

# *Dynamics of Crystallization and Melting under Pressure*

*Vitali Prakapenka*

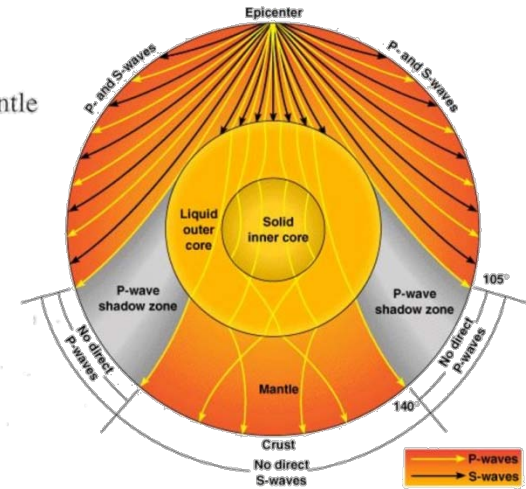
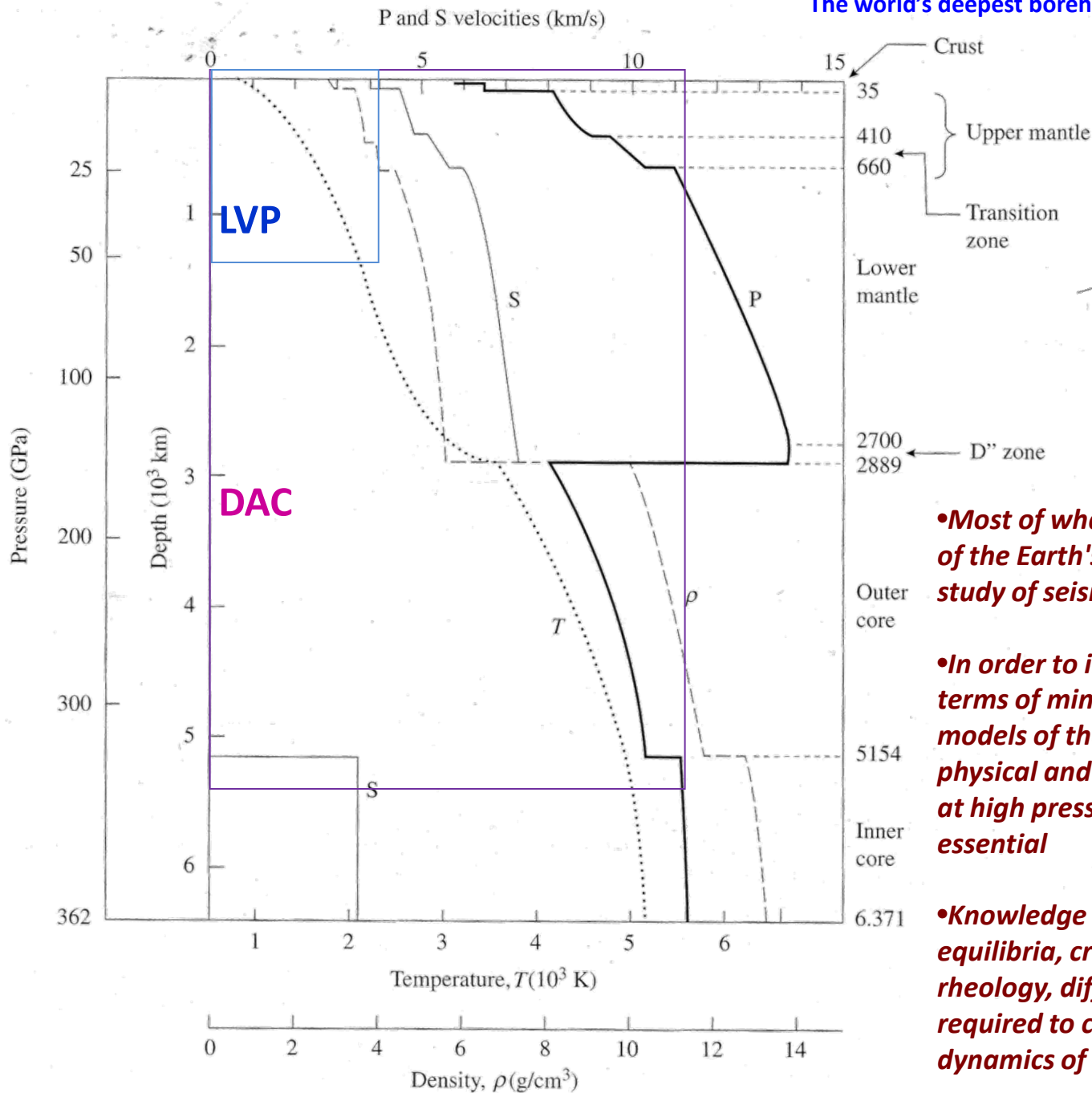


*GSECARS, University of Chicago, Chicago*



[prakapenka@cars.uchicago.edu](mailto:prakapenka@cars.uchicago.edu)

The world's deepest borehole in Sakhalin is 12,345 m (40,502 ft)



• *Most of what we know about the structure of the Earth's deep interior comes from the study of seismic wave velocities*

• *In order to interpret such measurements in terms of mineralogical/compositional models of the Earth's interior, data on the physical and chemical properties of minerals at high pressures and temperatures are essential*

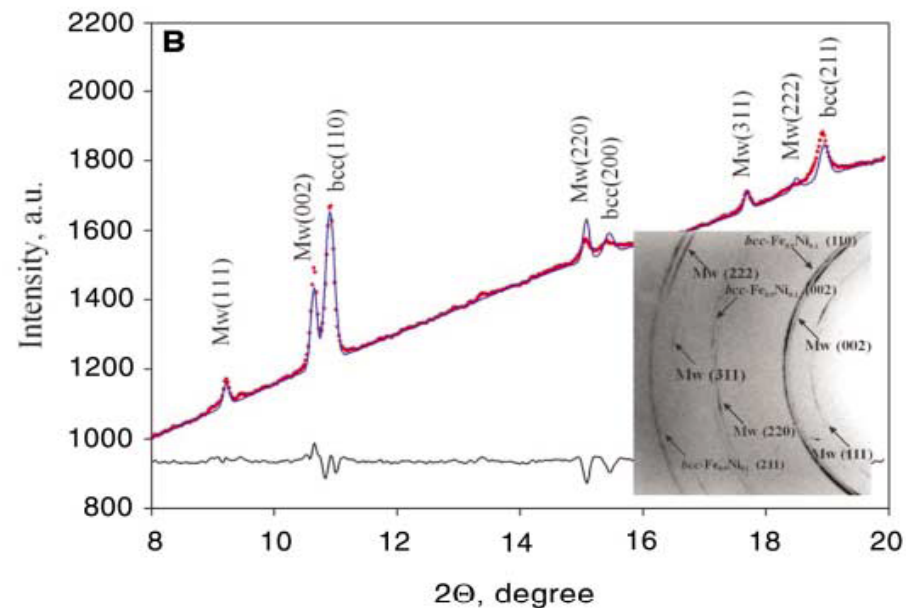
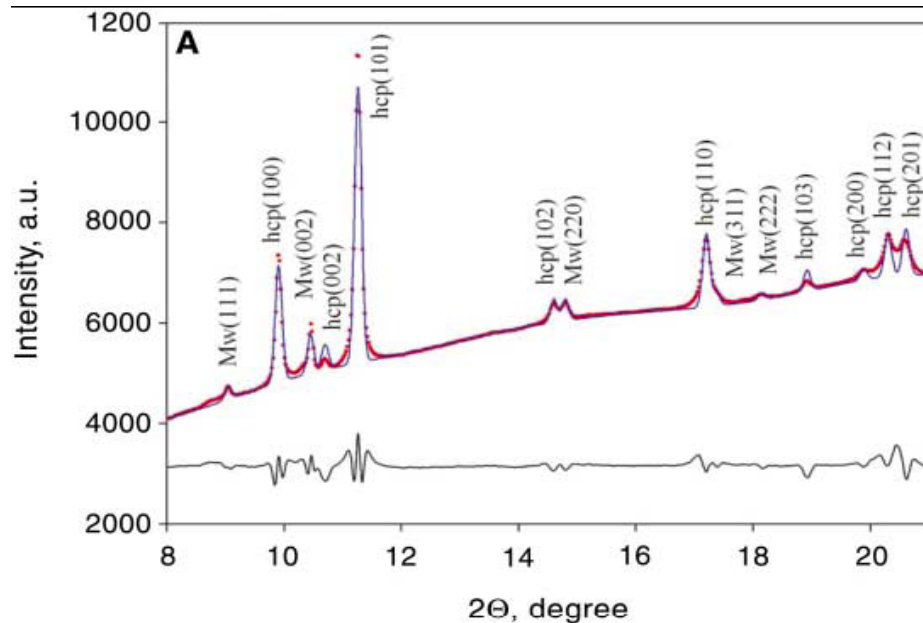
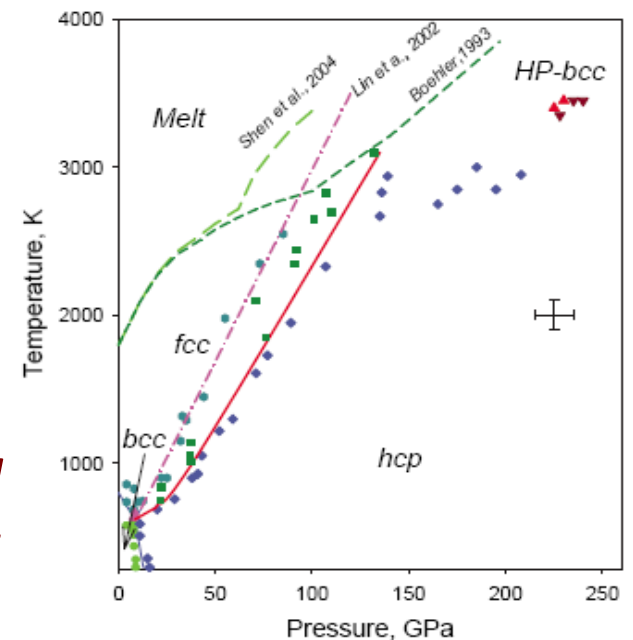
• *Knowledge of thermodynamics, phase equilibria, crystal chemistry, crystallography, rheology, diffusion and heat transport are required to characterize the structure and dynamics of the Earth's deep interior*

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

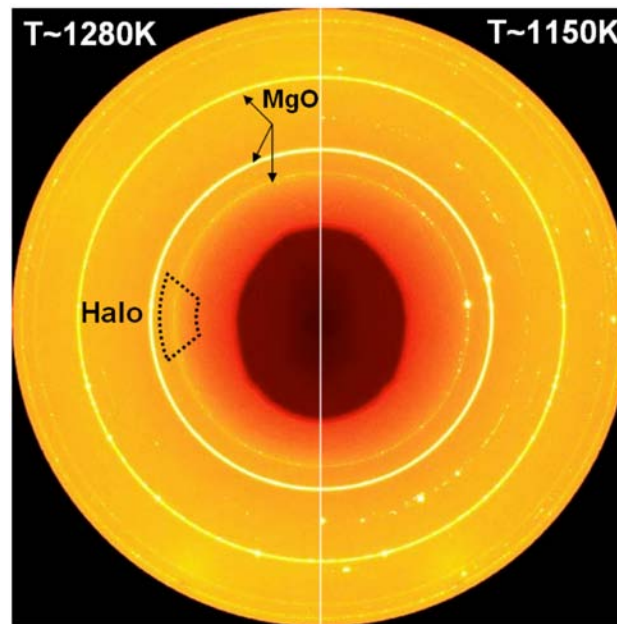
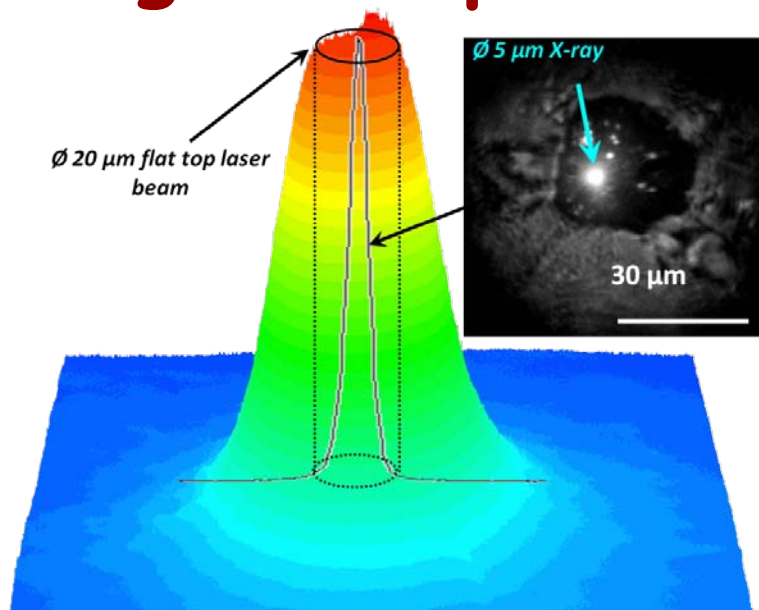
L. Dubrovinsky,<sup>1</sup> N. Dubrovinskaia,<sup>2</sup> O. Narygina,<sup>1</sup> I. Kantor,<sup>1</sup> A. Kuznetsov,<sup>3</sup> V. B. Prakapenka,<sup>3</sup> L. Vitos,<sup>4,5,6</sup> B. Johansson,<sup>4,5</sup> A. S. Mikhaylushkin,<sup>6,7</sup> S. I. Simak,<sup>7</sup> I. A. Abrikosov<sup>7</sup>

*Science* 316, 1880 (2007), Times Cited: [53](#)

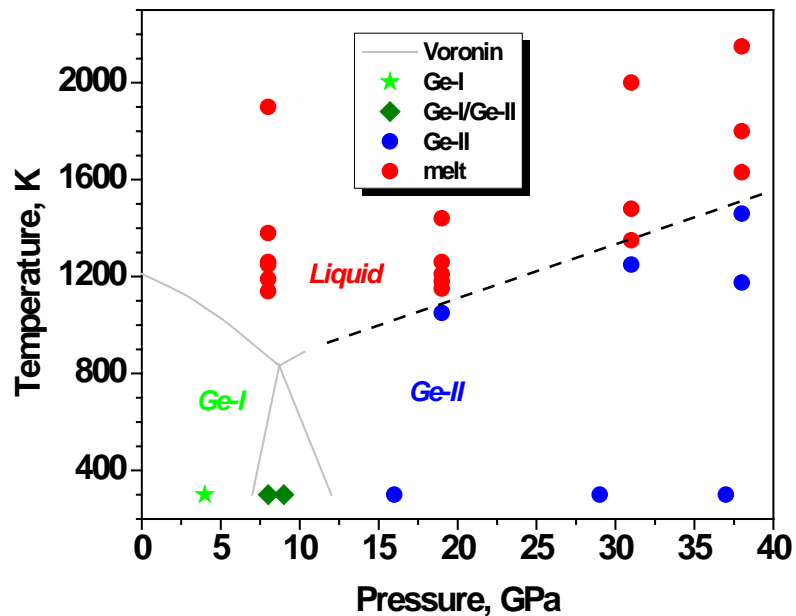
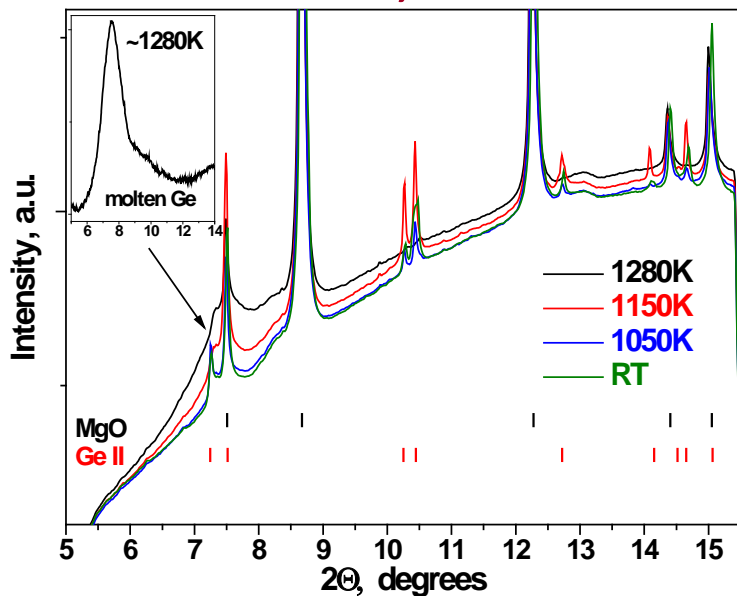
*At pressures above 225 gigapascals and temperatures over 3400 kelvin, Fe<sub>0.9</sub>Ni<sub>0.1</sub> adopts a body-centered cubic structure*

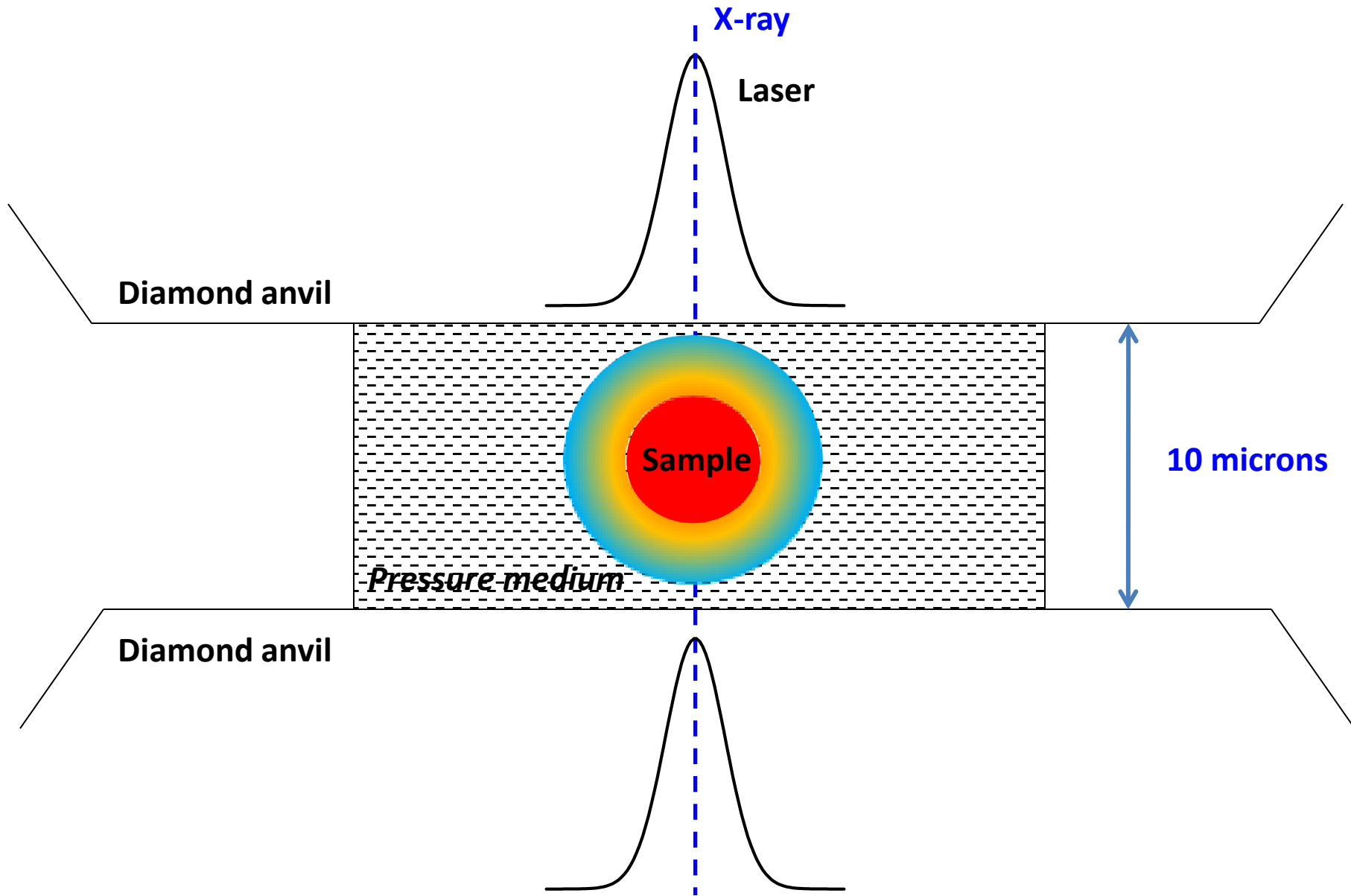


# High P-T phase diagram of Germanium



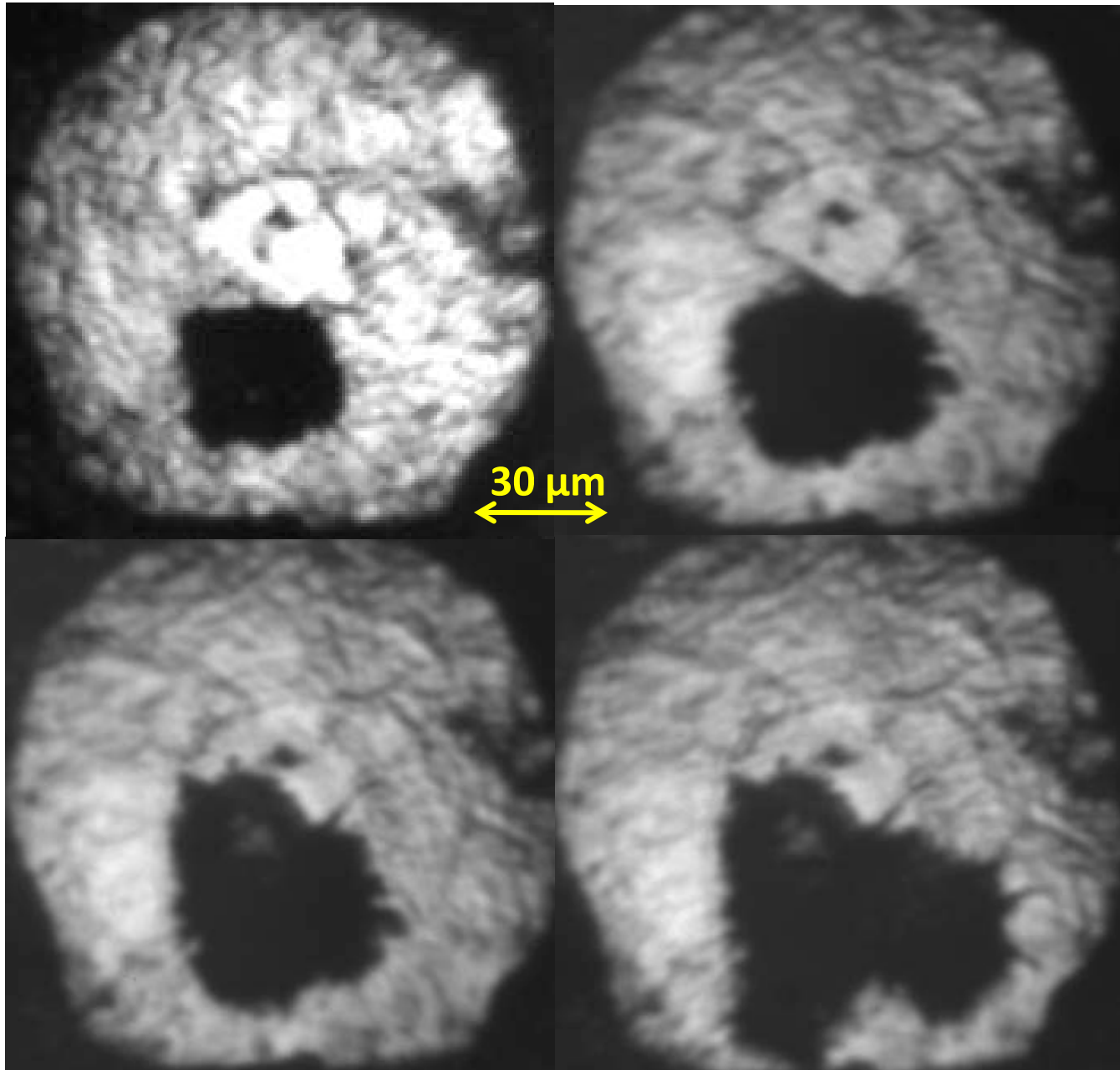
Ge, 19 GPa



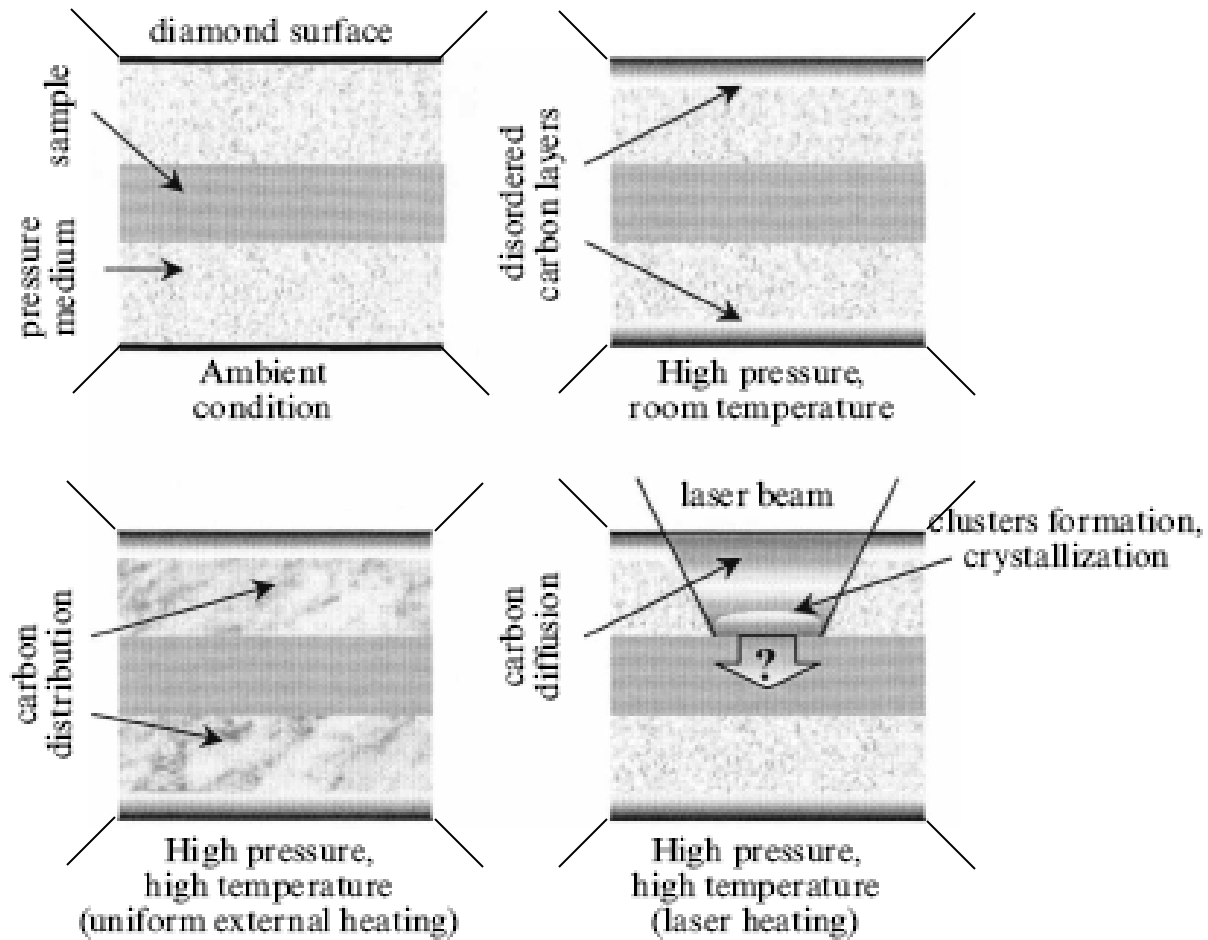


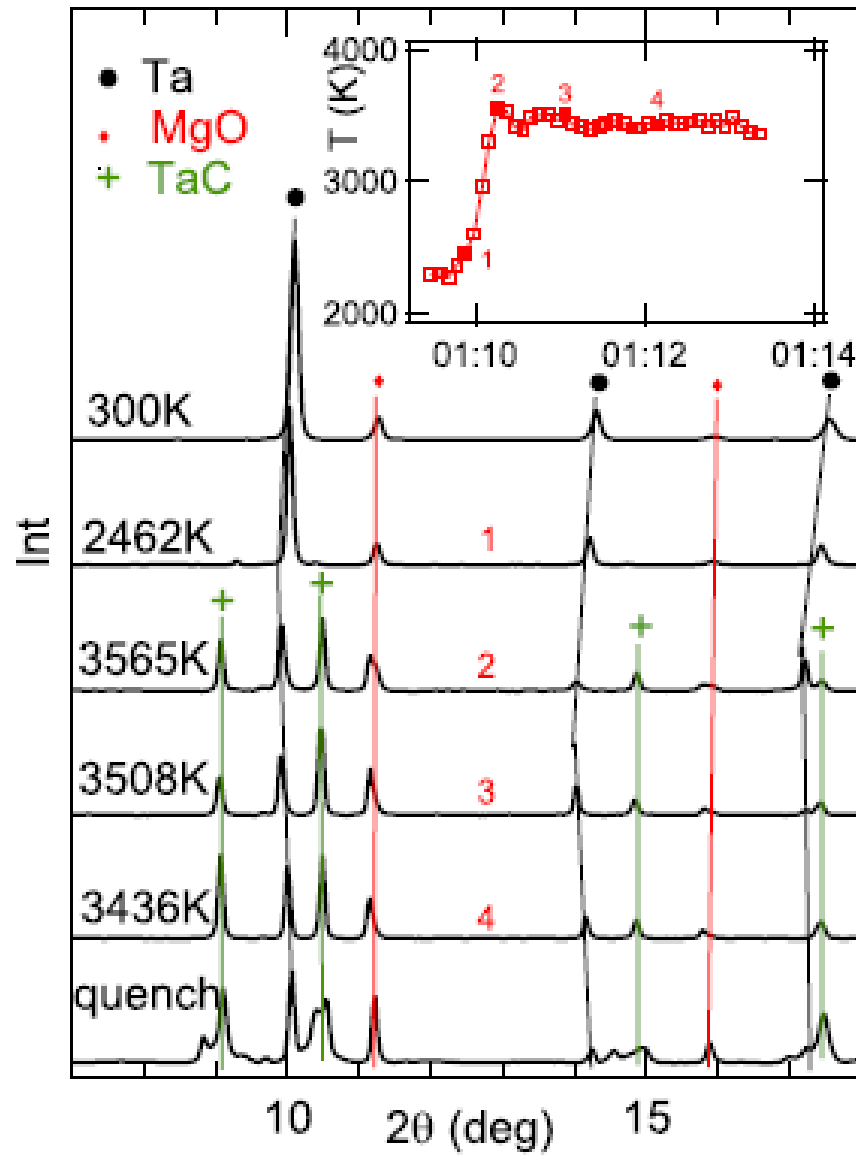
*hot spot in the confined space*

**Fe : SiO<sub>2</sub>, 38 GPa,**

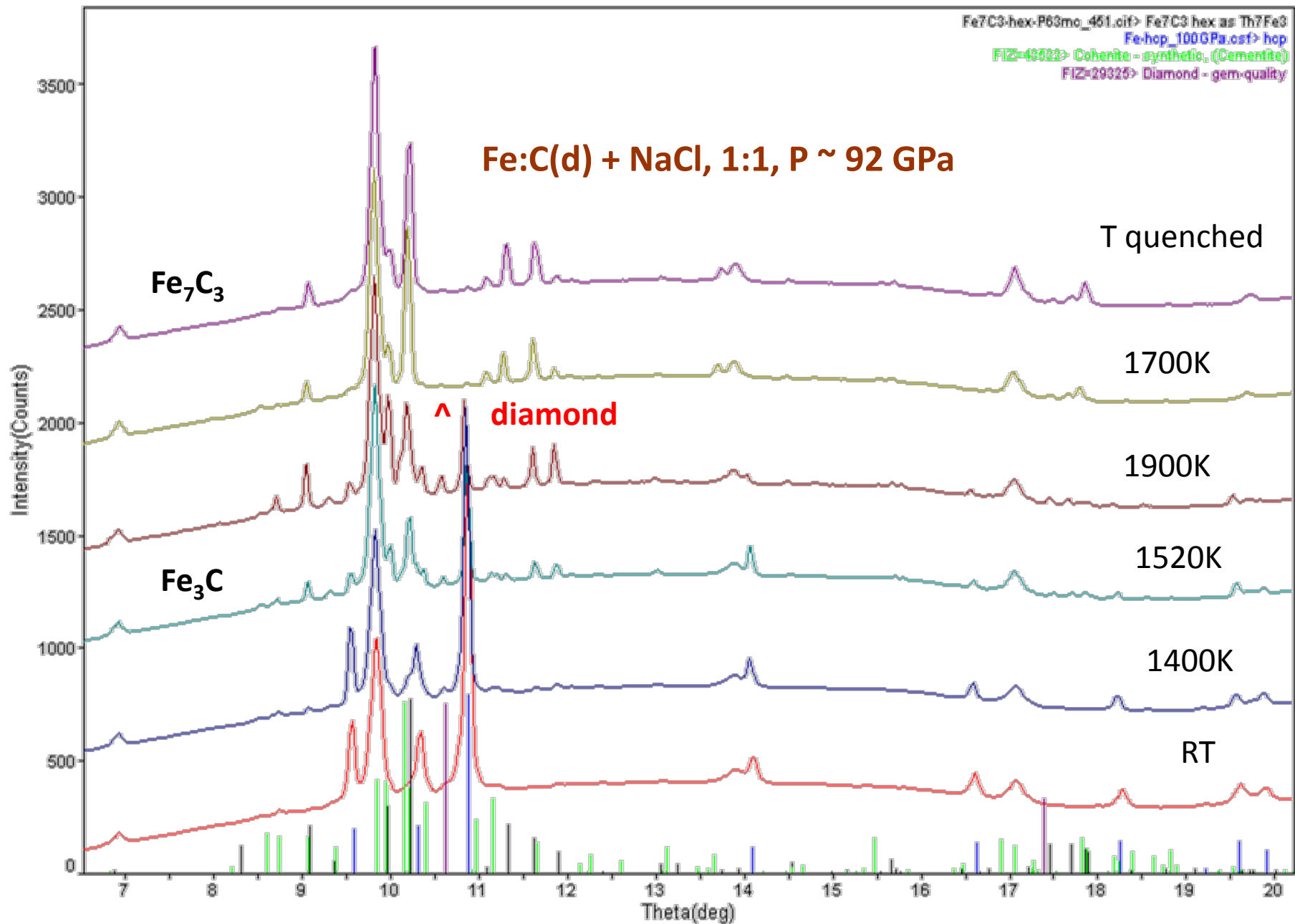


# Carbon transport in diamond anvil cells

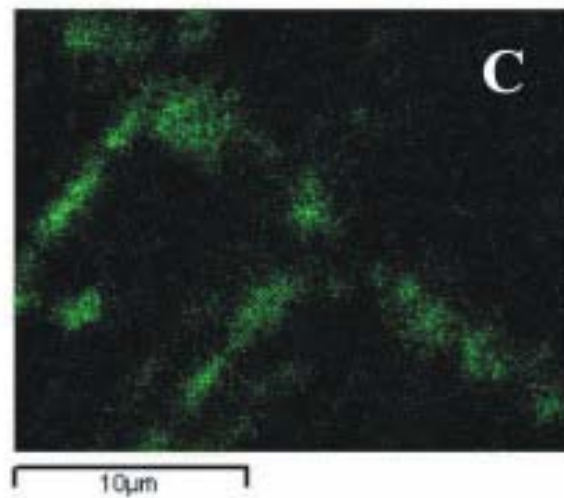
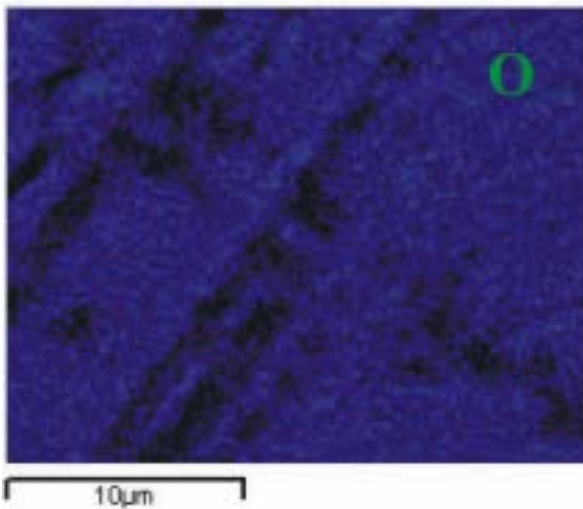
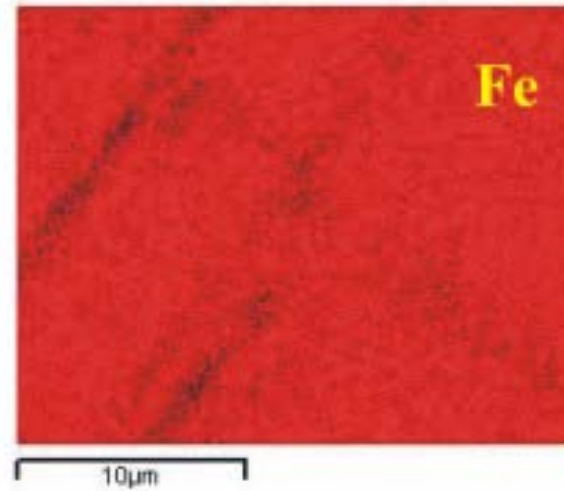
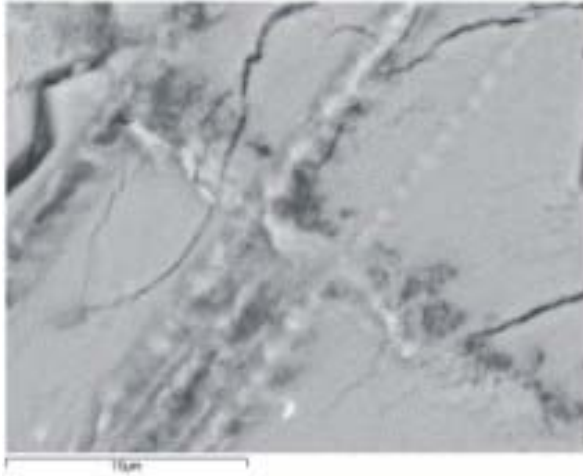




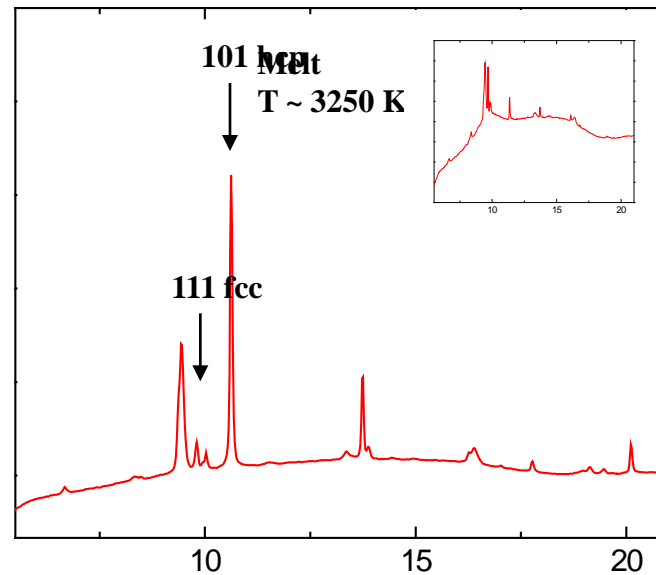
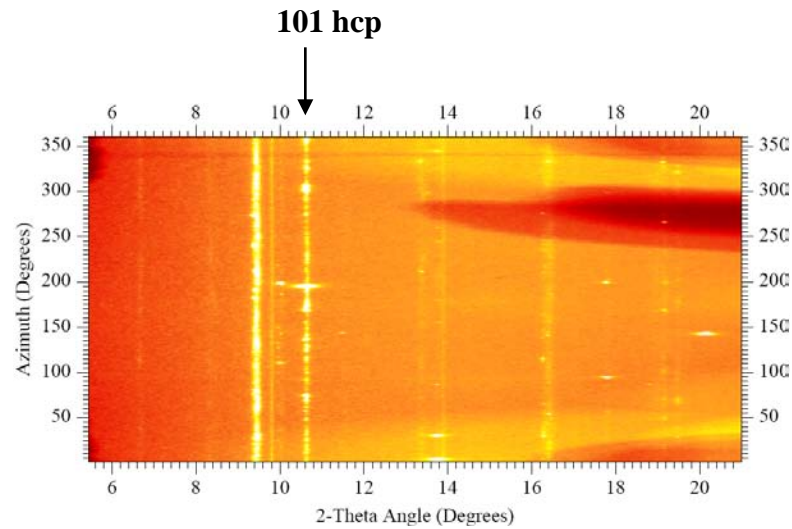




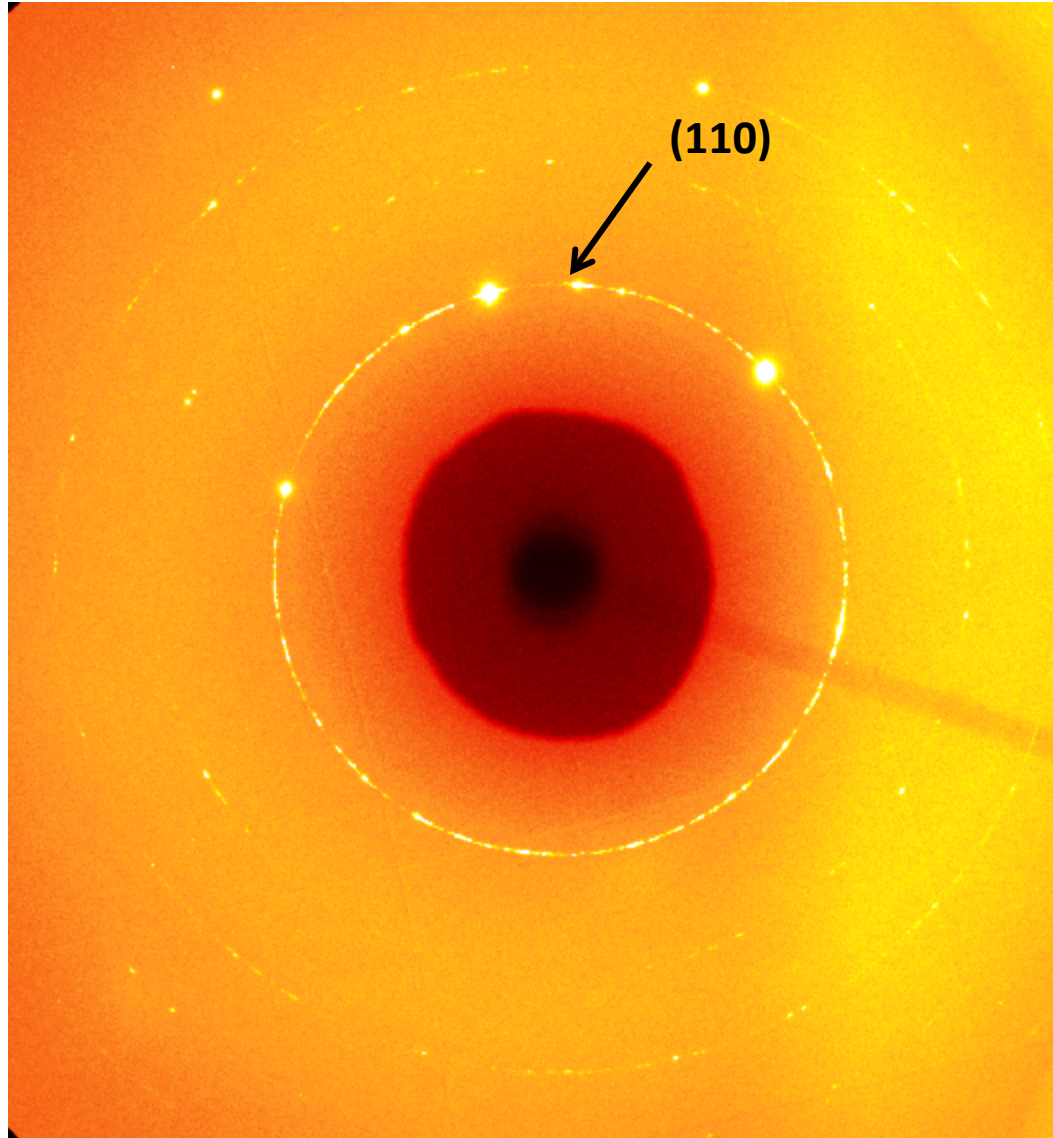
# SEM images of FeO sample after laser heating



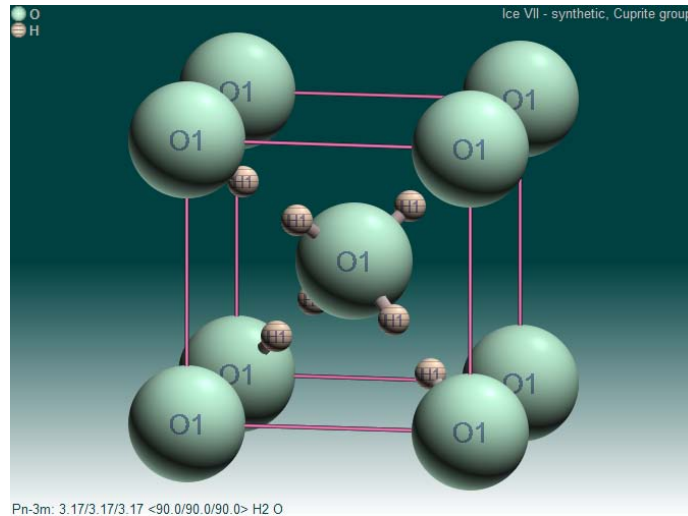
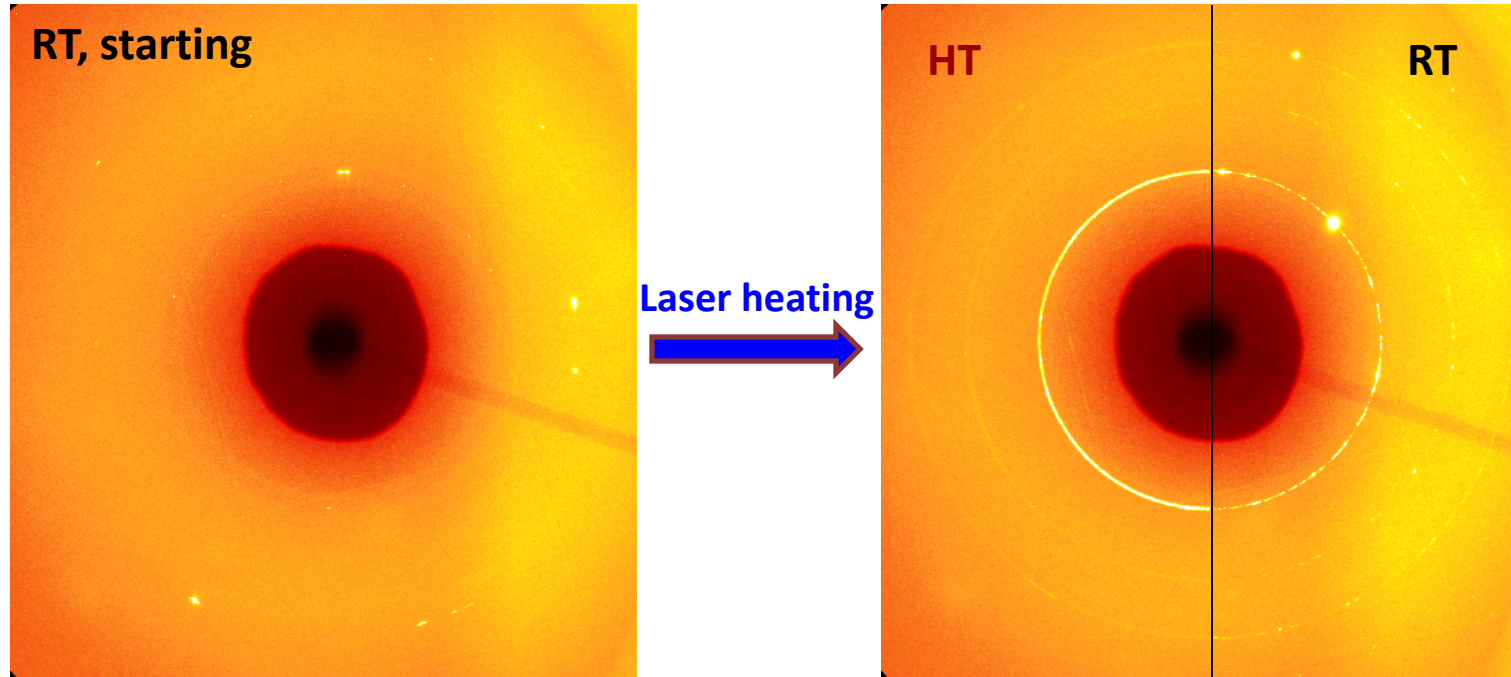
# Melting of Fe-Ni alloy at 60 GPa

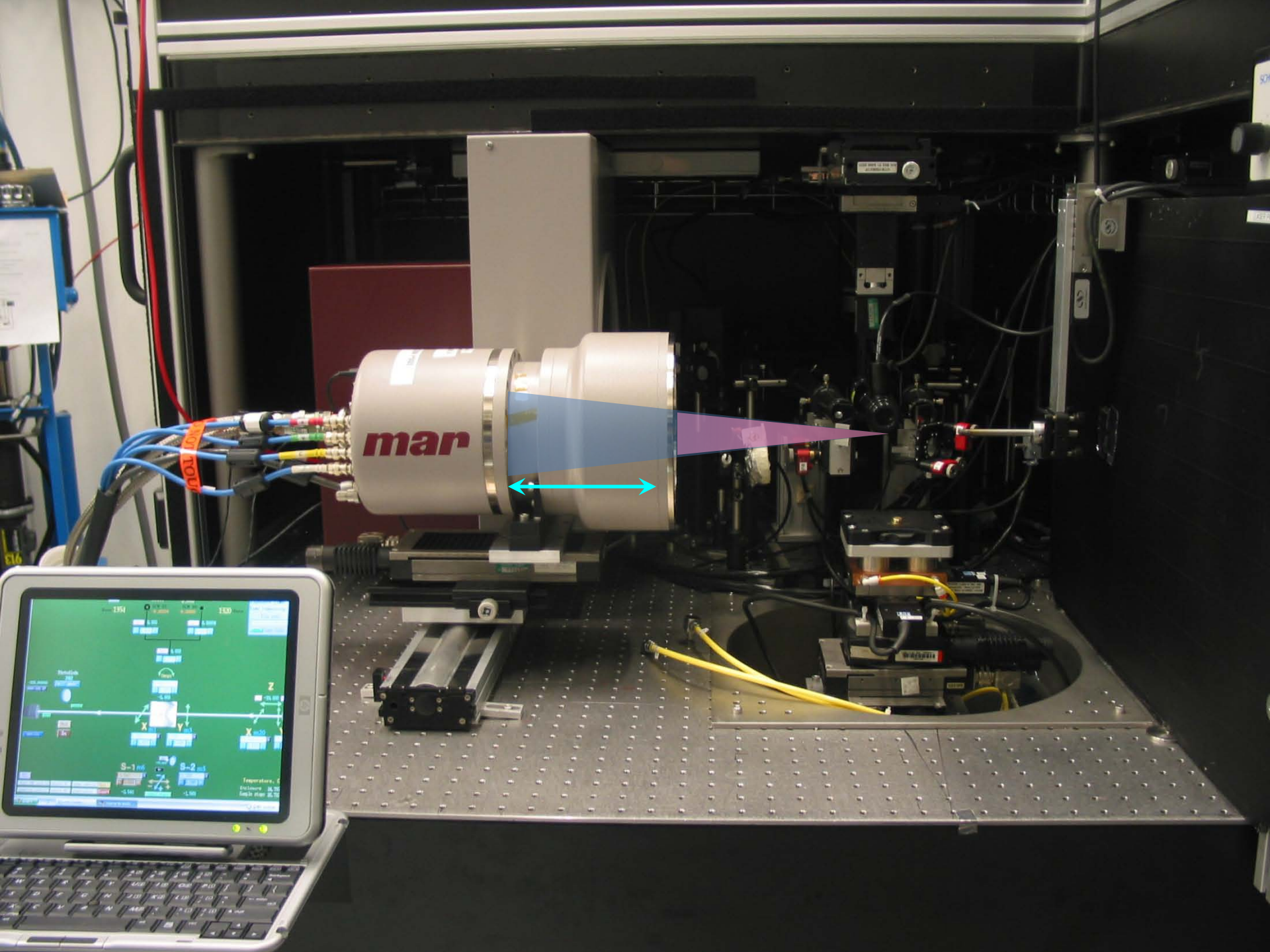


**Ice VII, 7 GPa,  $T_m \sim 600\text{K}$**

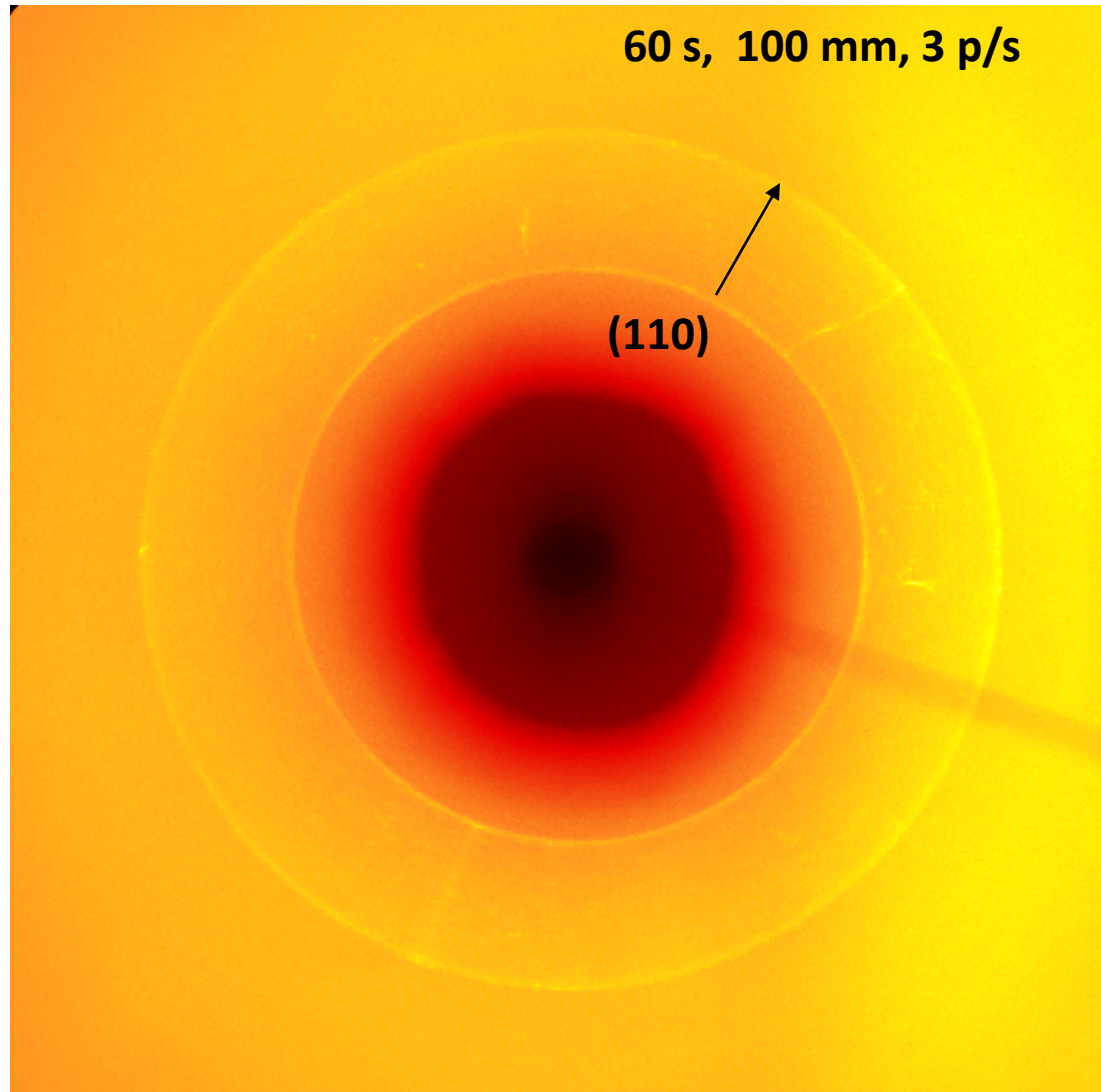


# Ice VII, 7 GPa, $T_m \sim 600\text{K}$



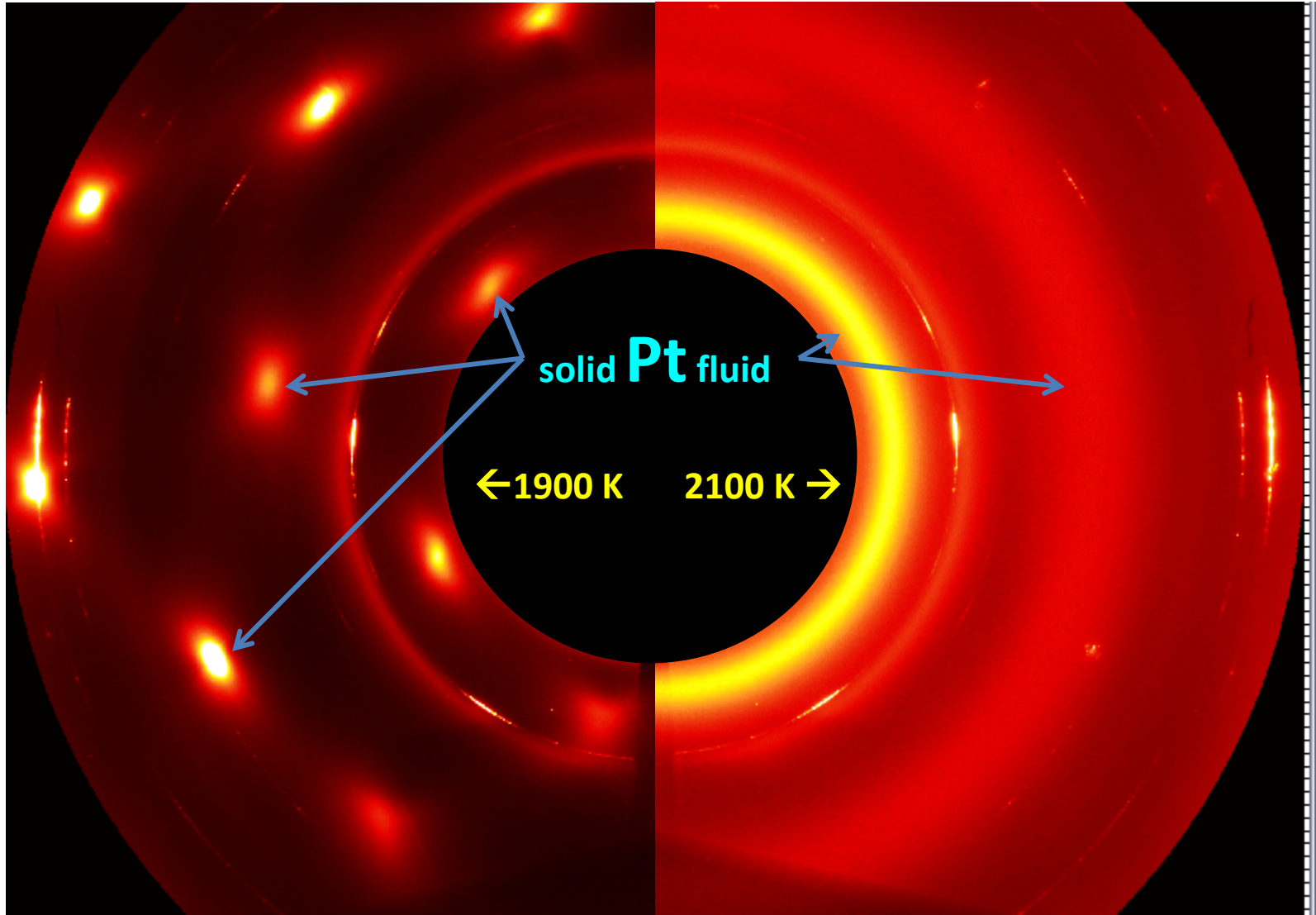


**Ice VII, 7 GPa,  $T_m \sim 600\text{K}$**



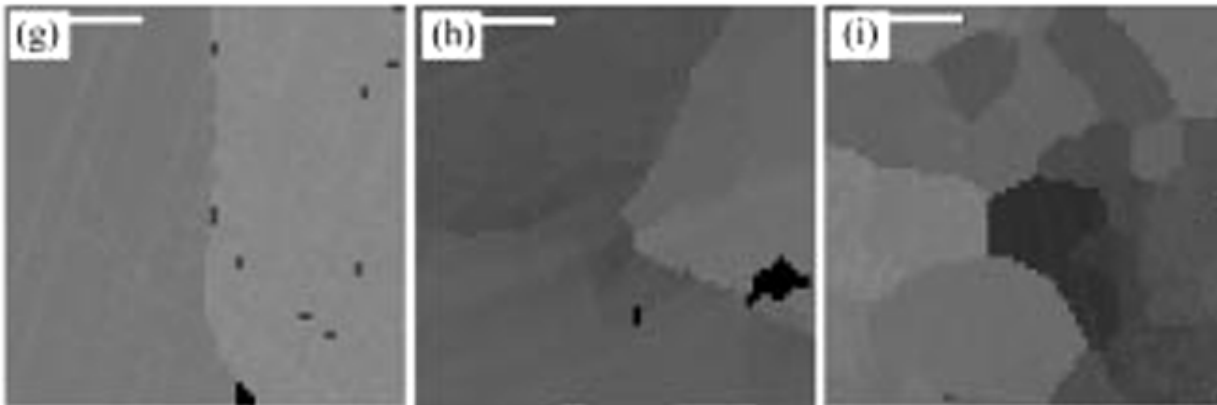
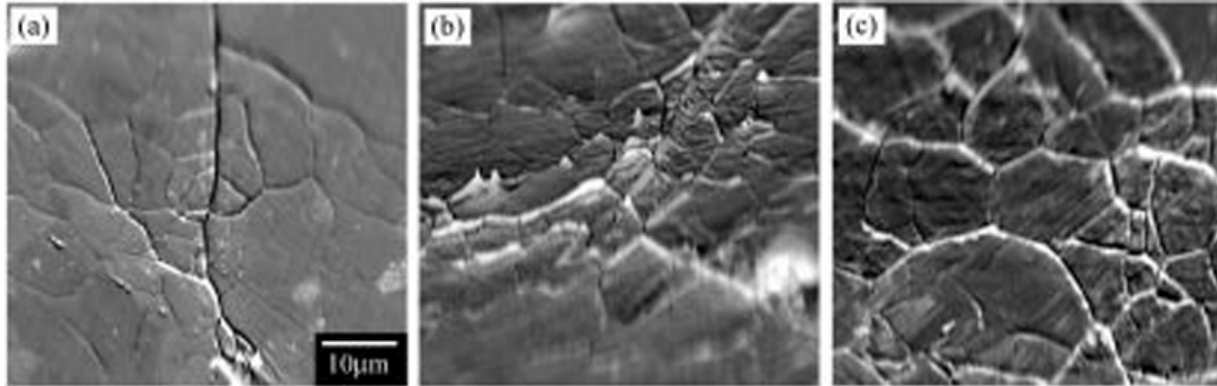
**Laser effect?**

# Pt foil: melting at ambient pressure with double sided laser heating



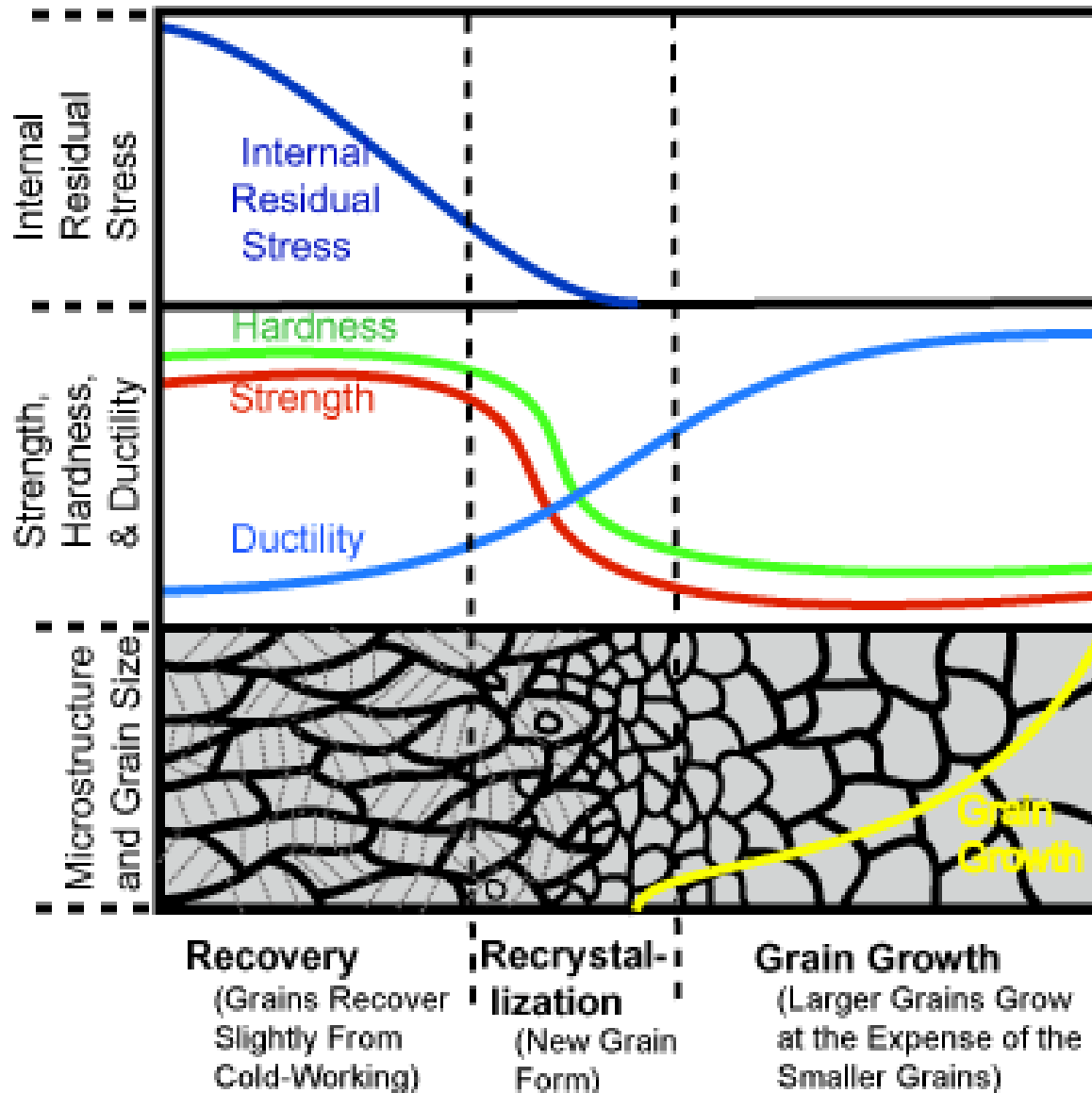


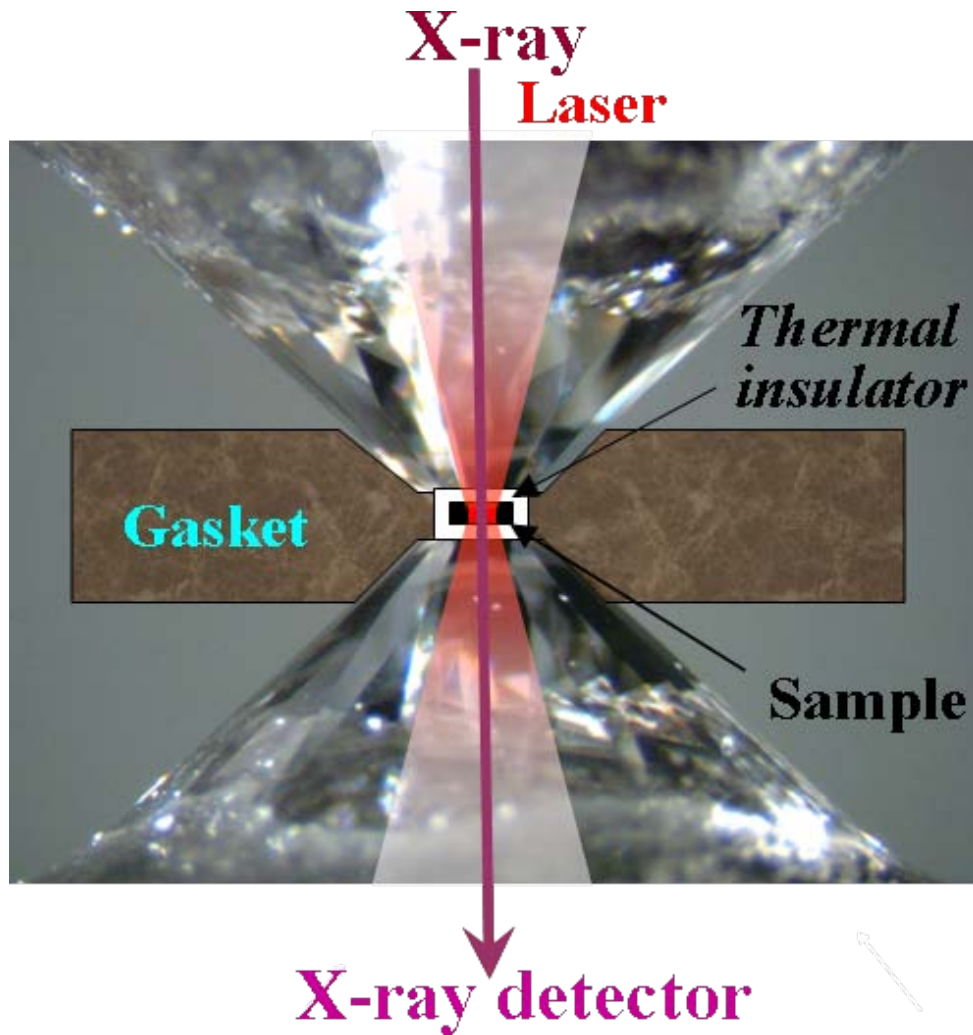
**Images of electron back scattered diffraction at 1000°C and 0.5/sec under various strains; (a) 10%, (b) 50% and (c) 500%**



**Grain boundary misorientation maps of electron back scattered diffraction**

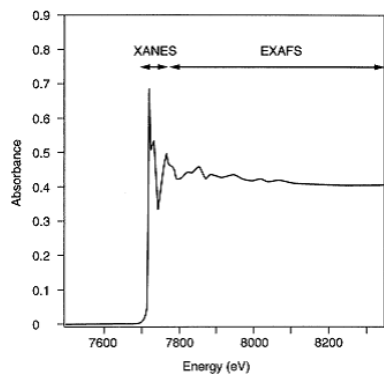
# Dynamic recrystallization





- **Chemistry**
- **Sample diffusion**
- **Crystallization**

# Probing fundamental ultrafast processes



*Atomic vibrations*

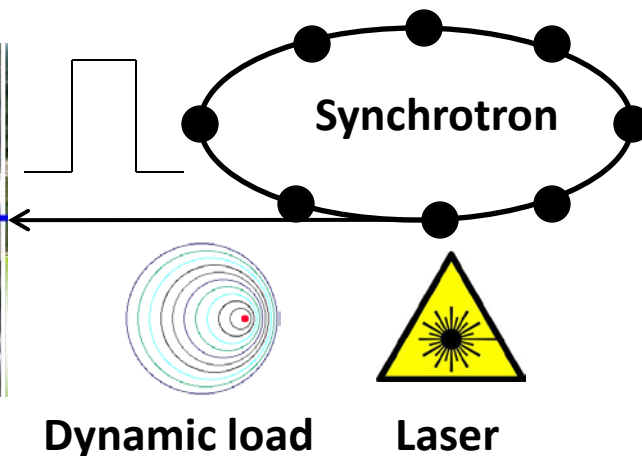
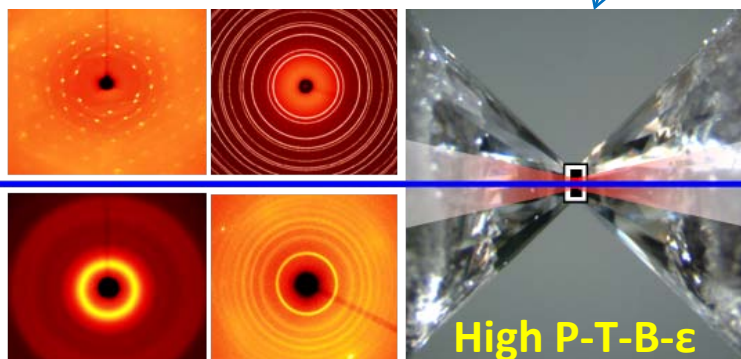
*Phase Transformation*  
*Dislocation nucleation*

*recrystallization*

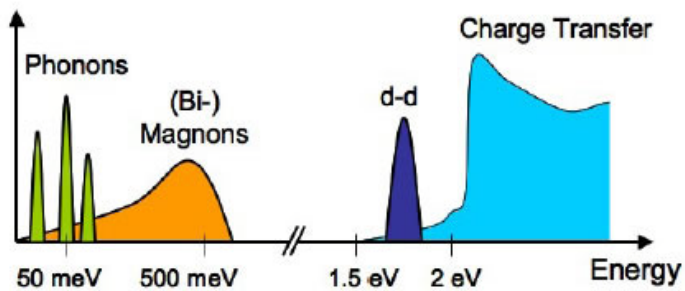
*diffusion*

*Thermally activated reaction dynamics*

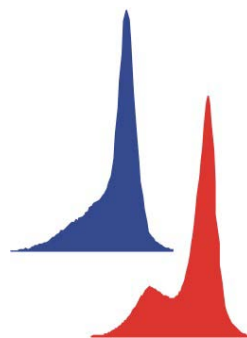
*Static experiment*



Inelastic scattering

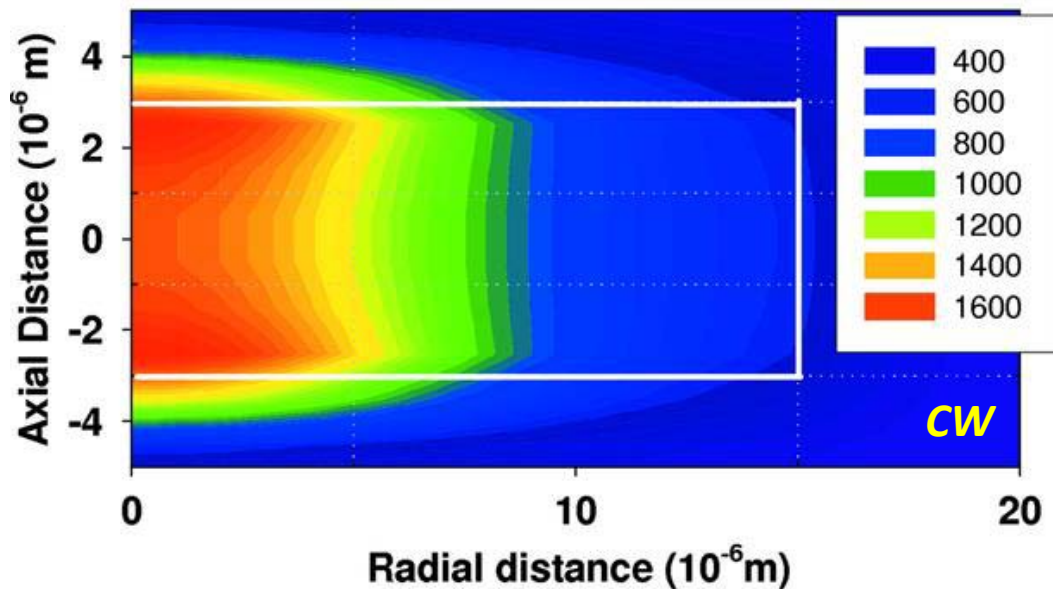


XES

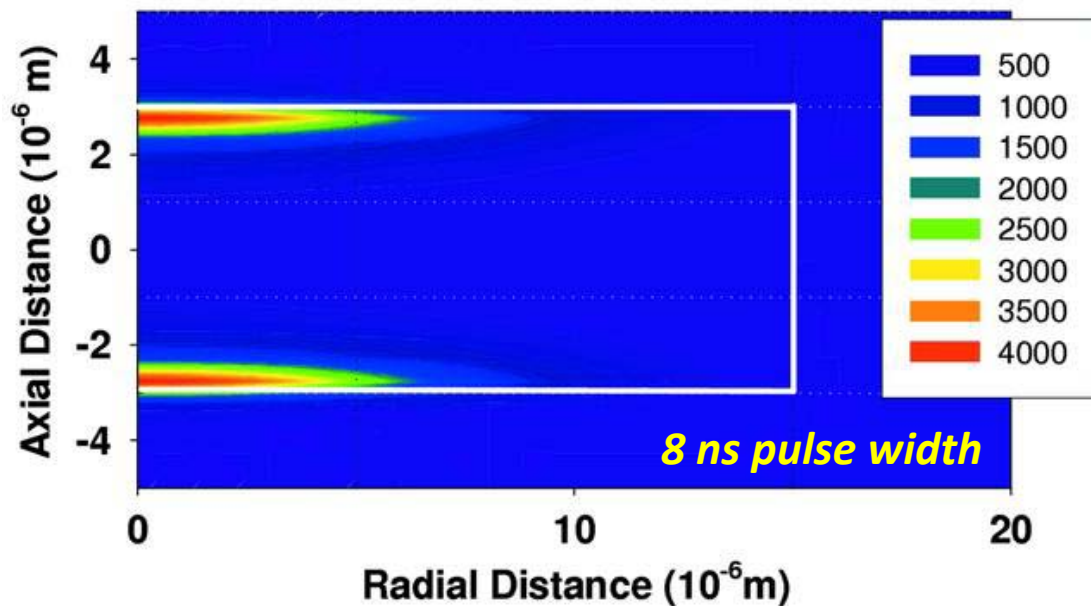


- phase transition kinetics
- structural dynamics & deformation
- chemical reaction dynamics
- transport properties (diffusion)
- electronic properties

# Finite-element calculations of the temperature profiles in the DAC cavity

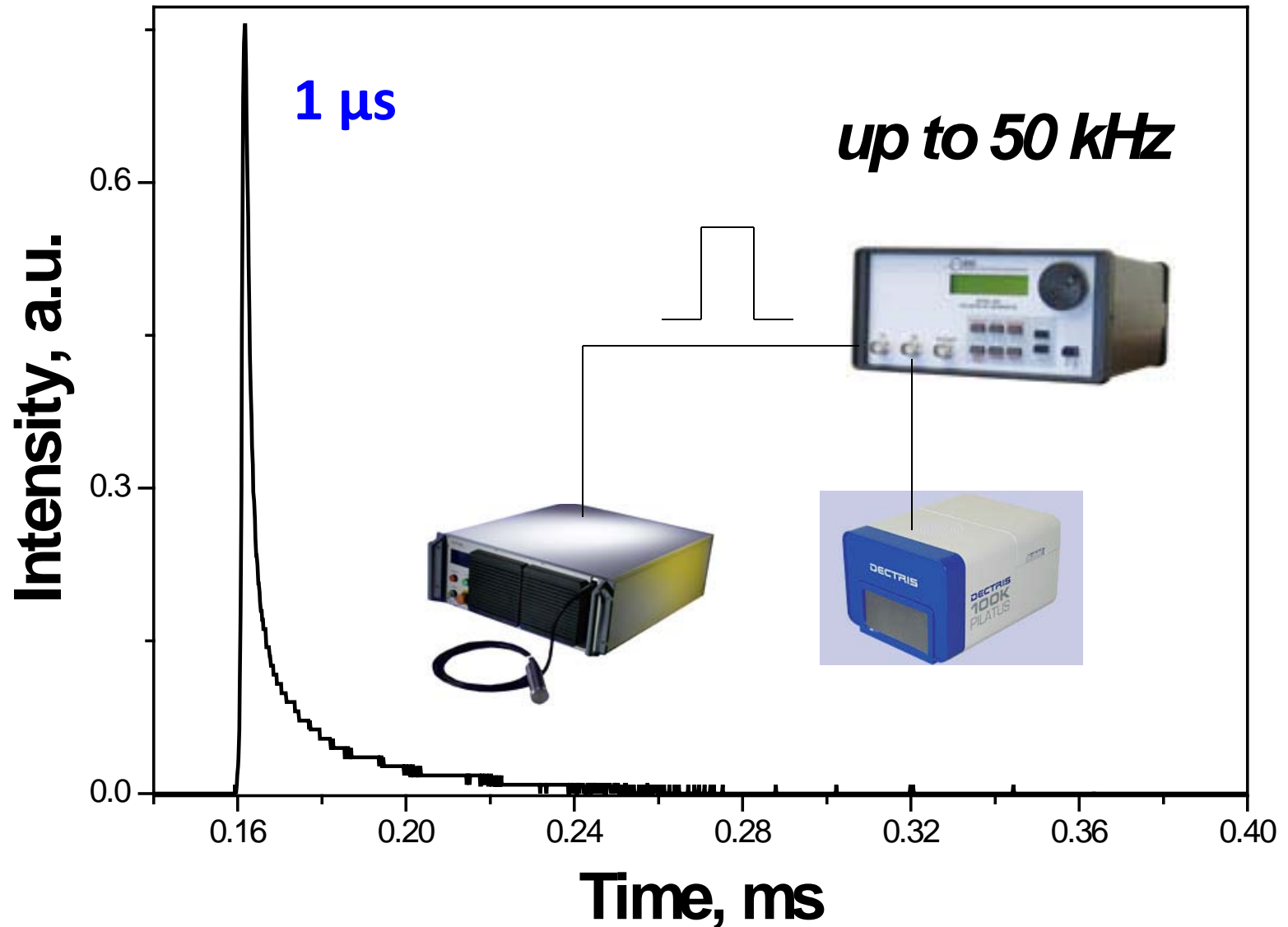


(a)

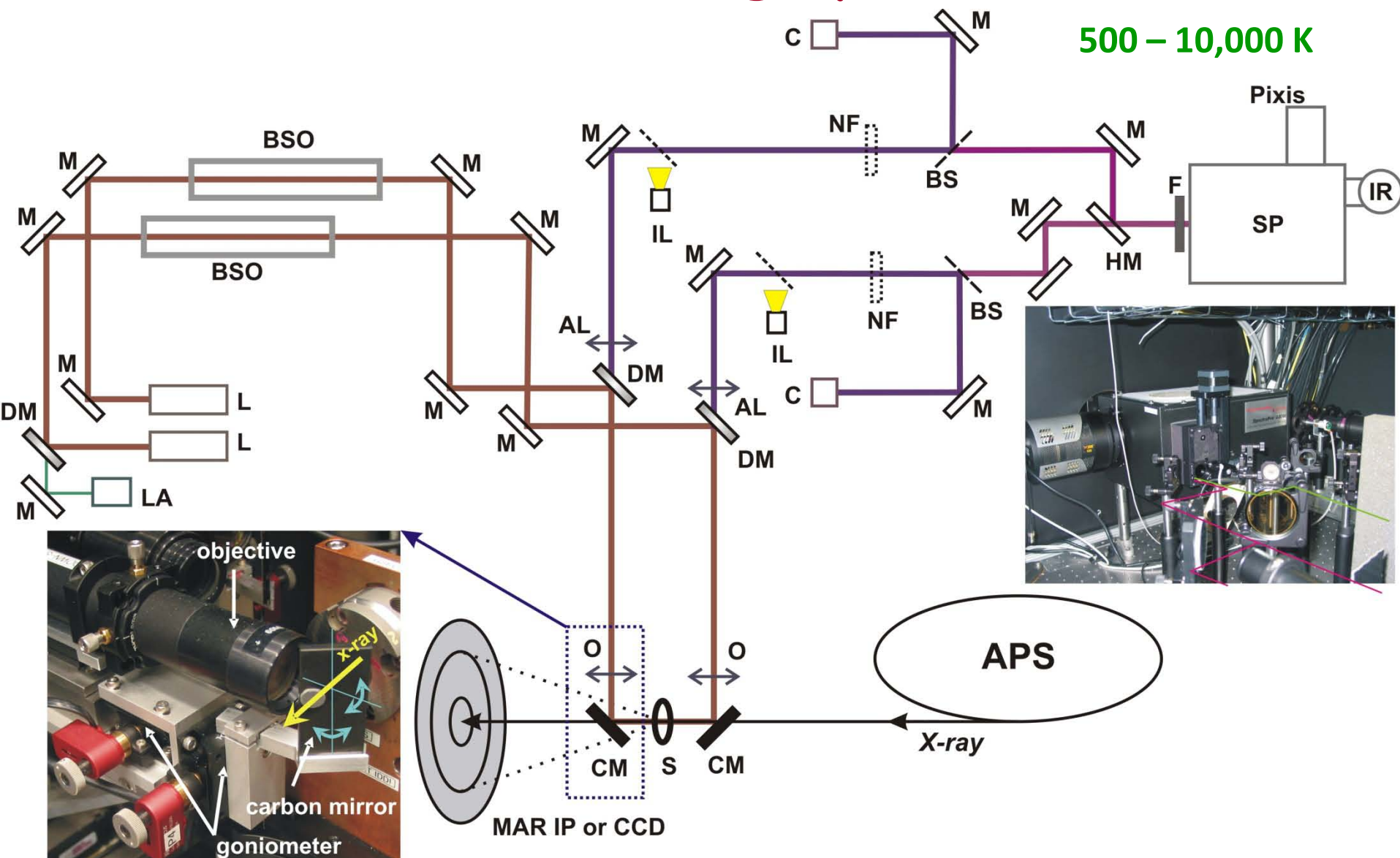


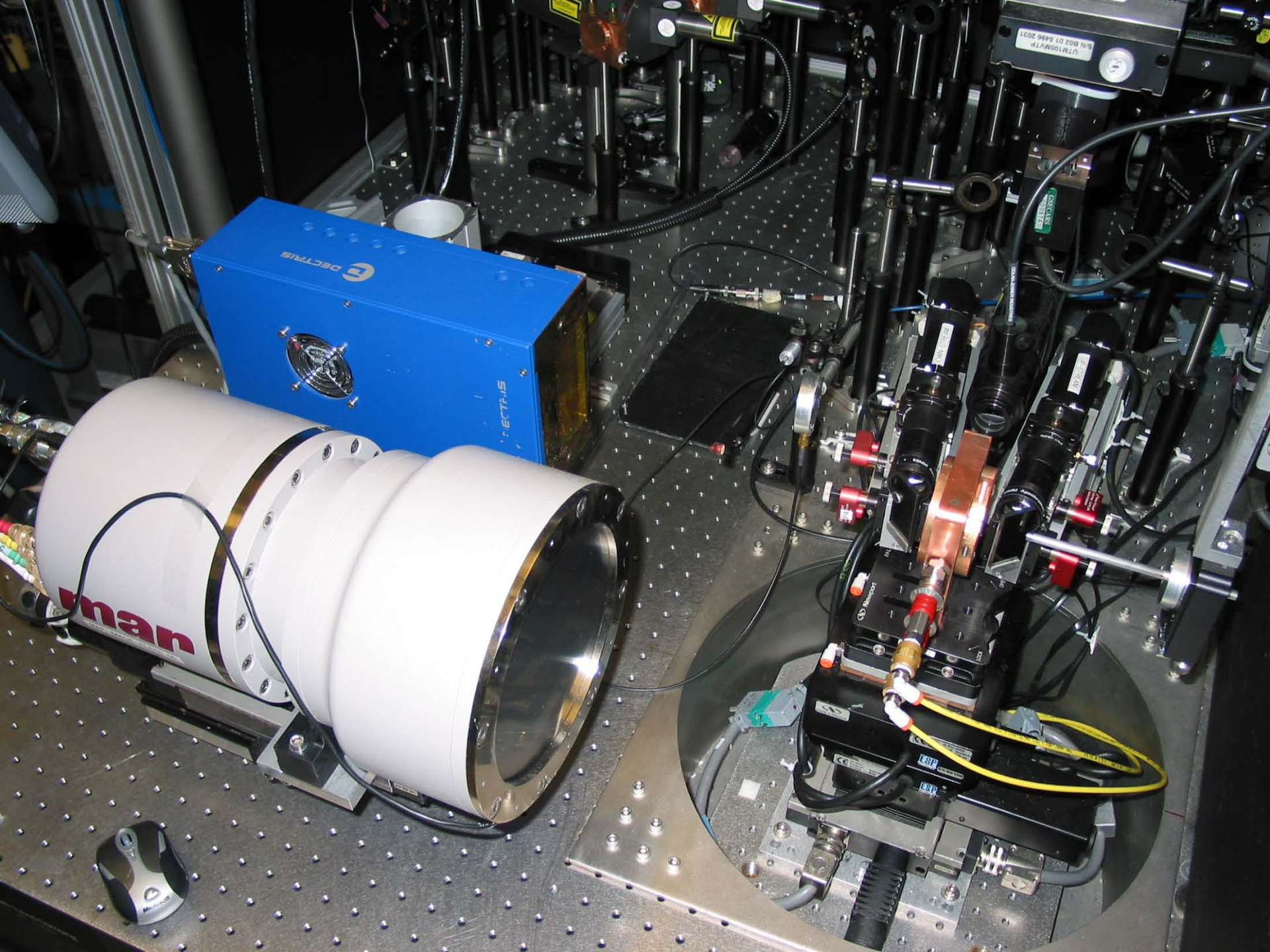
(b)

# Pulse laser heating



# Optical schema of the on-line, flat top, double-sided laser heating system at GSECARS



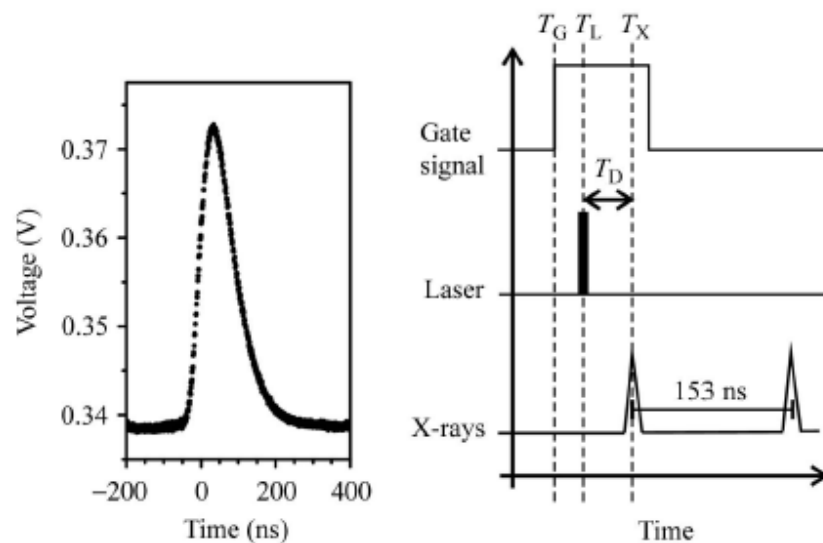




# Picosecond time-resolved laser pump/X-ray probe experiments using a gated single-photon-counting area detector

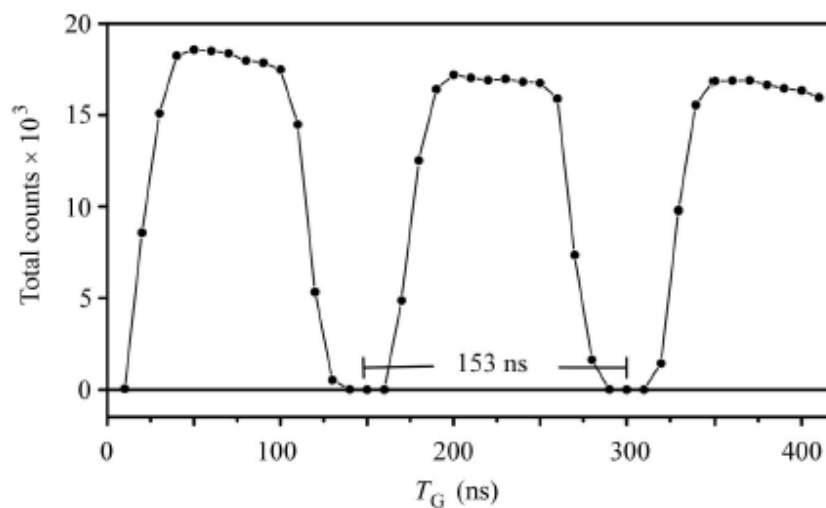
Received 9 December 2008  
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T. Ejdrup,<sup>a\*</sup> H. T. Lemke,<sup>a</sup> K. Haldrup,<sup>a</sup> T. N. Nielsen,<sup>a</sup> D. A. Arms,<sup>b</sup> D. A. Walko,<sup>b</sup>  
A. Miceli,<sup>b</sup> E. C. Landahl,<sup>c</sup> E. M. Dufresne<sup>b</sup> and M. M. Nielsen<sup>a</sup>



(a)

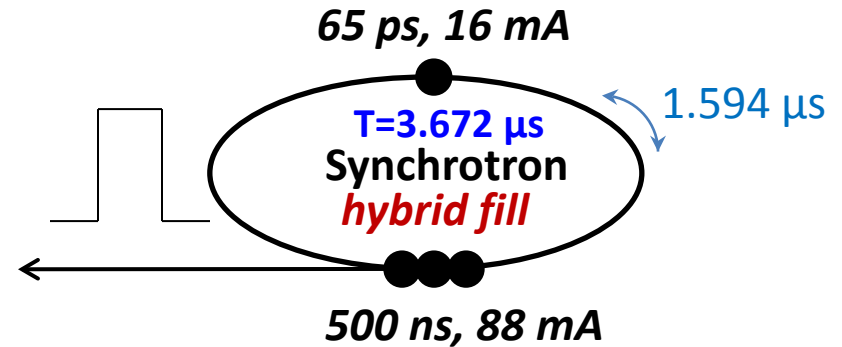
(b)



(c)

### **Standard Operating Mode, top-up**

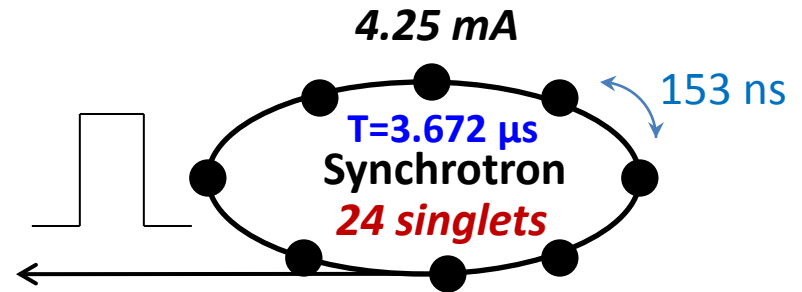
102 mA in 24 singlets (single bunches) with a nominal current of 4.25 mA and a spacing of 153 nanoseconds between 40 ps singlets.



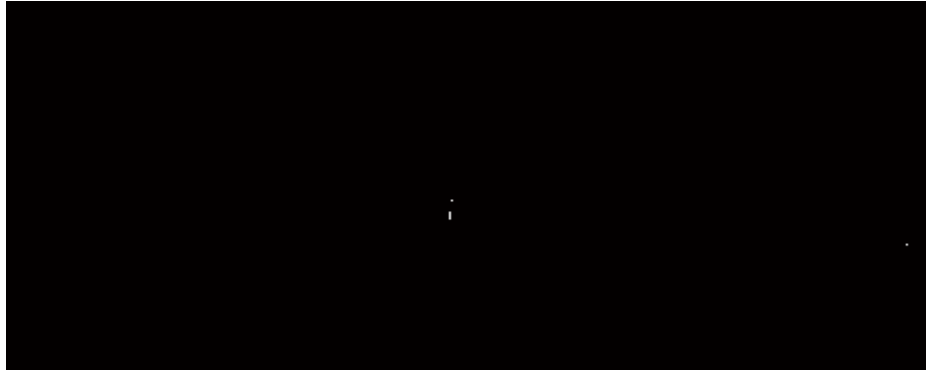
### **Special Operating Mode - hybrid fill, top-up**

Total current is 102 mA. A single bunch containing 16 mA isolated from the remaining bunches by symmetrical 1.594 microseconds gaps.

The remaining current is distributed in 8 group of 7 consecutive bunches with a maximum of 11 mA per group. The total length of the bunch train is 500 ns.

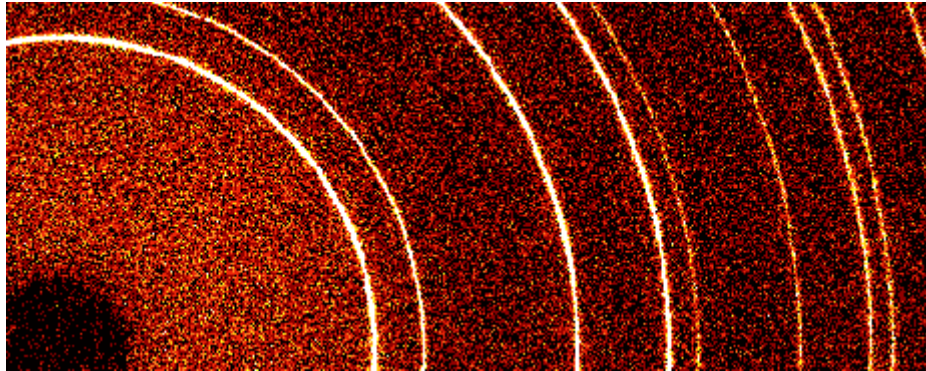


# Pt, 69 GPa

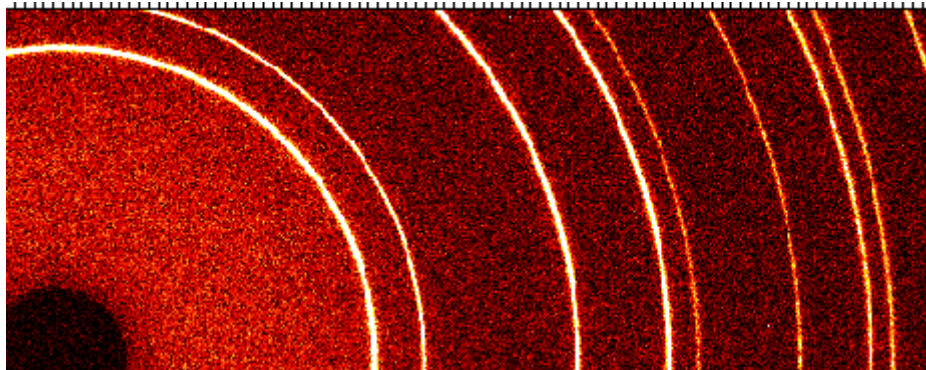


Pilatus, 10000 pls  
600 ns width

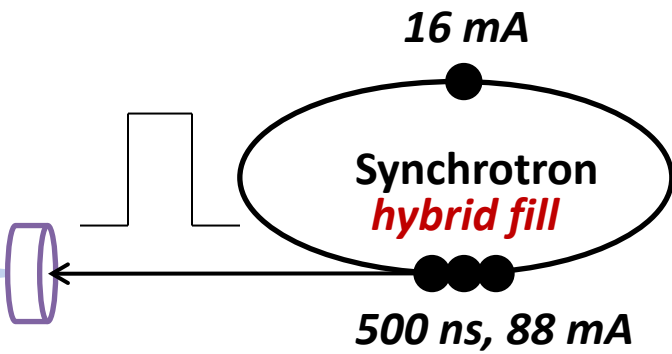
0 ns delay



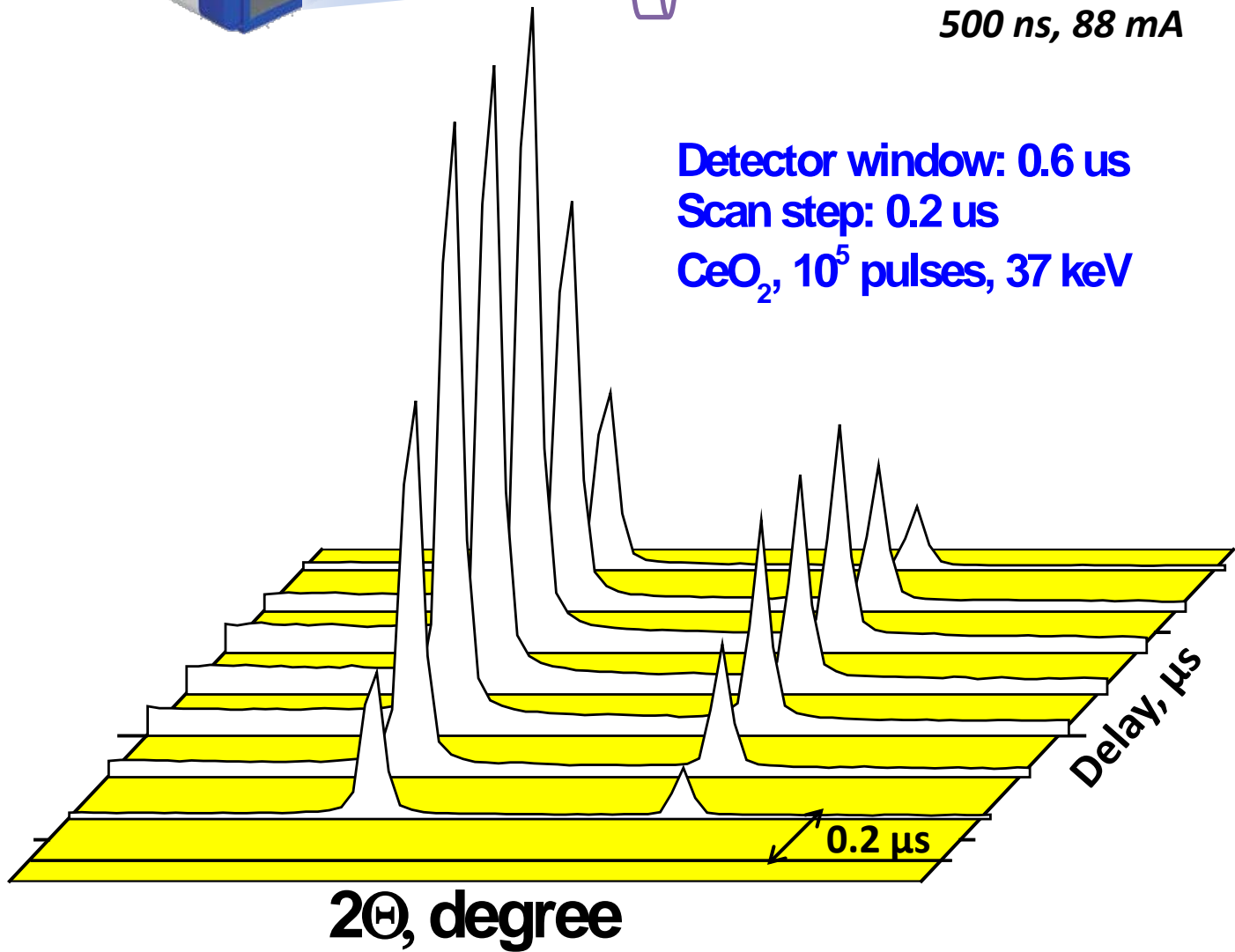
200 ns delay

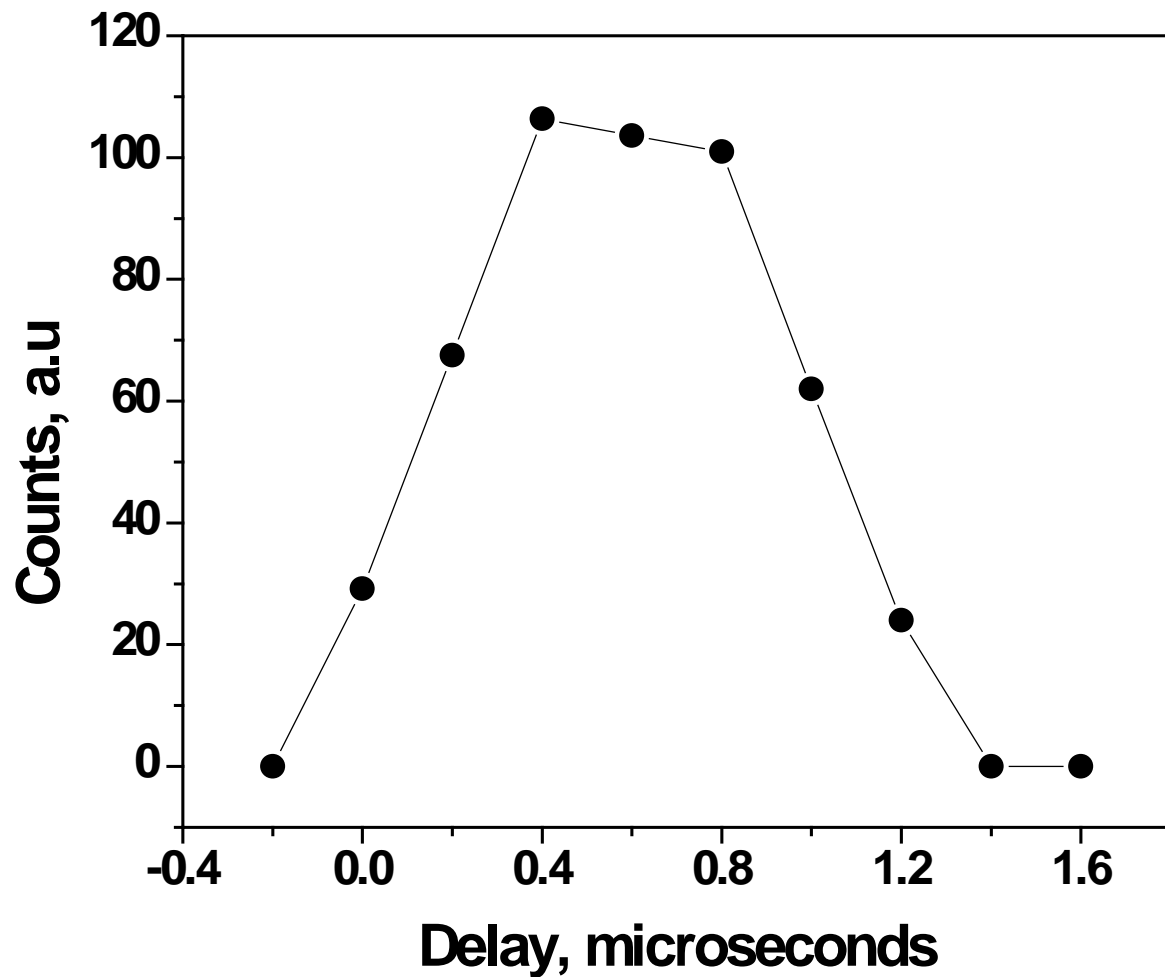
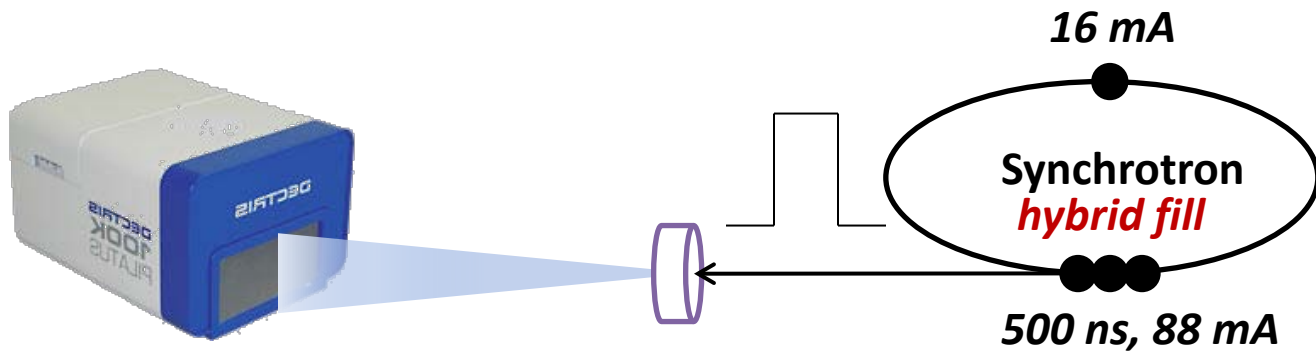


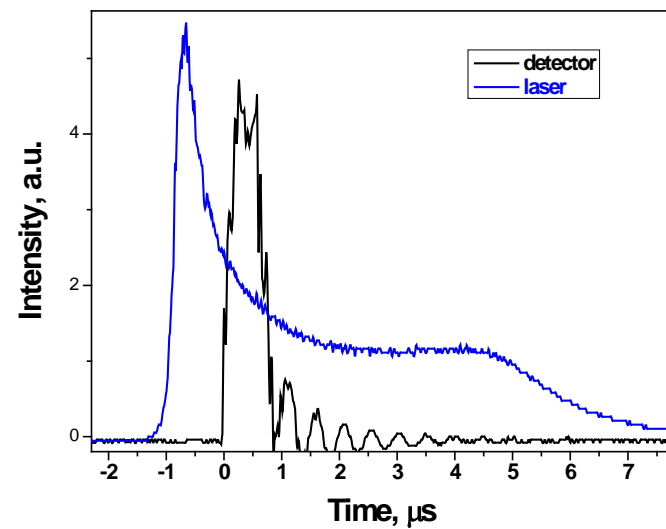
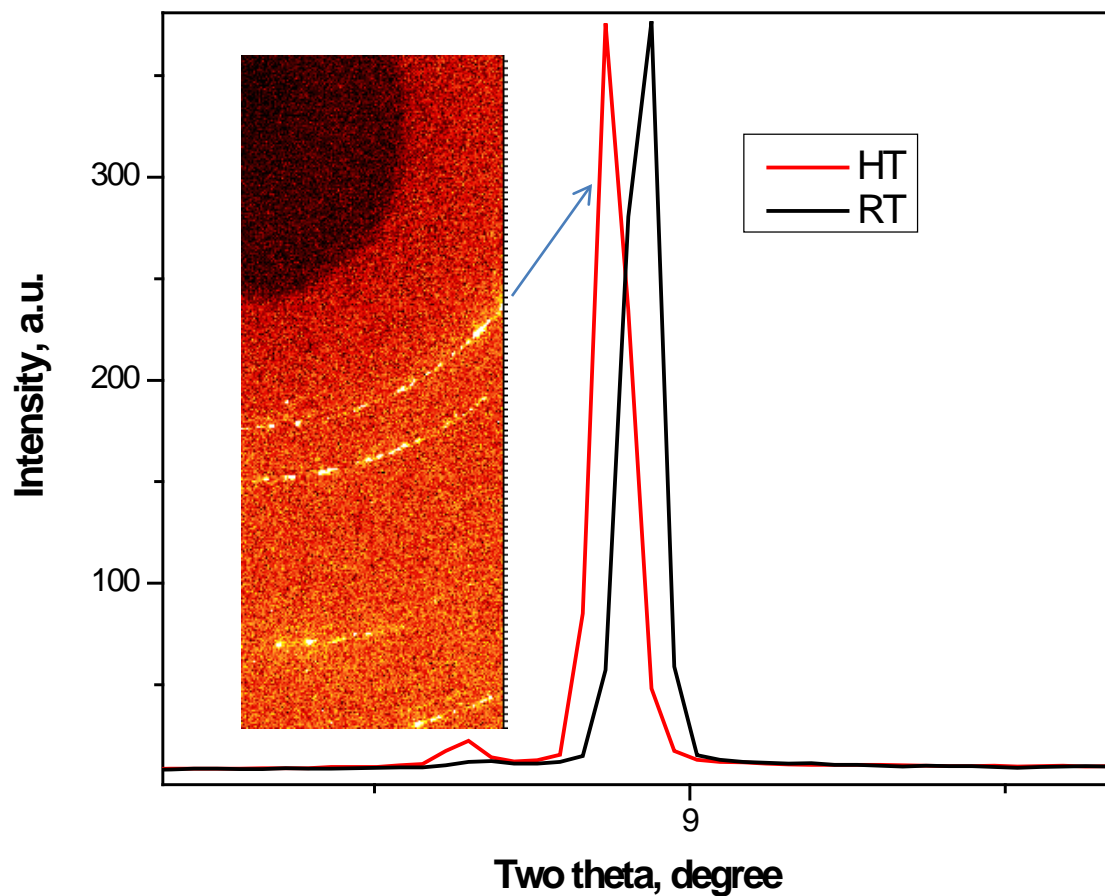
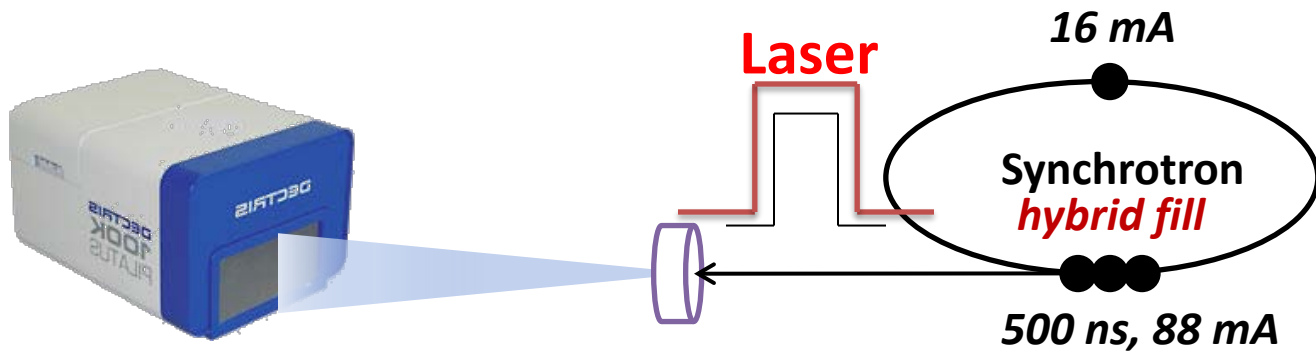
600 ns delay



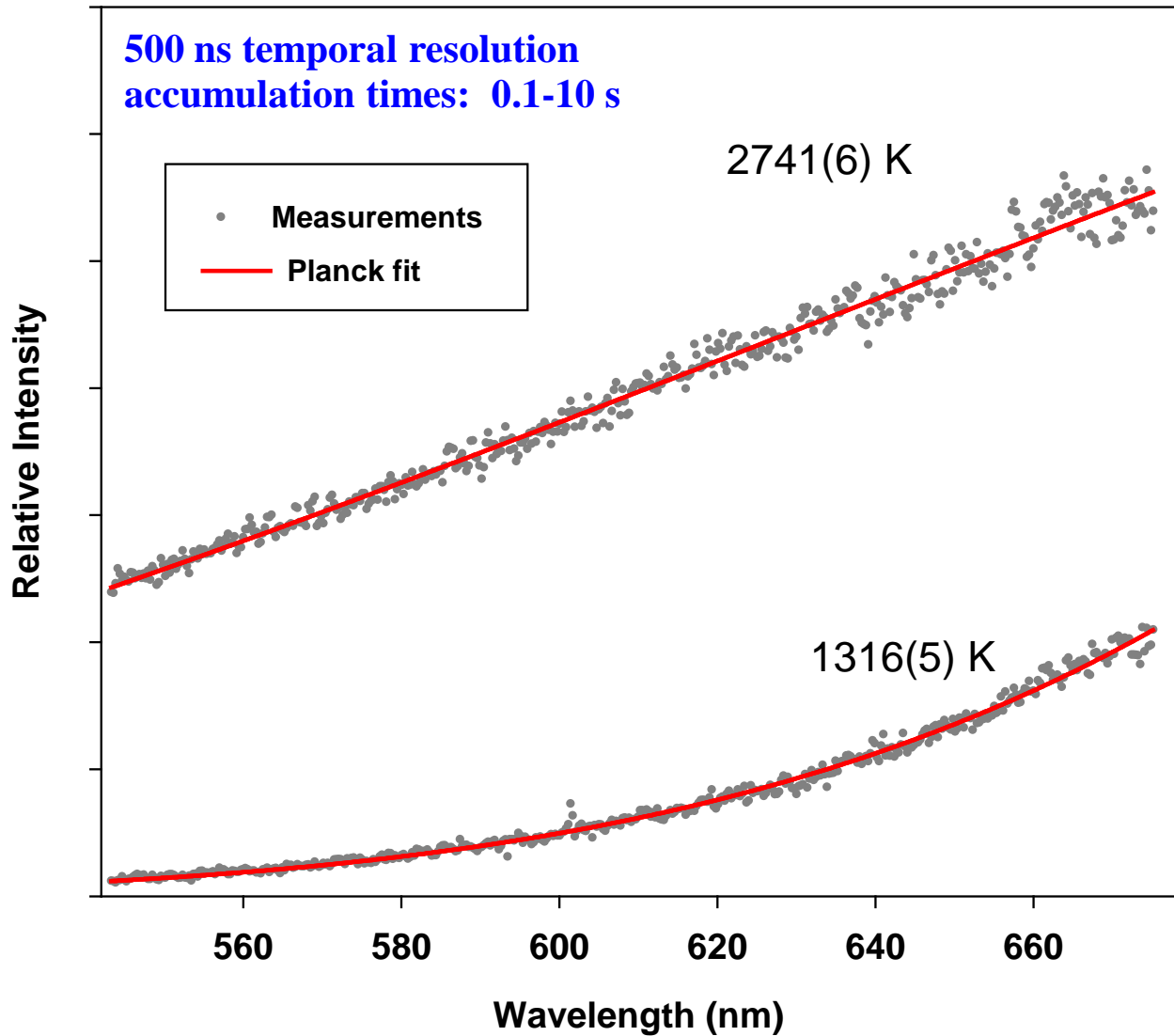
Detector window: 0.6  $\mu$ s  
Scan step: 0.2  $\mu$ s  
CeO<sub>2</sub>, 10<sup>5</sup> pulses, 37 keV



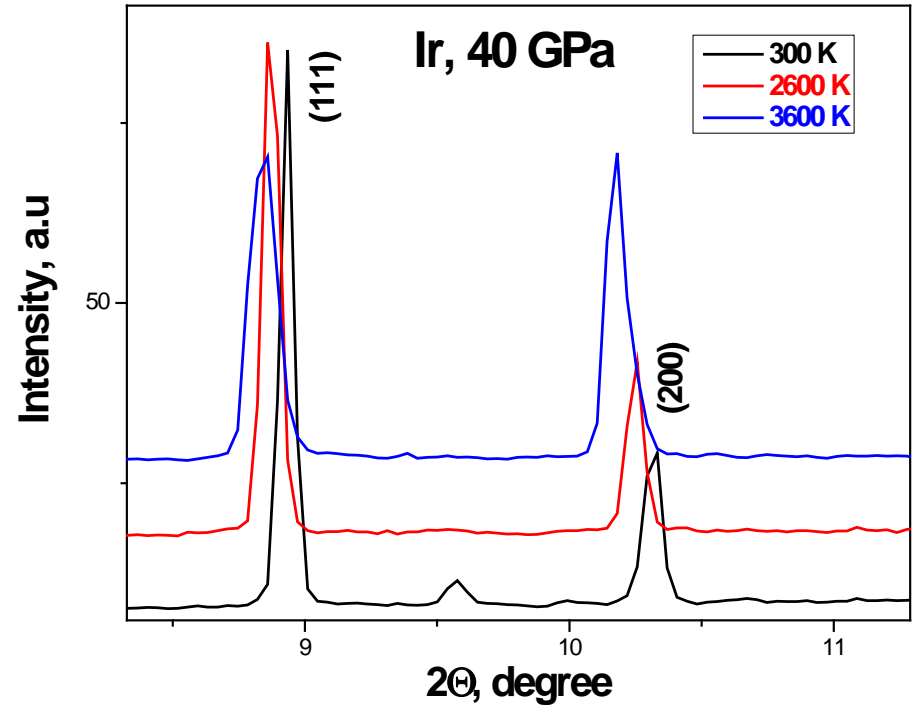
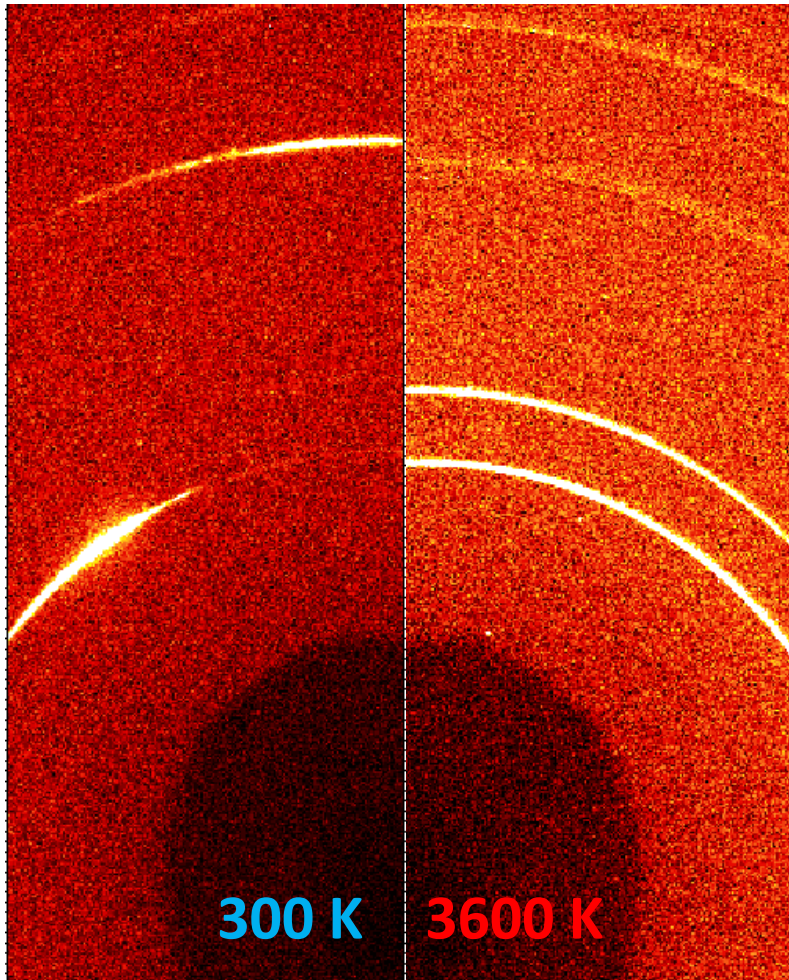




# The time-resolved radiometric measurements of temperature

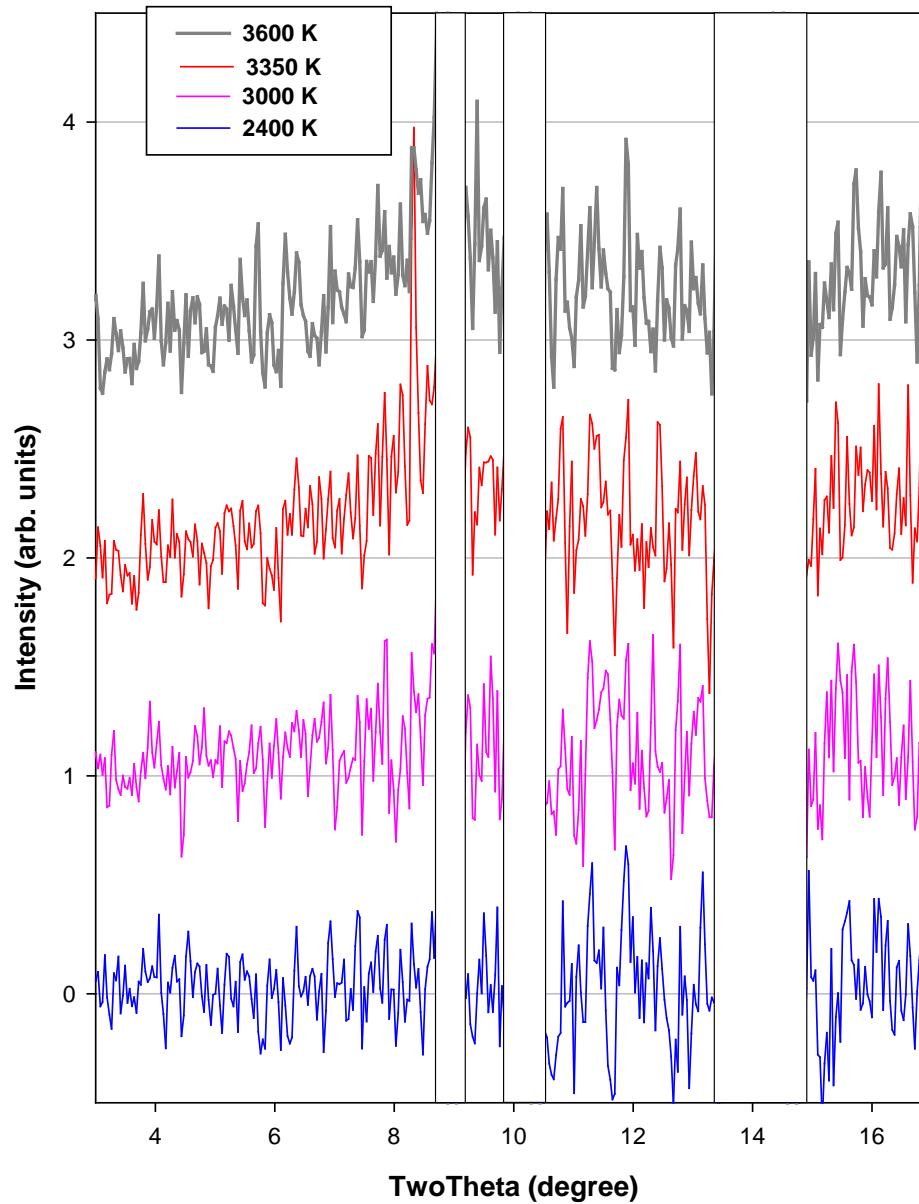


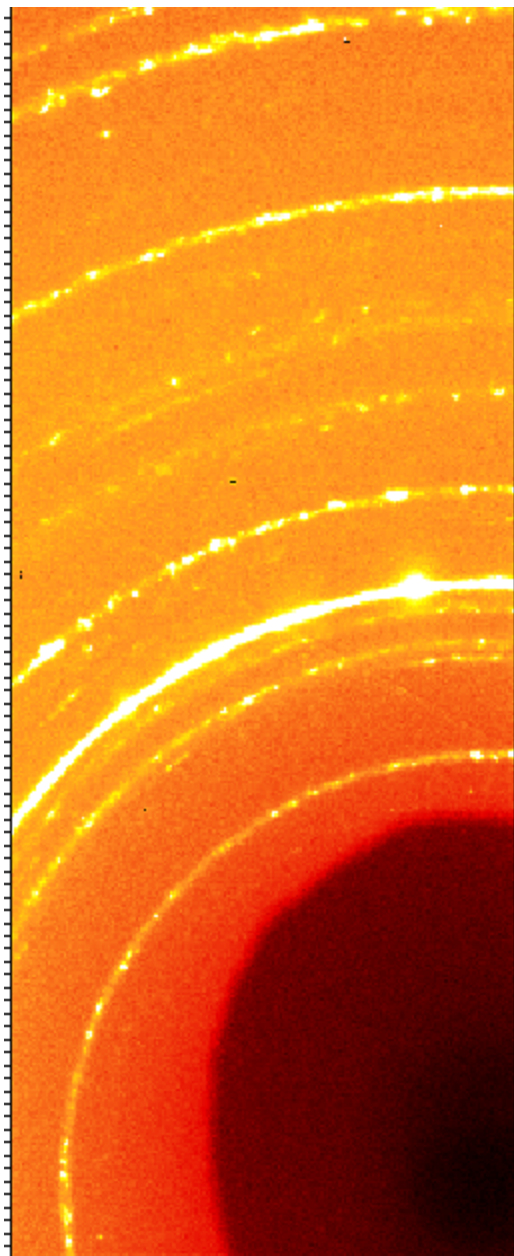
# Pulsed laser heating Ir at 40 GPa



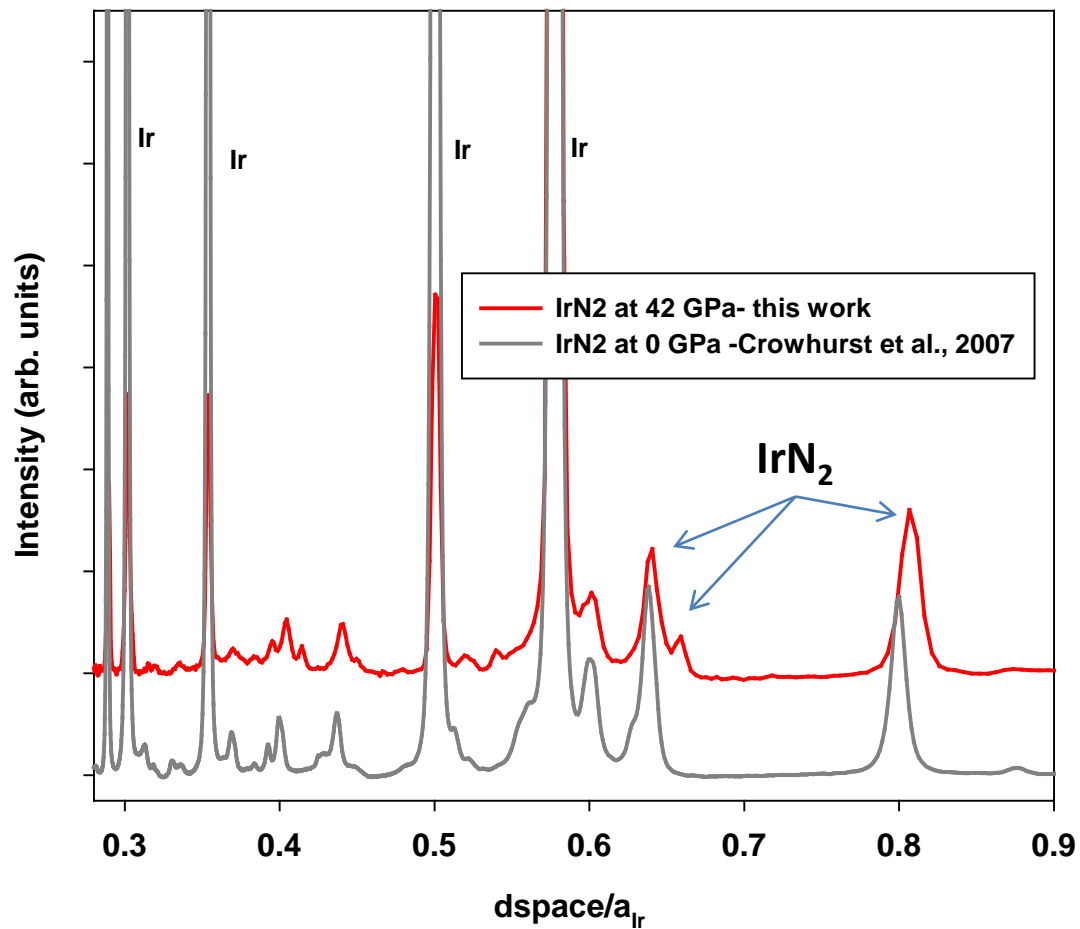


- Melting can be detected by observing a diffuse diffraction ring



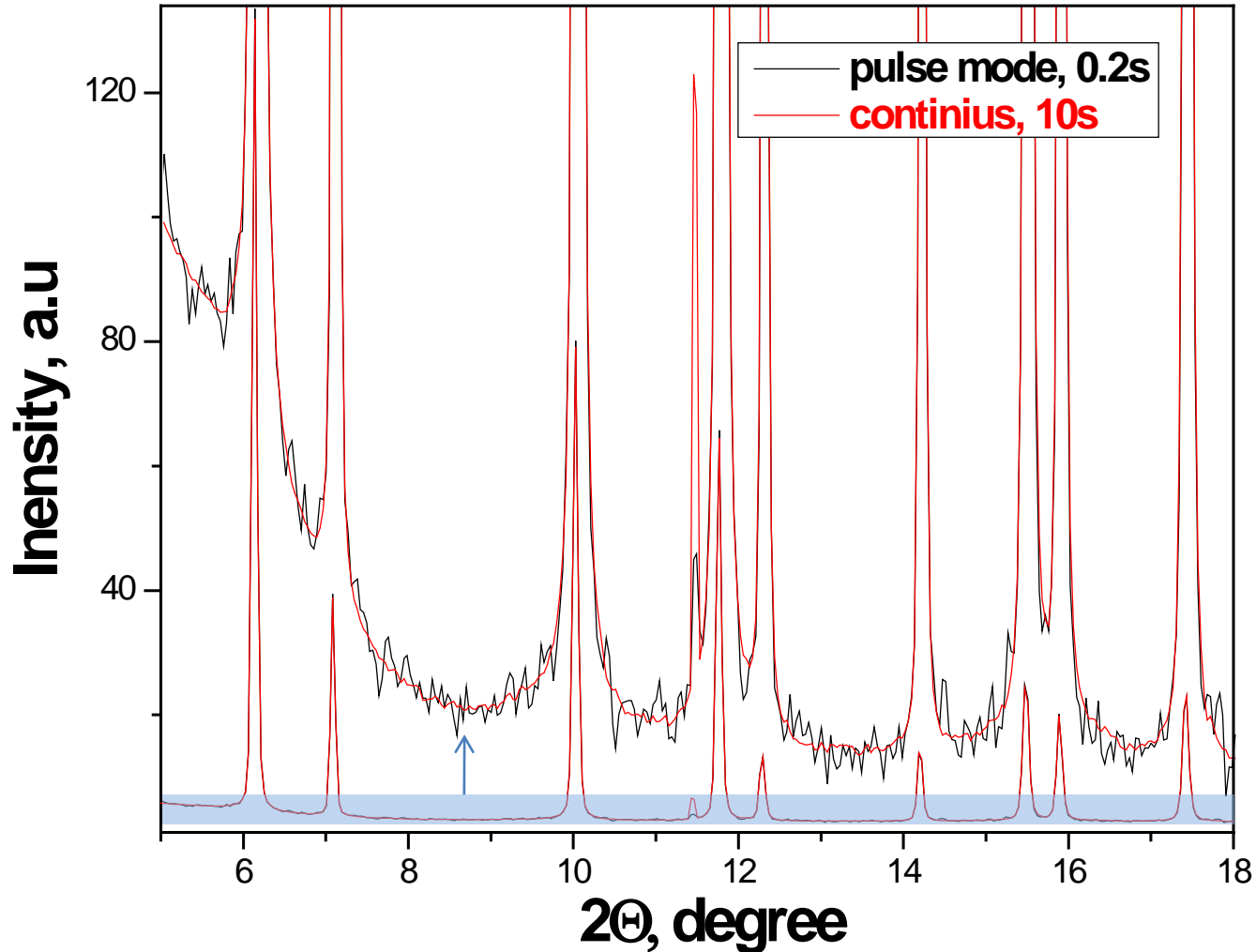


- *Chemical reactivity is very fast in pulsed heating experiments*
- *New possibilities for studying of chemical reactions*



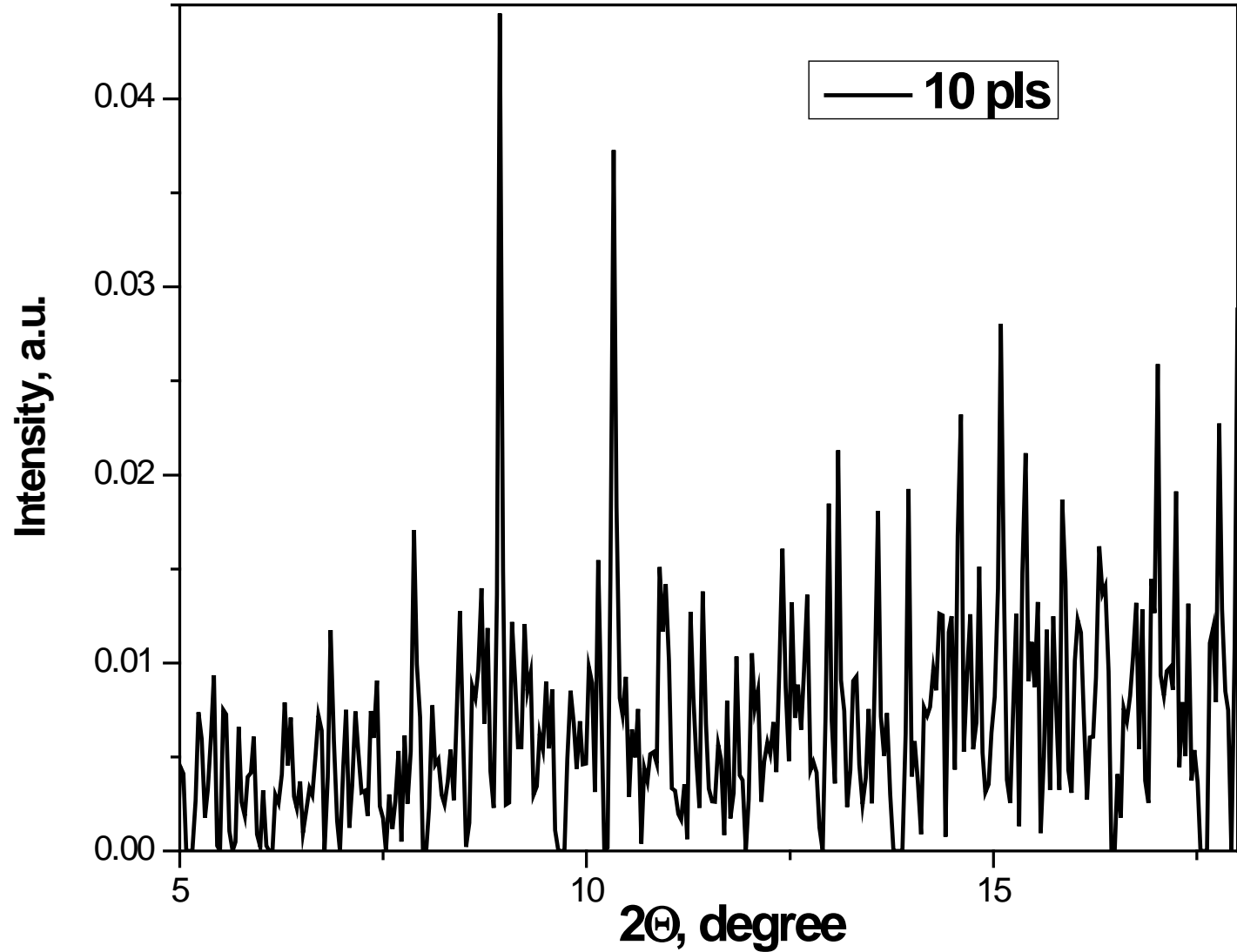
Compare XRD for  $\text{CeO}_2$  collected with PILATUS detector  
for different exposure time:

continues 10s (divided by 50) and 0.2s averaged over  $10^5$ pls, 2us window



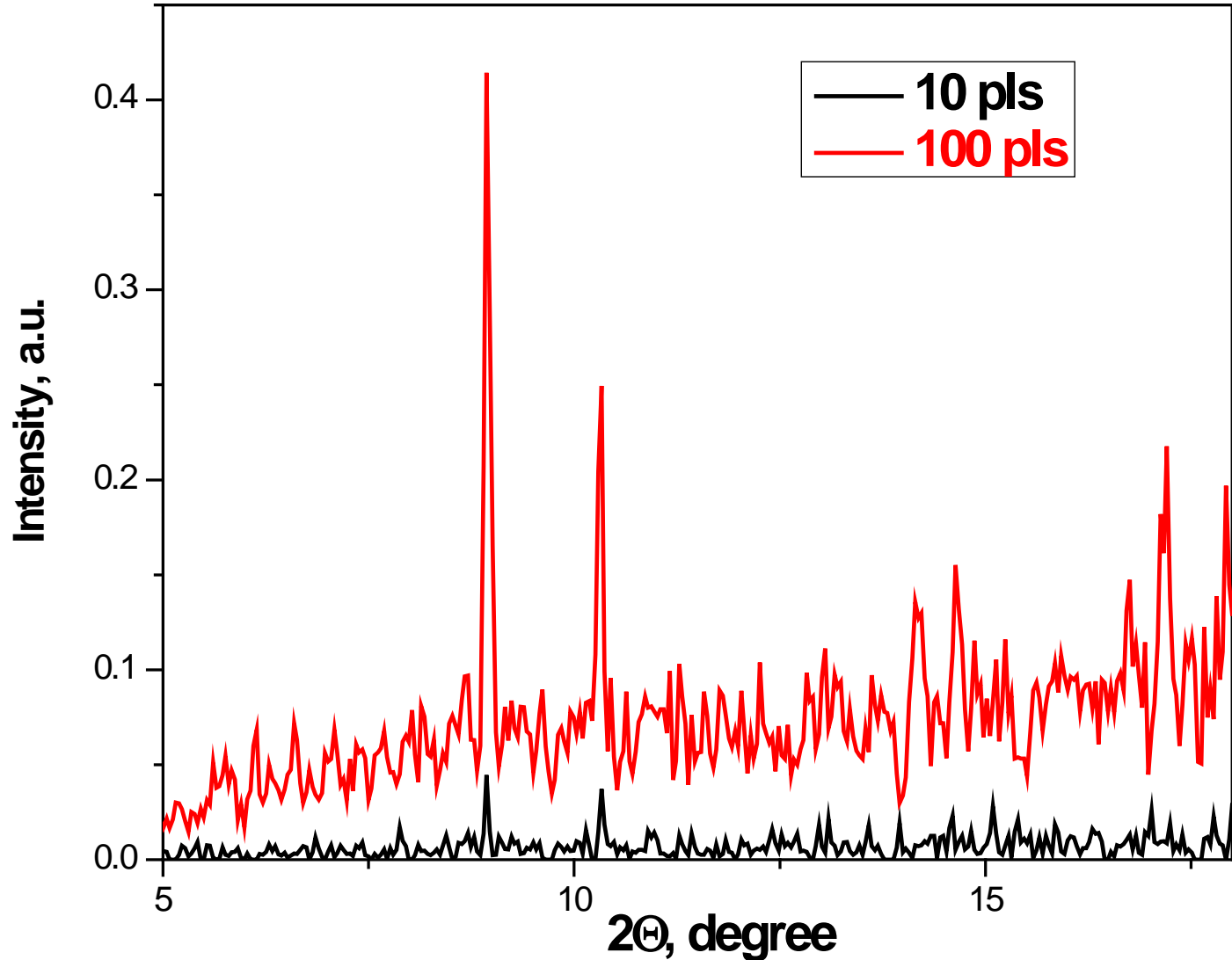
*Standard Operating Mode, top-up: 102 mA in 24 singlets (single bunches) with a nominal current of 4.25 mA and a spacing of 153 nanoseconds between 40 ps singlets*

**Detector window: 2 us or ~12 single bunches or ~2x10<sup>5</sup> photons**



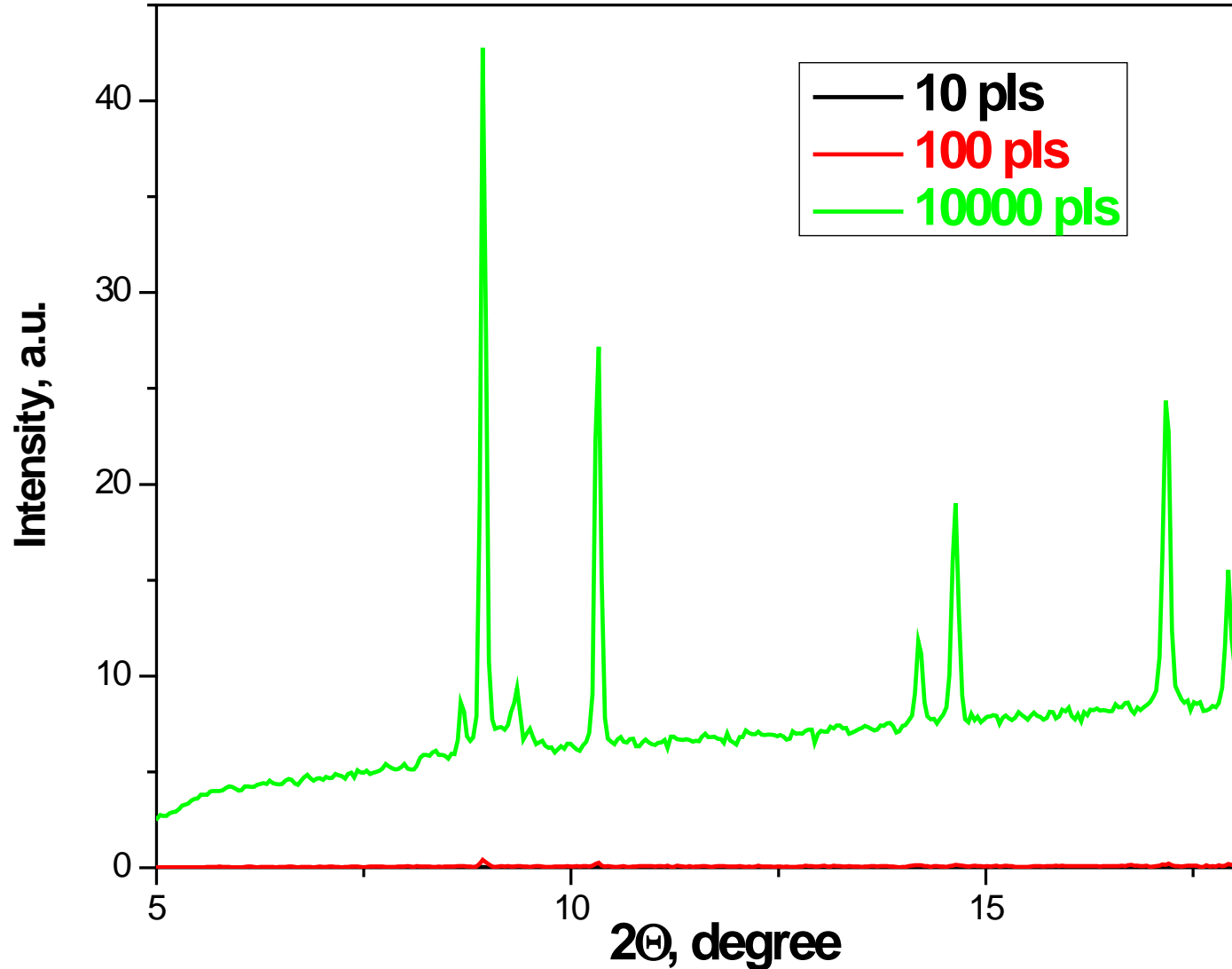
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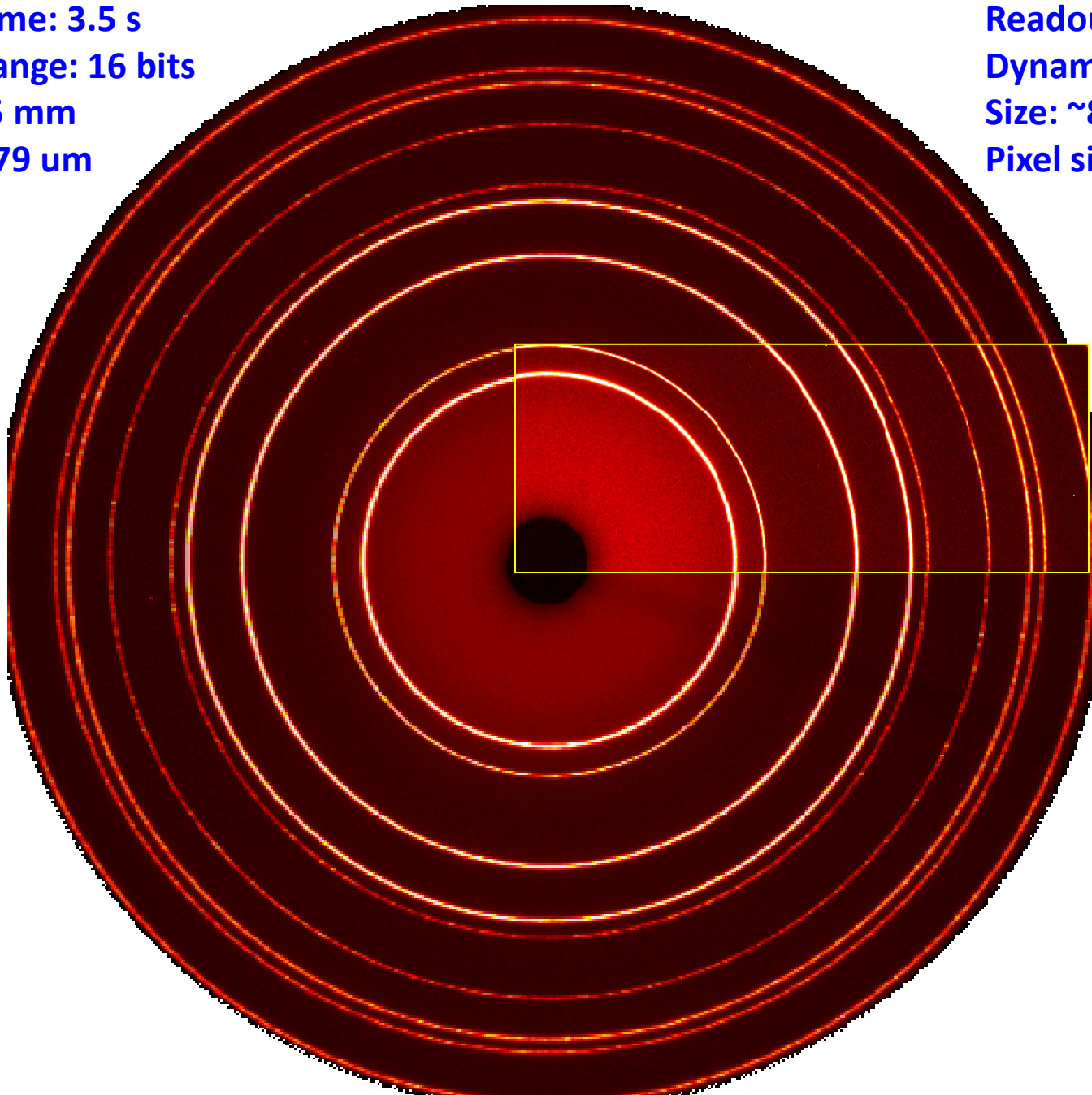
## MAR-CCD

Readout time: 3.5 s

Dynamic range: 16 bits

Size:  $\varnothing$ 165 mm

Pixel size: 79  $\mu$ m



## PILATUS 100K

Readout time: 2.7 ms

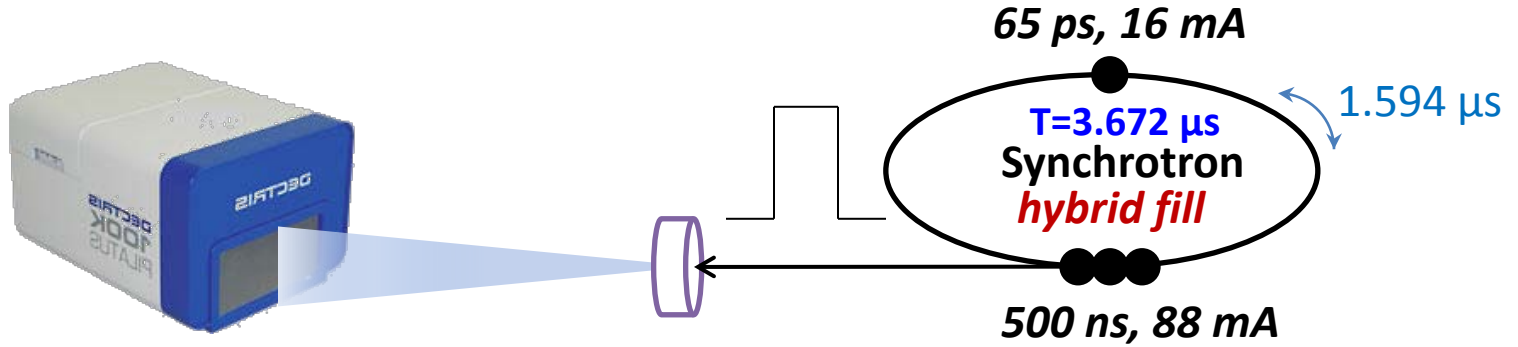
Dynamic range: 20 bits

Size:  $\sim$ 84x33 mm<sup>2</sup>

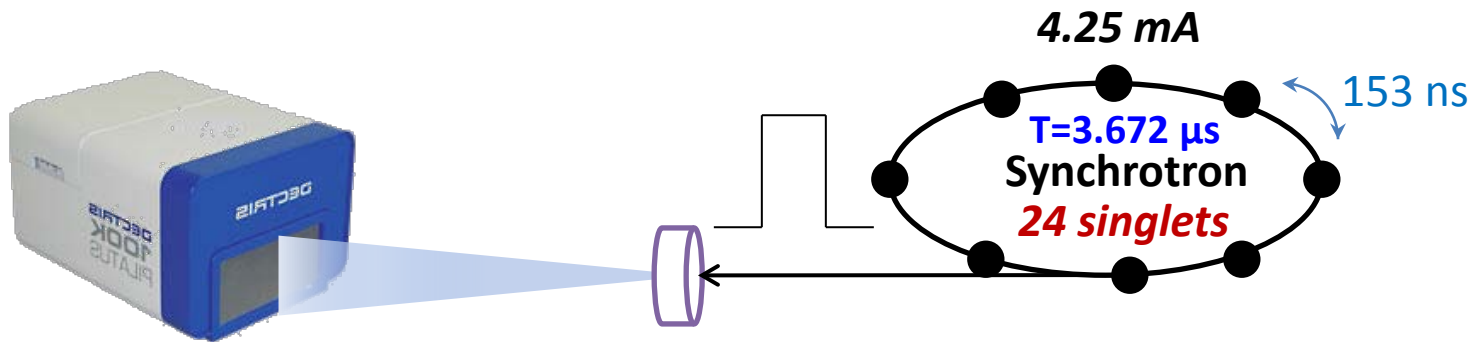
Pixel size: 172  $\mu$ m

# Dynamic x-ray probe optimization:

< 1  $\mu$ s:



2 -100  $\mu$ s:



General:

*Detector: larger area, higher efficiency above 30 keV*

*Sample: thick, high Z, single crystal*



*Pulse laser heating and optical spectroscopy combined with  
time-resolved x-ray probe*



**Reliable experimental conditions at higher than static T and P**

Probing fundamental ultrafast processes

- high temperature EOS
- phase transition kinetics
- structural dynamics & deformation
- chemical reaction dynamics
- transport properties (*e.g.*, diffusion)
- electronic properties



**GSECARS: Mark Rivers, Steve Sutton,  
Yanbin Wang, Peter Eng, Matt  
Newville, Przemek Dera, Nancy  
Lazarz, Fred Sopron**

**CIW :  
A. Goncharov  
V. Struzhkin**

**ESRF :  
I. Kantor**