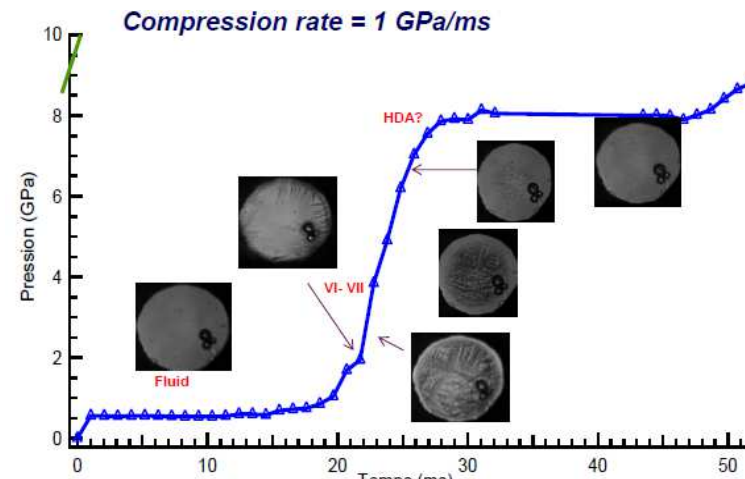
Pressure increase  
0.1 to 1 GPa/ms

Proposal HC-2183 on ID27: Dynamic compression of water probed by XRD (30 keV, 5x5μm)

Monitoring of P(t) by Ruby luminescence



Monitoring of the sample evolution with a rapid camera (2000 frames/s)



# ms time resolved EXAF measurement of the $\alpha \rightarrow \omega$ transition in Zr

The  $\alpha \rightarrow \omega$  phase transformation in zirconium followed with ms-scale time-resolved X-ray absorption spectroscopy

A. Dewaele, R. André, F. Occelli, O. Mathon, S. Pascarelli, T. Irifune & P. Loubeyre  
Pages 237-249 | Received 21 Apr 2016, Accepted 04 Jun 2016, Published online: 02 Aug 2016

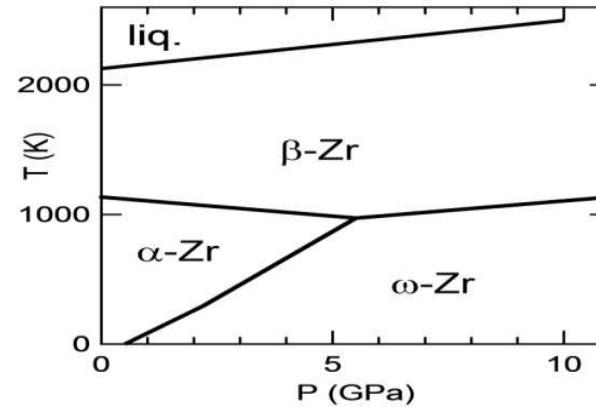


## Earlier work:

Zr polycrystal of 99.98 % purity  
Non-hydrostatic or quasi-hydro-static  
in methanol+ethanol pressure medium

MK Jacobsen *et al.*, J Appl. Phys. **118**, 025902 (2015).

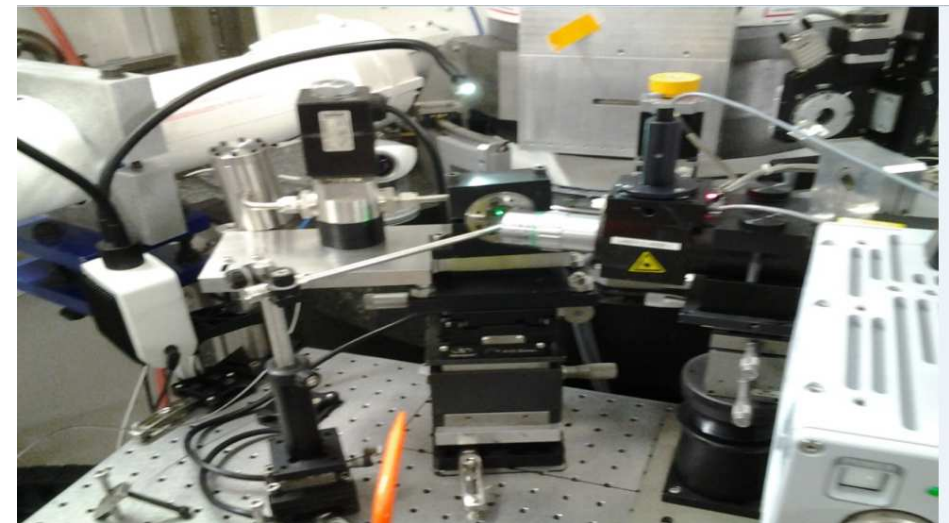
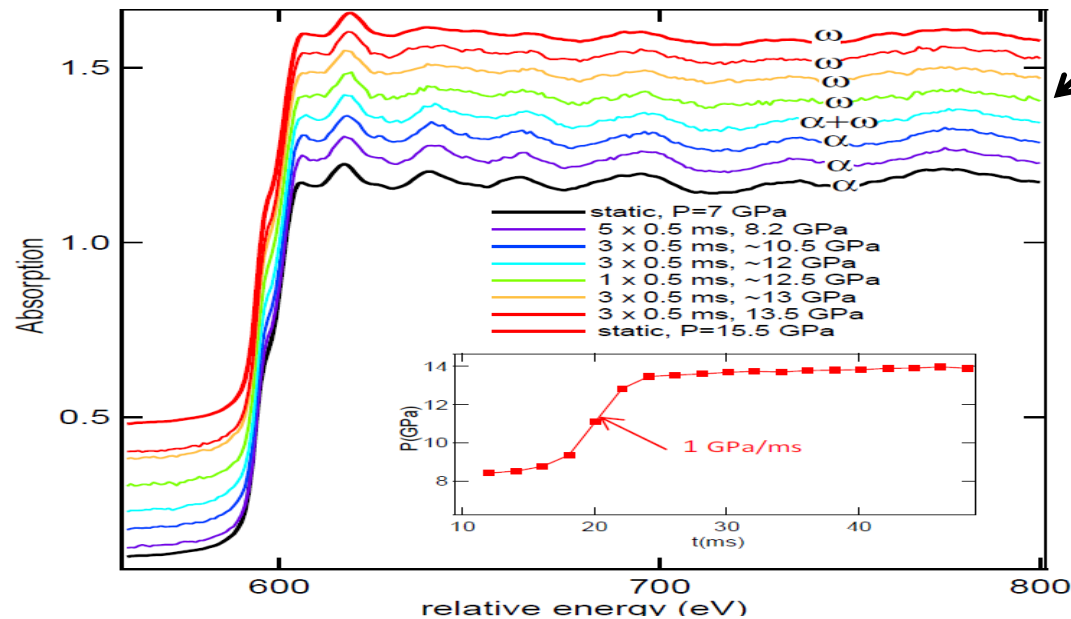
Sluggish kinetics with transition times reaching several minutes



## Experiment performed on ID24

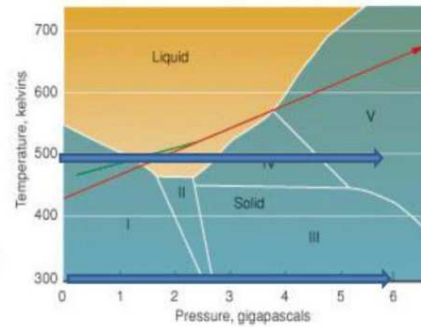
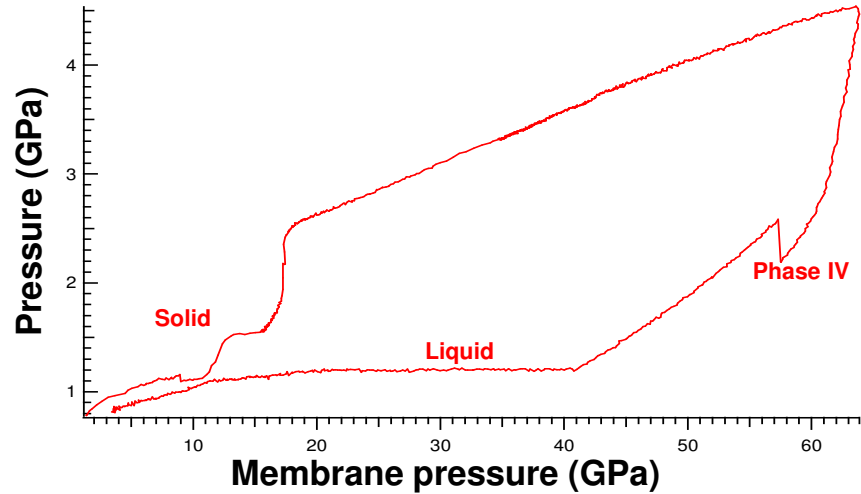
Zr polycrystal of 99.9+ purity  
Hydrostatically compressed in neon  
Zr Kedge (18,0 keV)  
Nanopolycrystal DAC  
Frelon detector

$P_{\alpha \rightarrow \omega} = 11 \text{ GPa (static) vs } 12 \pm 1 \text{ GPa (dynamic)}$   
The transition is achieved in less than a few ms !

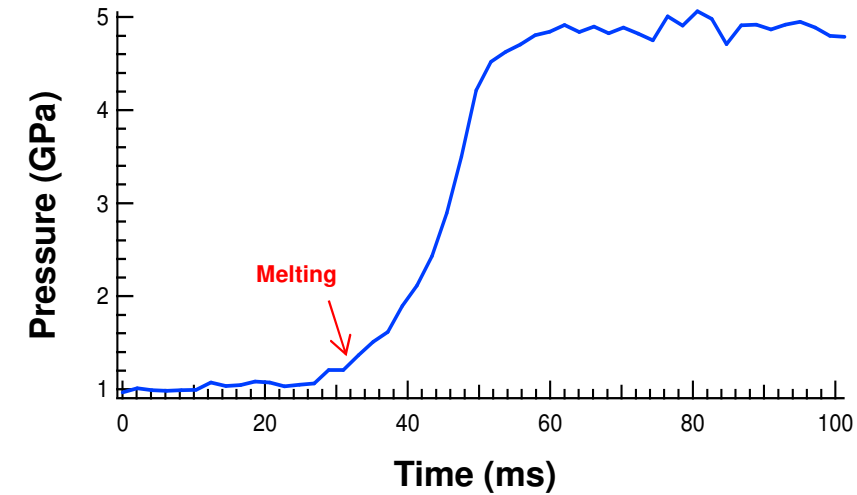


# First trials on Bi

## Static DAC experiment @ 498 K



## Dynamic DAC experiment @ 498 K



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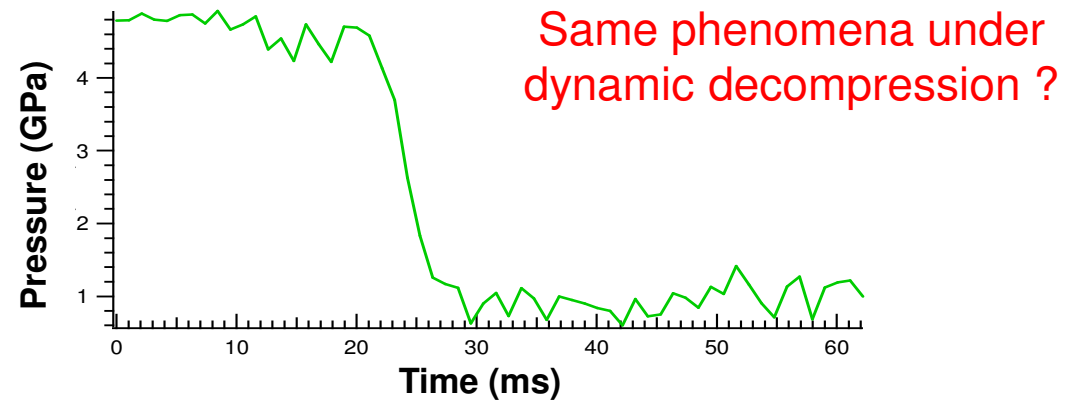
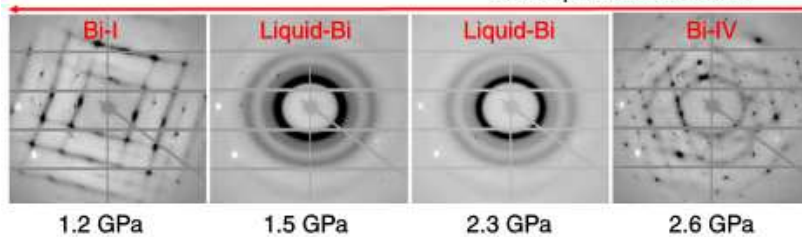
DOI: 10.1038/ncomms14260

OPEN

## A metastable liquid melted from a crystalline solid under decompression

Chuanlong Lin<sup>1</sup>, Jesse S. Smith<sup>1</sup>, Stanislav V. Sinogeikin<sup>1</sup>, Yoshio Kono<sup>1</sup>, Changyong Park<sup>1</sup>, Curtis Kenney-Benson<sup>1</sup> & Guoyin Shen<sup>1</sup>

### Decompression at 489 K



Ability to probe different isotherms of Bi both under compression and release



# $\mu$ s Ohmic ramps

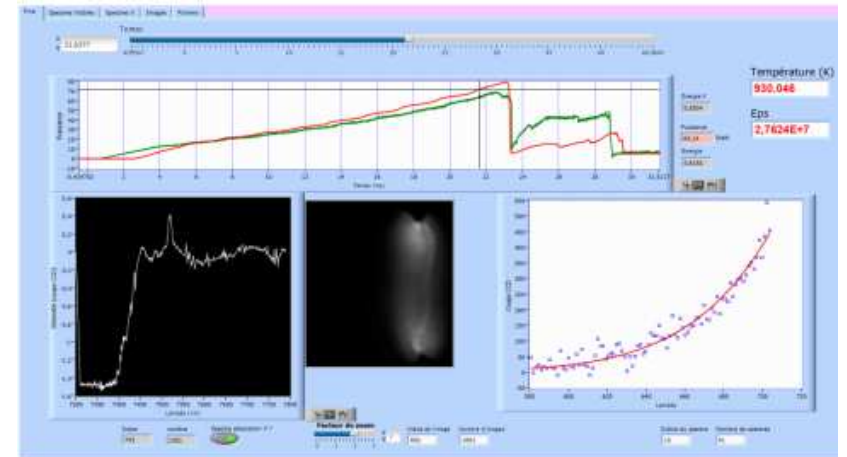
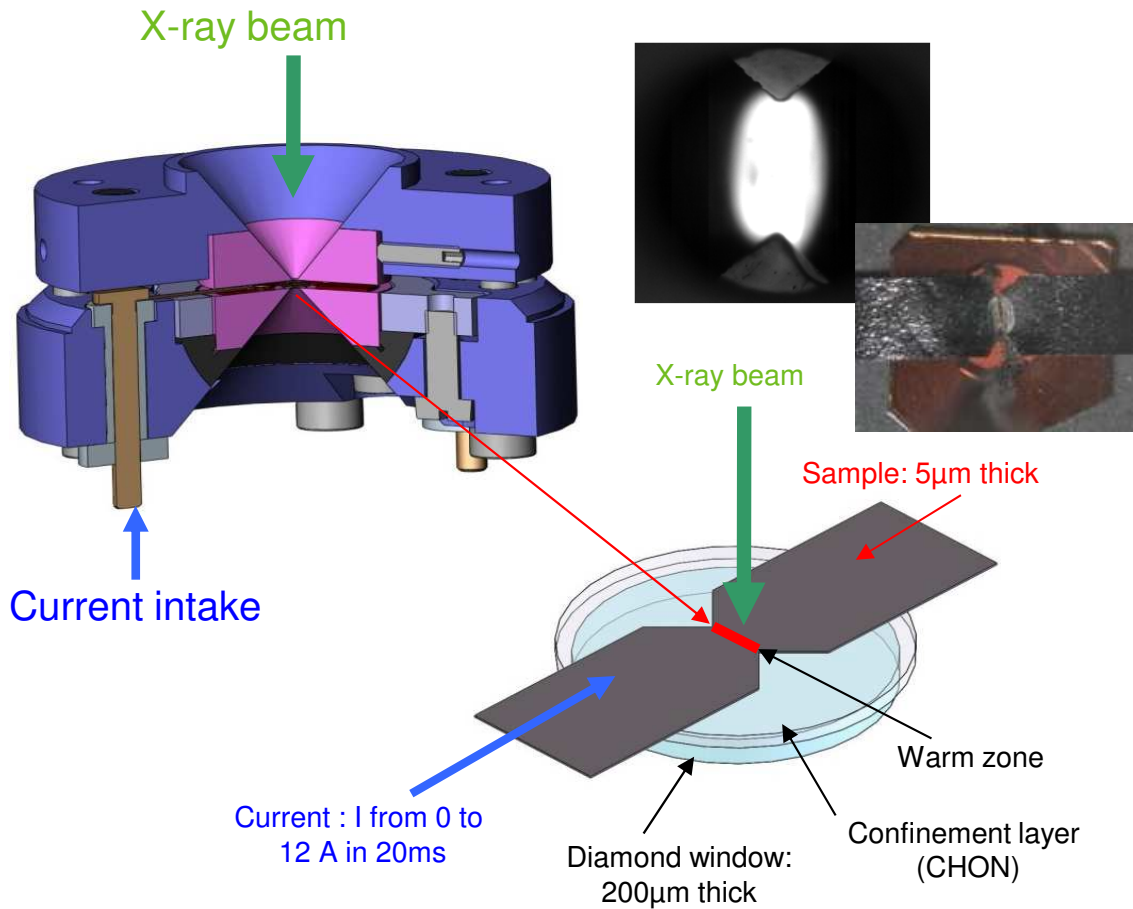
JOURNAL OF APPLIED PHYSICS 115, 093513 (2014)

## A microsecond time resolved x-ray absorption near edge structure synchrotron study of phase transitions in Fe undergoing ramp heating at high pressure

C. Marini,<sup>1</sup> F. Occelli,<sup>2</sup> O. Mathon,<sup>1</sup> R. Torchio,<sup>2</sup> V. Recoules,<sup>2</sup> S. Pascarelli,<sup>1</sup> and P. Loubeyre<sup>2</sup>

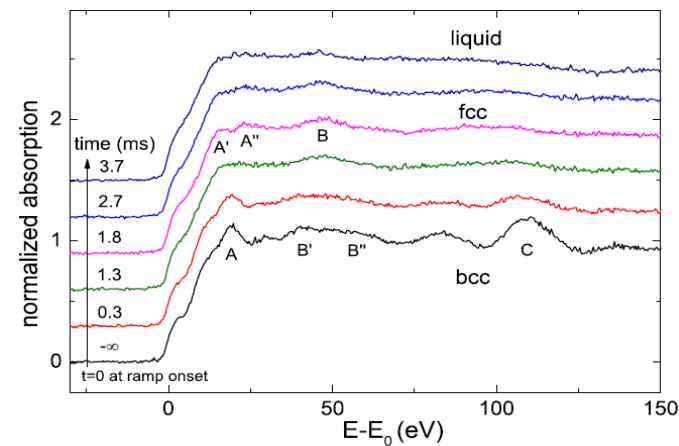
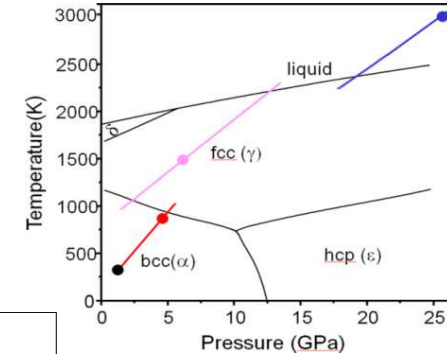
<sup>1</sup>European Synchrotron Radiation Facility, 6 Rue Jules Horowitz, BP220, 38043 Grenoble Cedex, France

<sup>2</sup>CEA, Bruyeres le Chatel, 91297 Arpaion Cedex, France



Monitoring of T(t) by time resolved spectral pyrometry

## Quasi-isochoric heating



# ns Laser shock compression

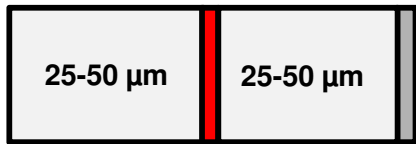
## Portable high power laser systems



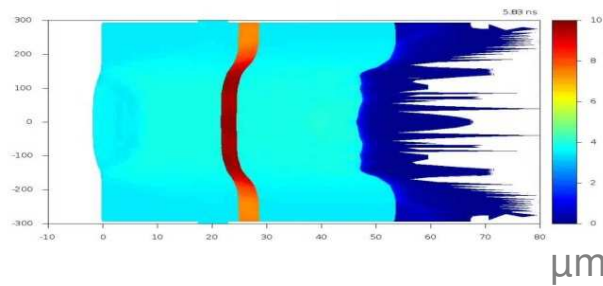
$E \sim 20-50 \text{ J}, \tau \sim 4-100 \text{ ns}$

Up to 3 Mbar with ablator and small diameter PZP

Diamond Diamond CH



Sample  
A few  $\mu\text{m}$

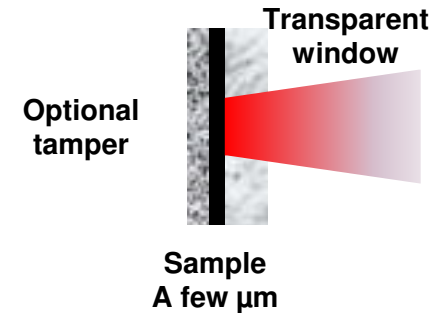


$\sim 2\text{D}$ , scalable pressure, poor reproducibility

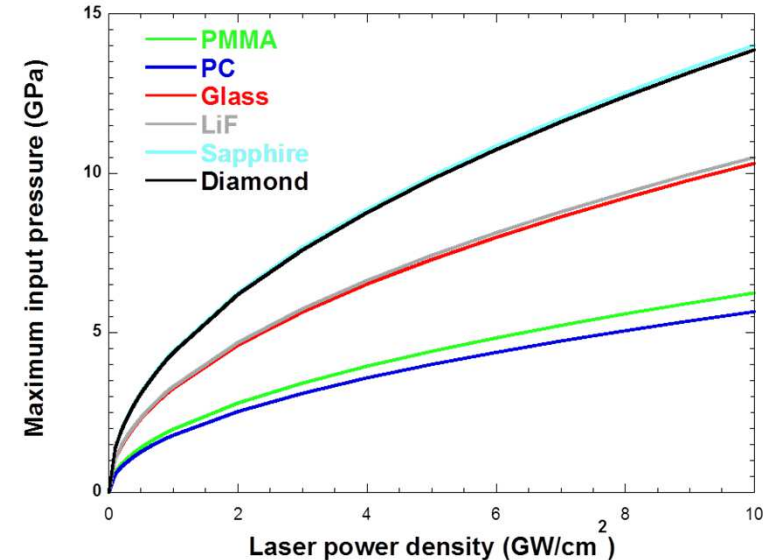
## Tabletop low power laser systems

$E < 1 \text{ J}, \tau \sim 5-10 \text{ ns}$

Up to 10 GPa in confined geometry



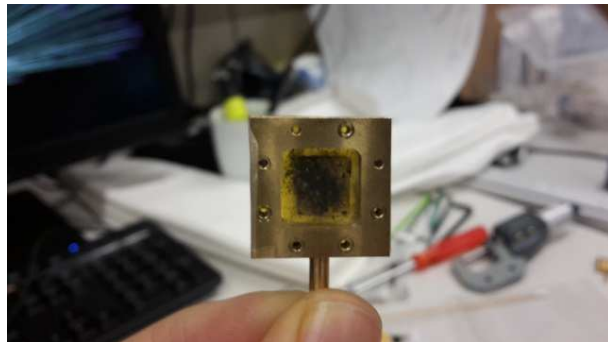
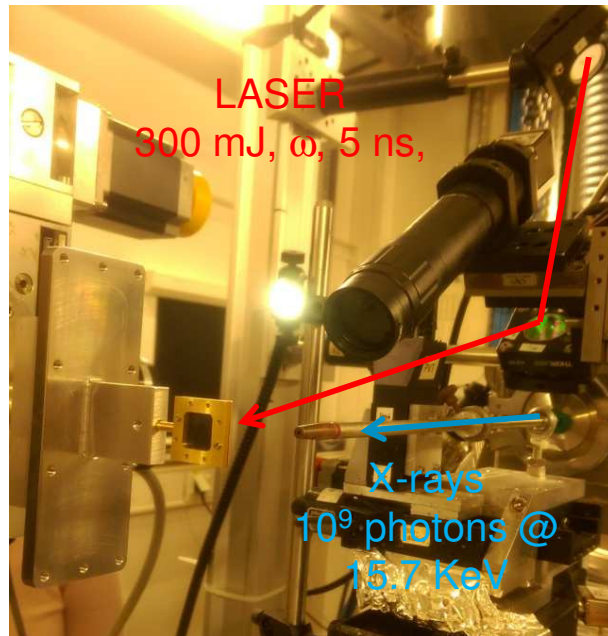
Bi, Fabbro's model,  $\alpha=0,25$



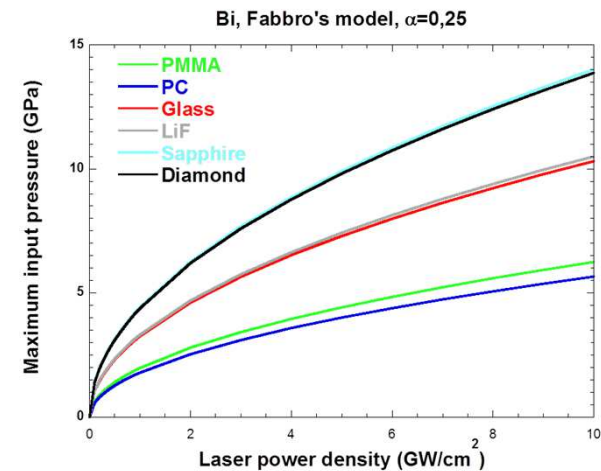
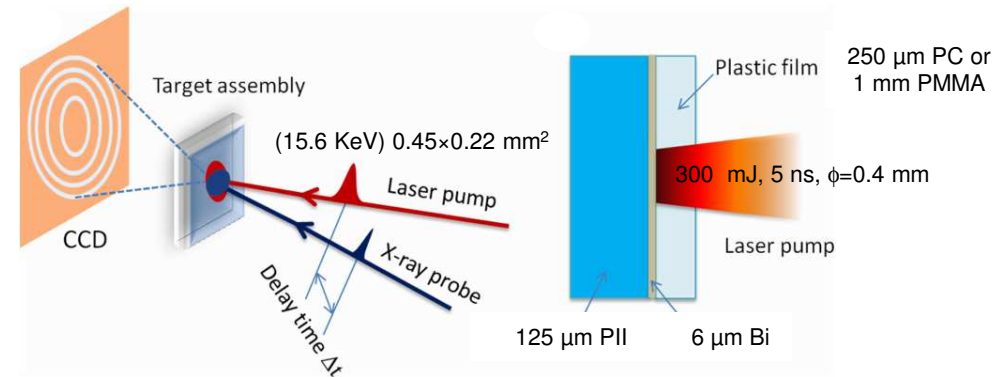
1D, scalable pressure, very reproducible

# Bi laser shock compression on ID09

Experiment HC 2848 (Nov 2016)



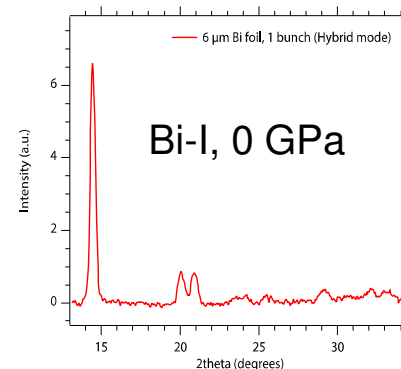
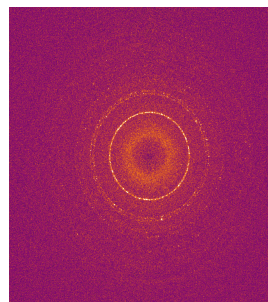
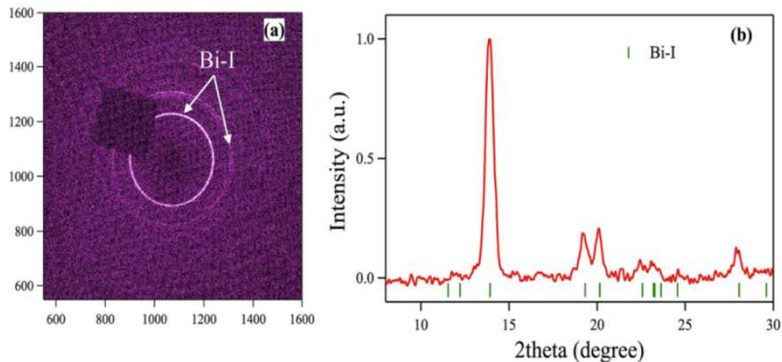
Confined geometry setup with the inline EKSPLA ns laser system



For each target (~ 12 x 12 mm), between 6 and 10 laser shots could be fired

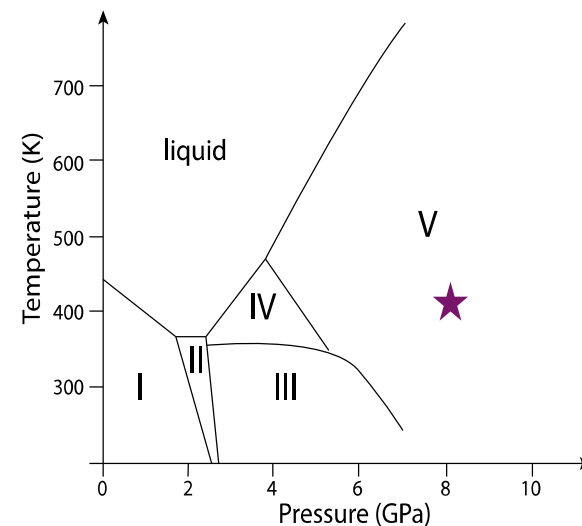
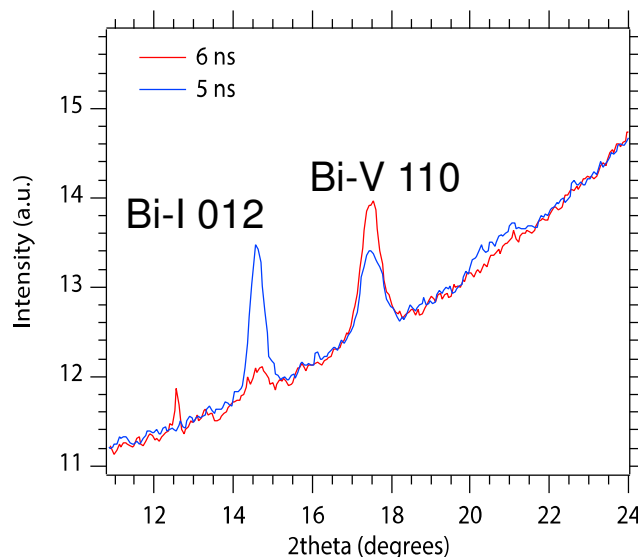
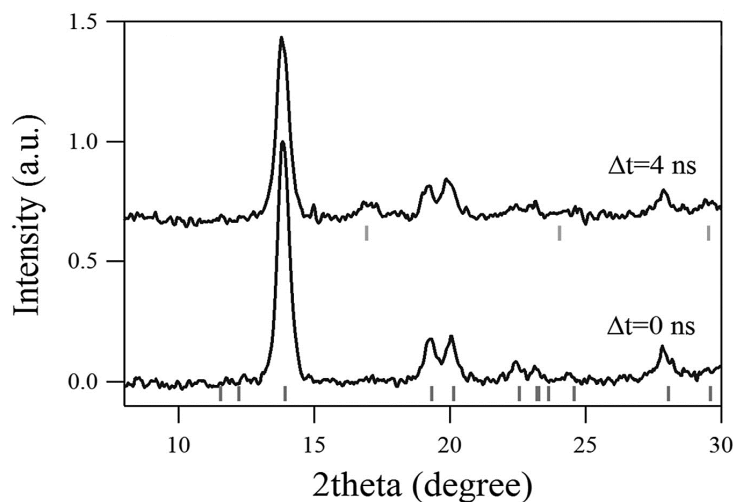
# Bi laser shock compression on ID09

## Reference Bi-I pattern



ID09, single bunch exposure

## Shock compression @ P ~ 8 GPa

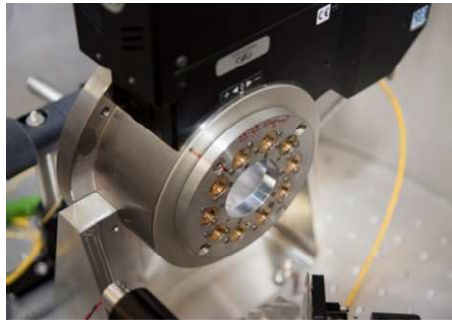


J. Hu et al., Complex structural dynamics of bismuth under laser-driven compression. Appl. Phys. Lett. **103**, 161904 (2013).

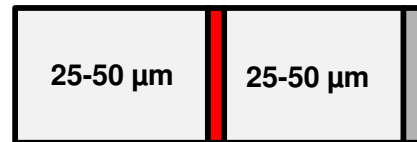
Bi-V (bcc) forms after 5 ns with a compression ( $V/V_0$ ) corresponding to a pressure of 8 GPa. At 6 ns, the majority of the 6 μm sample is compressed in the high pressure state.



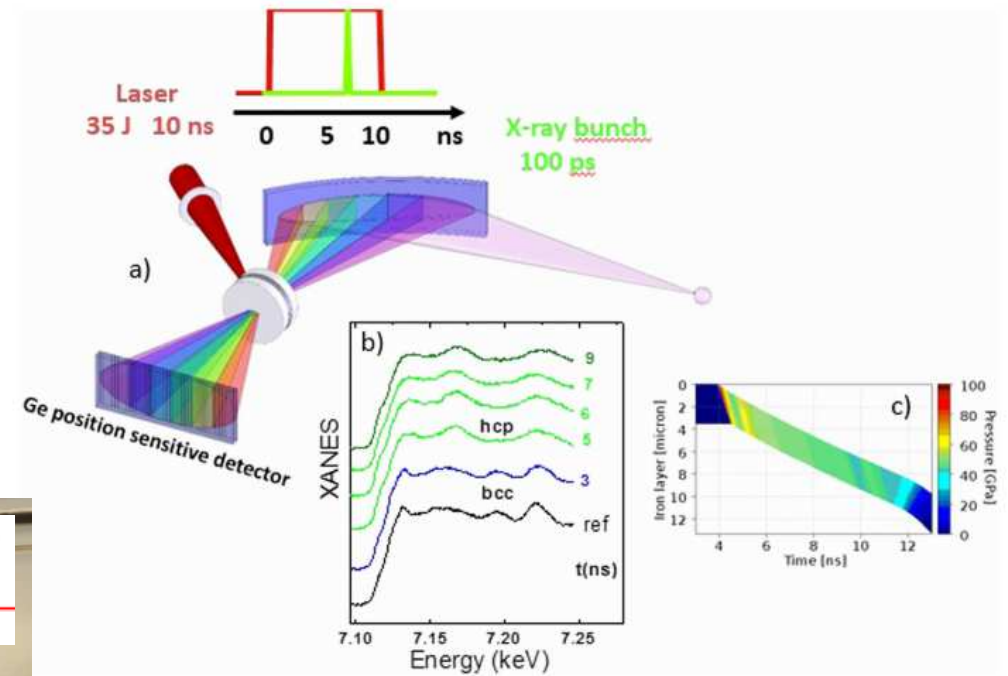
# Fe laser shock compression on ID24



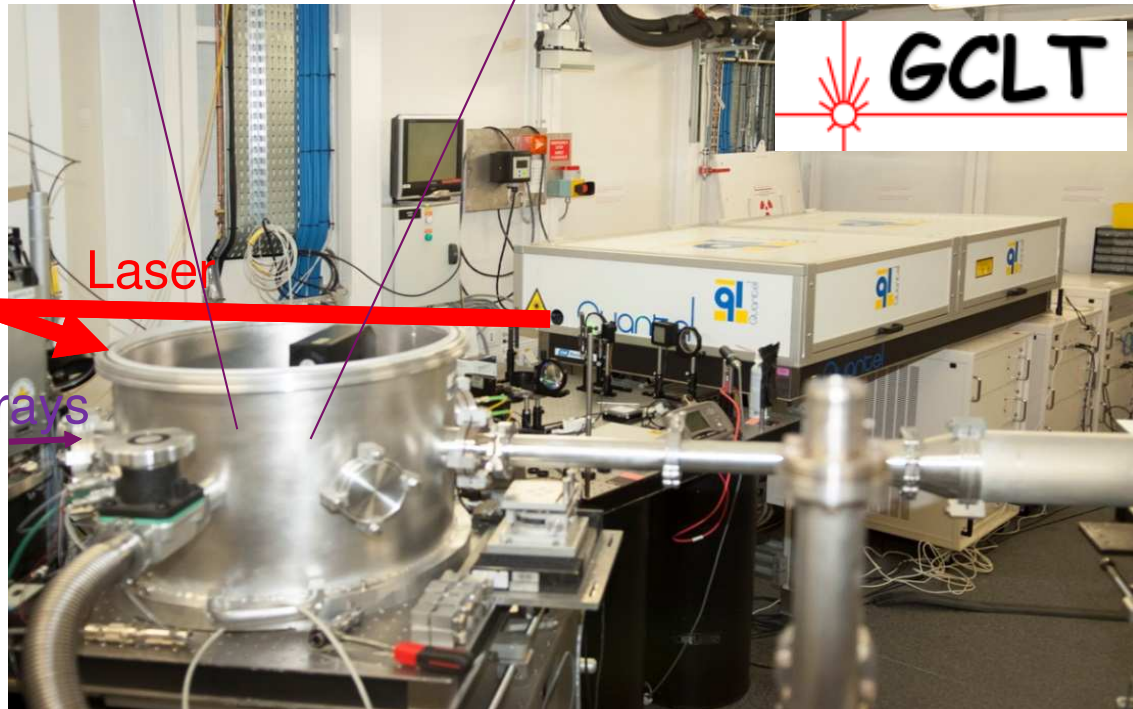
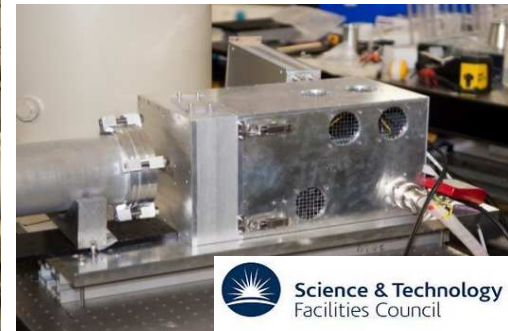
Diamond Diamond CH



Sample  
A few μm



XH detector





**OPEN** Probing local and electronic structure in Warm Dense Matter: single pulse synchrotron x-ray absorption spectroscopy on shocked Fe

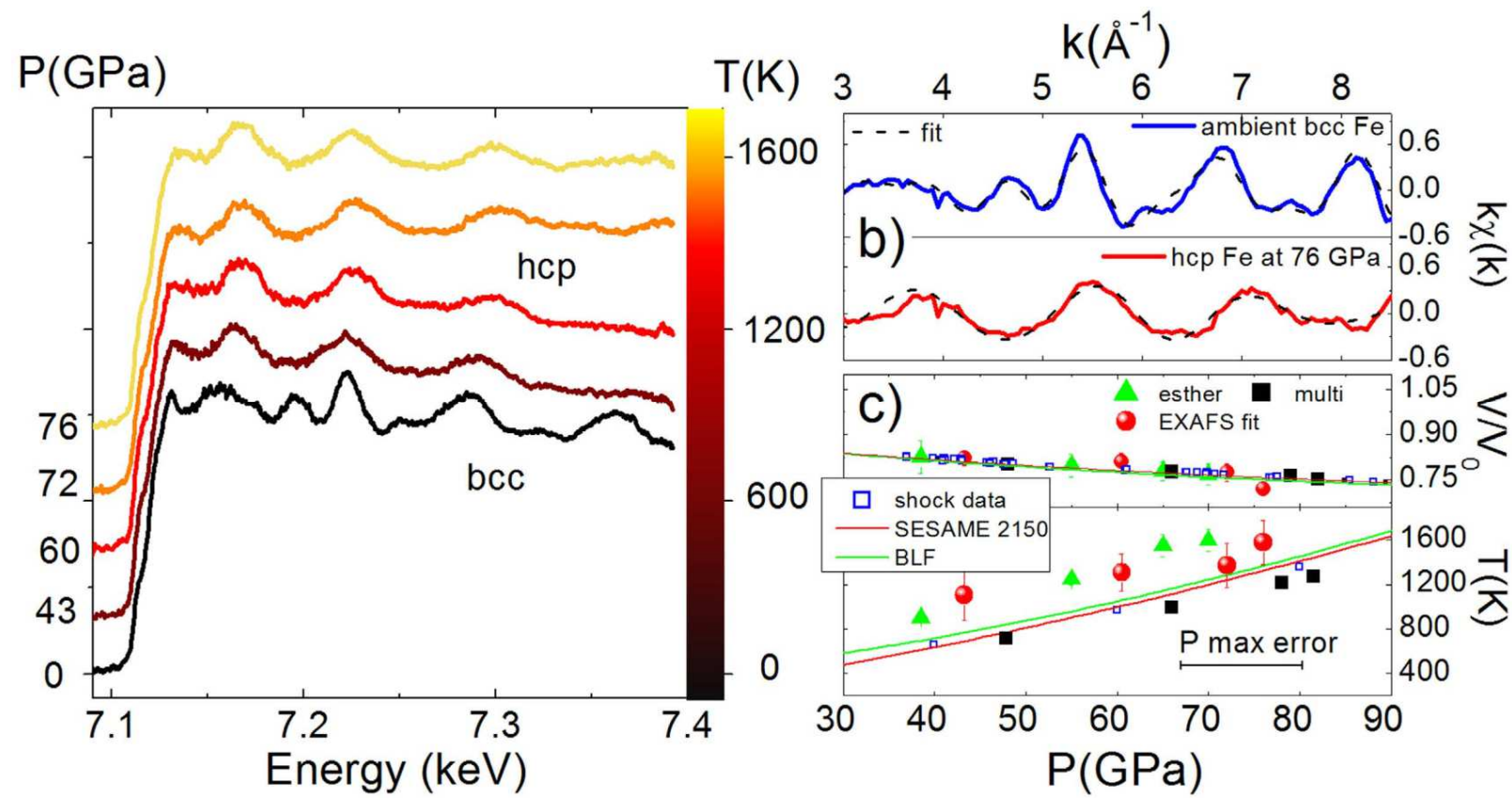
Received: 05 October 2015  
Accepted: 28 April 2016  
Published: 01 June 2016

Raffaella Torchio<sup>1,2</sup>, Florent Occelli<sup>1</sup>, Olivier Mathon<sup>2</sup>, Arnaud Sollier<sup>1</sup>, Emilien Lescoute<sup>1</sup>, Laurent Videau<sup>1</sup>, Tommaso Vinci<sup>3,4</sup>, Alessandra Benuzzi-Mounaix<sup>3,4</sup>, Jon Headspith<sup>5</sup>, William Helsby<sup>5</sup>, Simon Bland<sup>6</sup>, Daniel Eakins<sup>5,6</sup>, David Chapman<sup>6</sup>, Sakura Pascarelli<sup>2</sup> & Paul Loubeyre<sup>1</sup>

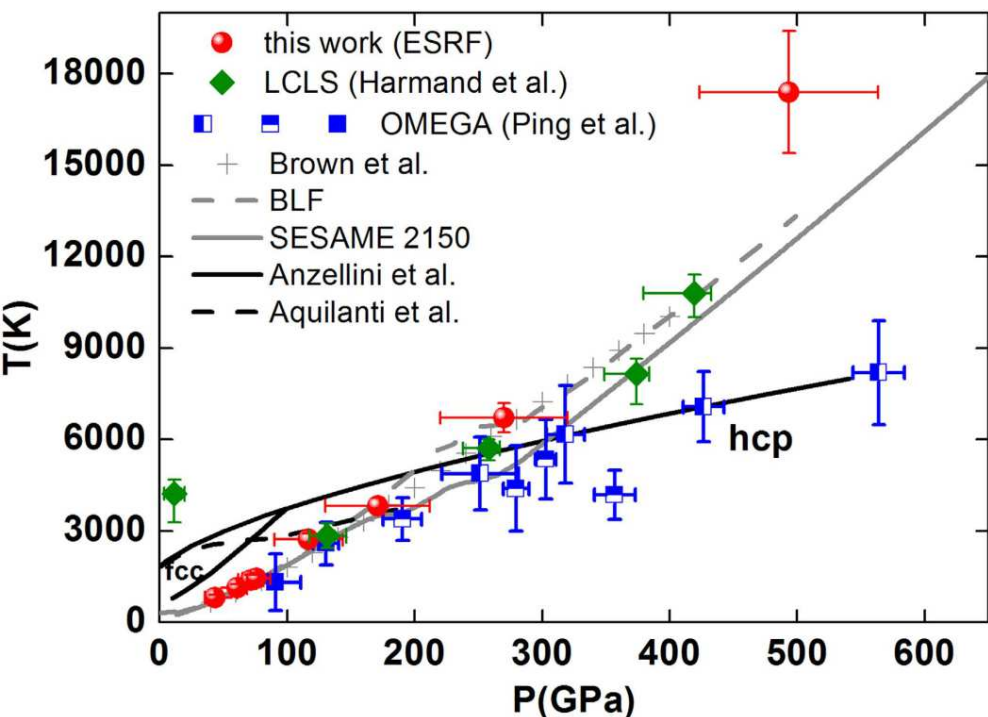
**Laser parameters**  
 $E = 23 \text{ J}$ ,  $\tau = 10 \text{ ns}$ ,  $\phi \sim 380 \mu\text{m}$  (PZP)  
 $I \sim 2 \cdot 10^{12} \text{ W/cm}^2$   
**X-rays**  $5 \times 90 \mu\text{m}$

The bcc-hcp transition is clearly observed  
 The reproducibility is good but not perfect

**Low pressure data**  
 Single pulse acquisitions

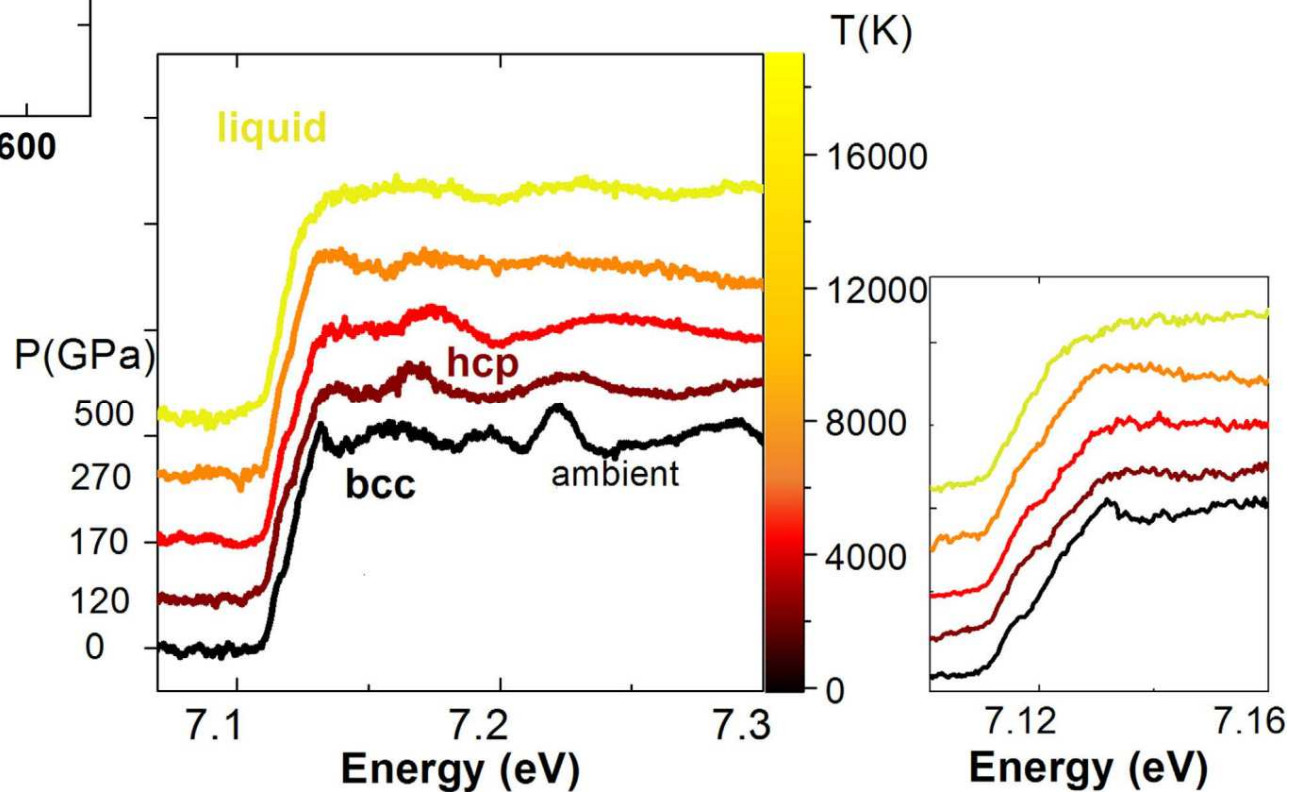


# Fe laser shock compression on ID24



**Laser parameters**  
 $E = 31 \text{ J}$ ,  $\tau = 10 \text{ ns}$ ,  $\phi \sim 90 \mu\text{m}$   
 $I_{\text{max}} \sim 5 \cdot 10^{13} \text{ W/cm}^2$   
**X-rays**  $5 \times 7 \mu\text{m}$

**High pressure data**  
 Single pulse acquisitions  
 Lower quality because of X-ray focusing optics



# Conclusion

- Various dynamic loading facilities available or in development at ESRF
  - Different loading paths
  - Different strain rates
- A unique opportunity to study various aspects of the dynamic behavior of materials in a large P/T range
- Collaborative work to go from proof of principle experiments to systematic fine microscopic measurements!
- But a lot of issues still need to be fixed
  - Integration into the beamlines
  - Adaptation of the existing beamlines to time-resolved conditions
  - New XR diagnostics (fast gate, high frame rate, high sensitivity)
  - New P/T diagnostics at all the relevant time scales

# Strong collaboration required



Imperial College  
London



Science & Technology  
Facilities Council



ÉCOLE POLYTECHNIQUE  
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ACO-CHOCOLAS

Matériaux sous  
Hautes Vitesses de Déformation

Groupe de recherche

*You are all welcome ...*