INTRODUCTION TO THE LH-DAC AND ITS APPLICATION AT SYNCHROTRON RADIATION FACILITIES

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TABLE OF CONTENT

- Interest in HP-HT and how do we produce it in laboratories

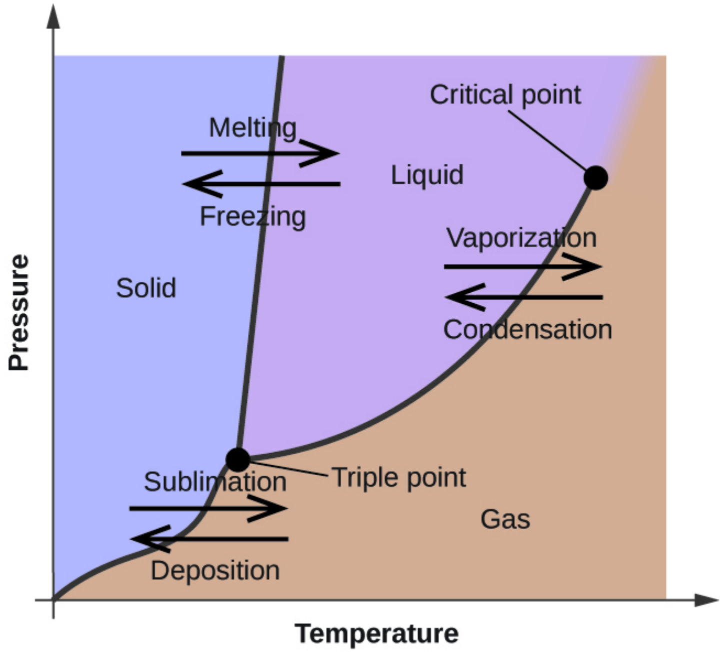
- Why LH-DAC ?

- Principle of the LH-DAC:
  Sample preparation
  Description of the three paths for imaging, laser-heating and T measurement
  LH-DAC metrology

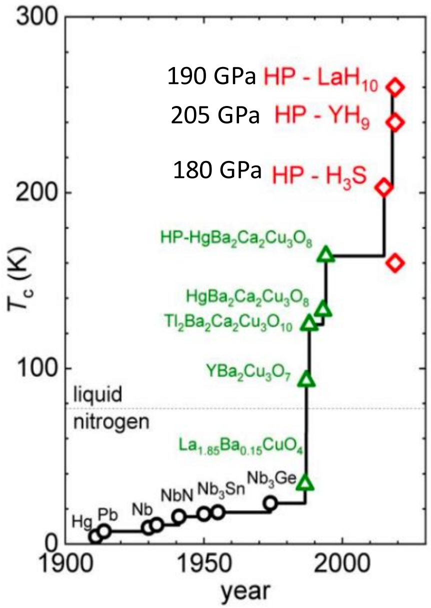
- LH-DAC at Synchrotron facilities
  - Coupled with XRD
  - Couple with XAS
  - Multi-techniques

WHY HIGH PRESSURE AND TEMPERATURE (1)

Basic Physics



Synthesis of new materials

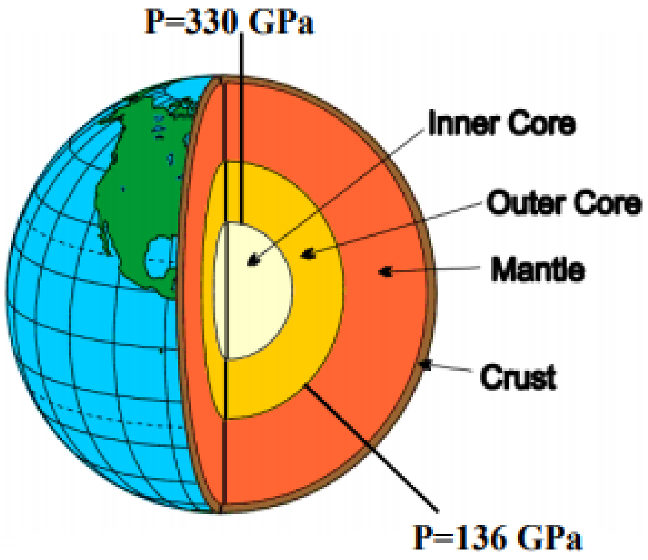


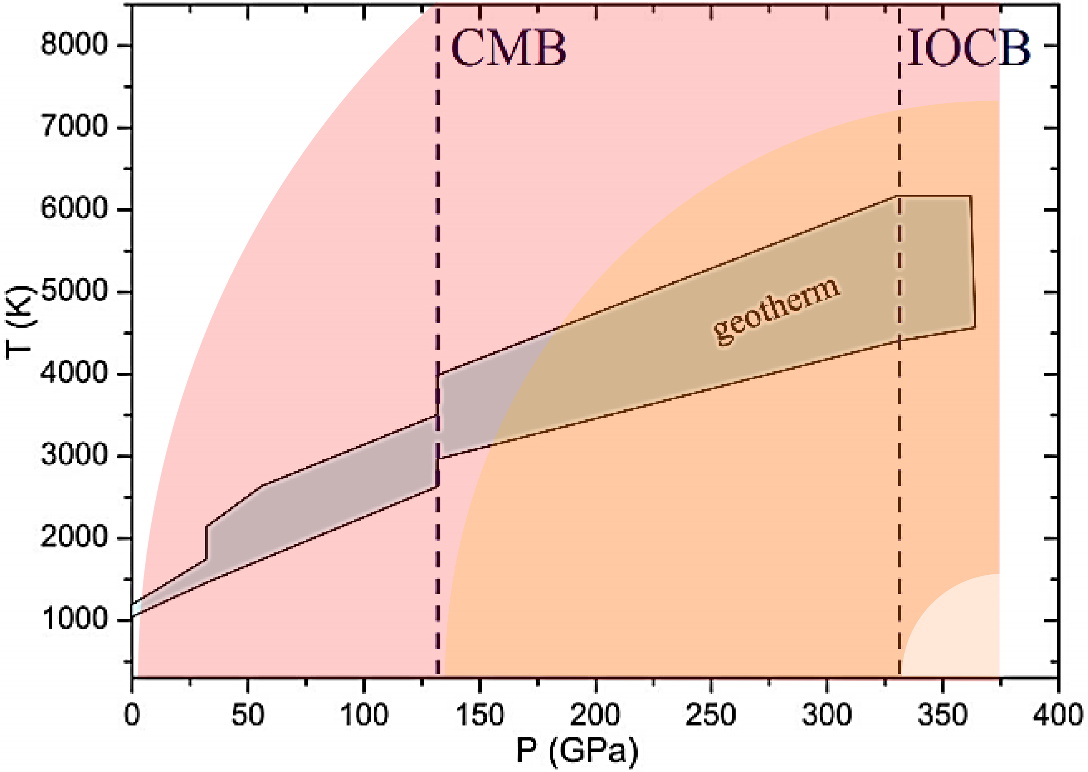
ambient temperature SC

ultra-hard materials

WHY HIGH PRESSURE AND TEMPERATURE (2)

Geophysical interest





HOW HIGH PRESSURE AND TEMPERATURE

static

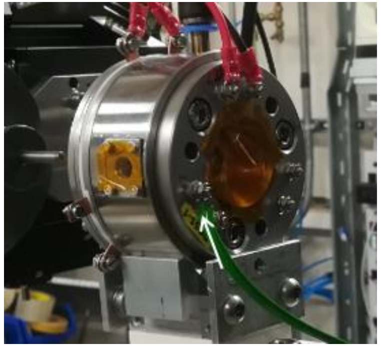
Large Volume Presses

Paris-Edinburgh

Multi-anvils

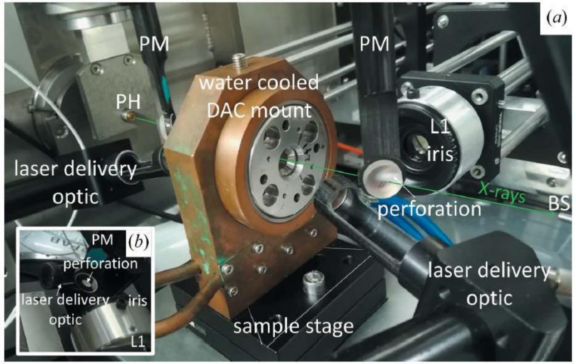
DAC

resistively-heated



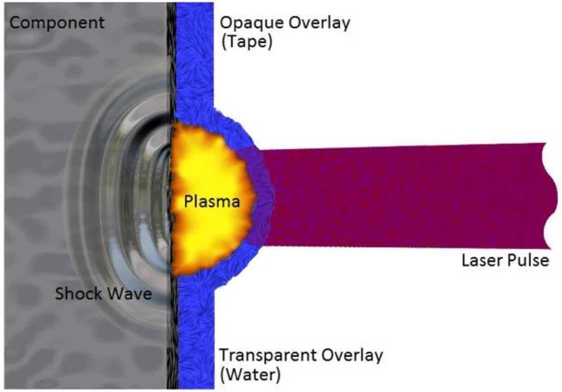
Louvel *et al.*, *J. Synch. Rad.* **27**, 529 (2020)

laser-heated



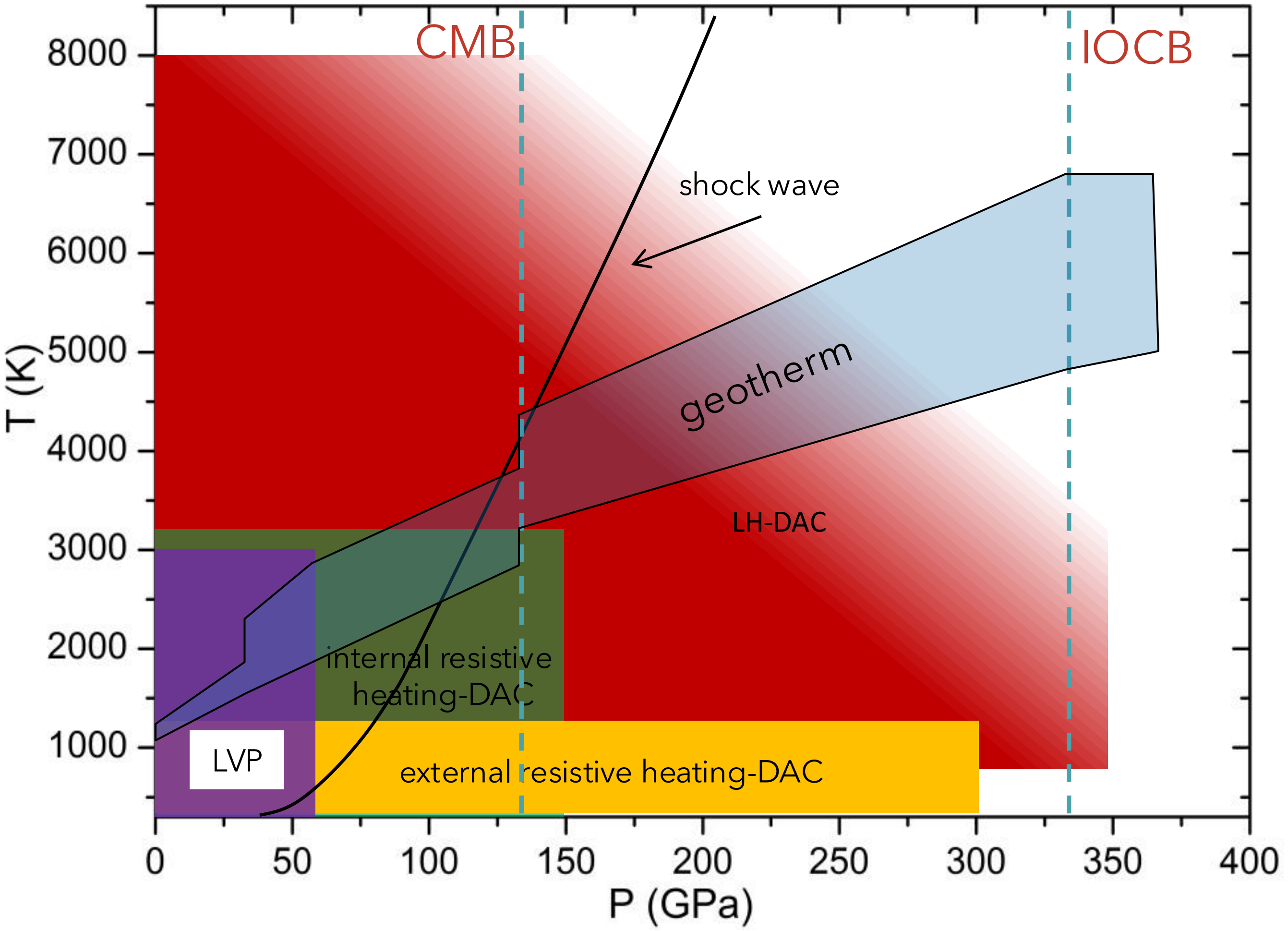
Anzellini *et al.*, *J. Synch. Rad.* **25** (2018)

dynamic



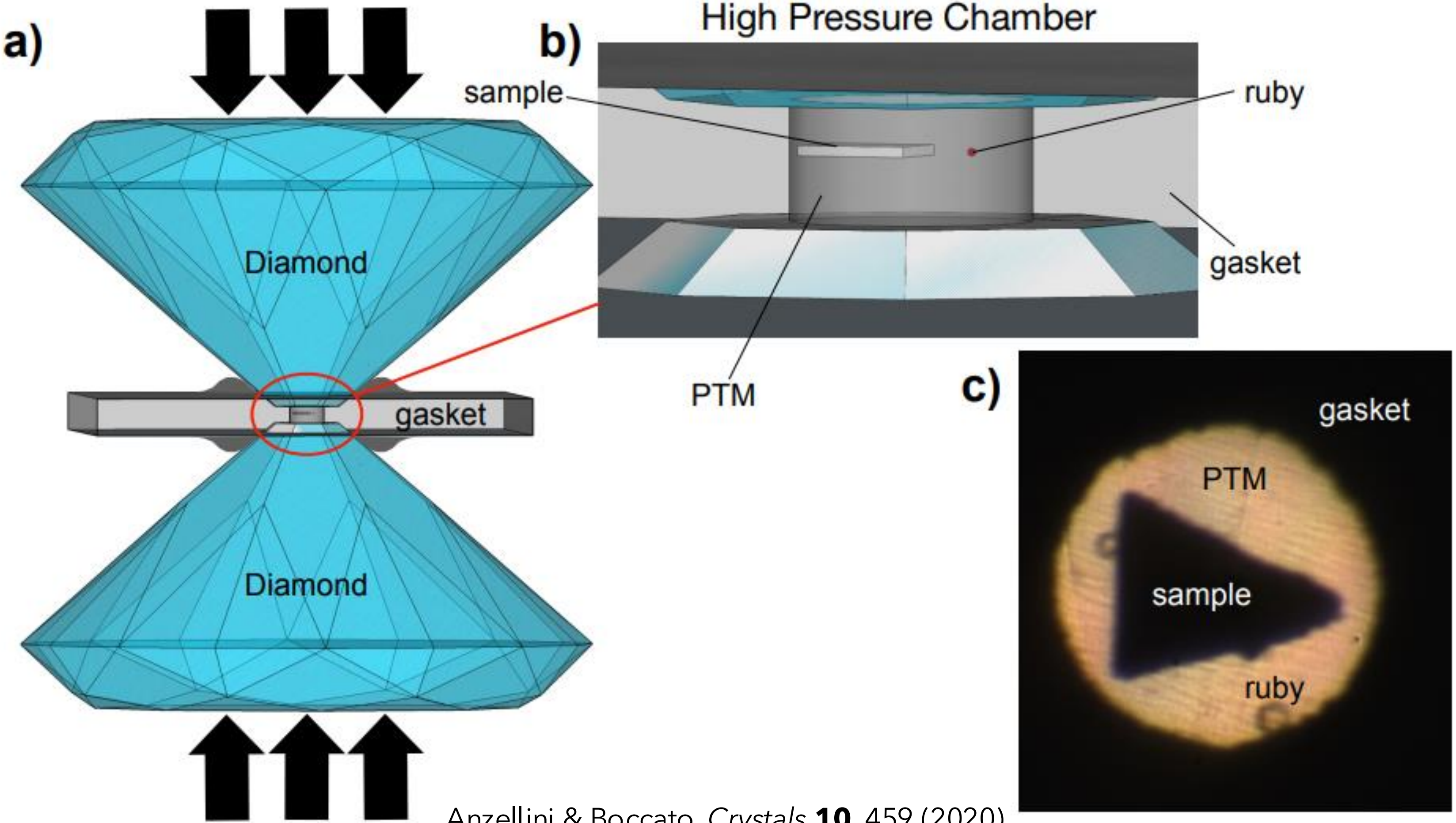
shock

WHY LH-DAC ?



CMB
IOCB
shock wave
geotherm
LH-DAC
internal resistive heating-DAC
LVP
external resistive heating-DAC
T (K)
P (GPa)
8000
7000
6000
5000
4000
3000
2000
1000
0
50
100
150
200
250
300
350
400

PRINCIPLE OF DAC

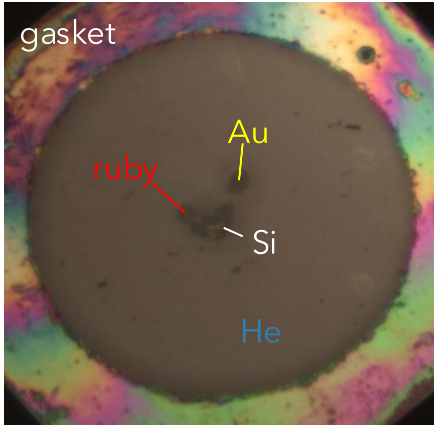


Anzellini & Boccato, *Crystals* **10**, 459 (2020)

SAMPLE PREPARATION

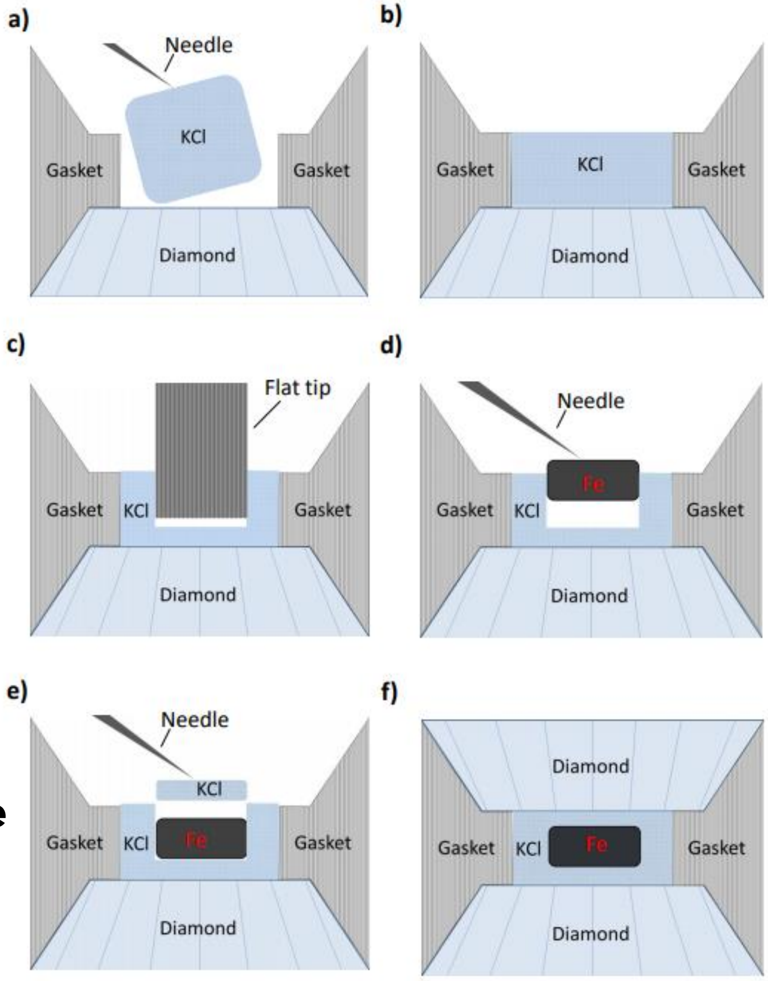
PTM generally chosen from low shear strength material

gases (He,Ne,…) are generally used for experiment at ambient T



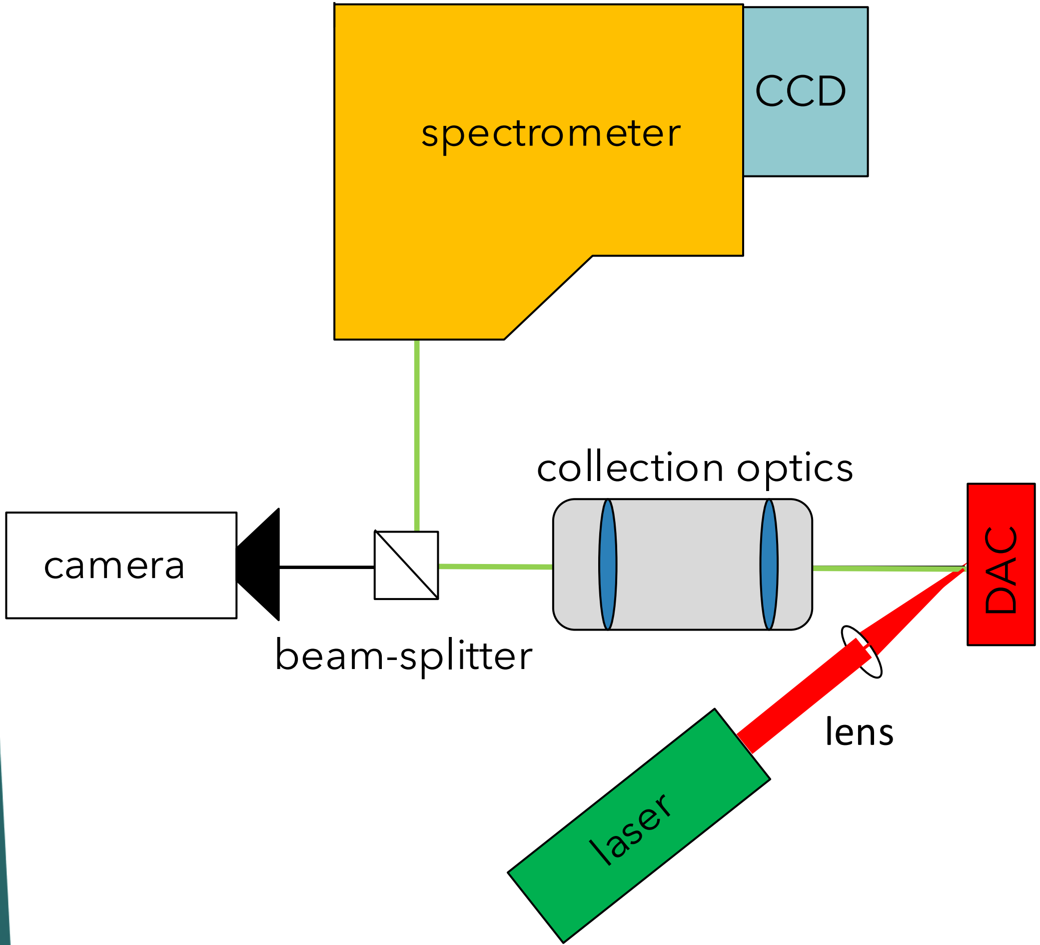
Diamonds are extremely thermally conductive

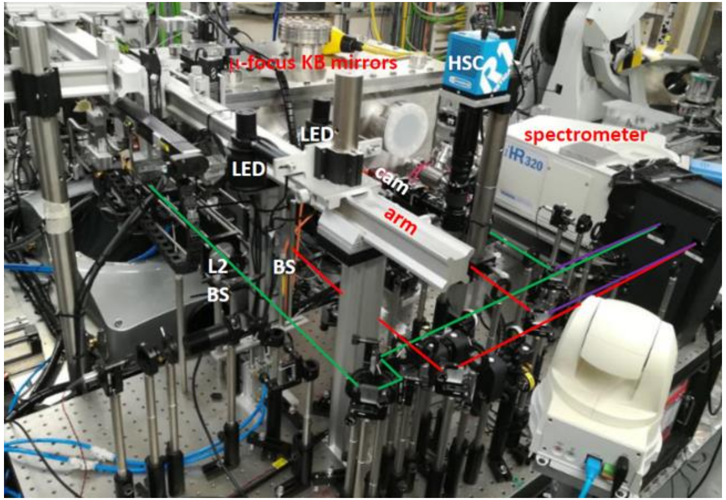
PTM used to thermally and chemically insulate the sample from the diamonds



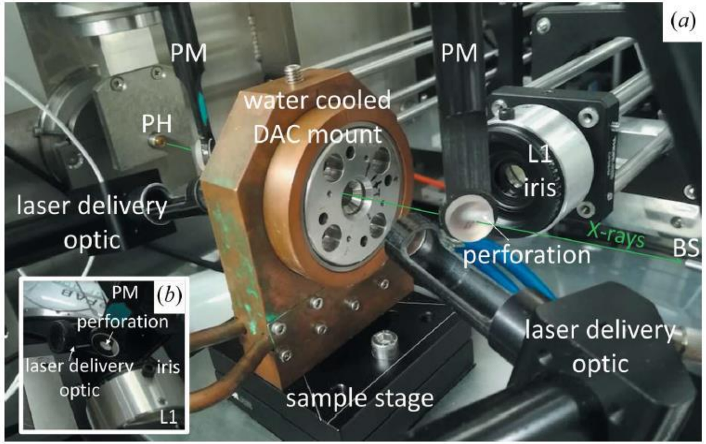
Anzellini PhD thesis (2014)

PRINCIPLE OF LH-DAC (1)

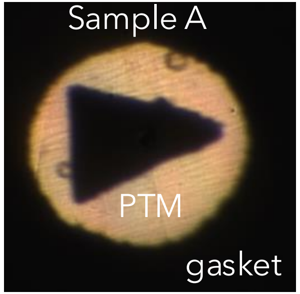




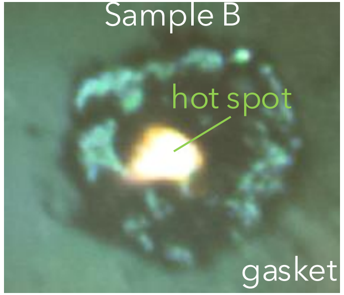
Anzellini *et al.*, *J. Synch. Rad.* **25** (2018)



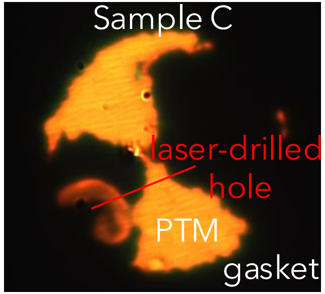
IMAGE



before heating

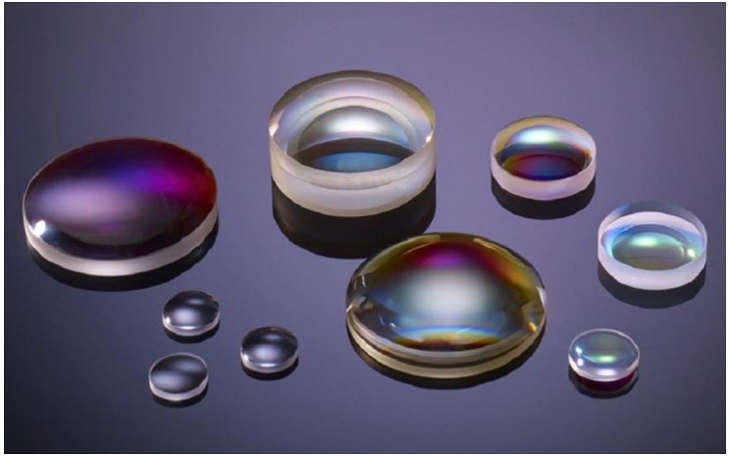


during heating

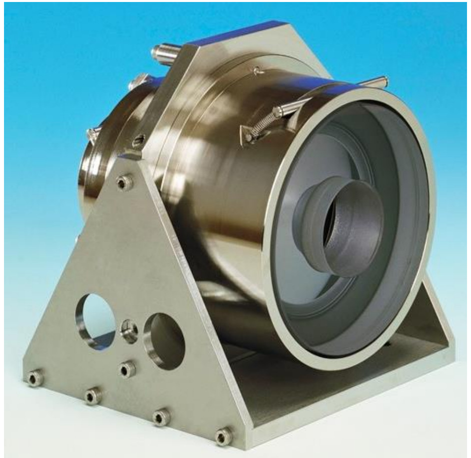


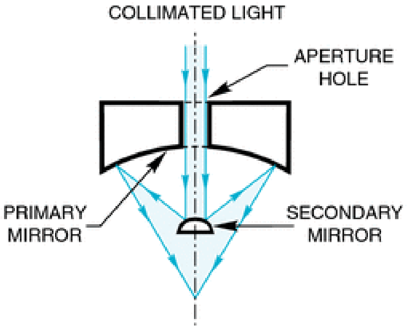
after heating

Refractive Optics



Reflecting Optics



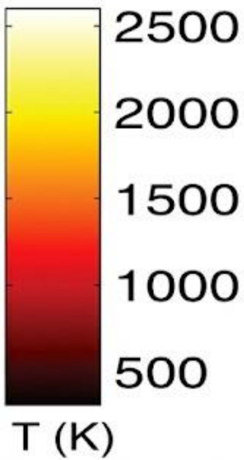


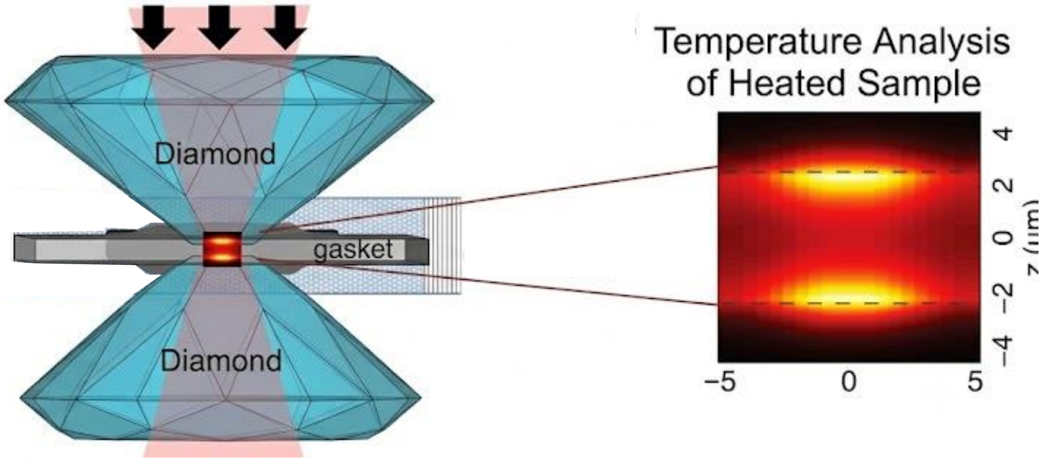
LASER PART (1)

Metals and semiconducting materials

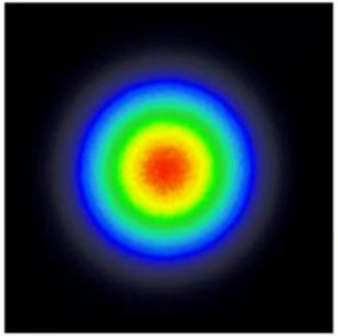
YAG or YLF lasers (λ=1.064 μm) – mainly interacting with the electrons → weakly absorbed by the material

axial





radial



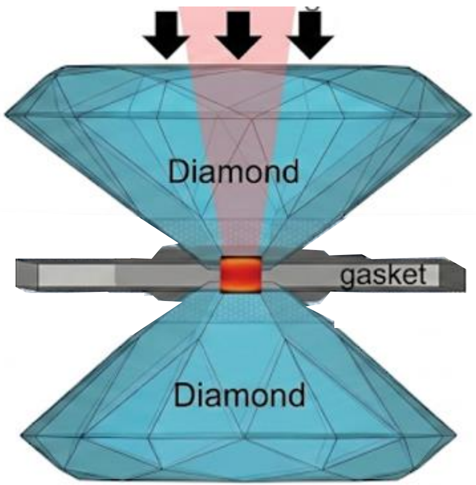
Big thermal gradients!!

LASER PART (2)

Optical transparent materials

CO_2 lasers (λ=10.6 μm) – light directly absorbed by phonons → large penetration depth into the material

Less thermal gradients

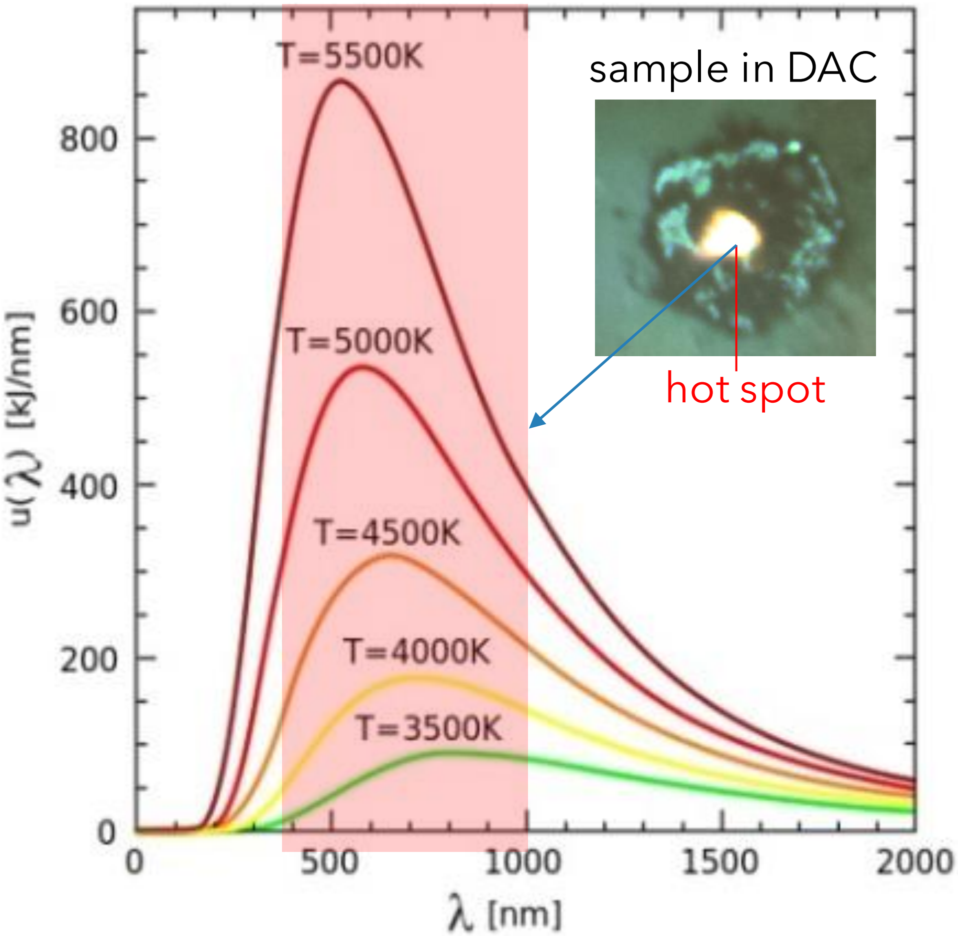


single side heating

- Big focal spot!!
- Special Optics needed

both kind of lasers can be used in continuous or pulsed modes.

TEMPERATURE (1)

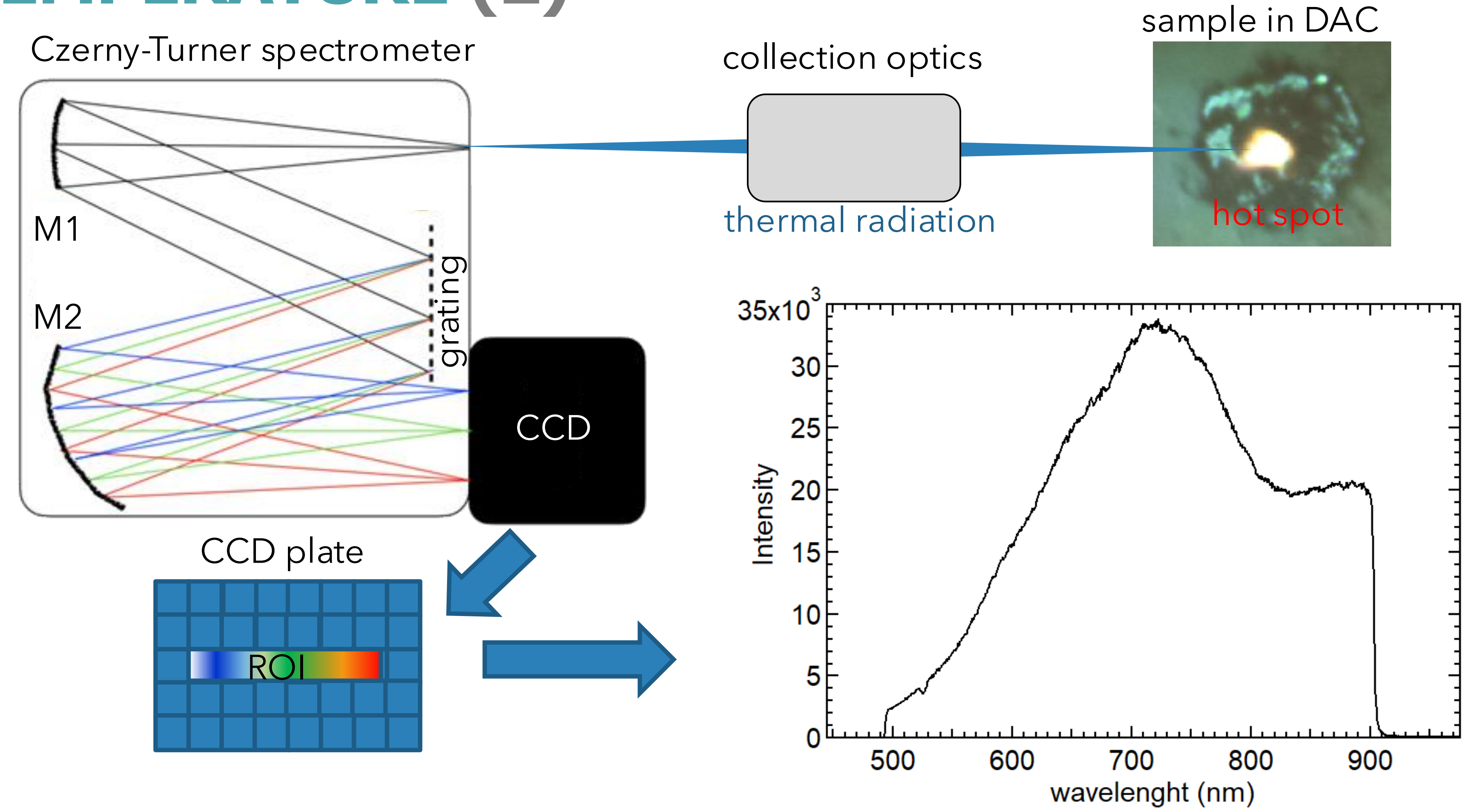


$$Planck : I(\lambda,T) = \frac{\varepsilon 2\pi h c^2}{\lambda^5}\left[\exp\left(\frac{hc}{kT\lambda}\right)-1\right]^{-1}$$

The fitting is done in grey-body approx:

$\varepsilon \neq \varepsilon(\lambda)$

TEMPERATURE (2)



TEMPERATURE (3)

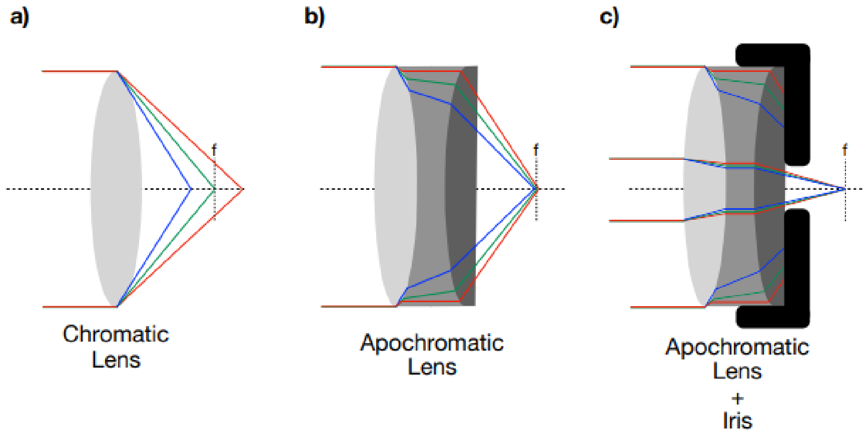
Walter & Koga, *Phys. Earth Planet. Int.* **143**, 541 (2004)

Sources of erroneous T readings

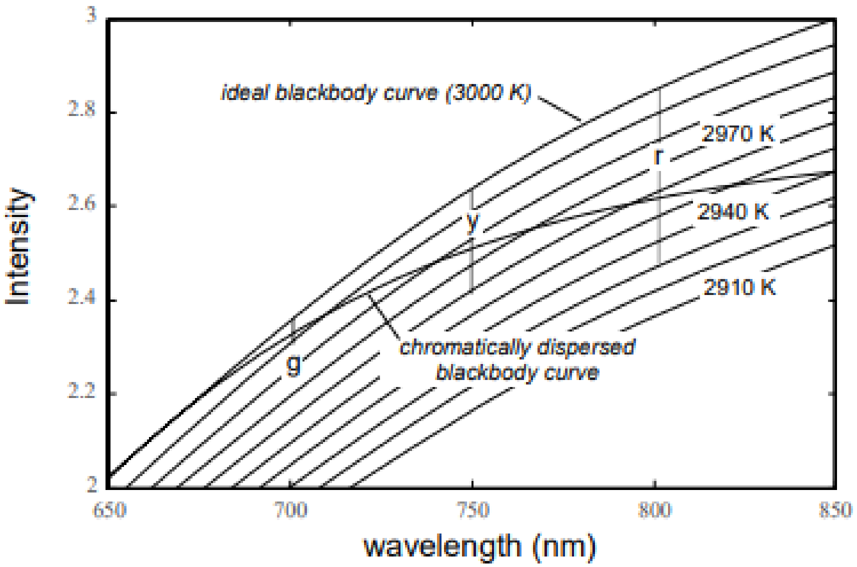
1) Change in emissivity due to phase transition

2) Misalignments of the hotspot with the spectrometer entrance slit

3) Chromatic + Radial aberration (for refractive objectives)

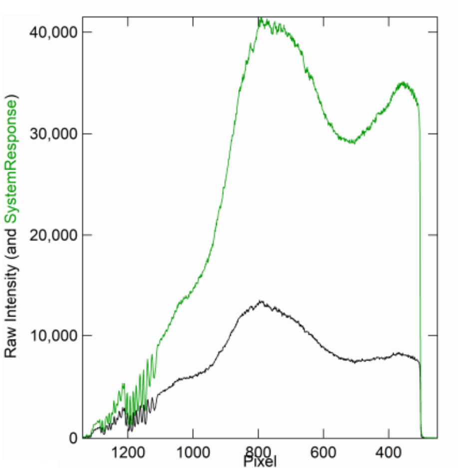


Anzellini & Boccato, *Crystals* **10**, 459 (2020)

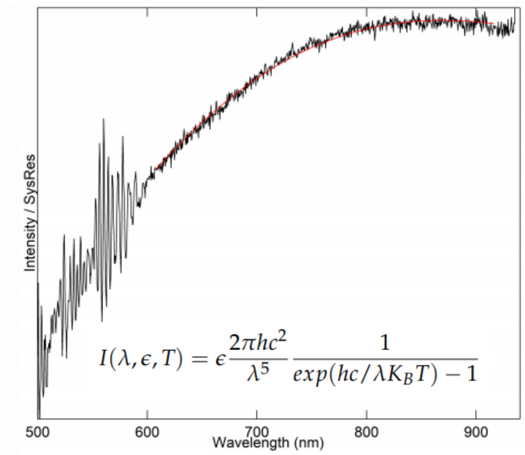


TEMPERATURE (4)

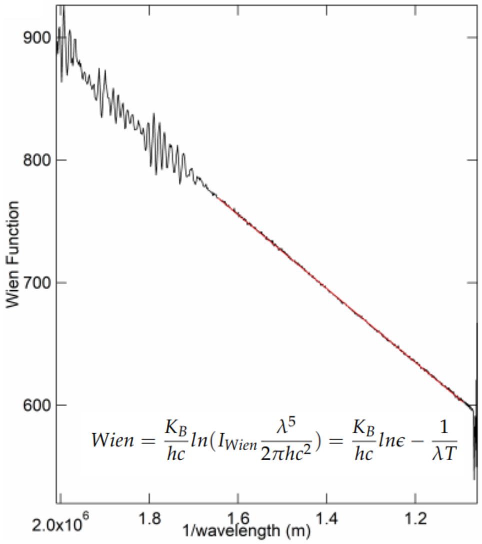
raw signals



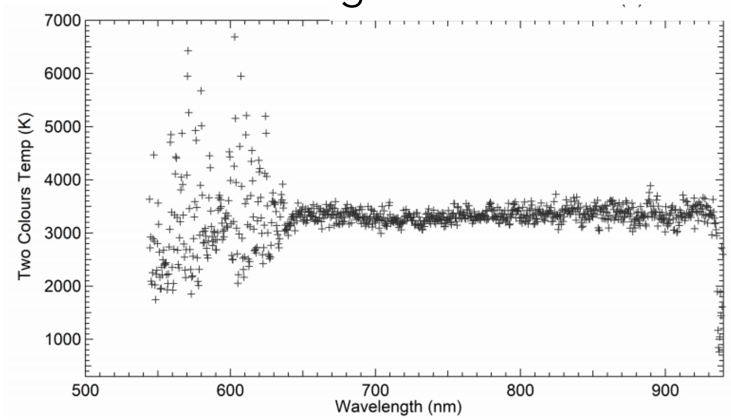
Planck



linearized Wien

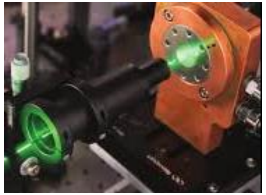


sliding two-colours

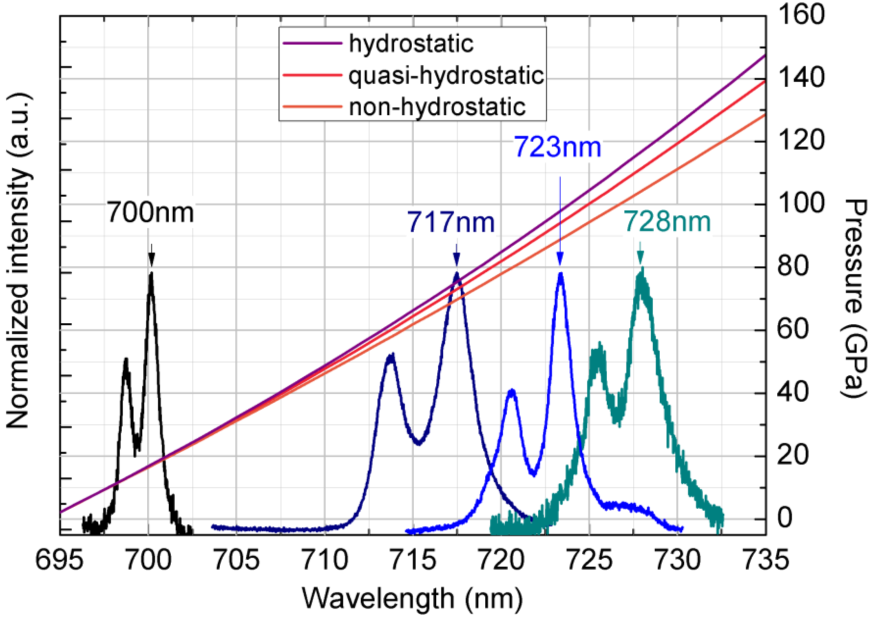


Anzellini & Boccato, *Crystals* **10**, 459 (2020)

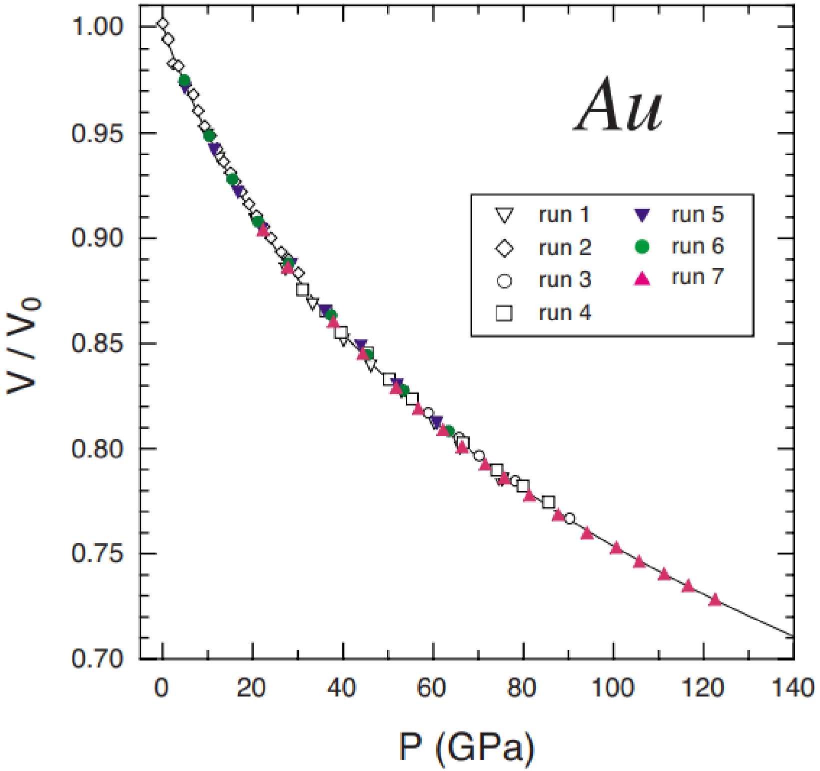
PRESSURE MEASUREMENT (ambient T)



Measured in a laboratory from:
-P-induced shift in ruby fluorescence peaks
-P-induced shift in Raman signal from the diamond culet



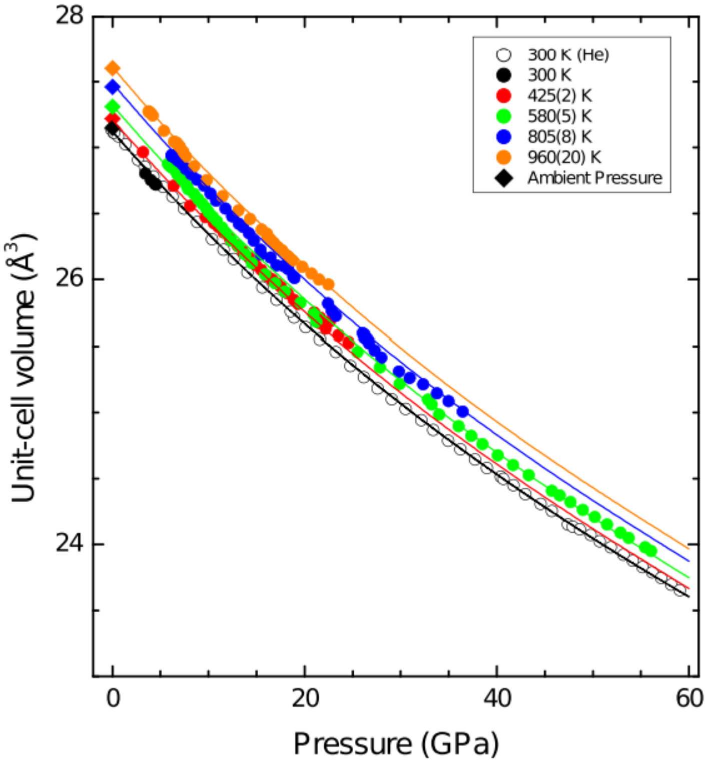
If XRD available
EoS of a calibrated standard



Takemura & Dewaele, *Physical Review B* **78**, 104119 (2008)

THERMAL PRESSURE

Anzellini *et al.*, *Scientific Reports* **9**, 14459 (2019)



XRD methods

Pressure can be measured from calibrated X-ray standard

Luminescence methods

Pressure can be measured only before and after heating

<u>Thermal pressure intrinsically linked to the PTM</u>→ It can be estimated empirically from previous XRD measurements

$$P_{th}(T) = \frac{0.03 \cdot P_{300K}}{T_m - T_0}(T - T_0).$$

COUPLING WITH SYNCHROTRONS


ALBA-Barcelona (ES)


APS-Lemont (IL-USA)


ALS-Berkeley (CA-USA)


Elettra-Trieste (IT)

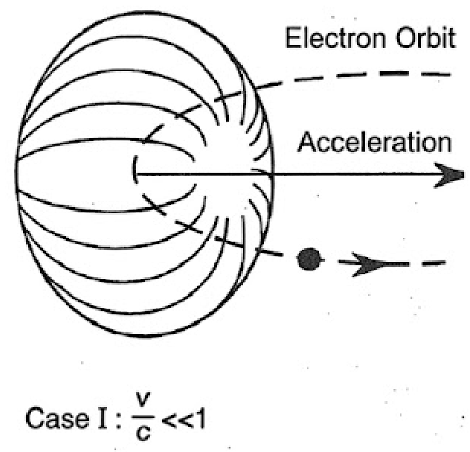

Spring-8-Sayo (JP)


ESRF-Grenoble (FR)

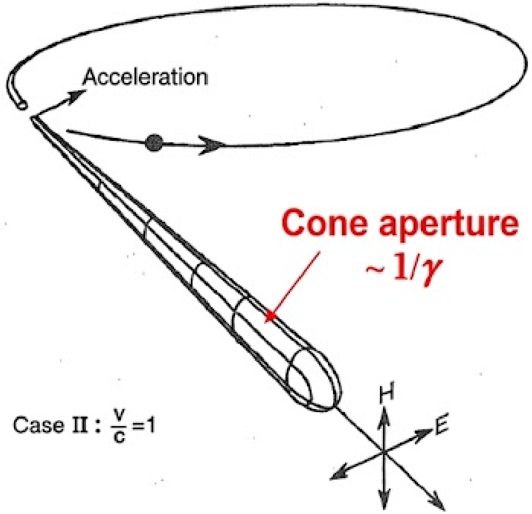

Diamond Light Source-Didcot (UK)

What is Synchrotron Radiation

relativistic effect on accelerated charged particles



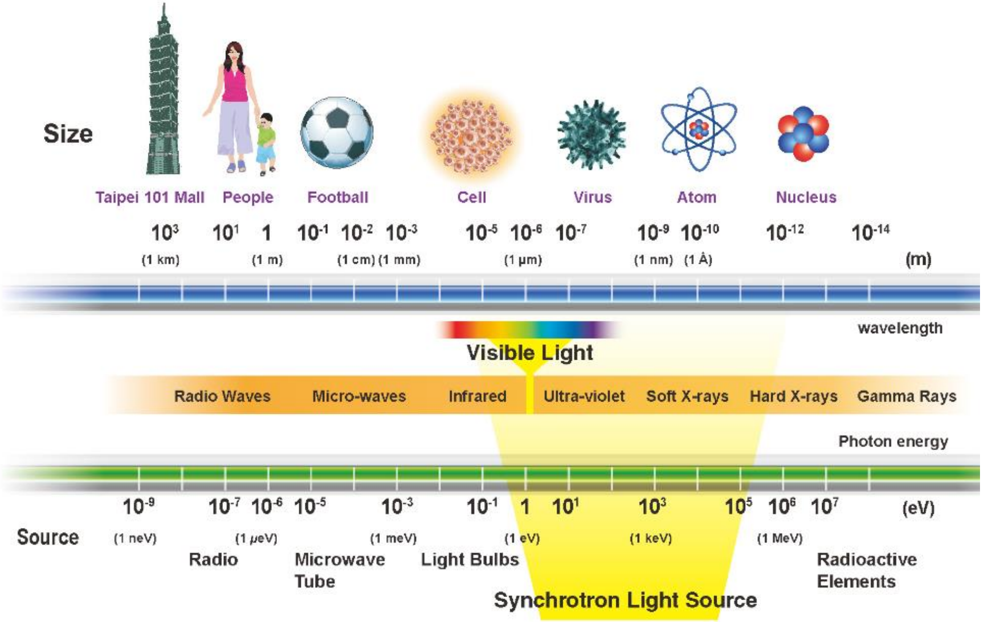
At low electron velocity (non-relativistic case) the radiation is emitted in a non-directional pattern

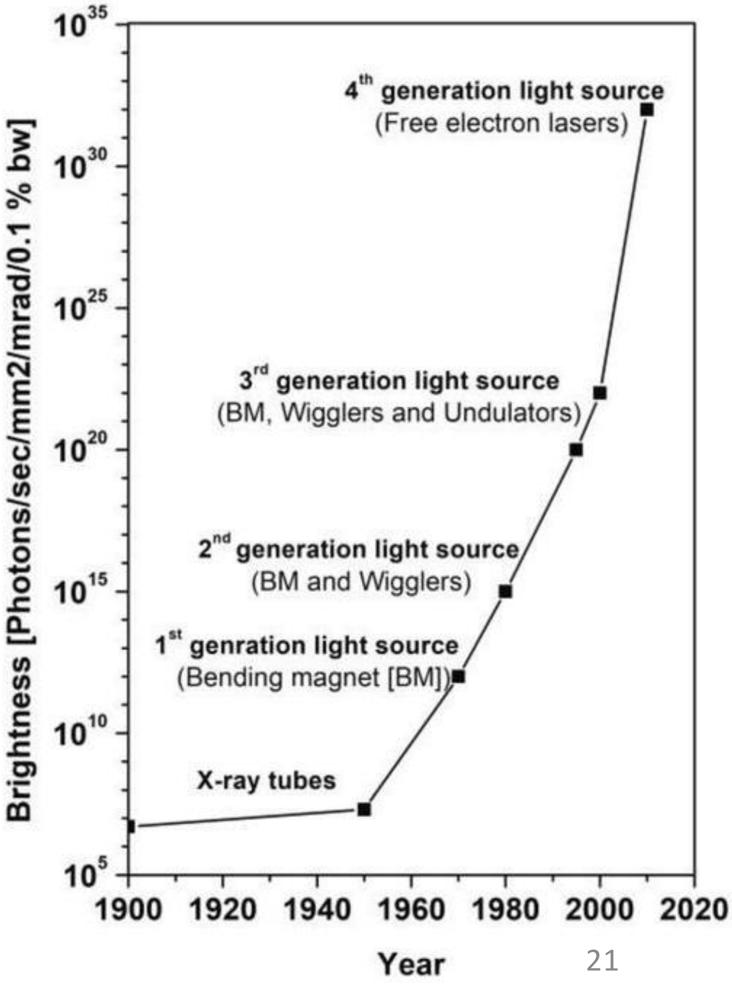


When the electron velocity approaches the velocity of light, the emission pattern is folded sharply forward.

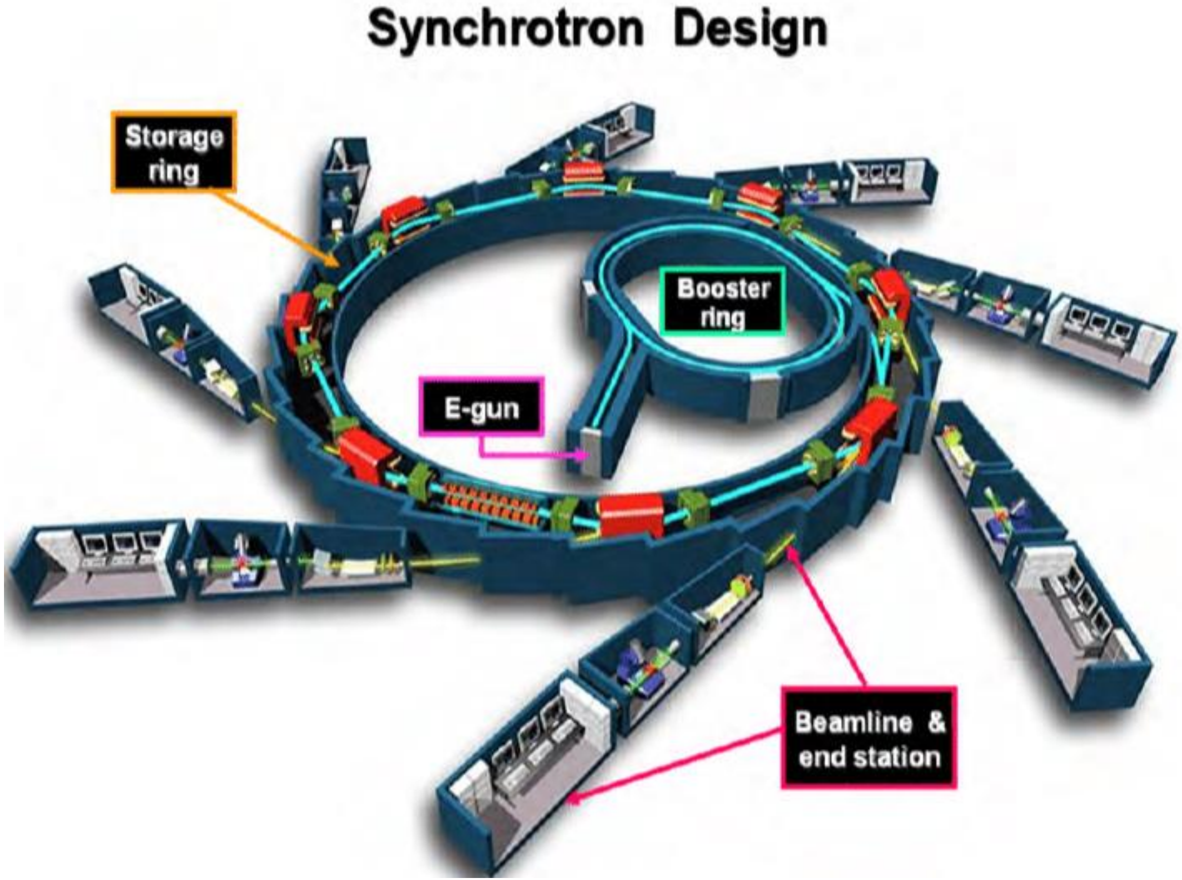
Characteristics of Synchrotron Radiation

- High Brightness
- Highly collimated
- Broad and continuous wavelength range





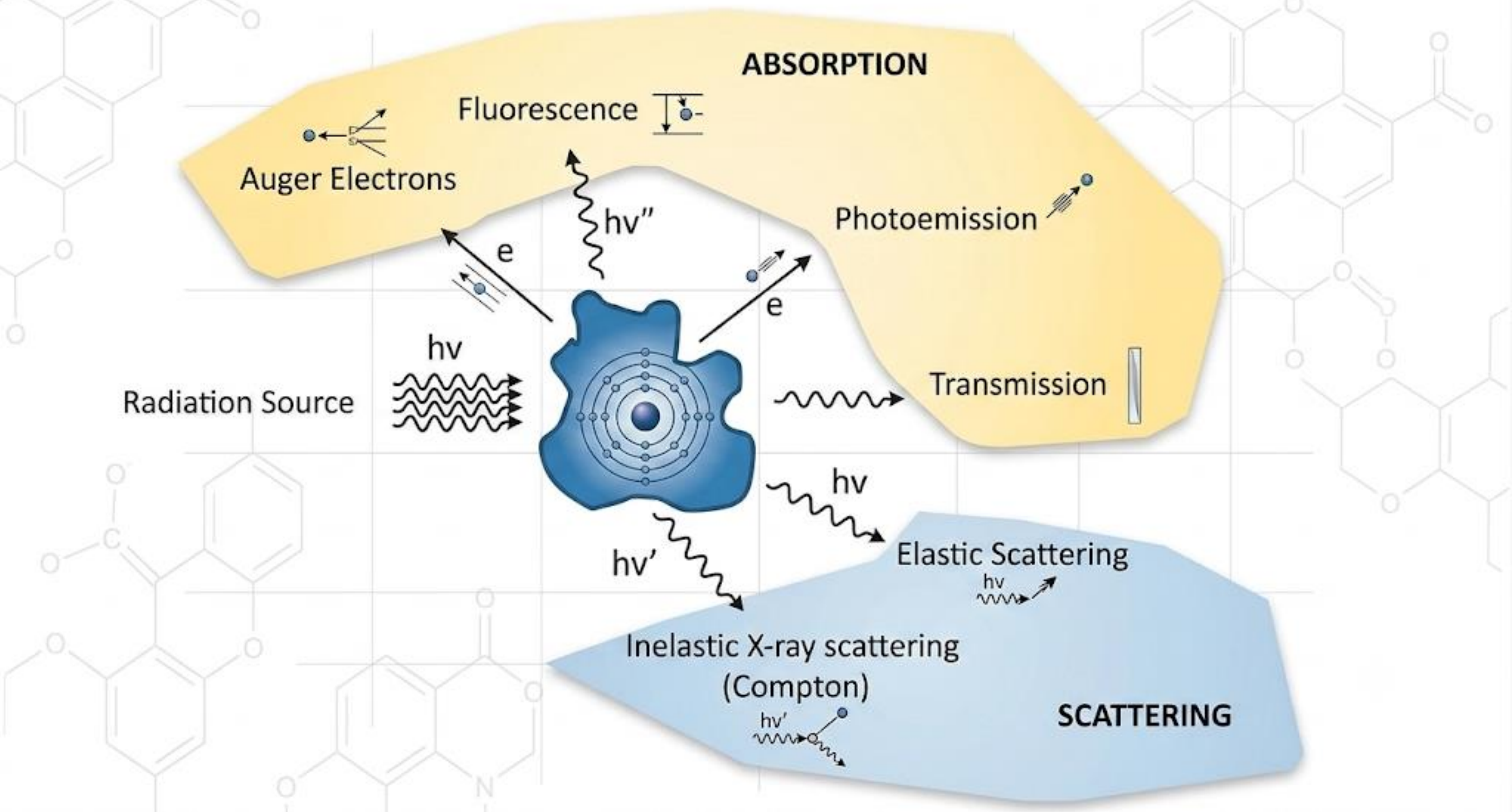
WHAT IS A SYNCHROTRON ?



Ali *et al.* (2011). Synch. Rad.: Applications in Diagnosis and Treatment of Malignant Brain Tumors, Diagnostic Techniques and Surgical Management of Brain Tumors, Dr. Ana Lucia Abujamra (Ed.)

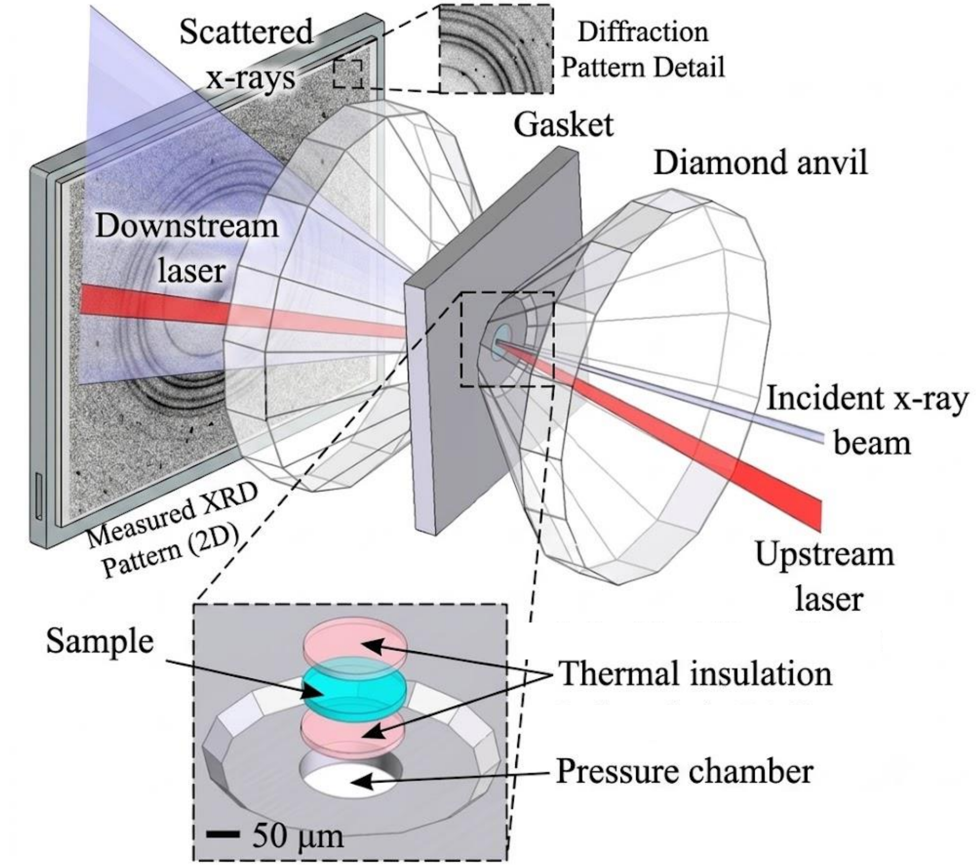
Interaction Matter - Radiation

Mainly two processes are involved in the UV and X-ray range:

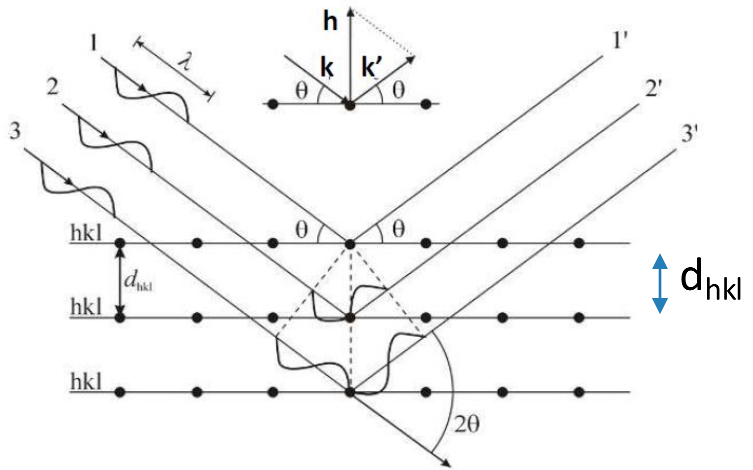


XRD

XRD (1)



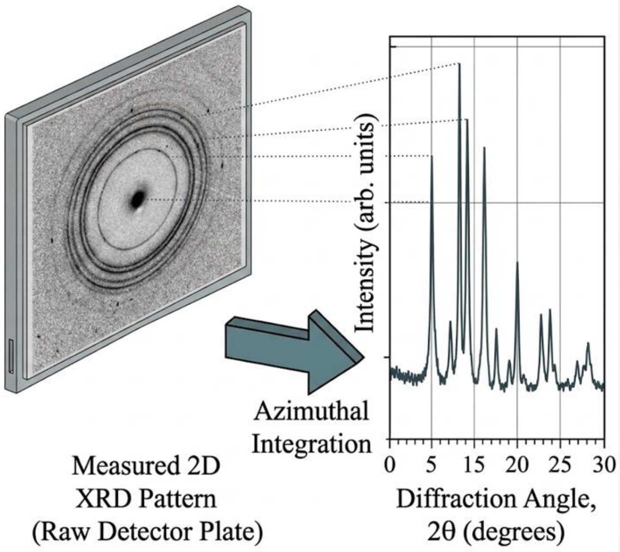
Bragg law: $\lambda = 2d_{hkl}\sin\theta$

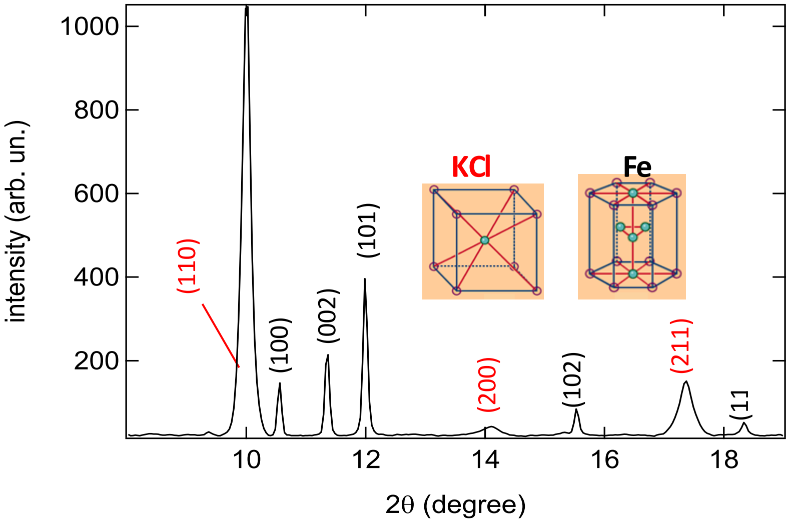


Information

- Structural
- Textural
- Chemical

X-ray Diffraction - XRD (2)





Position of the peaks:

-space group

-lattice parameters→ molar volume

Peaks profile:

-micro-strain

-crystallite size

Intensity of the peaks:

-Crystal structure

-Atomic positions

-Temperature factors

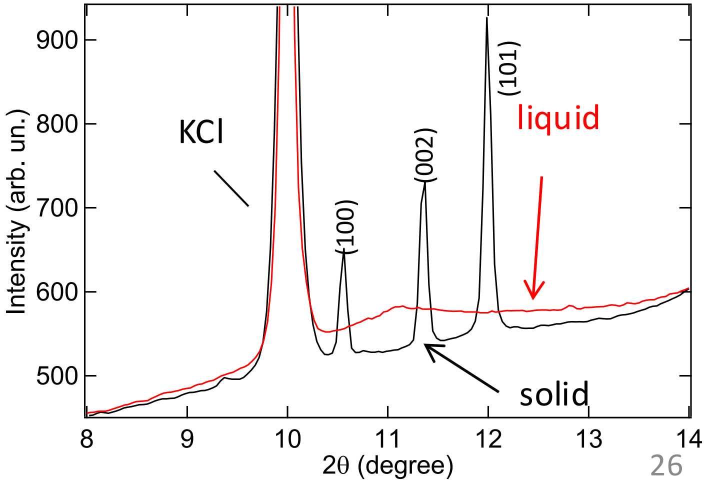
-Occupancy

-Preferred orientation

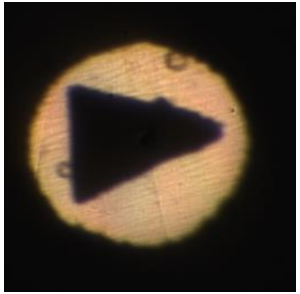
Information on the state of the sample

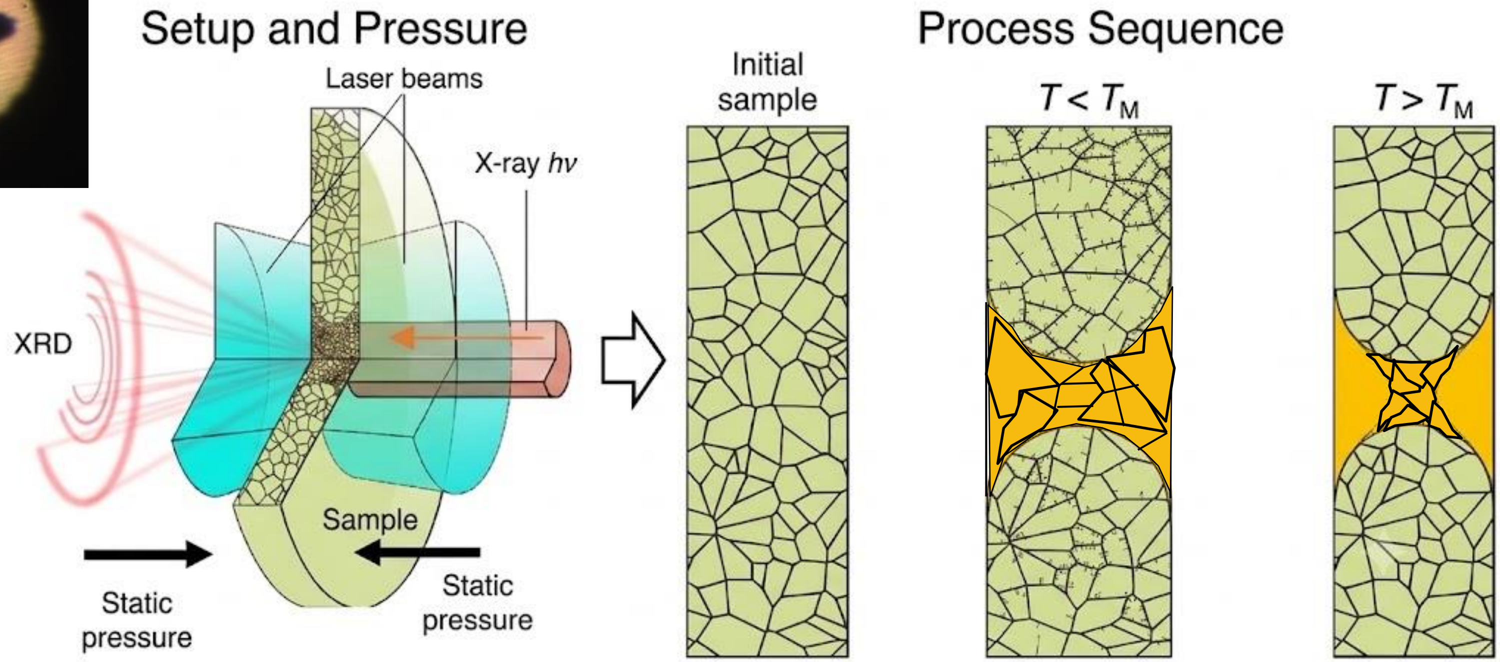
-Crystalline

-Amorphous



XRD (3)





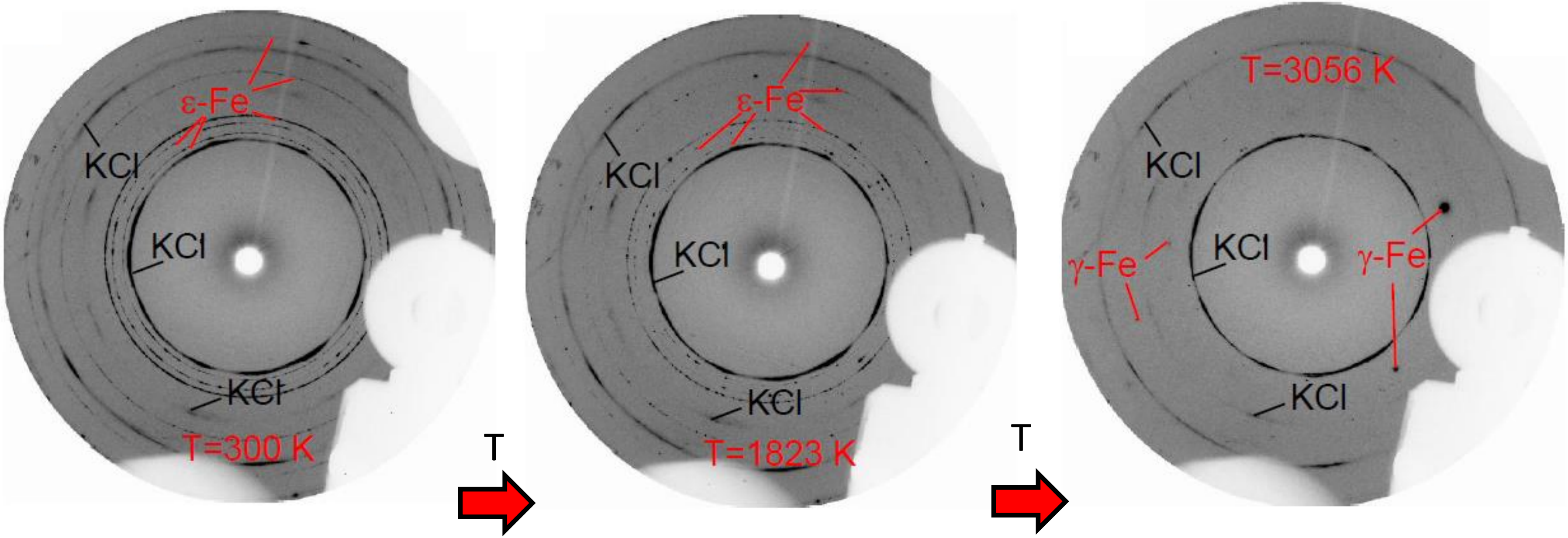
Hrubiak *et al.*, *Nature Communications* **8**, 14562 (2017)

XRD (4)

starting powder signal

temperature effects

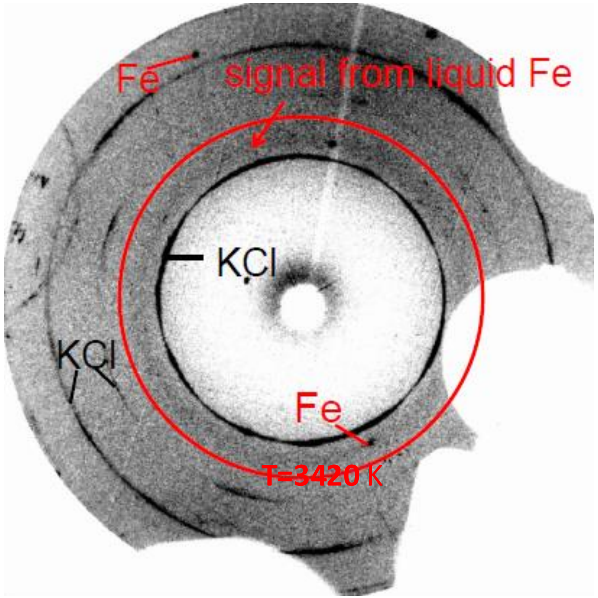
recrystallization

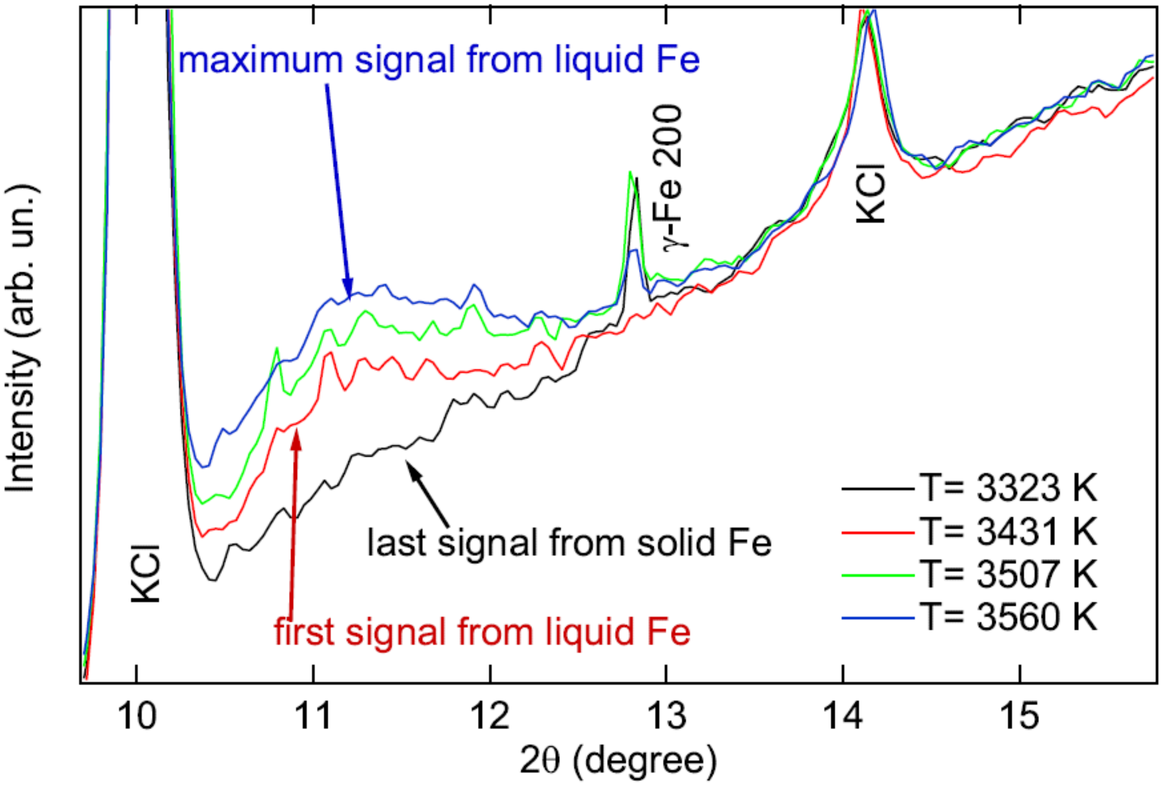


example of Fe sample loaded in KCl PTM

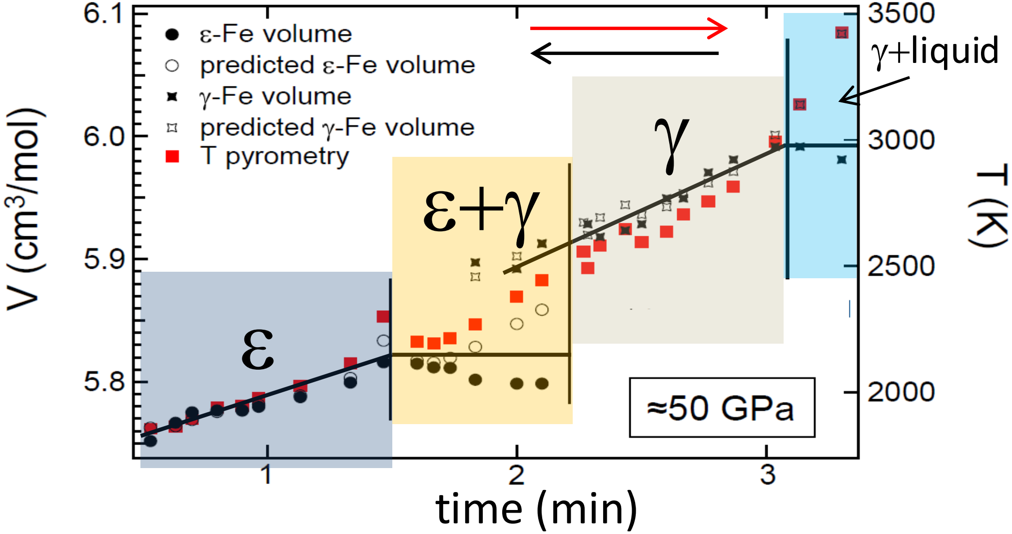
XRD (5)

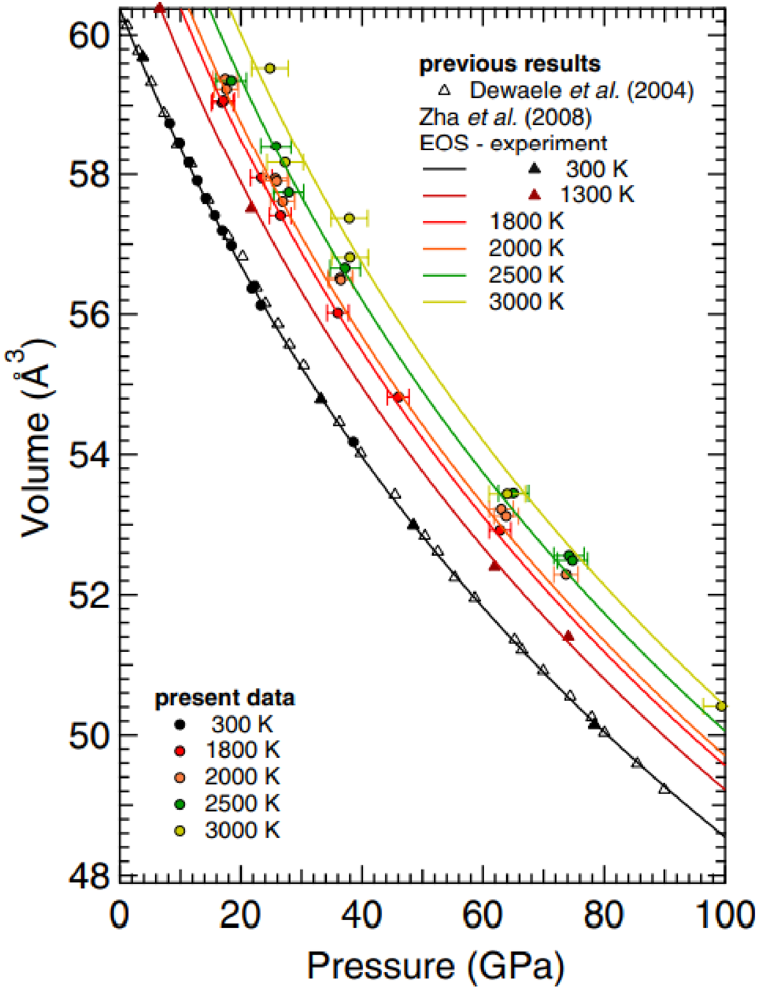
diffuse ring of molten sample





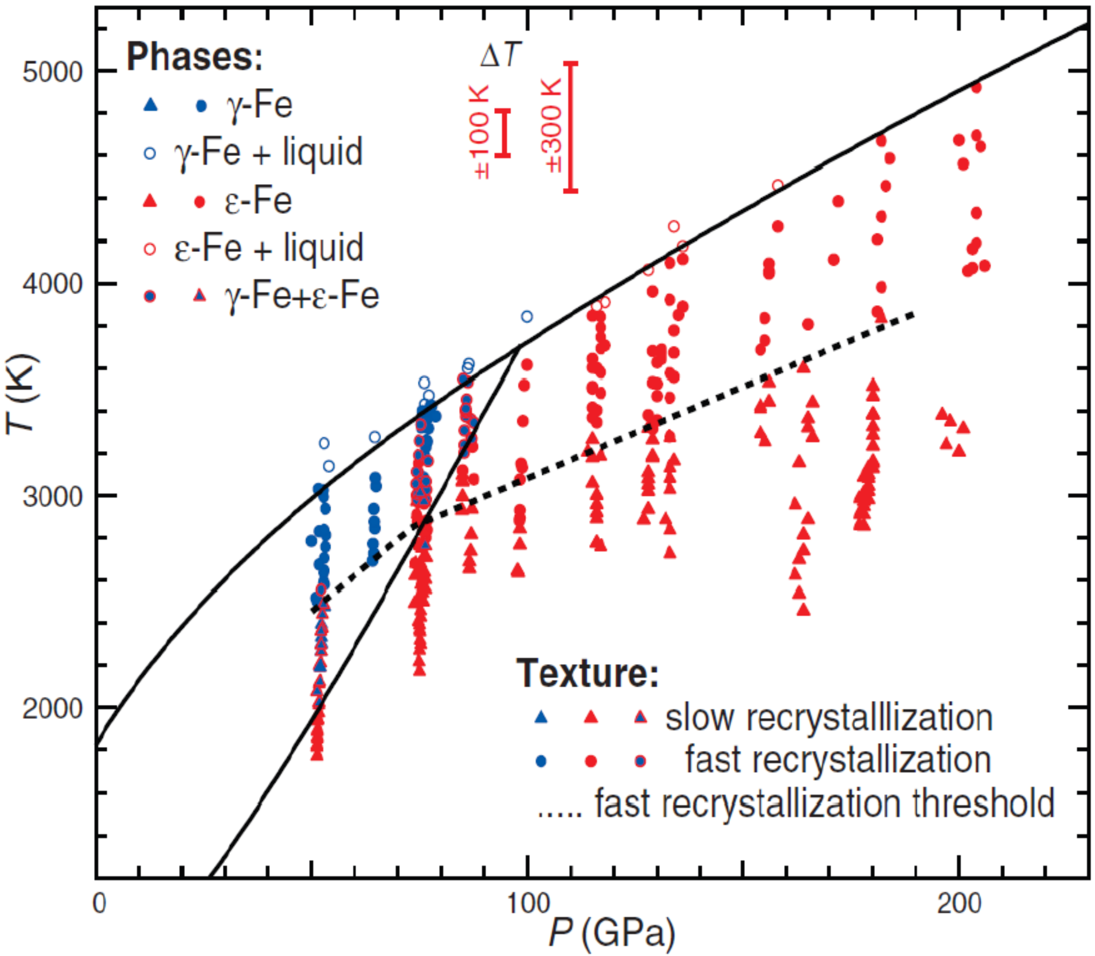
XRD (5)





Anzellini *et al.*, *Scientific Reports* **9**, 13034 (2019)

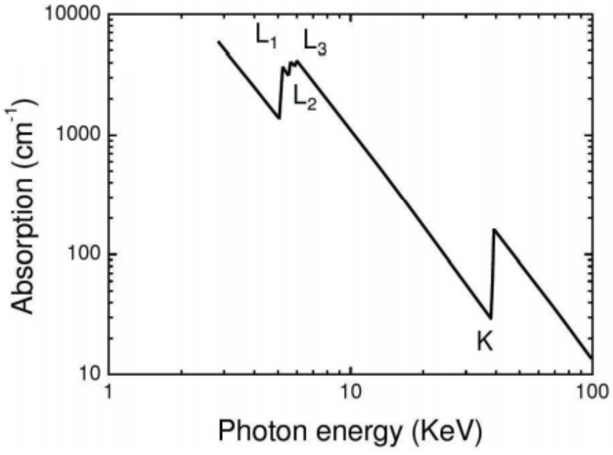
XRD (6)

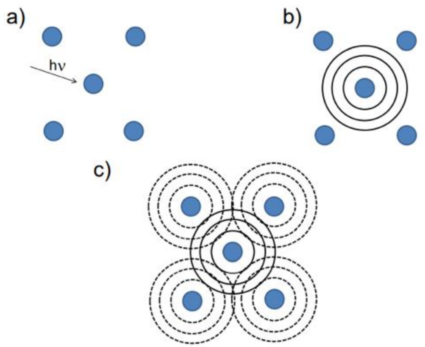


Anzellini *et al.*, *Science* **340**, 464 (2013)

XAS

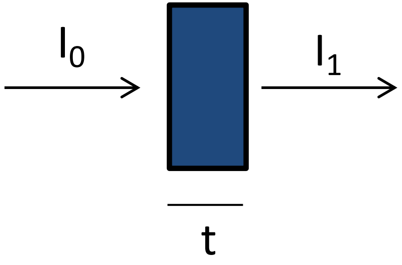
XAS (1)



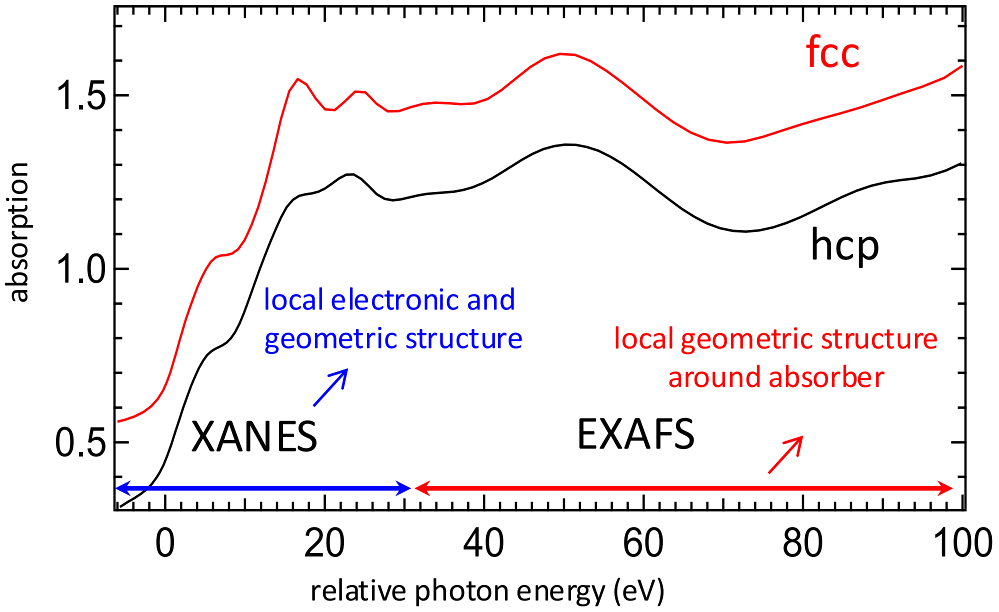


Beer-Lambert Law

$$I_1 = I_0 e^{-\mu t}$$

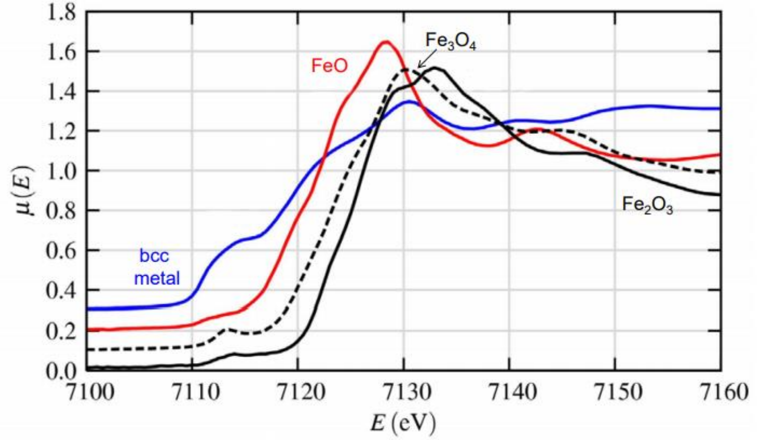


μ: linear absorption coefficient (E)



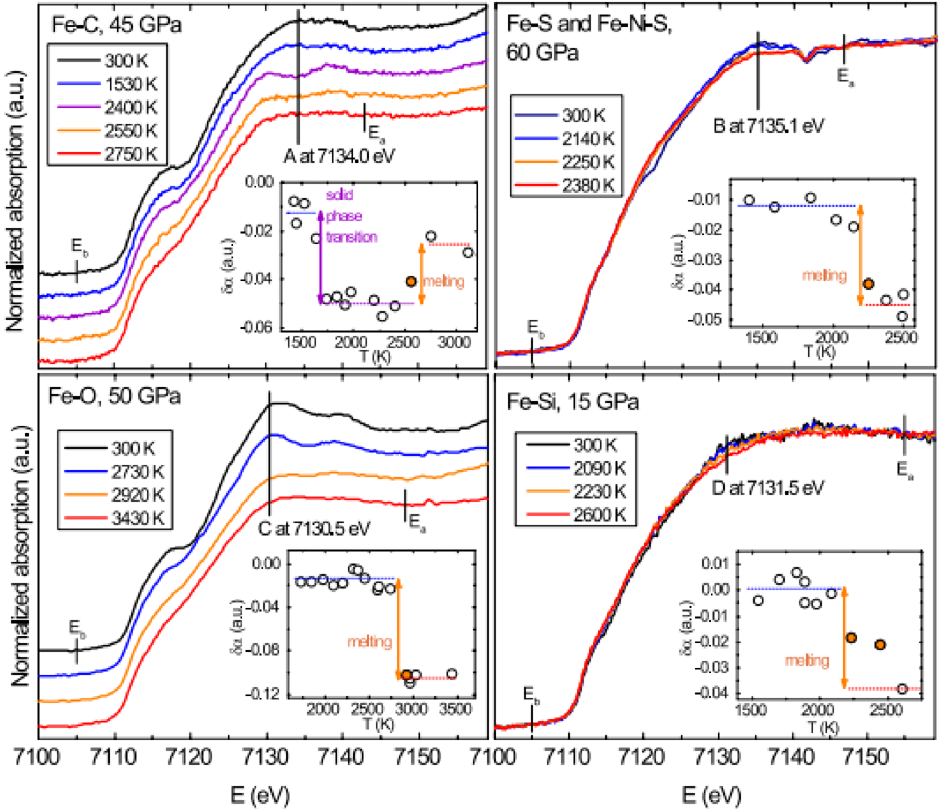
XAS (2)

Edge sensitive to oxidation state



M. Newville "*Foundamentals of XAFS*", Consortium for Advanced Radiation sources, University of Chicago (2004).

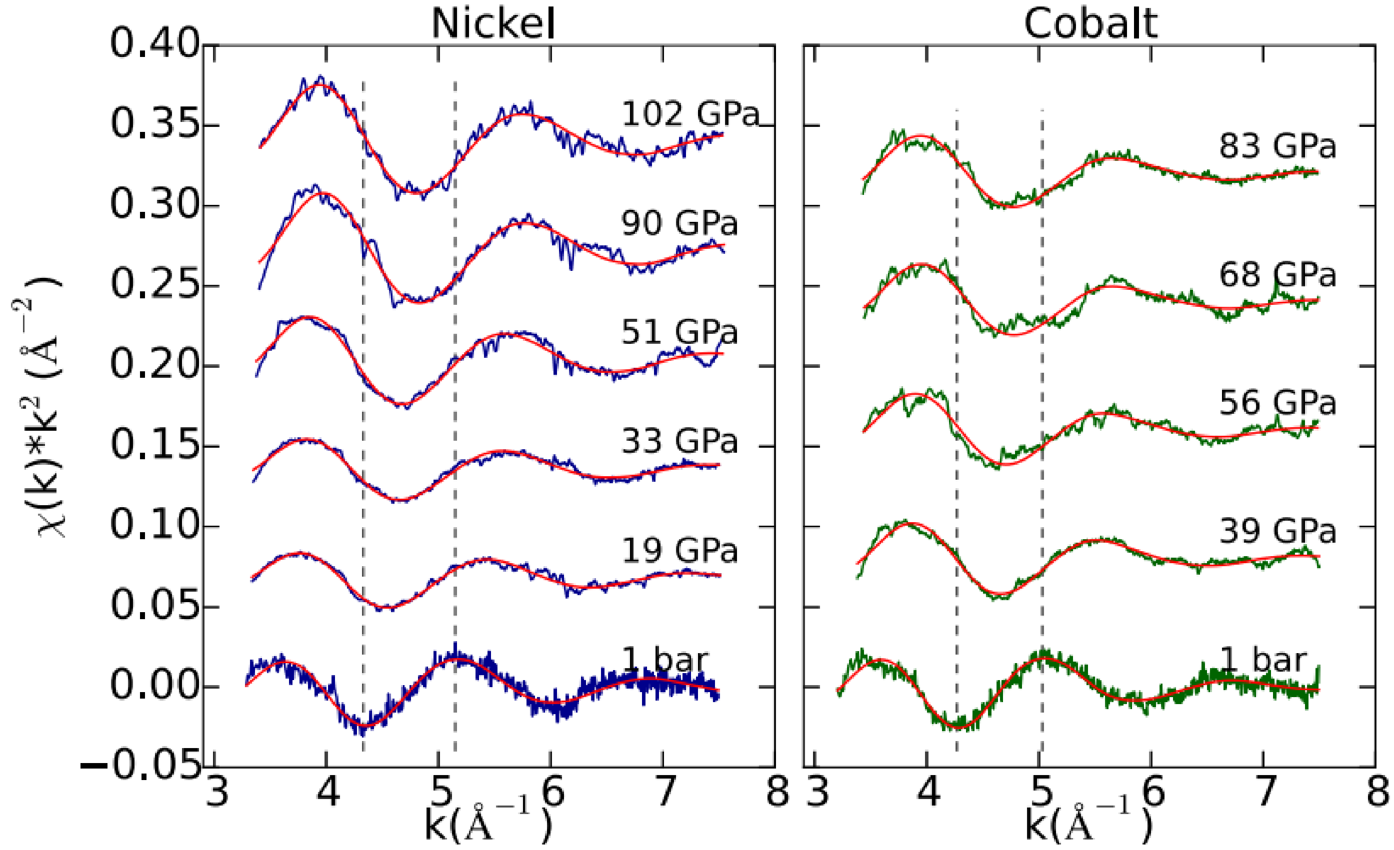
can detect onset of melting



Boccato *et al.*, *Scientific Reports*. **10**, 11663 (2020)

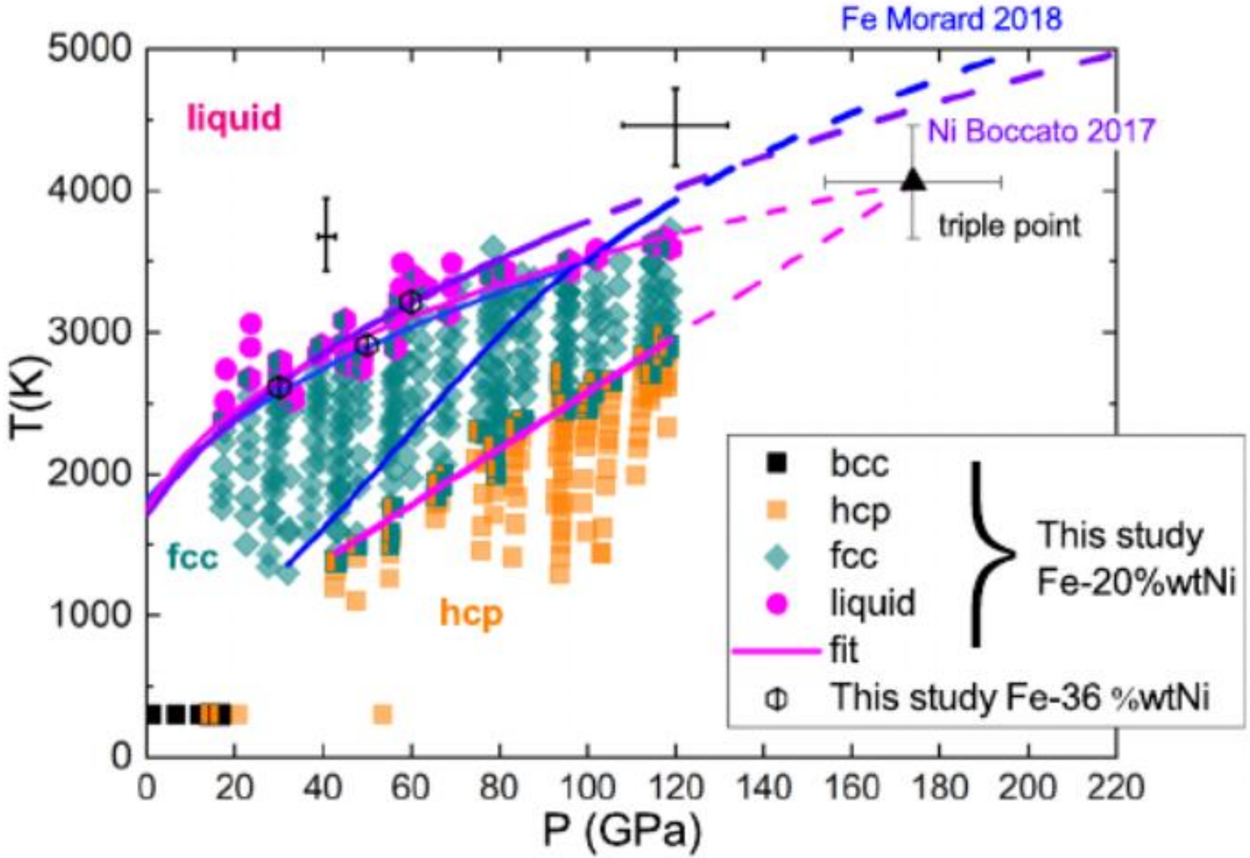
XAS (3)

Exploring compressional behaviour of liquids



Boccato *et al.*, *Phys. Rev. B* ***100***, 180101 (2019)

XAS (4)



Torchio *et al.*, *Geoph. Res. Lett.* **47** (2020)

MULTI-TECHNIQUES (4)

Portable double-sided laser-heating system for Mössbauer spectroscopy and X-ray diffraction experiments at synchrotron facilities with diamond anvil cells

Review of Scientific Instruments **83**, 124501 (2012); https://doi.org/10.1063/1.4772458

I. Kupenko[1,2], L. Dubrovinsky[1], N. Dubrovinskaia[3], C. McCammon[1], K. Glazyrin[1], E. Bykova[3], T. Boffa Ballaran[1], R. Sinmyo[1], A. Hanfland[2], W. Crichton[2], *and* M. Merlini[4]

ELSEVIER

Physics of the Earth and Planetary Interiors

Volume 174, Issues 1–4, May 2009, Pages 282-291

Development of *in situ* Brillouin spectroscopy at high pressure and high temperature with synchrotron radiation and infrared laser heating system: Application to the Earth's deep interior

Motohiko Murakami[a, b], Yuki Asahara[c], Yasuo Ohishi[c], Naohisa Hirao[c], Kei Hirose[d, e]

Full . Submitted: 20 July 2016 . Accepted: 08 December 2016 . Published Online: 28 December 2016

Combined laser ultrasonics, laser heating, and Raman scattering in diamond anvil cell system

Review of Scientific Instruments **87**, 123908 (2016); https://doi.org/10.1063/1.4972588

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SUMMARY

- Interest in HP-HT and how do we produce it in laboratories
- Why LH-DAC ?
- Principle of the LH-DAC:
  Sample preparation
  Description of the three paths for imaging, laser-heating and T measurement
  LH-DAC metrology
- LH-DAC at Synchrotron facilities
  - Coupled with XRD → Obtainable information
  - Couple with XAS → Obtainable information
  - Multi-techniques