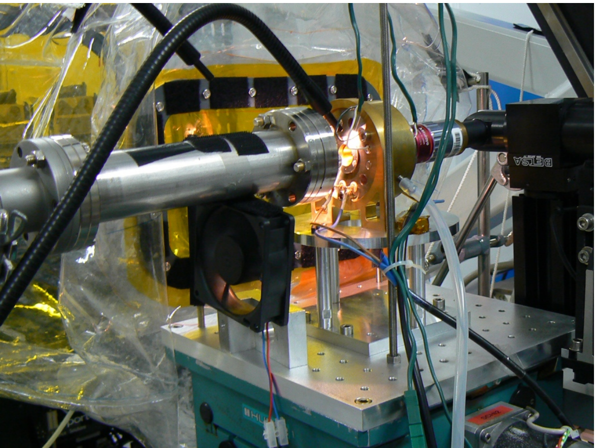
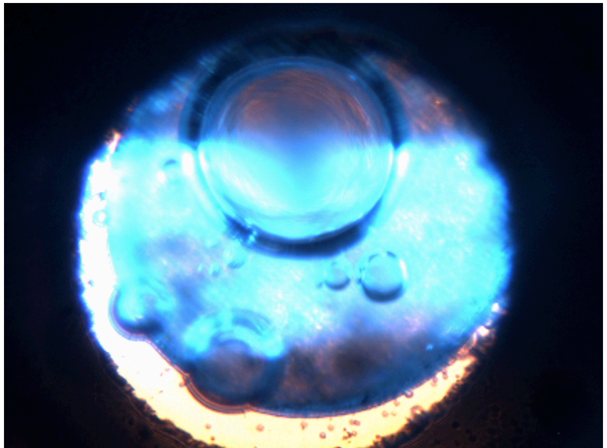
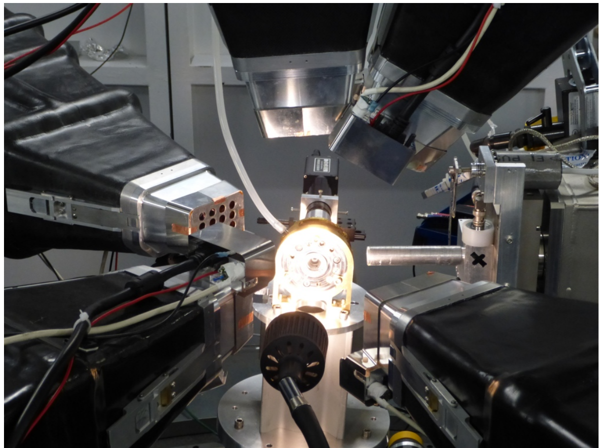
Introduction to resistively heated DAC techniques

Max Wilke

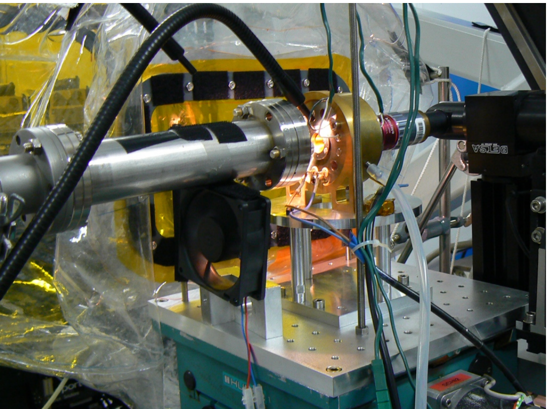
Universität Potsdam

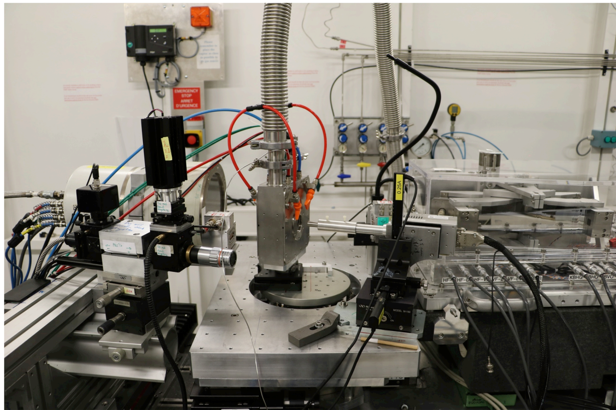
with material by C. Schmidt
& A. Rosa

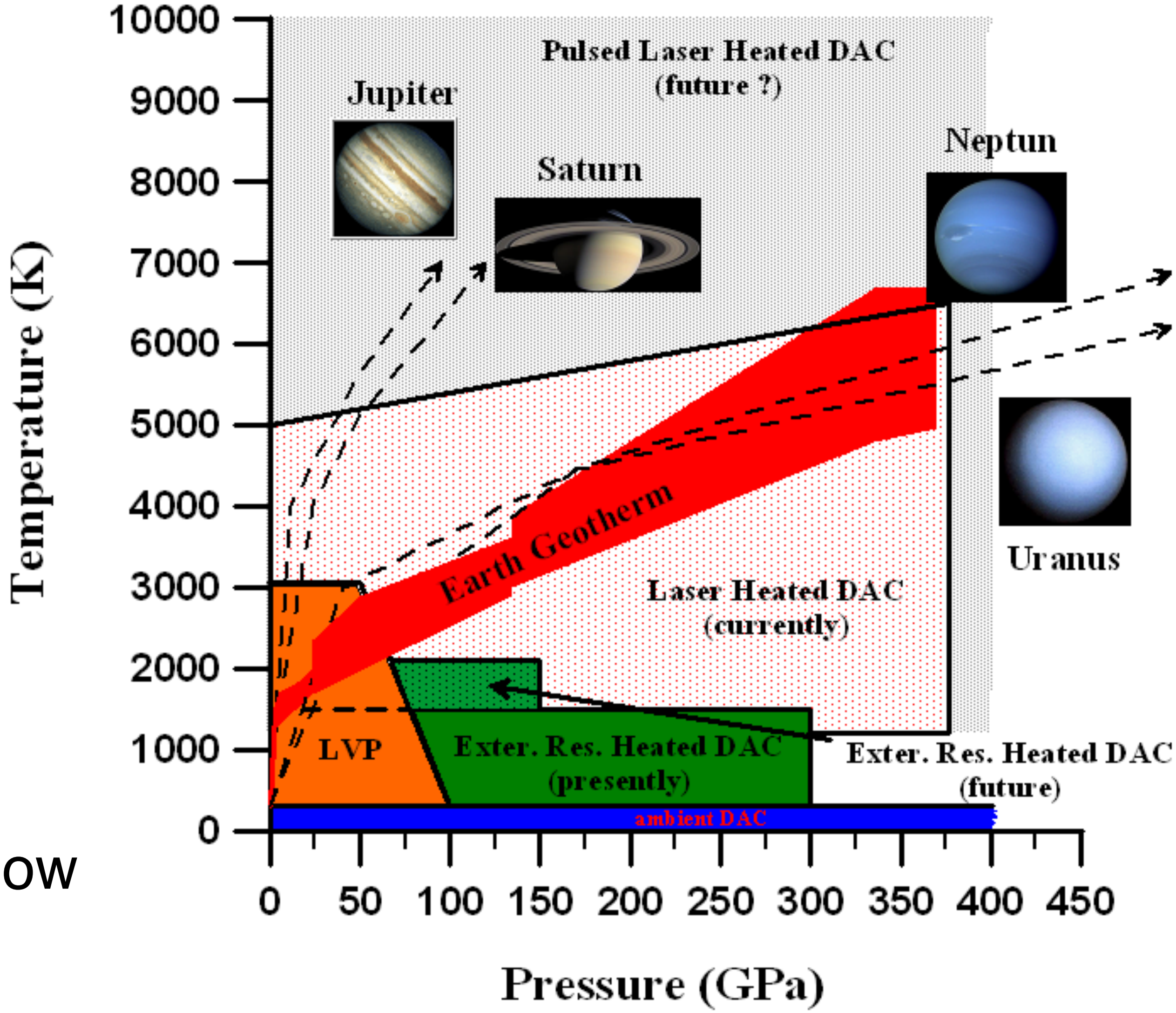
Outline

- Resistive Heating?
- Technical Concepts
    - Hydrothermal DAC
    - Resistively Heated DAC for Megabars
- Using resistively heated DACs
- Examples of Application





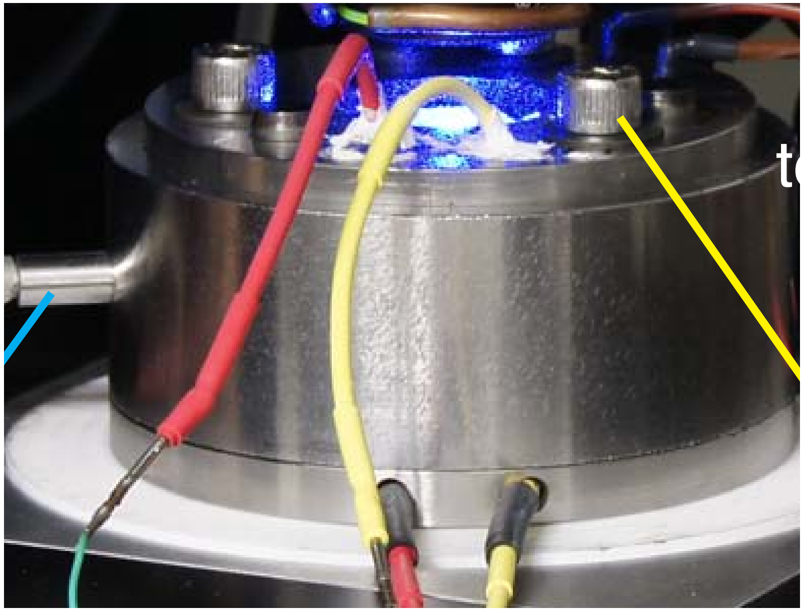
Why using resistive heating?



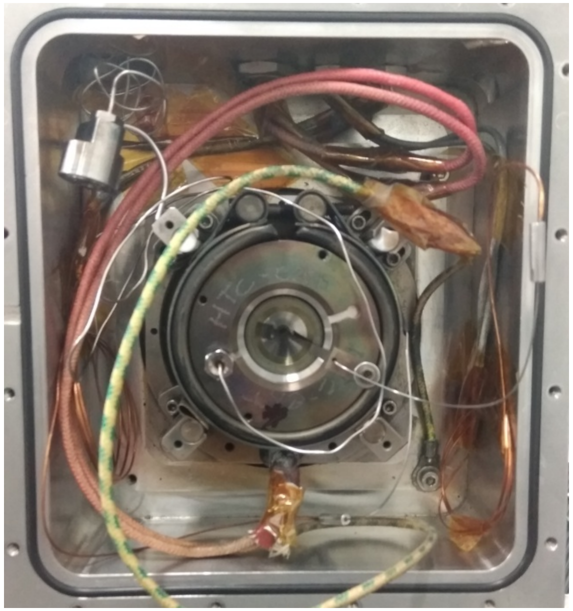
H.P. Liermann, DESY Hamburg

- Temperatures below 1500 K
- precise T-Control

Hydrothermal DAC vs. DAC f. Megabars



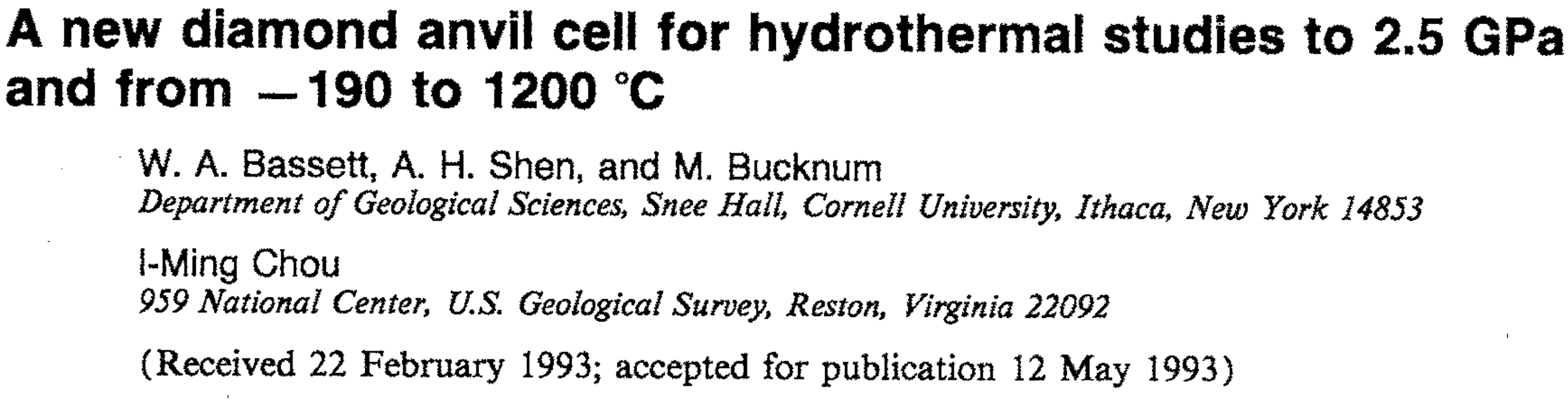
Hydrothermal DAC



ESRF-type DAC

Hydrothermal DAC - HDAC

Rev. Sci. Instr. 64, No. 8, August 1993



A new diamond anvil cell for hydrothermal studies to 2.5 GPa and from −190 to 1200 °C

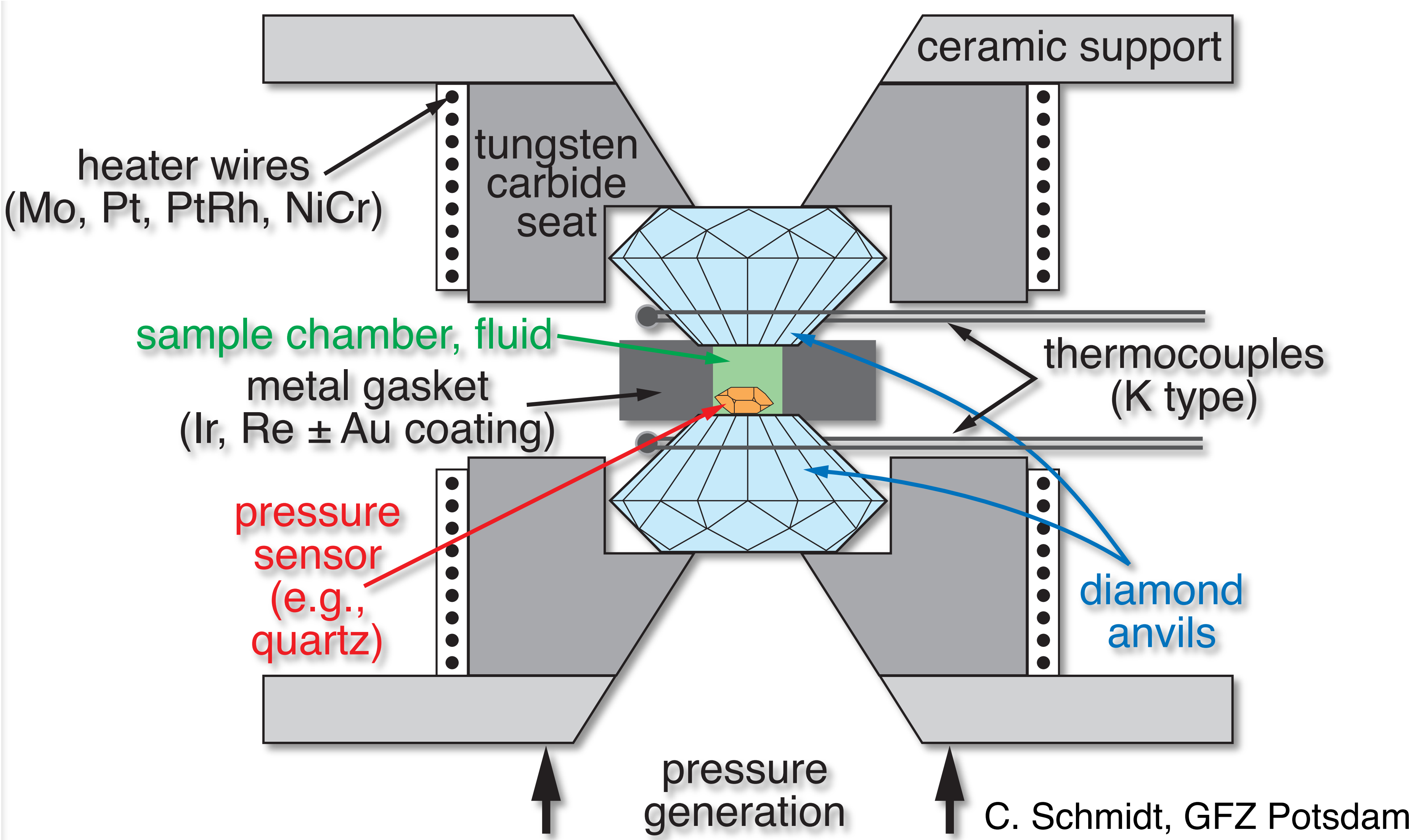
W. A. Bassett, A. H. Shen, and M. Bucknum
Department of Geological Sciences, Snee Hall, Cornell University, Ithaca, New York 14853

I-Ming Chou
959 National Center, U.S. Geological Survey, Reston, Virginia 22092

(Received 22 February 1993; accepted for publication 12 May 1993)

- particularly used for experiments with hydrous fluids
  e.g.
  23 GPa at 750 °C (Lin et al., 2004)
  1025±10 °C at ~2 GPa (Audétat & Keppler, 2005)

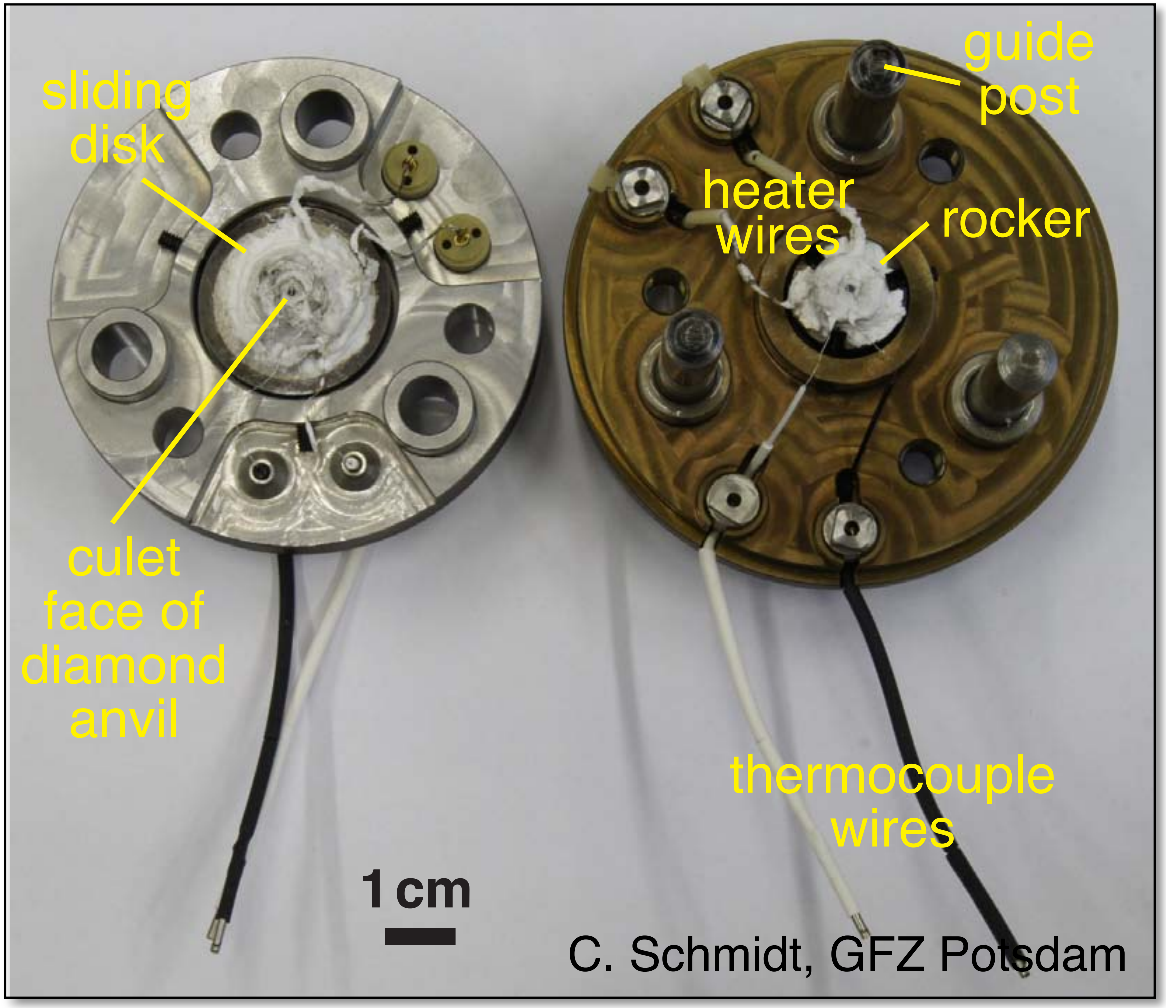
HDAC Construction



C. Schmidt, GFZ Potsdam

HDAC Construction

upper and lower platen



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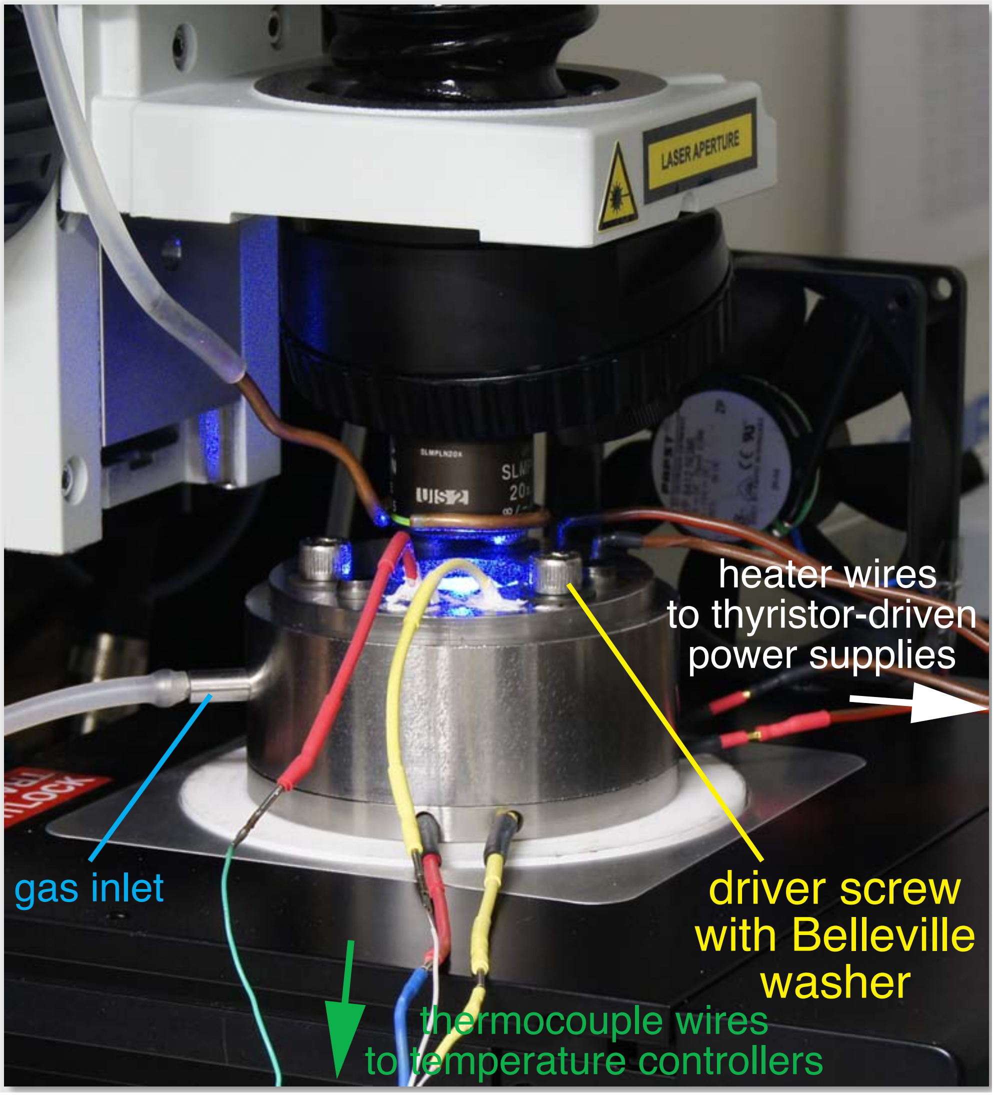
precise T control (± 0.1 K)
small T gradients
fast T stabilization
accurate T measurement

HDAC Construction



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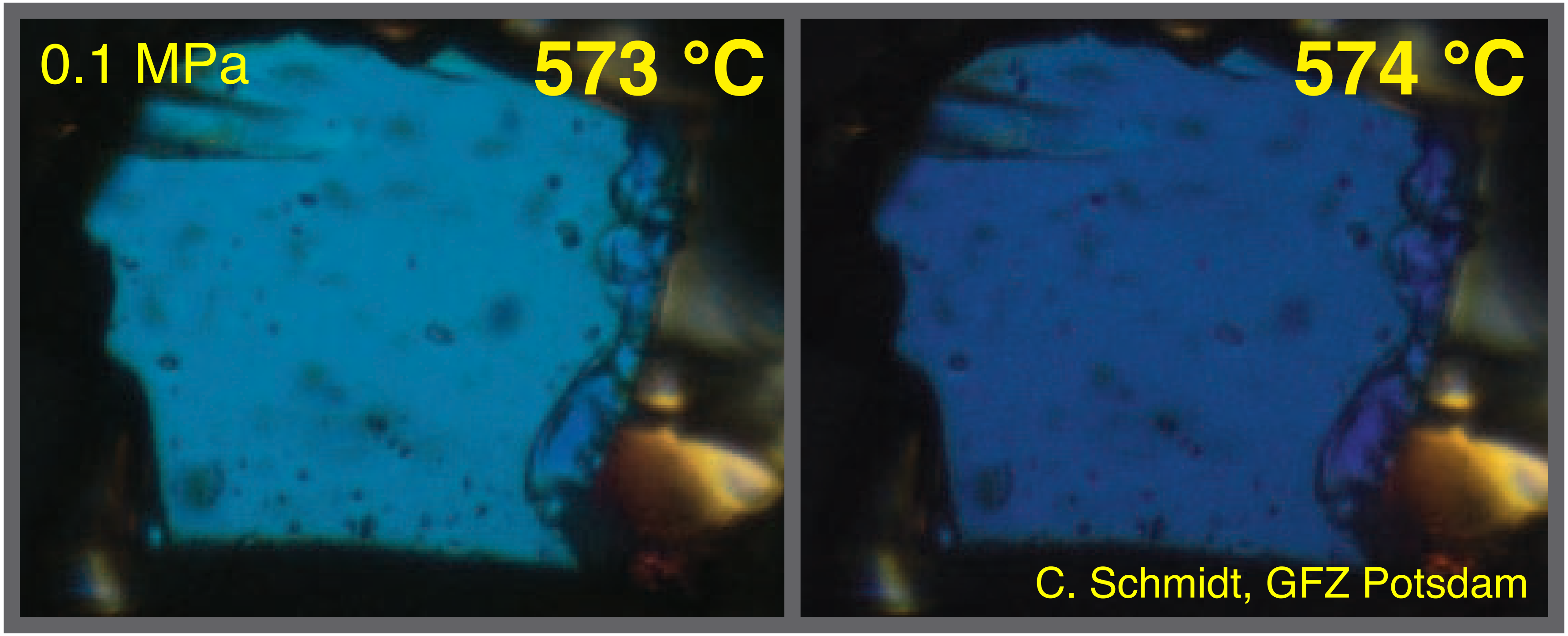
- HDAC on stage of Micro-Raman spectrometer
- Cell body is flushed with H_2-bearing gas (Ar, N_2) to prevent oxidation of parts

Temperature Calibration

- Melting points at ambient pressure, eg.
  NaCl, 800.7°C
  CsCl, 645°C
  $K_2Cr_2O_7$, 398°C
  $NaNO_3$, 306.8°C

- Triple point of H_2O
  Ice I + liquid + vapor at 0.1°C, 0.6 kPa
  Ice I + Ice III + liquid at -21.985°C, 209.9 MPa

Temperature Calibration



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- α-β quartz transition: displacive, little hysteresis
  observation using crossed polarizers
  cut II c-axis 75 *μ*m thick

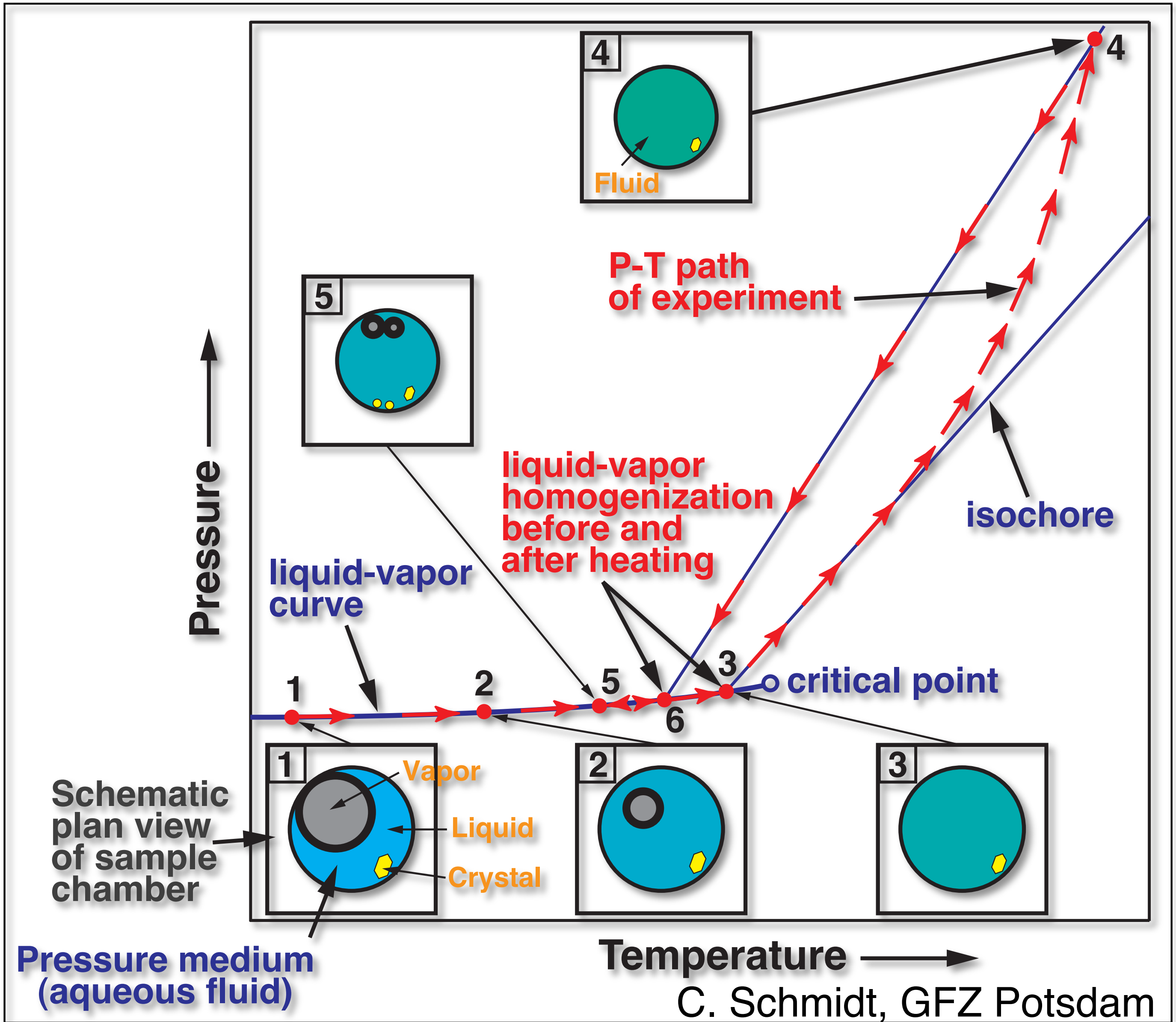
Pressure determination

- **Phase transitions and equation of state** of the pressure medium (fluid)

- **Pressure calibrants**
  X-ray diffraction: e.g. Au, Pt

  Raman or Fluorescence lines:
  fluorescence sensors: e.g. ruby, Sm:YAG
  Raman sensors: e.g. α-quartz, zircon

Pressure determination
phase transitions and EoS of fluid



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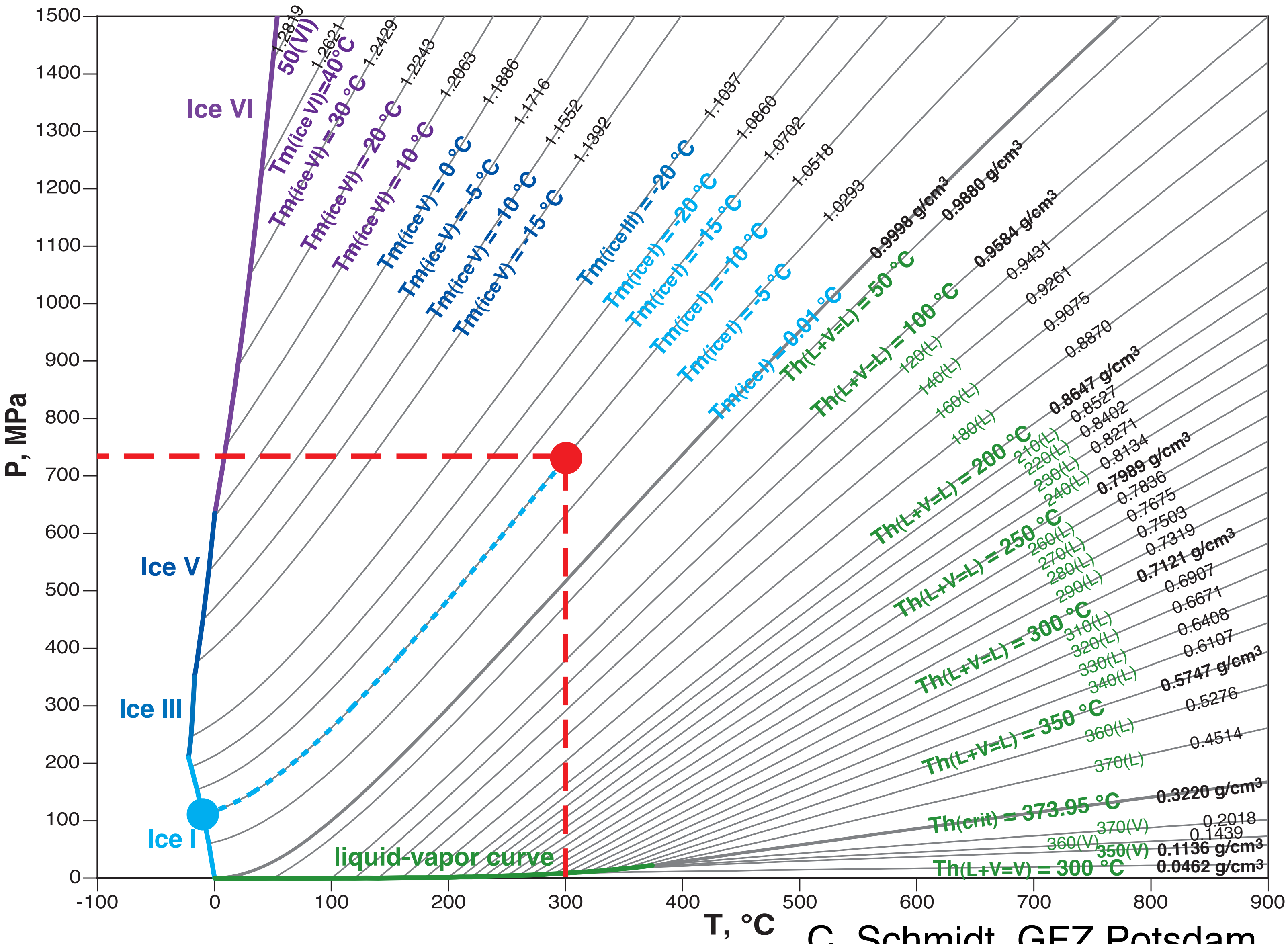
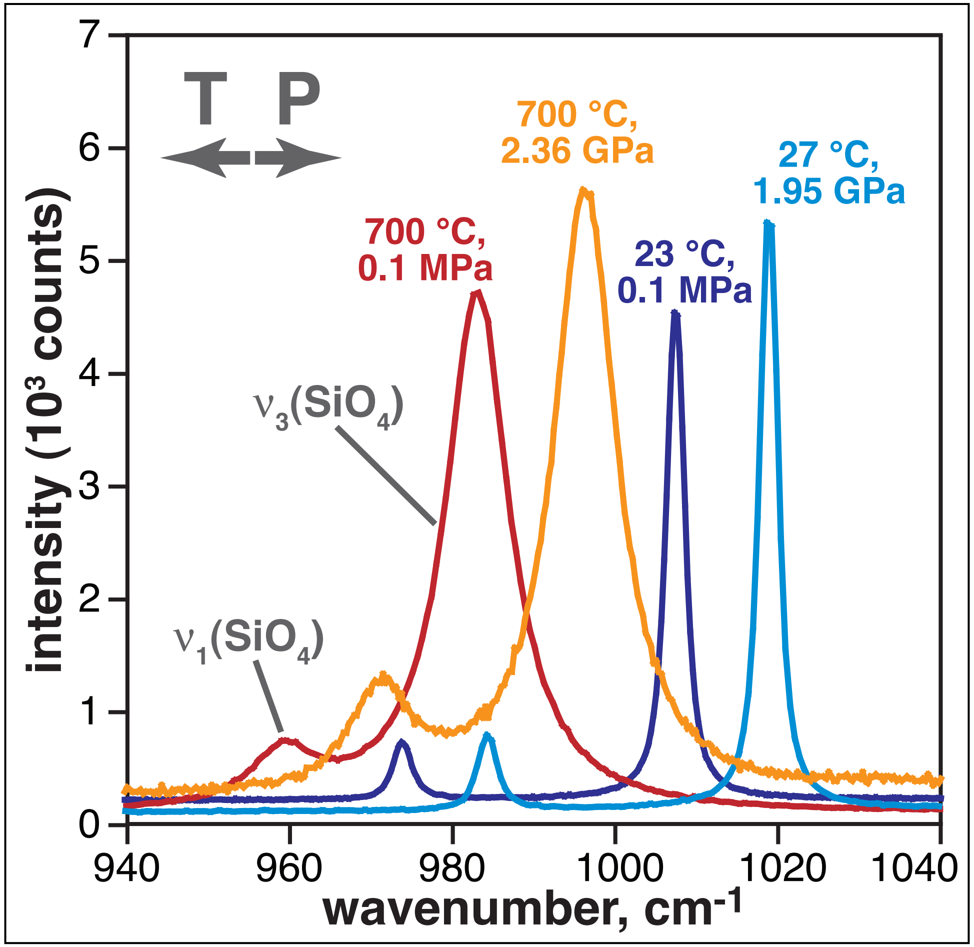
Pressure determination
EoS of H_2O for dilute solutions

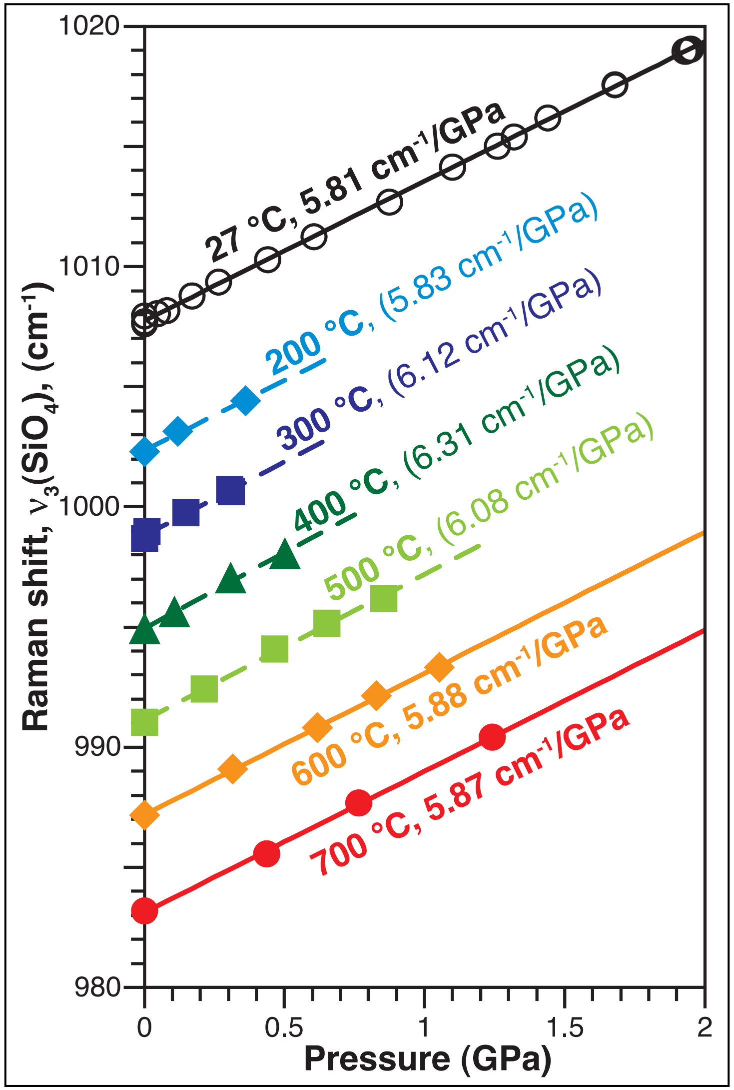


equation of state: Wagner and Pruß (2002)

C. Schmidt, GFZ Potsdam

Pressure determination
$\nu_3(SiO_4)$-band zircon ($ZrSiO_4$)





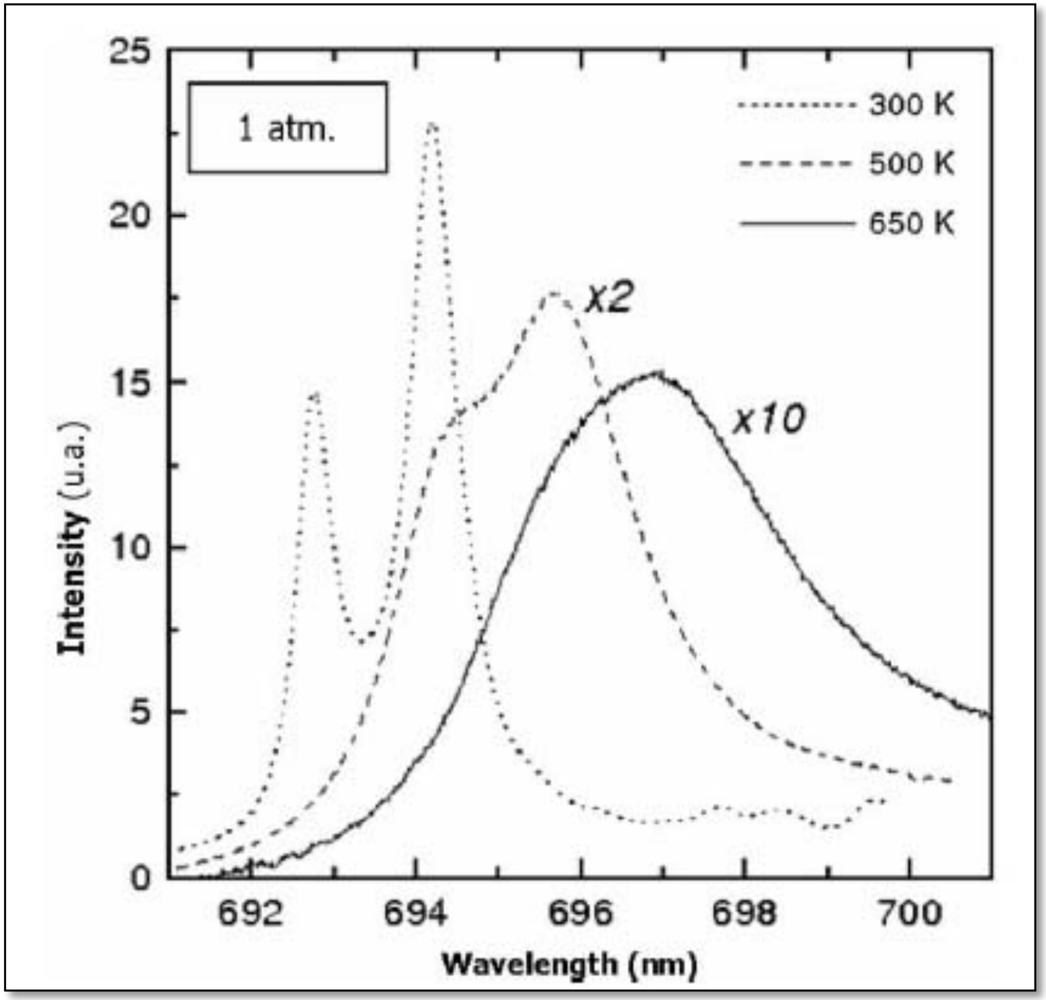
ν_{1008}: to ~1000 °C, to ~10 GPa

Schmidt et al. 2013

Ruby Fluorescence?

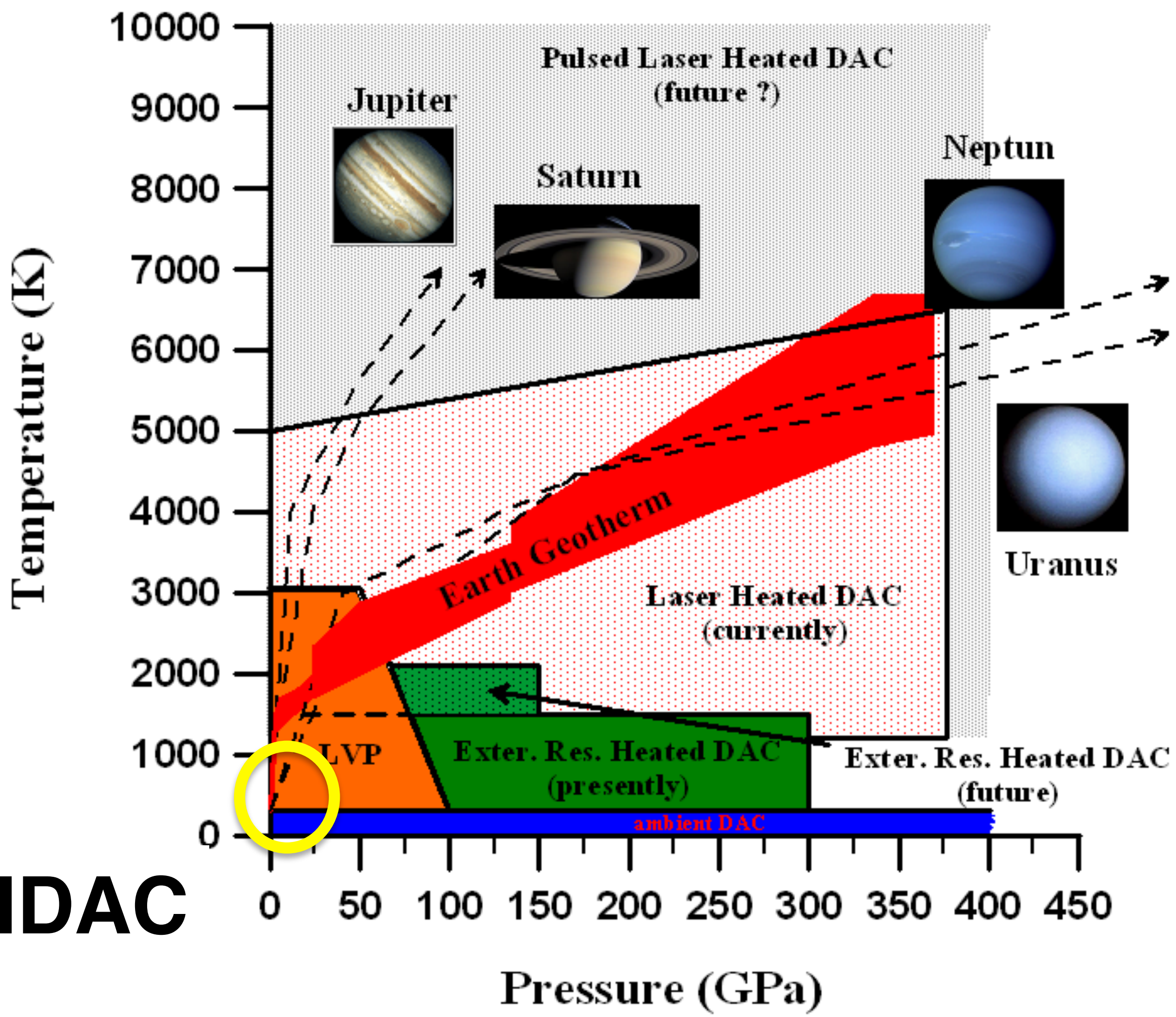
- at high T:
  two peaks merge
  intensity decreases
  → non-linear frequency shift with T

- above 300°C
  pressure determination difficult



Datchi et al. 2007

Reaching conditions beyond HDAC

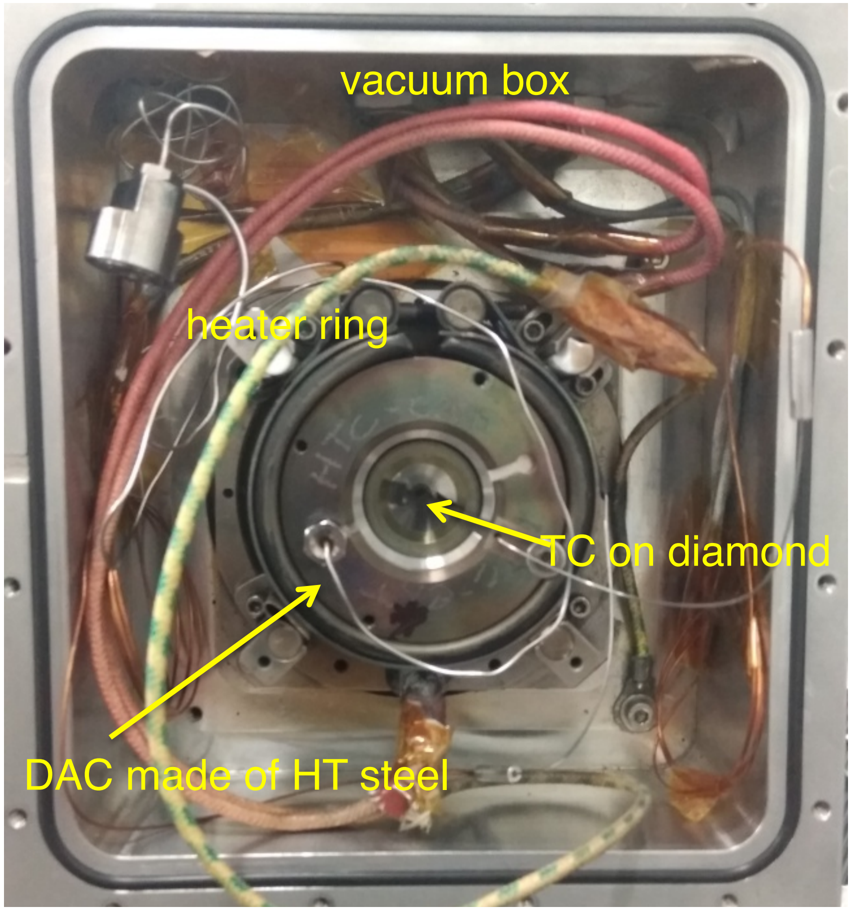


HDAC

H.P. Liermann, DESY Hamburg

Resistively Heated DAC for Megabars

External Heating



External heater ring

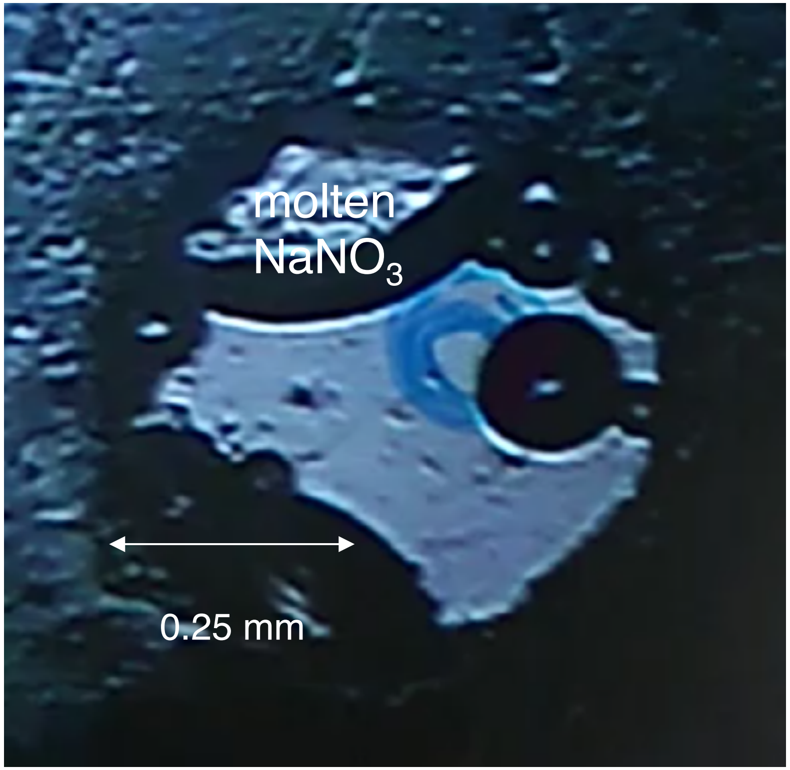
Angelika Rosa, ESRF

Resistively Heated DAC for Megabars

External Heating

- **T limited to 600^{o}C**
- **Slow T stabilisation, 20 min**
- **Accurate T measurement**

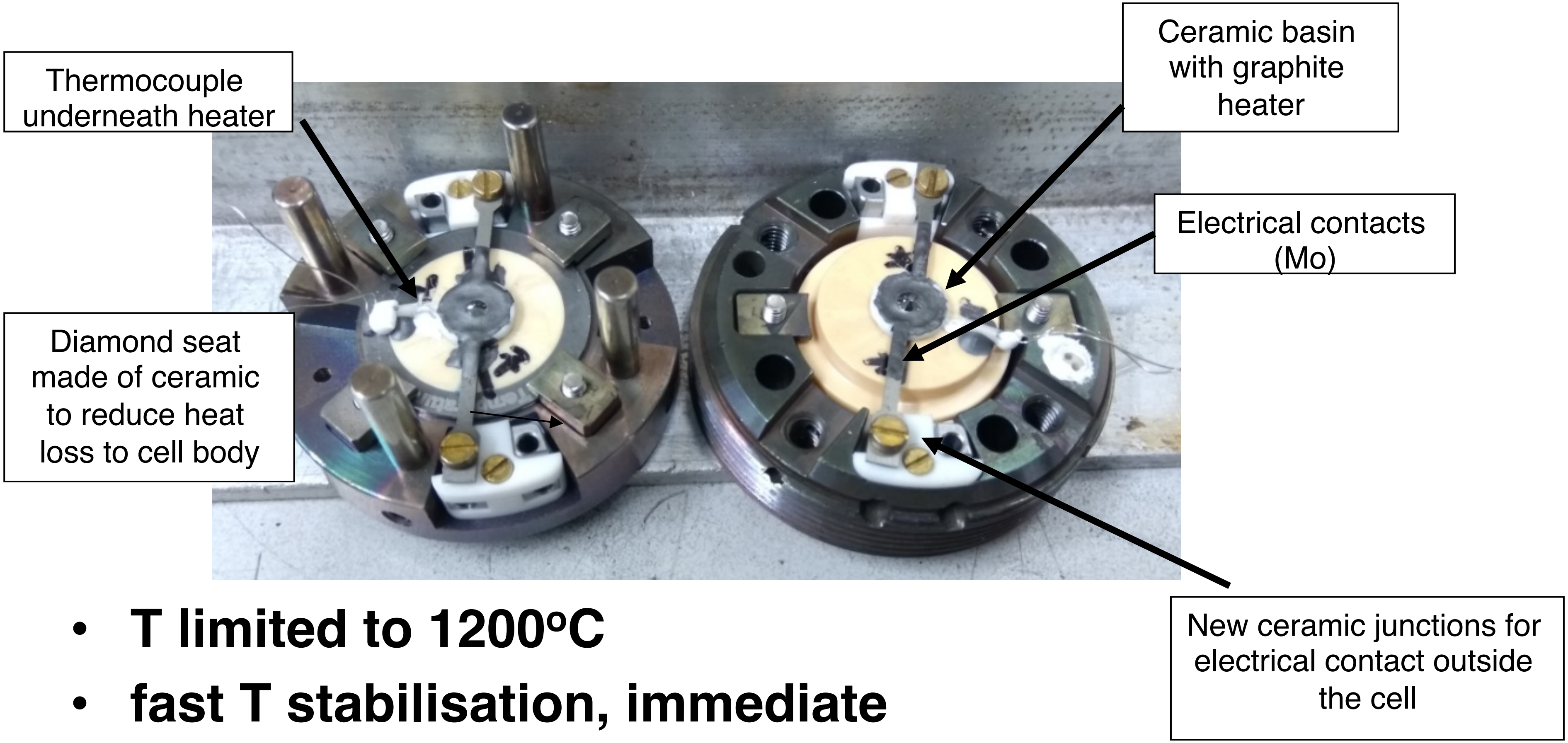
T diamond = 305°C
T sample = 306.4°C



With courtesy of R. Jarnias

Resistively Heated DAC for Megabars

Internal Heating


Thermocouple underneath heater

Diamond seat made of ceramic to reduce heat loss to cell body

Ceramic basin with graphite heater

Electrical contacts (Mo)

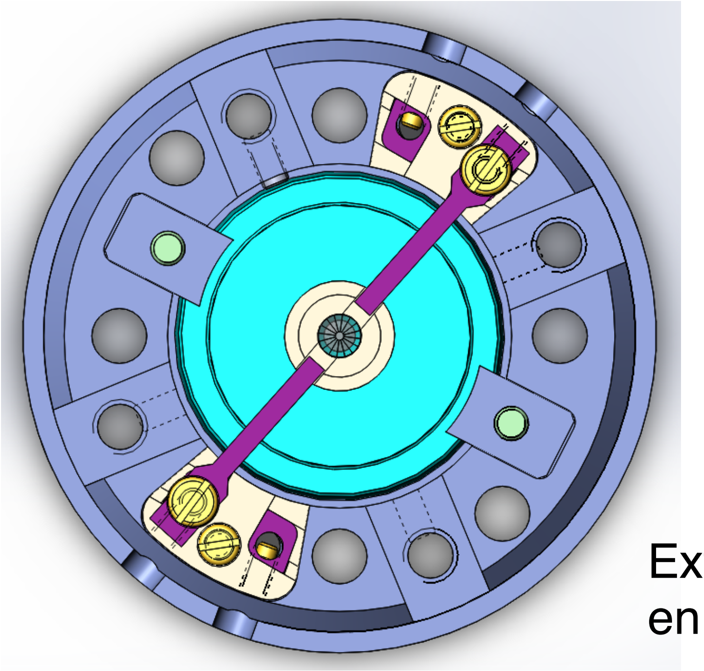
New ceramic junctions for electrical contact outside the cell

- **T limited to 1200ºC**
- **fast T stabilisation, immediate**
- **Accurate T measurement**

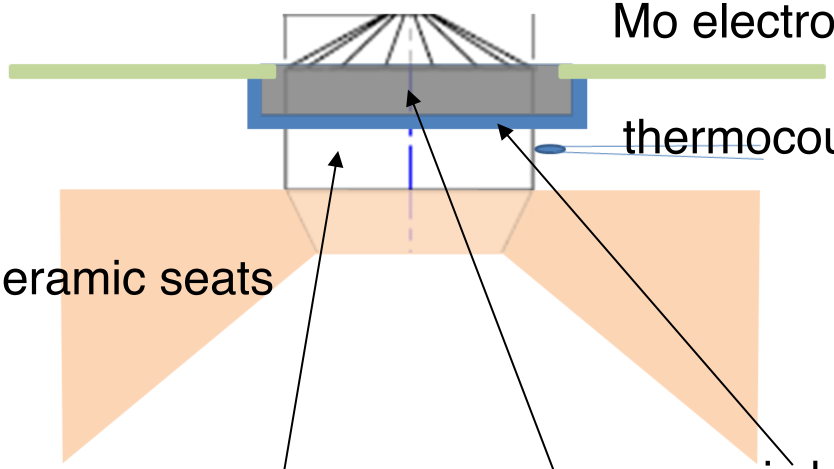
Resistively Heated DAC for Megabars

- New internal heating design
- liquid graphite, T-stability > 3000°C

TOP VIEW



SIDE VIEW



Mo electrodes

thermocouple

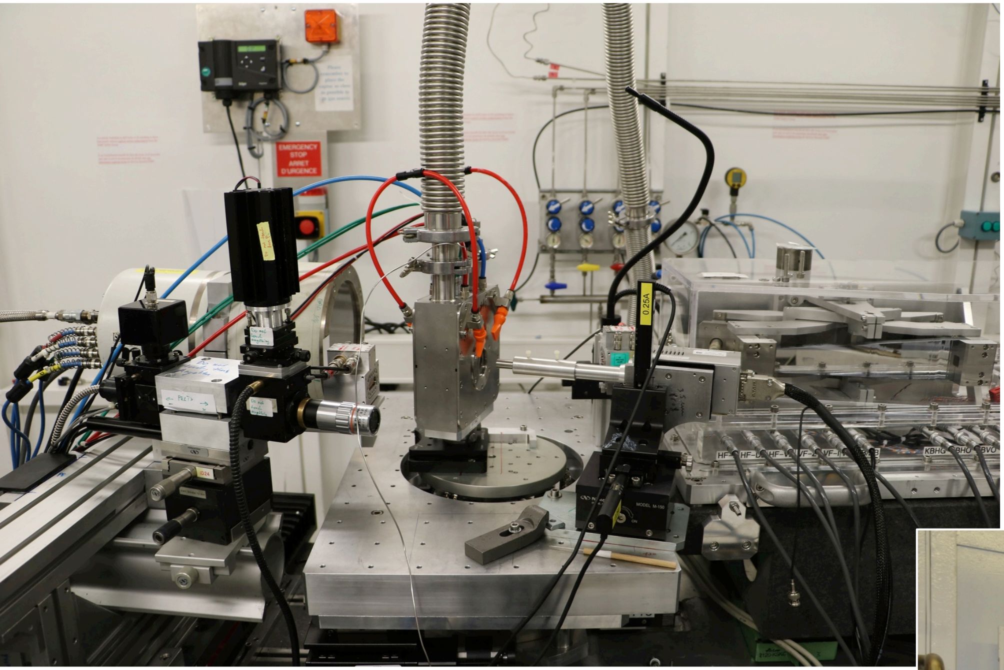
Ceramic seats

ceramic basin for heater

Extra high diamonds (1 mm) enlarged to fit TC and heater)

Graphite heater

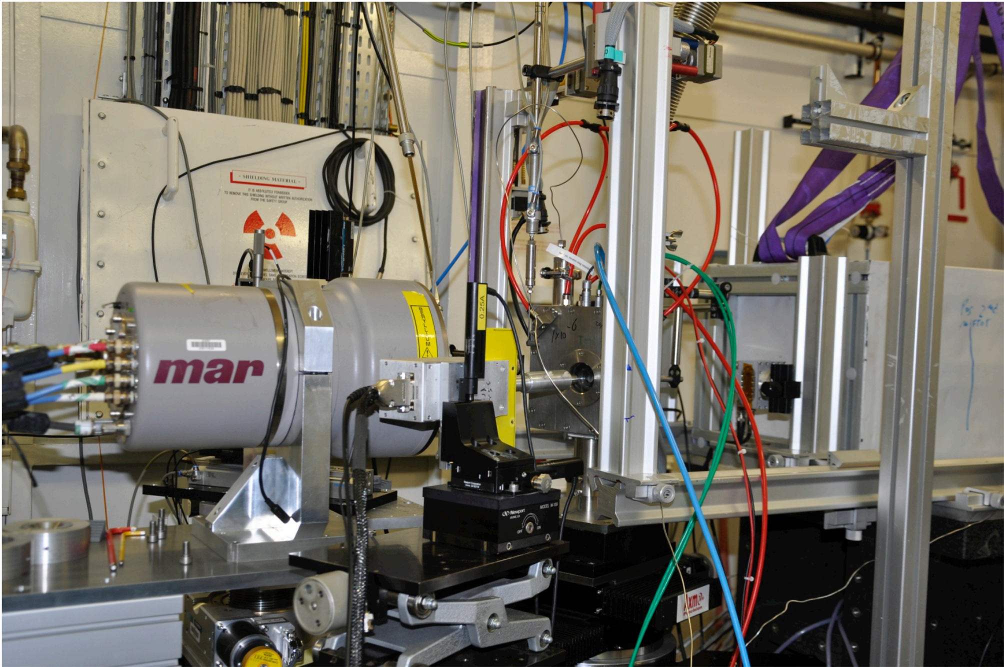
Implementation at Beam line



BM 23 (ID 24 DCM)

- ✓ XAS
- ✓ XRF
- ✓ XRD

- ✓ Beam size 3*3 μm^2
- ✓ 5-40 keV

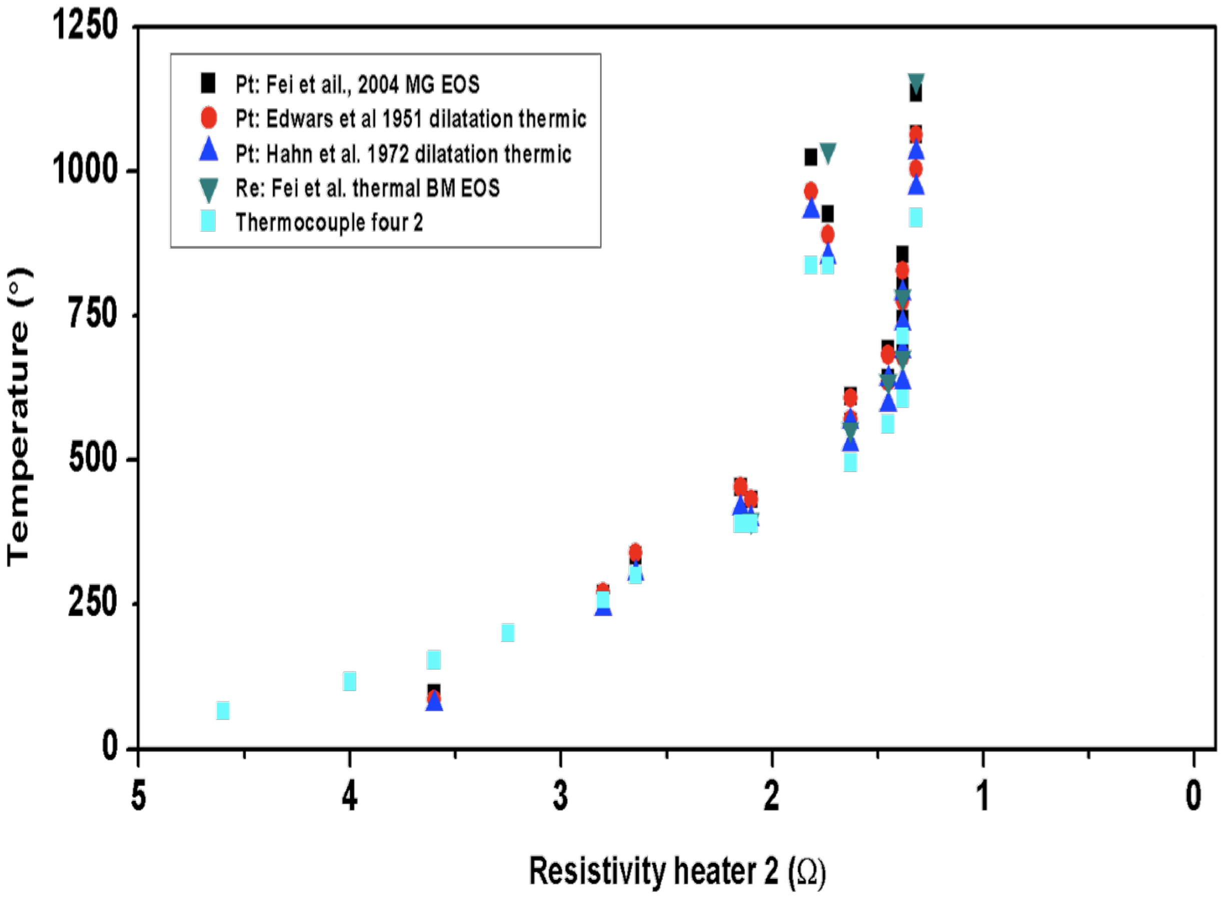


ID 27

- ✓ XRF
- ✓ XRD

- ✓ Beam size 1*1 μm^2 (-> 0.15 μm)
- ✓ 20-30 keV

Resistively Heated DAC for Megabars



With courtesy of A.D. Rosa and G. Garbarino

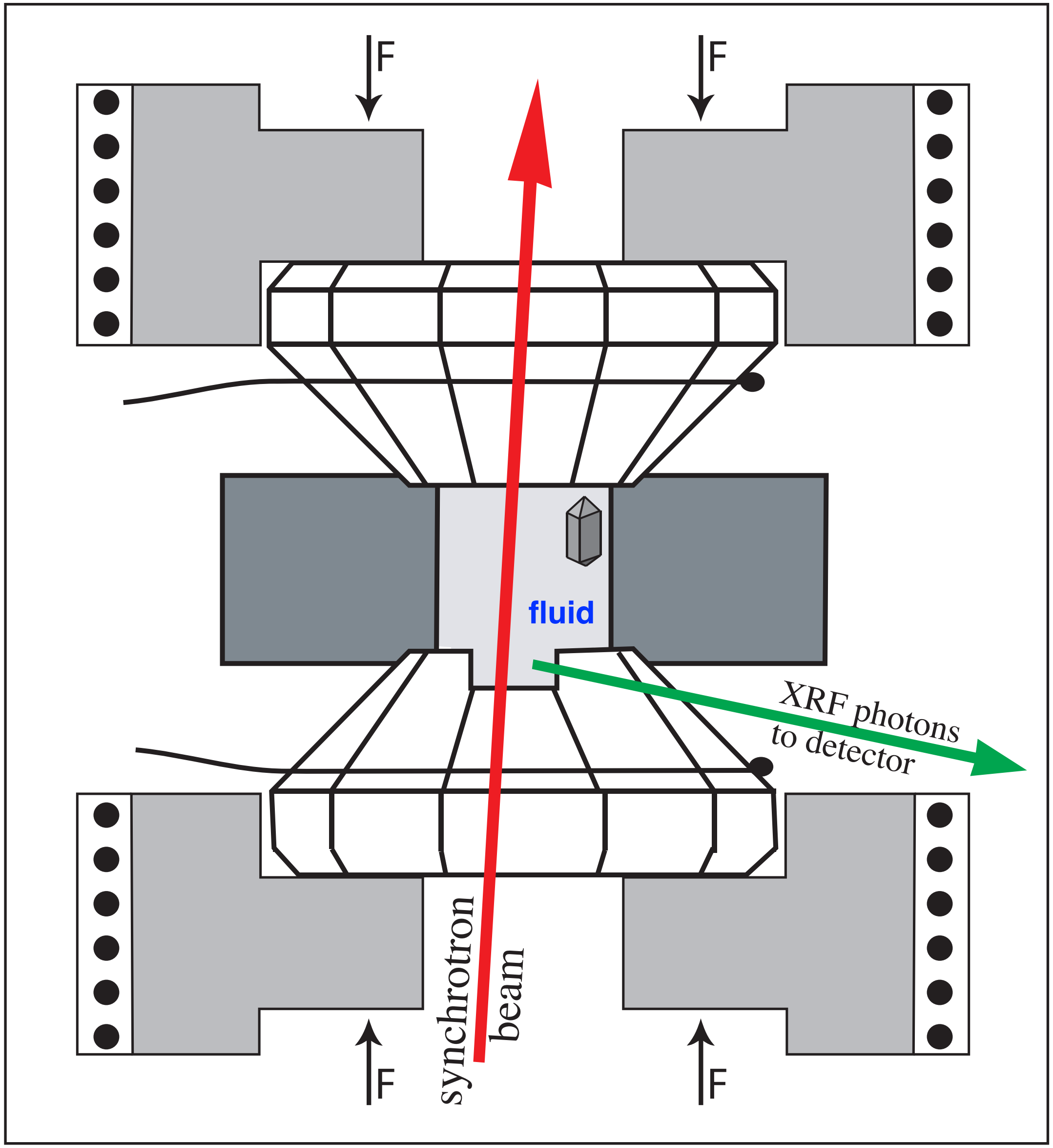
- first test runs at ID27 at ambient pressure using hex-BN and Pt as test samples

Applications of HDAC and RH-DAC

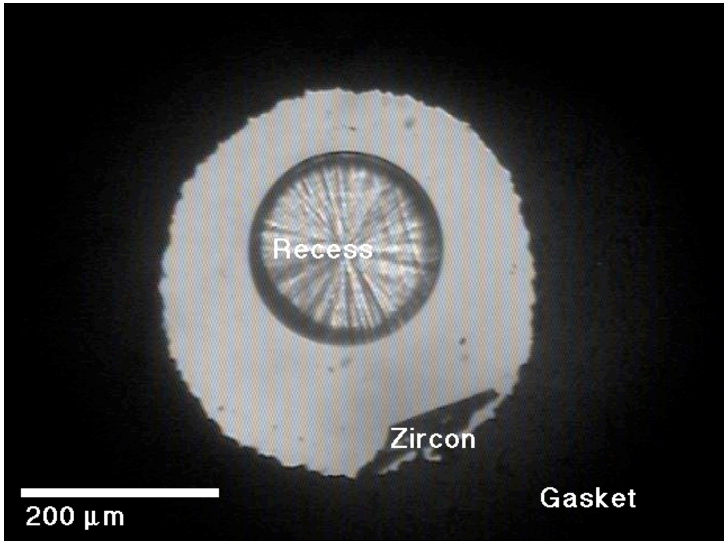
- optical microscopy, microthermometry
- Raman & IR spectroscopy
- synchrotron radiation X-ray fluorescence and absorption spectroscopy
- inelastic X-ray scattering (X-ray Raman spectroscopy)
- Brillouin spectroscopy
- electrical conductance
- XRD

- Zircon solubility and Zr complexation in fluids at high P&T
- H_2O at high P&T
- Silicate Melts/Glasses at extreme pressure

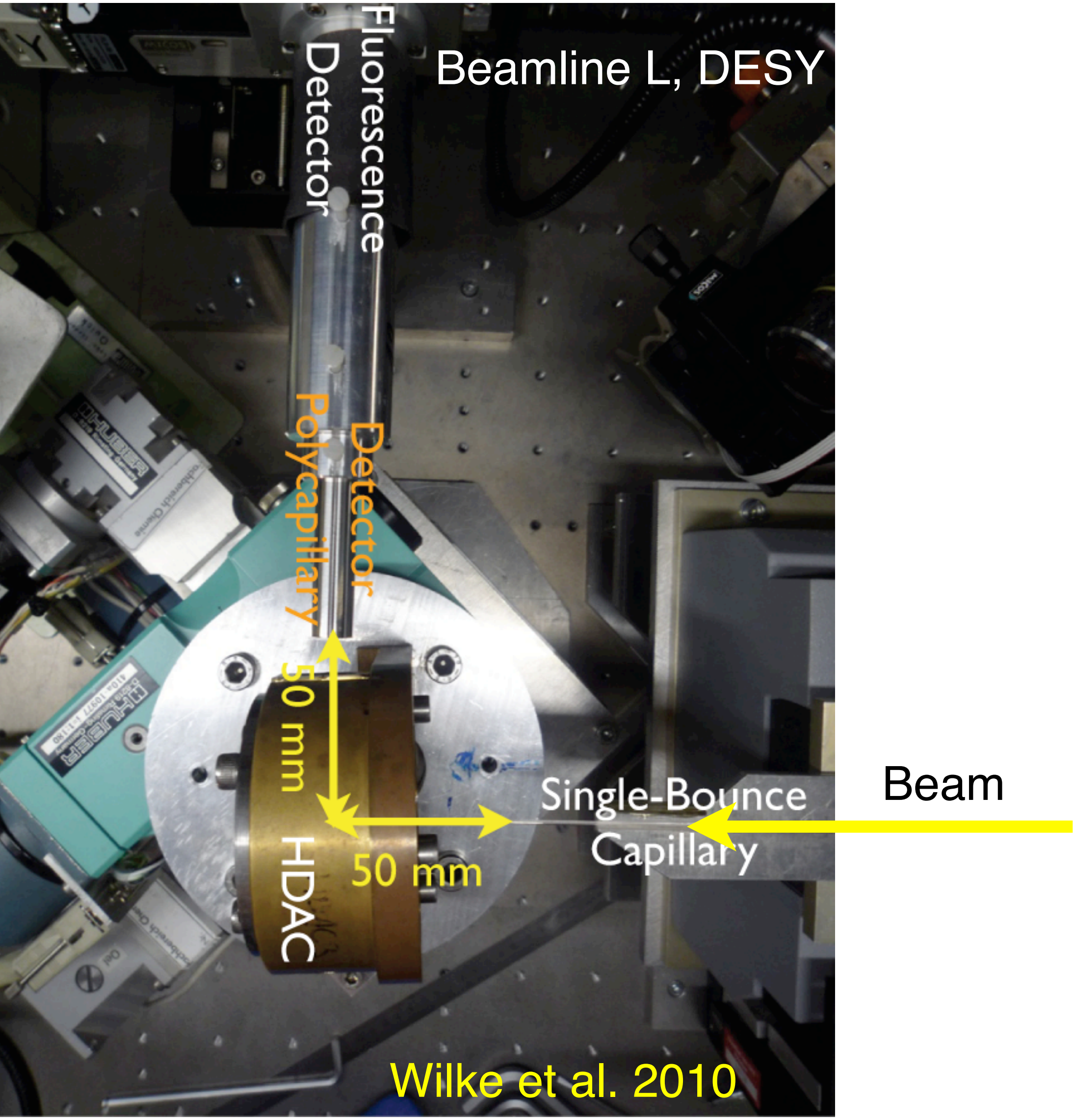
X-ray spectroscopy with HDAC



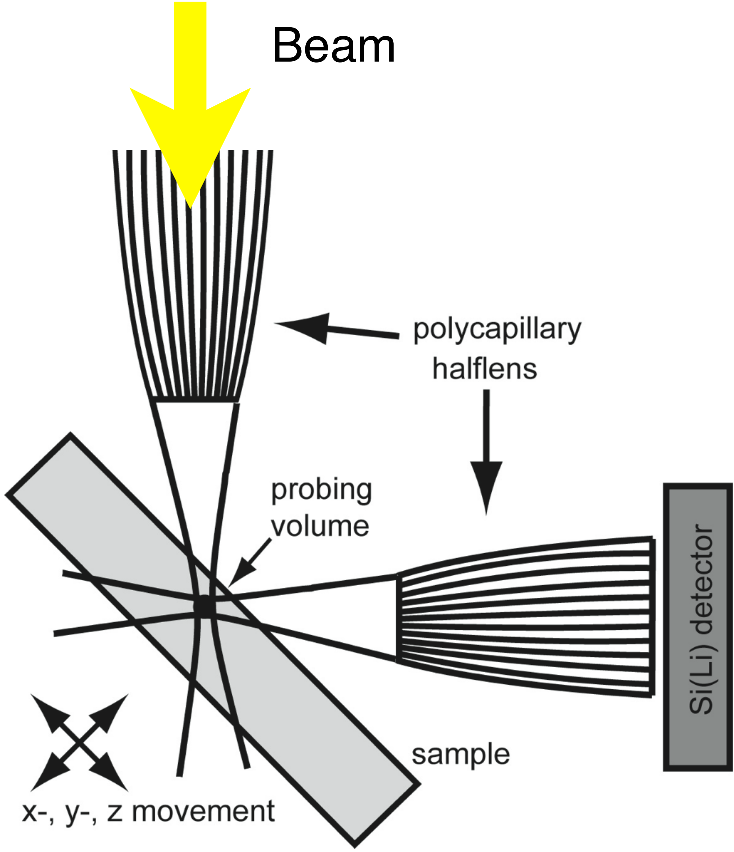
modified after Schmidt & Rickers 2003, Schmidt et al. 2007



Con-focal XRF detection



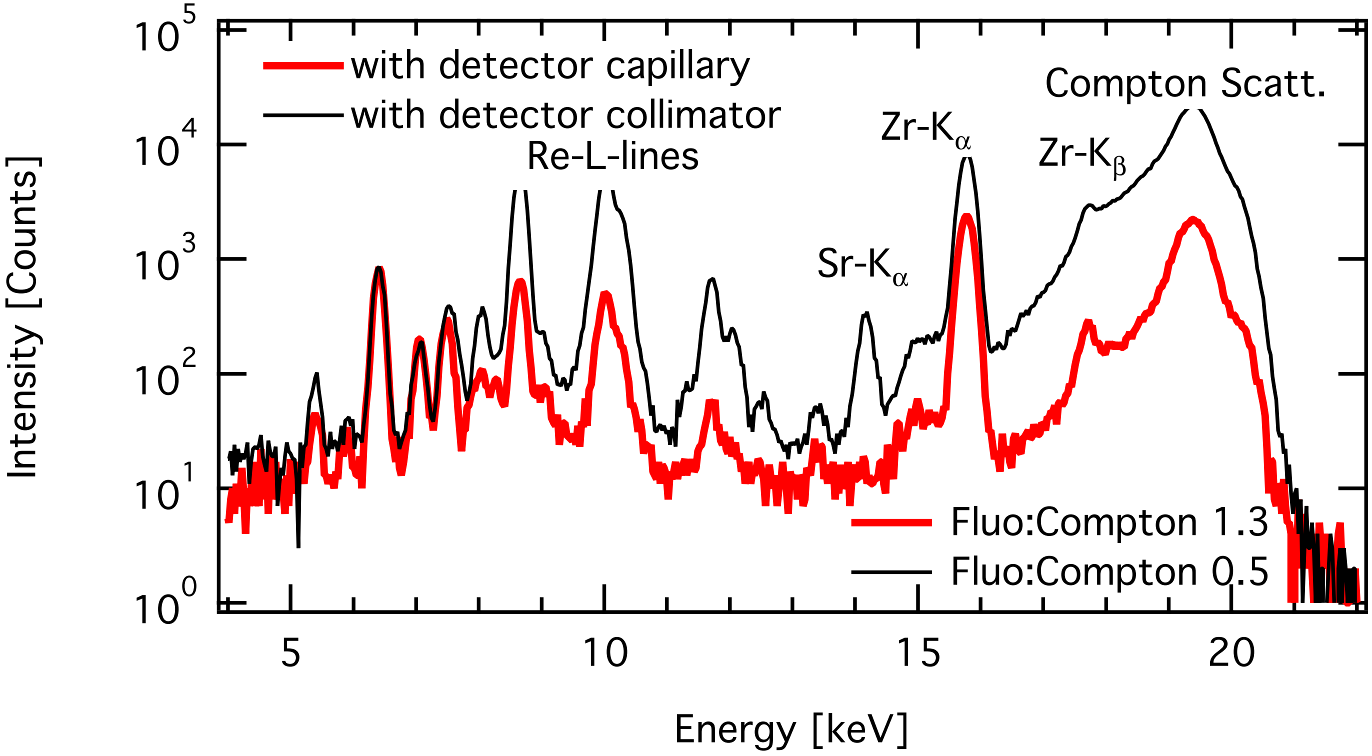
Wilke et al. 2010



Schmitz et al. 2009

Confocal XRF: Calibration

Standard Solution in HDAC with 1300 ppm Zr
21 keV excitation

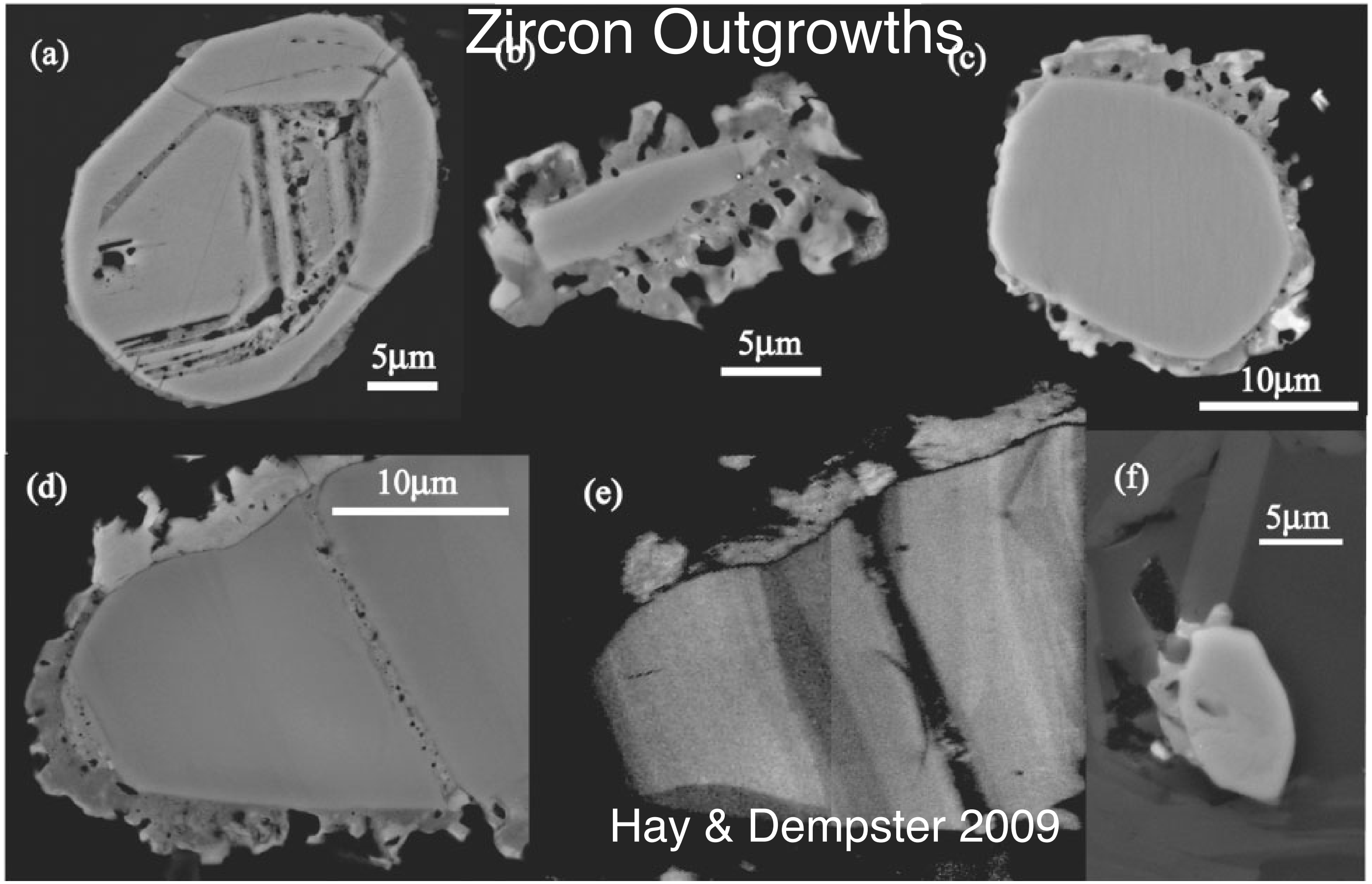


Detection limit < 1 ppm for Zr

Wilke et al. 2010

Mobilisation and Transport of Zr

- Zircon ($ZrSiO_4$) in low-grade (350°C) meta-sediments

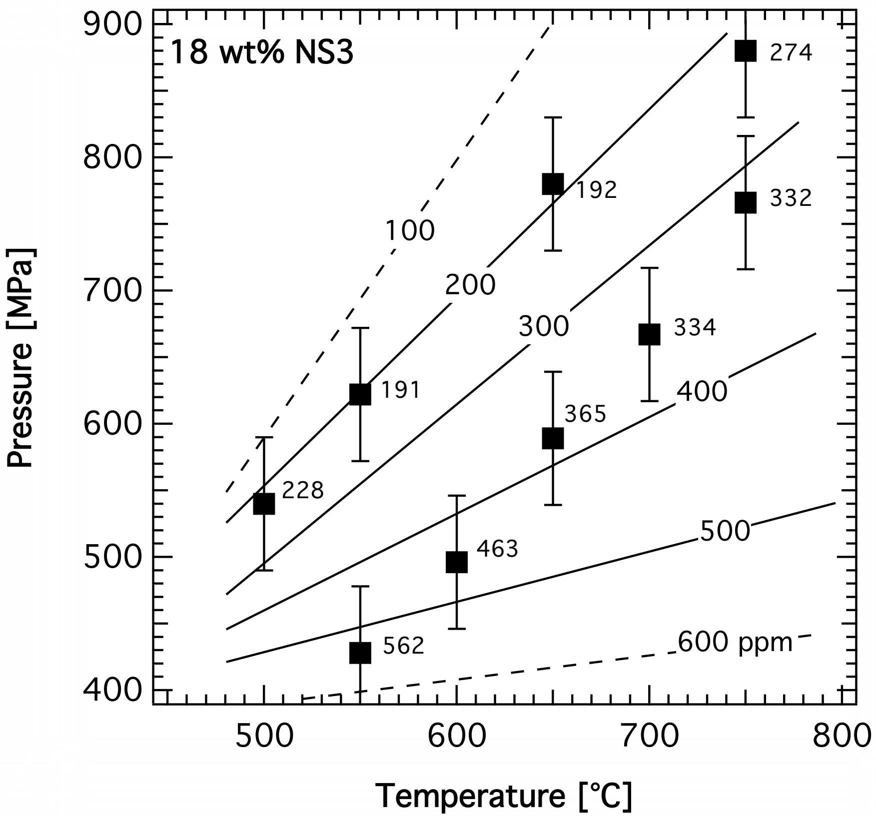


Zircon Outgrowths

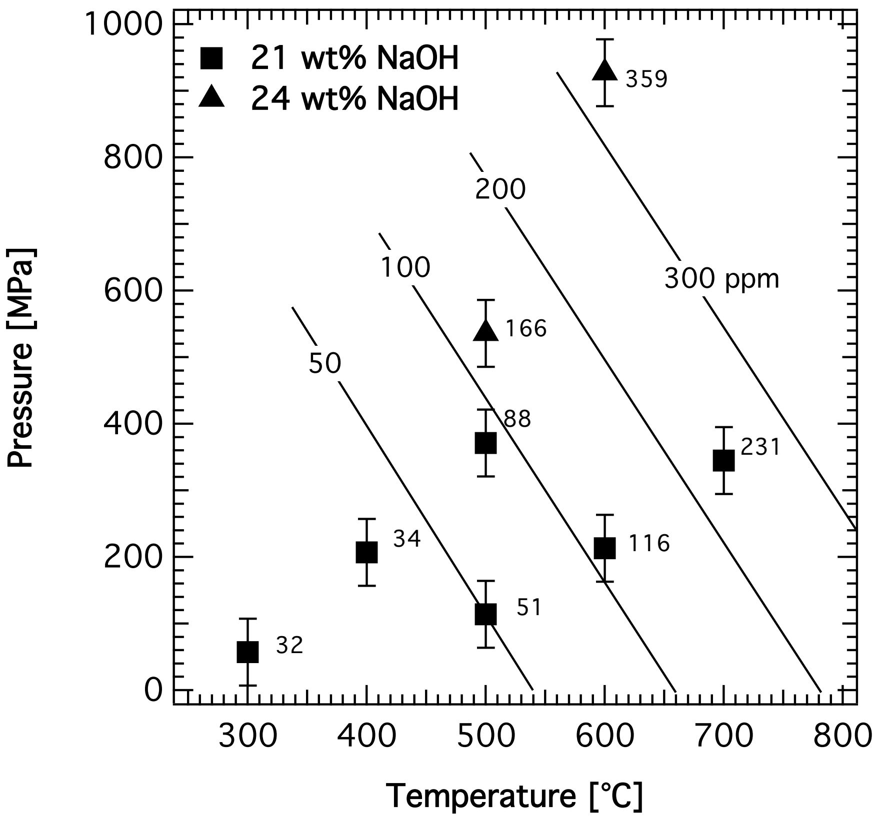
Hay & Dempster 2009

Zircon solubility in aqueous fluids

Aqueous Fluids containing $Na_2Si_3O_7$



Aqueous Fluids containing NaOH (baddelyite)

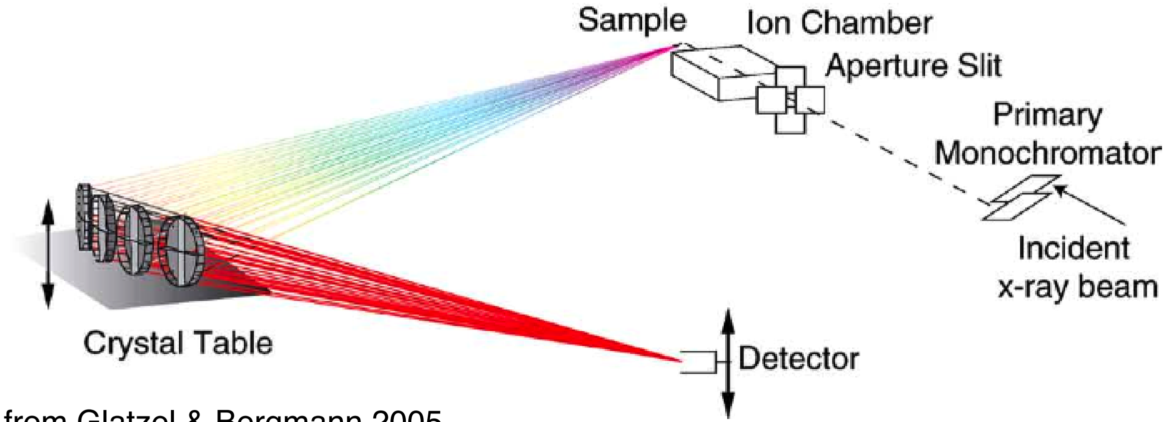


Zr-complexation in fluid?

Wilke et al. 2012

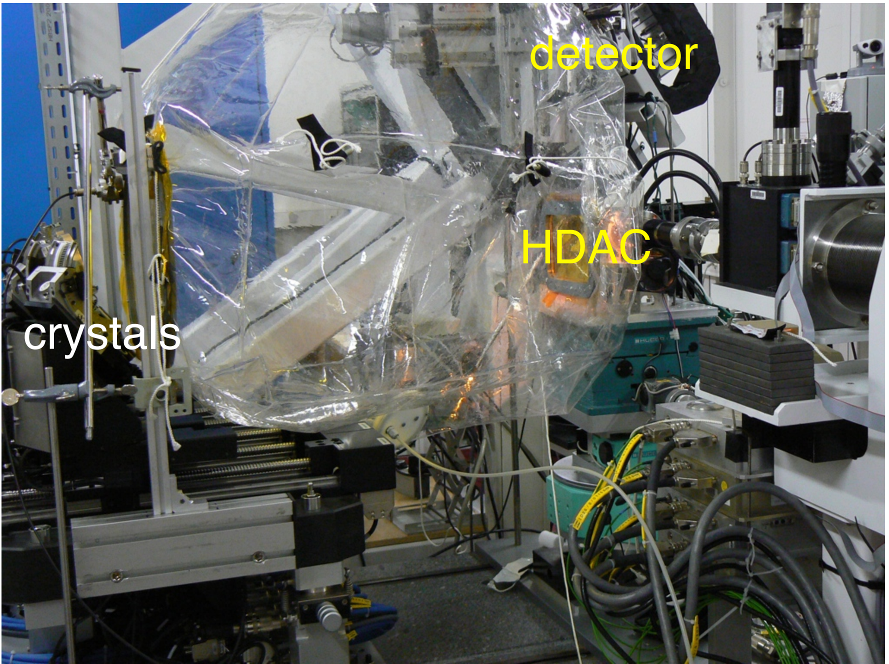
RIXS or HERFD-XAS on trace-elements in aqueous fluids at P & T

Rowland Spectrometer at ID26 @ ESRF

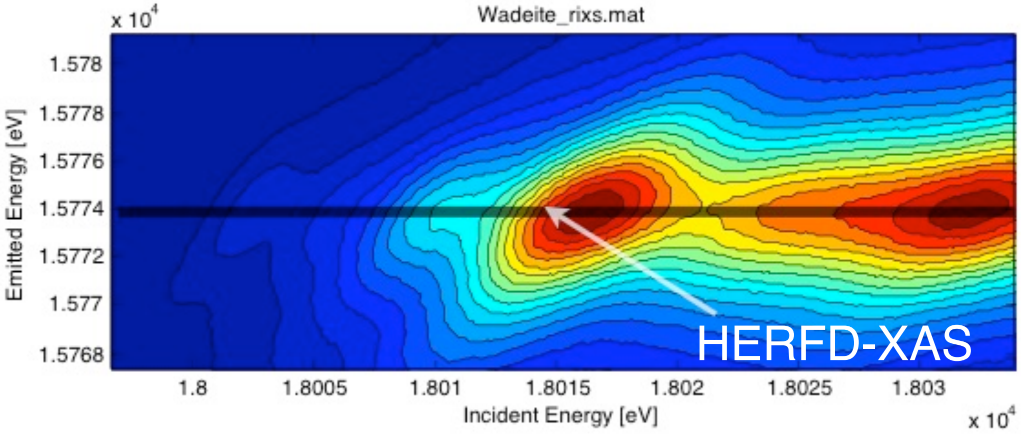


from Glatzel & Bergmann 2005

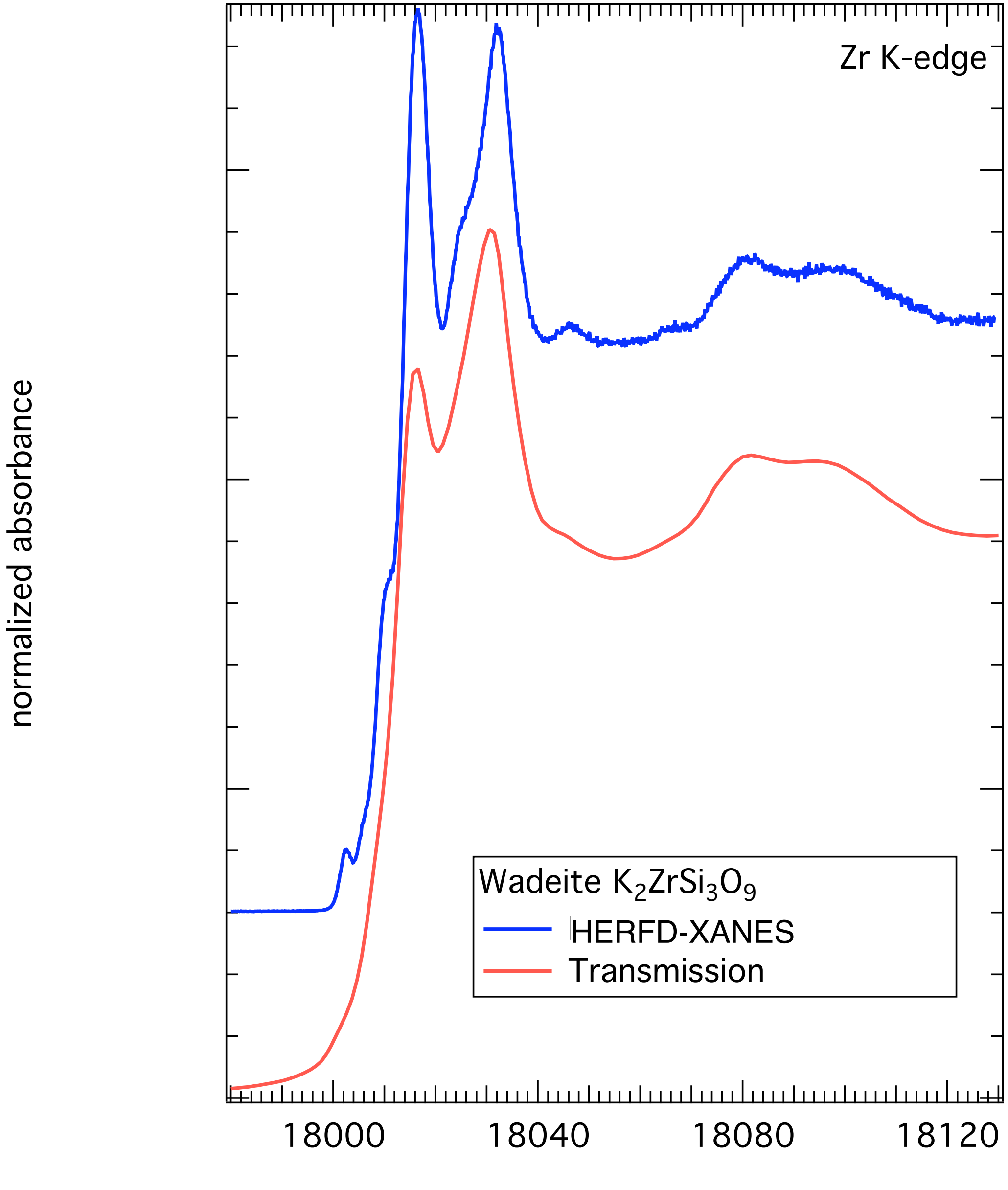
RIXS:
Resonant Inelastic X-ray Scattering
HERFD-XAS:
high energy resolved fluo detected - XAS



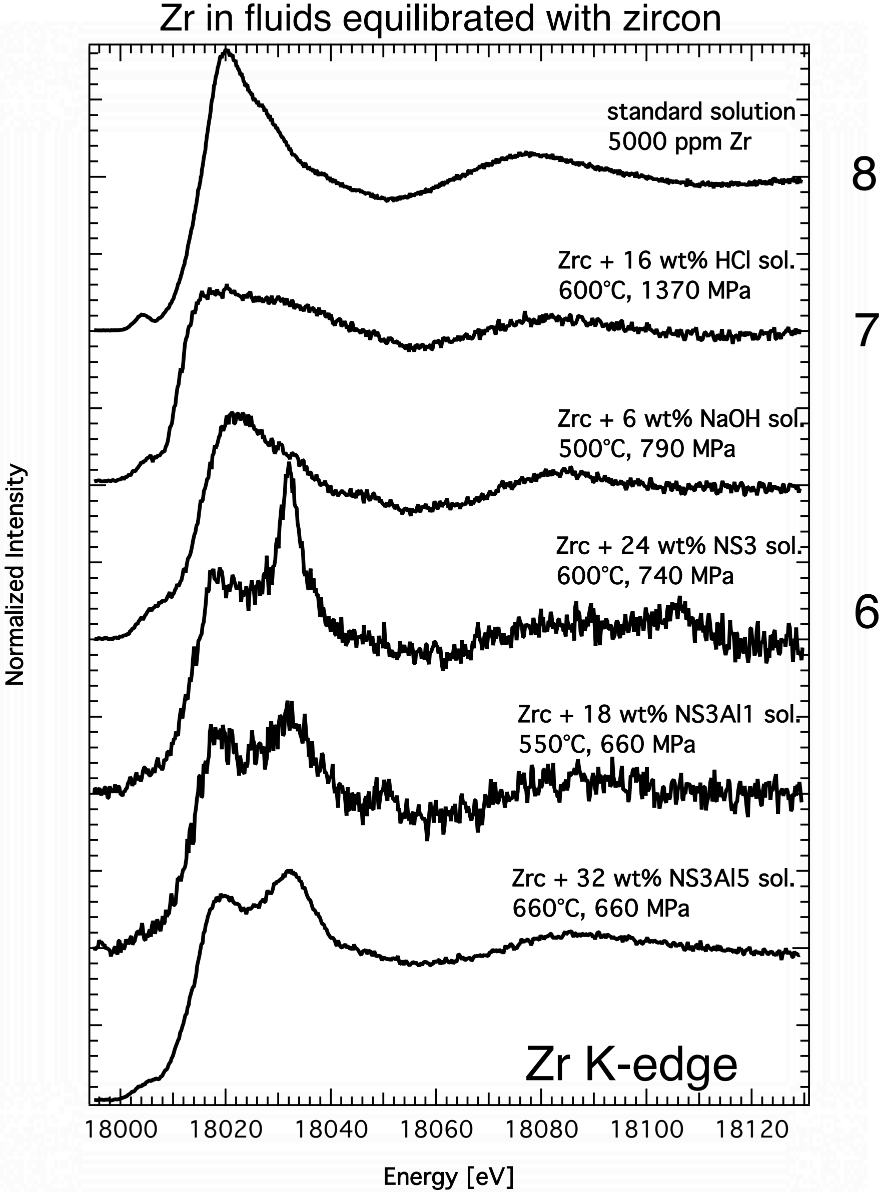
Zr 1s2p RIXS plane



HERFD-XANES vs. normal XANES



Zr Complexation from HERFD-XANES

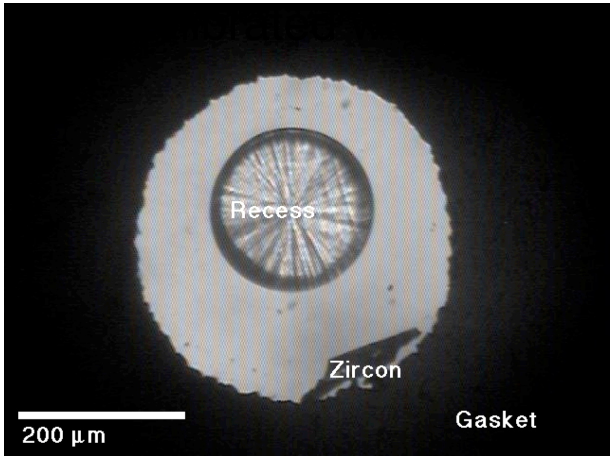


8-fold coordinated Zr

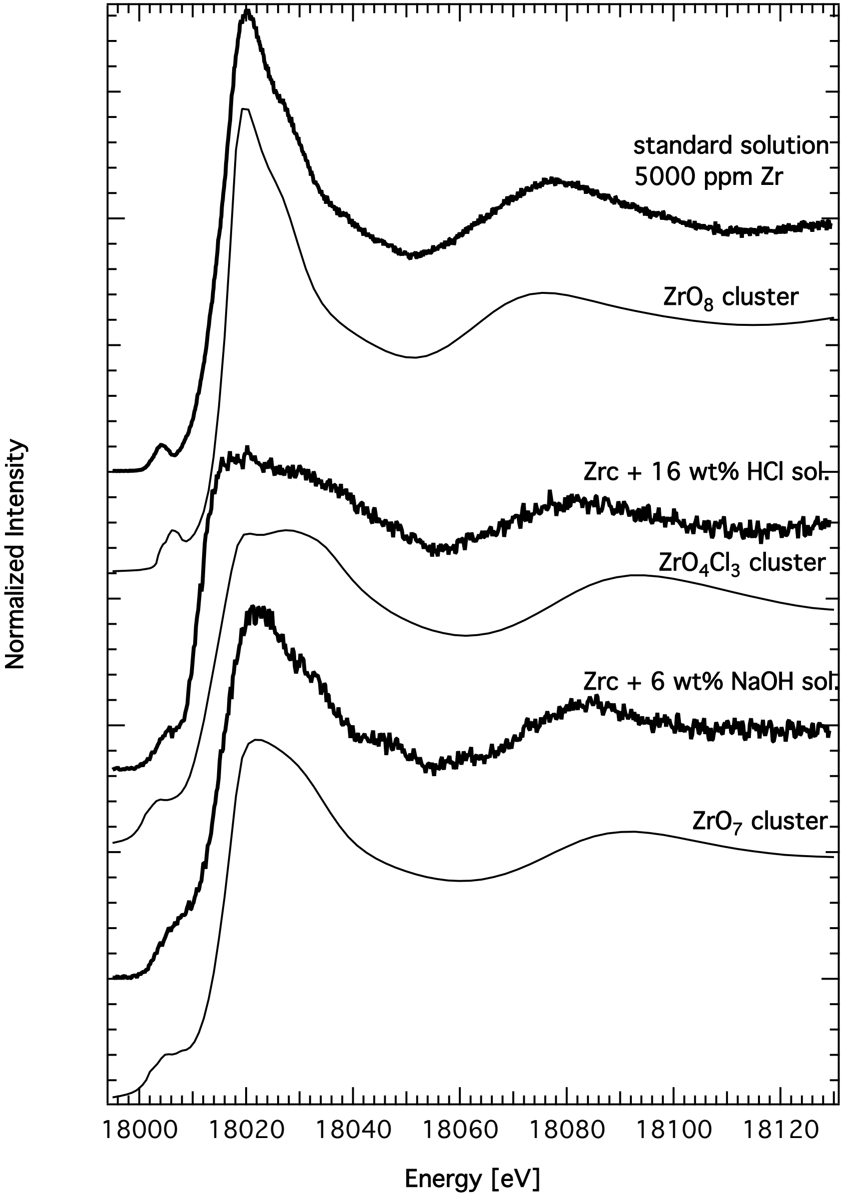
7-fold coordinated Zr

6-fold coordinated Zr

XANES taken on Fluids at P and T



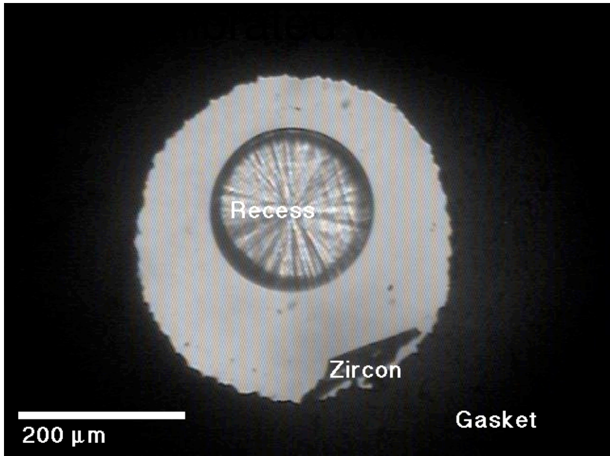
Zr Complexation from HERFD-XANES



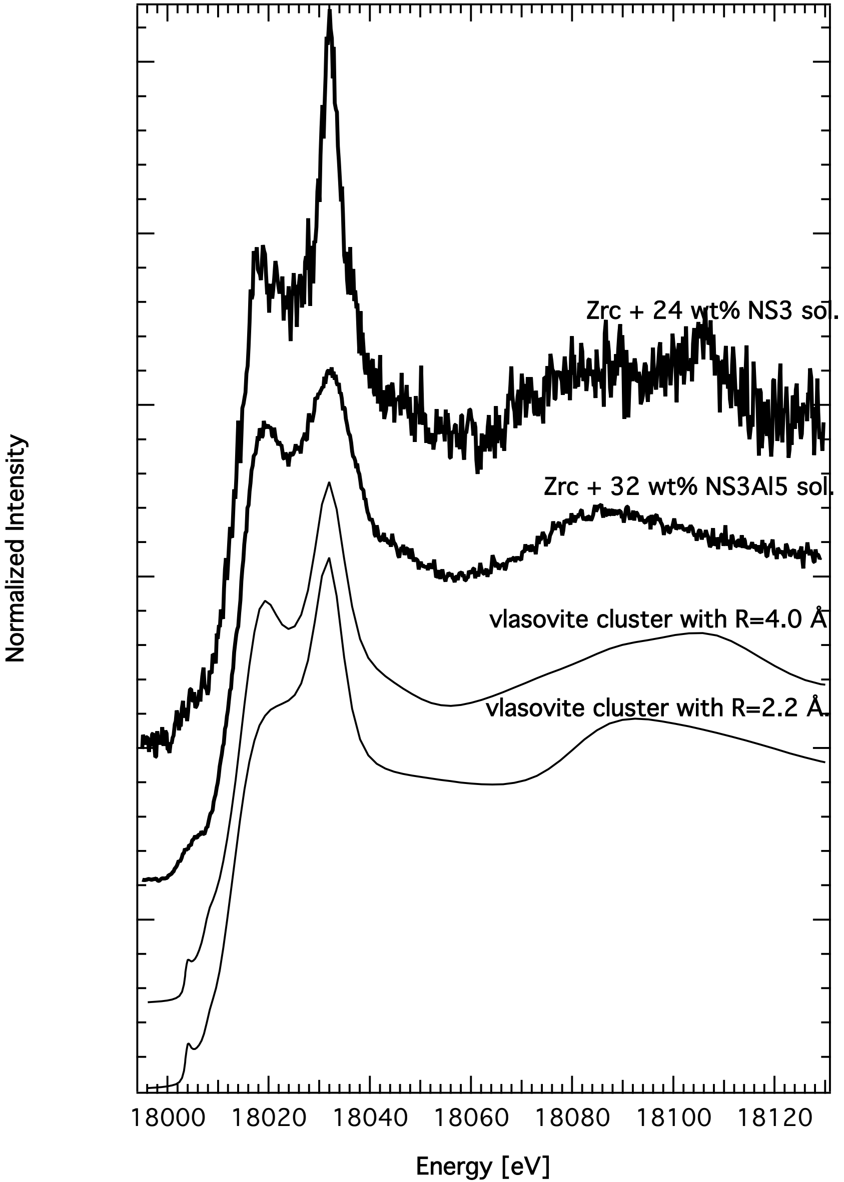
calculated with Feff

Wilke et al. 2012

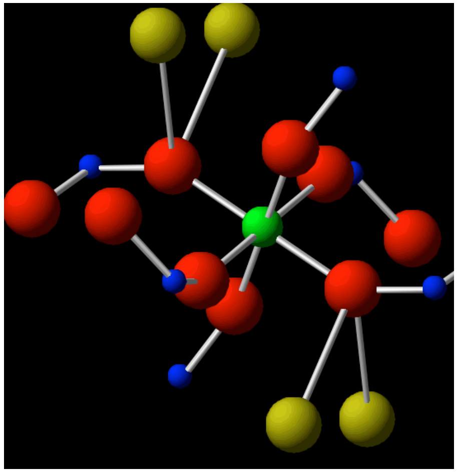
XANES taken on Fluids at P and T



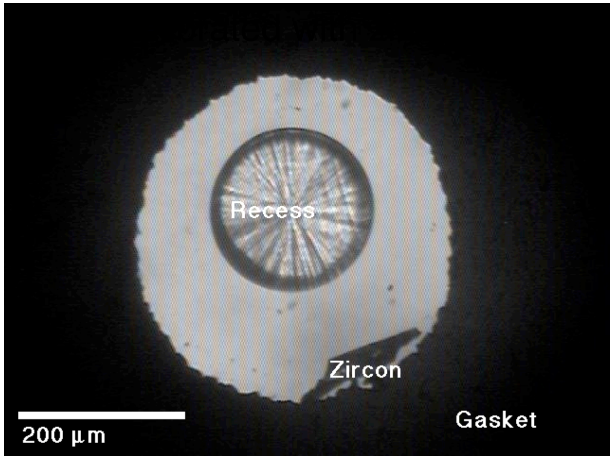
Zr Complexation from HERFD-XANES



Cluster based on Vlasovite $Na_2ZrSi_4O_{11}$



XANES taken on Fluids at P and T

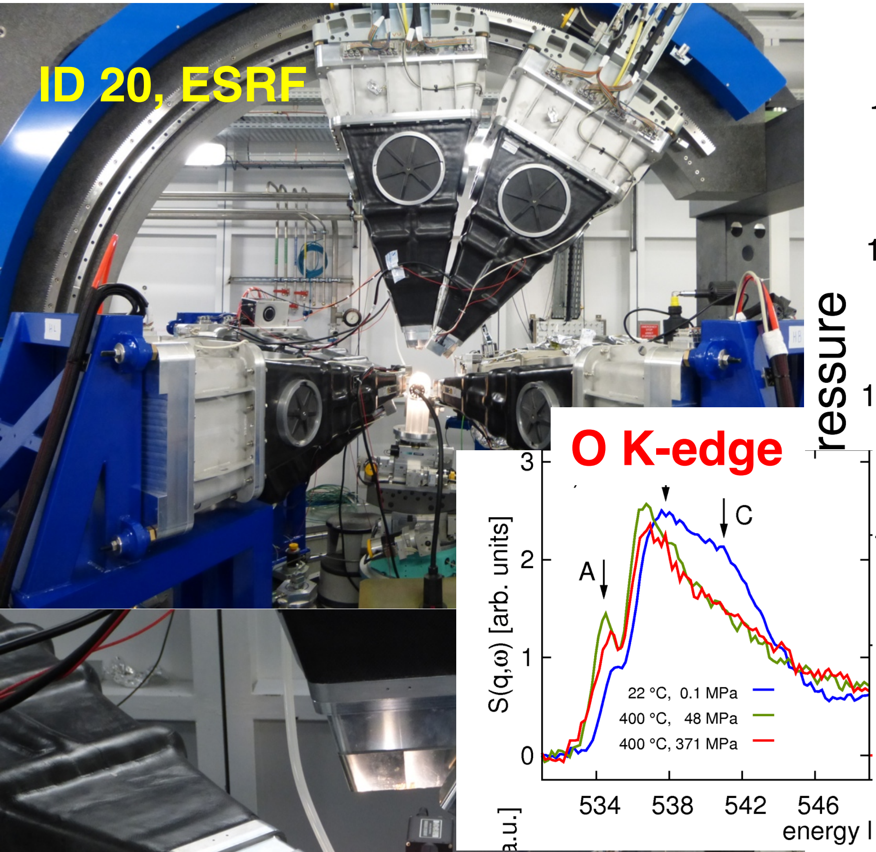


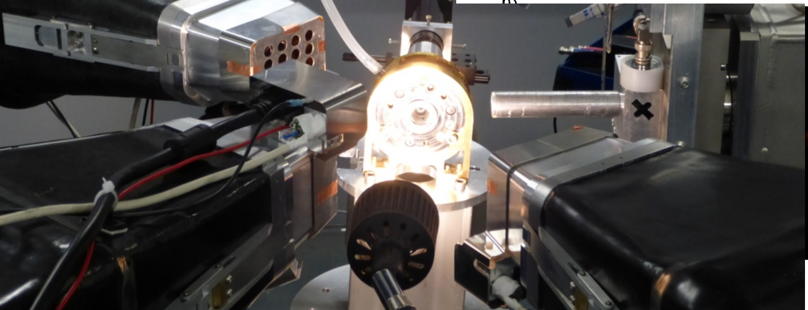
Wilke et al. 2012

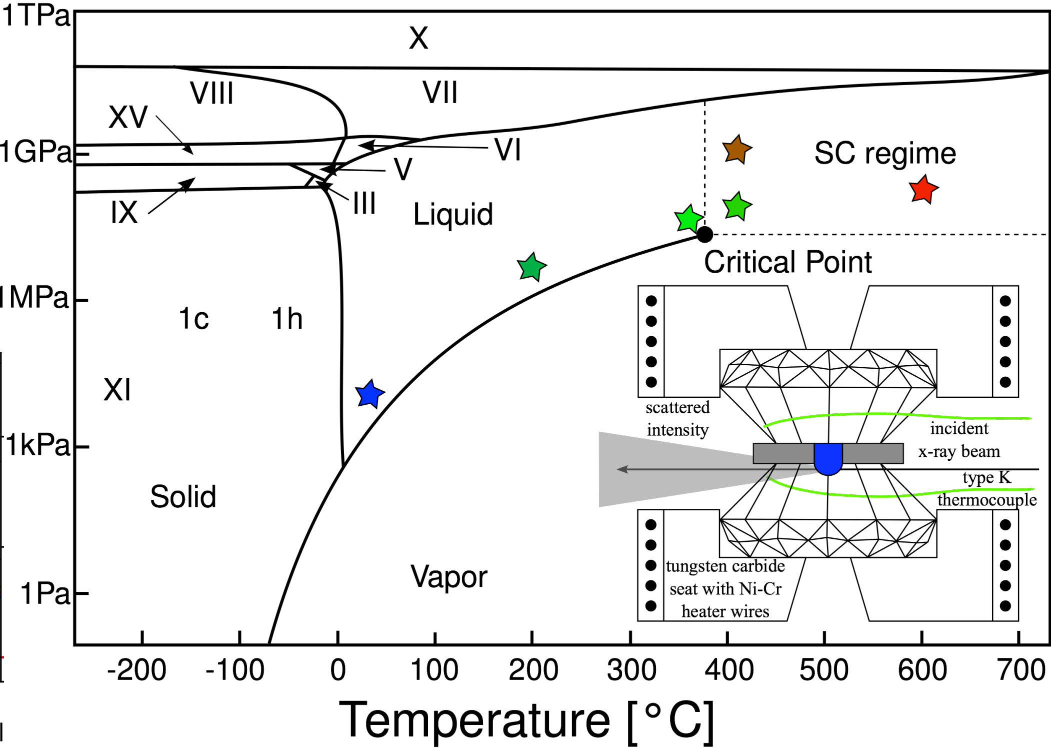
X-ray Raman Scattering with HDAC

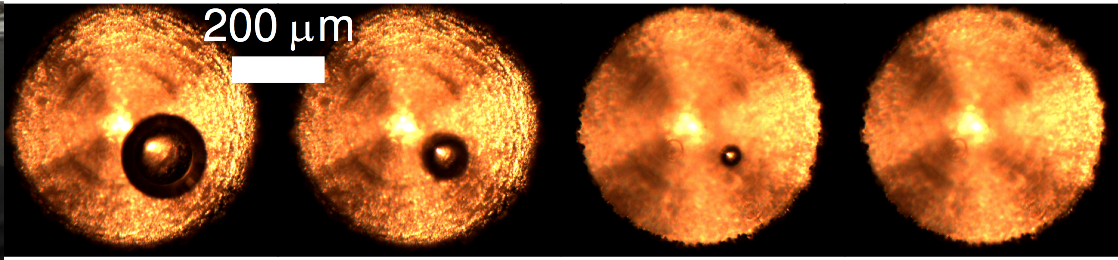
Structure of supercritical water
H-bonding ---> O K-edge

Sahle et al. (2013) PNAS, doi: 10.1073/pnas.1220301110







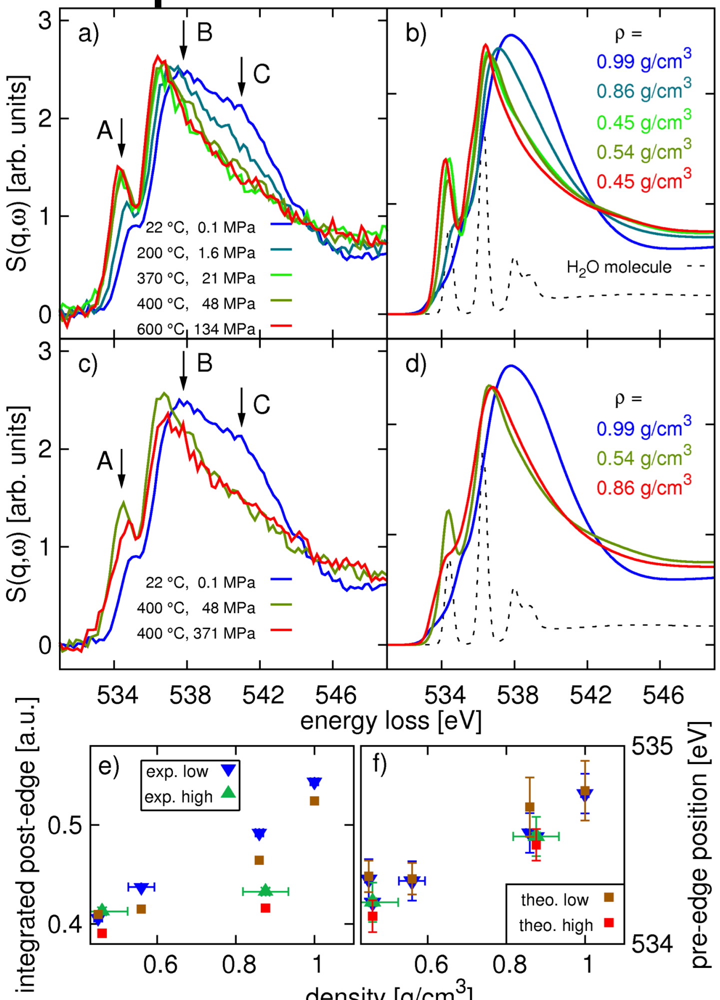


RT T=150 °C T=160°C T=163°C

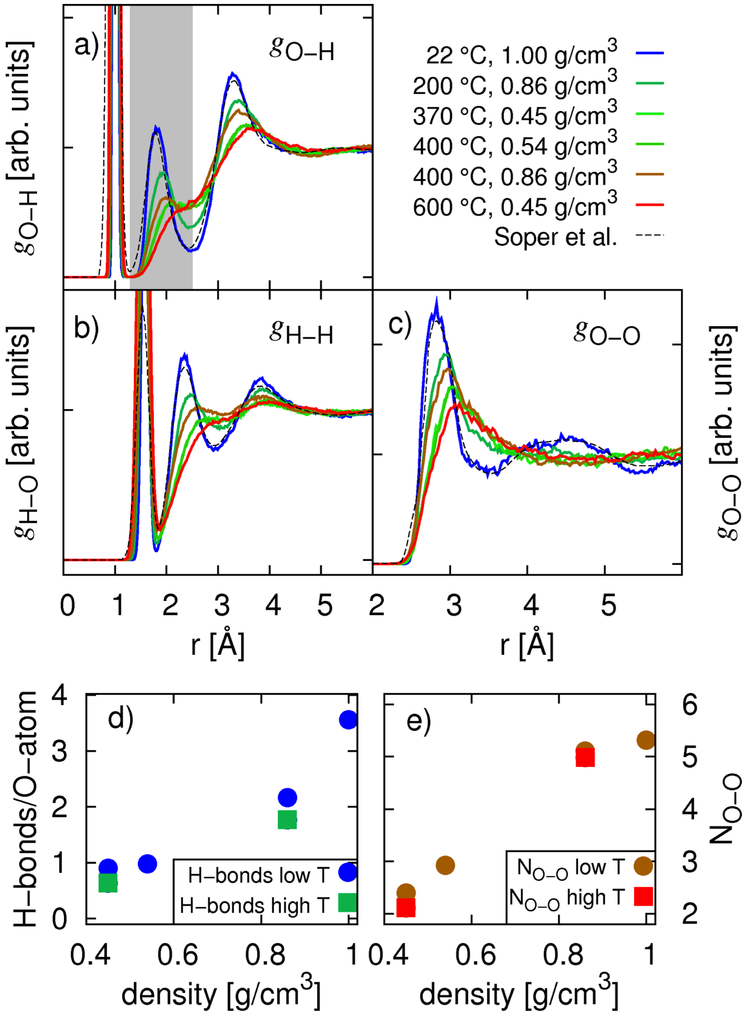
Structure of supercritical H2O H-bonding

O K-edge

experiment **simulation**



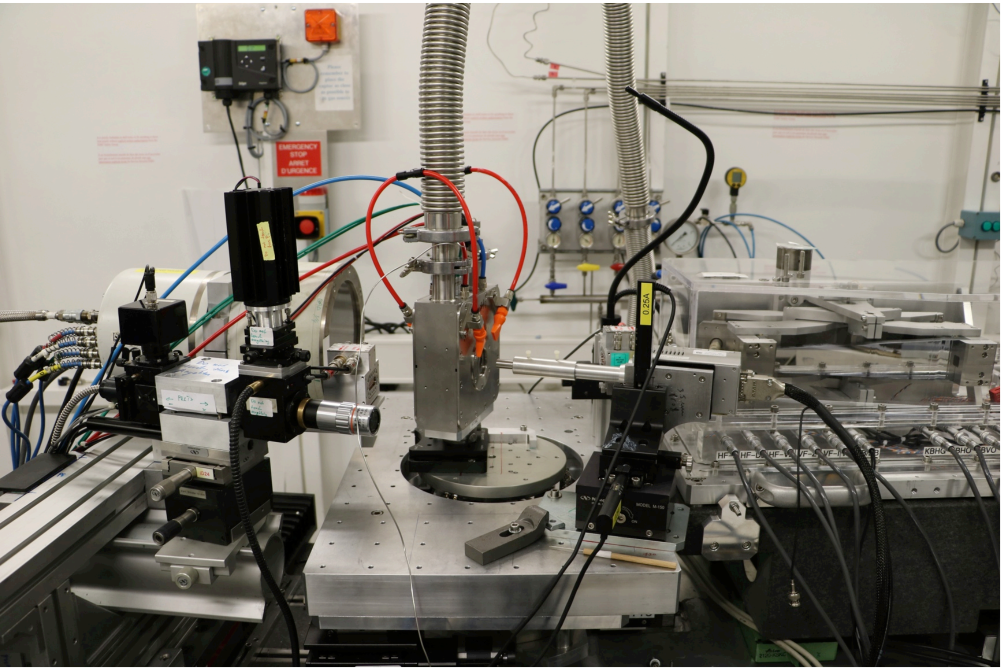
Structure from Molecular Dynamics



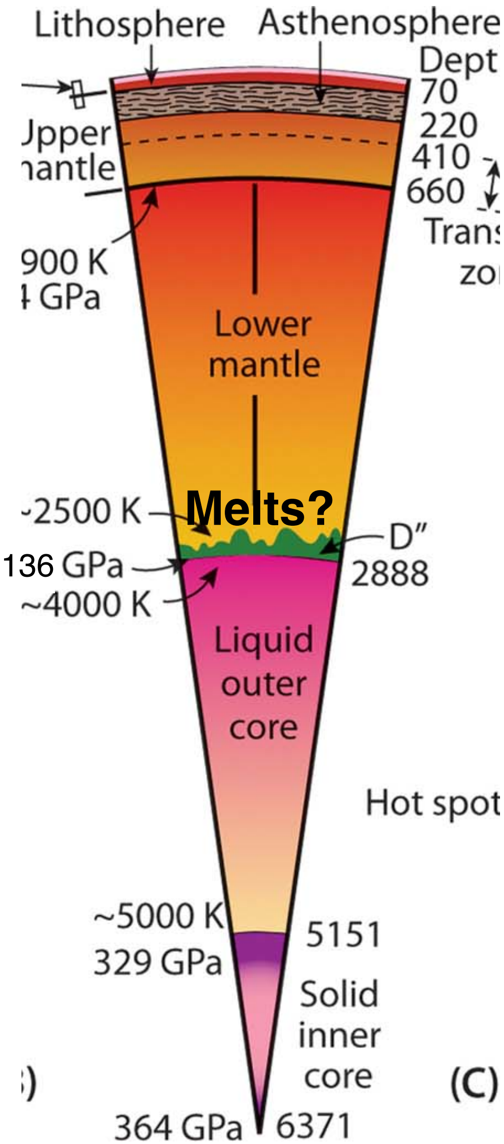
Sahle et al. (2013) PNAS, doi: 10.1073/pnas.1220301110

Silicate Glass/Melt at extreme pressures

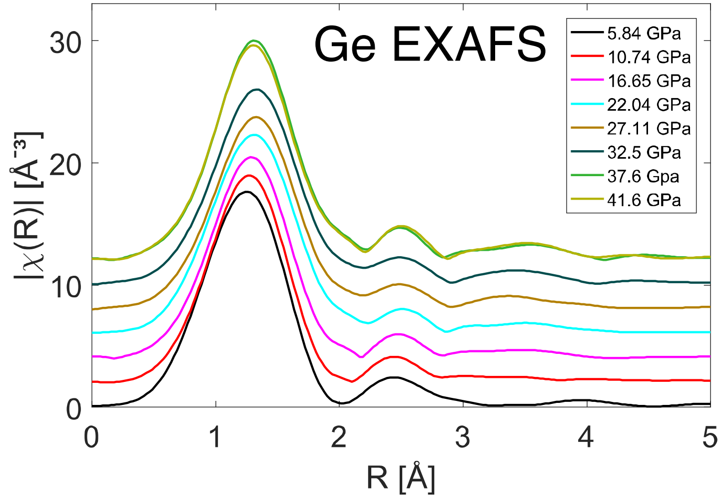
BM 23, externally heated DAC

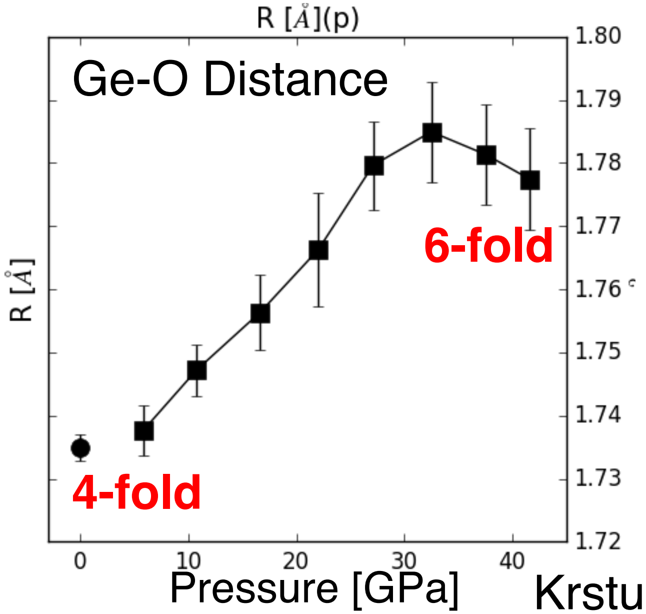


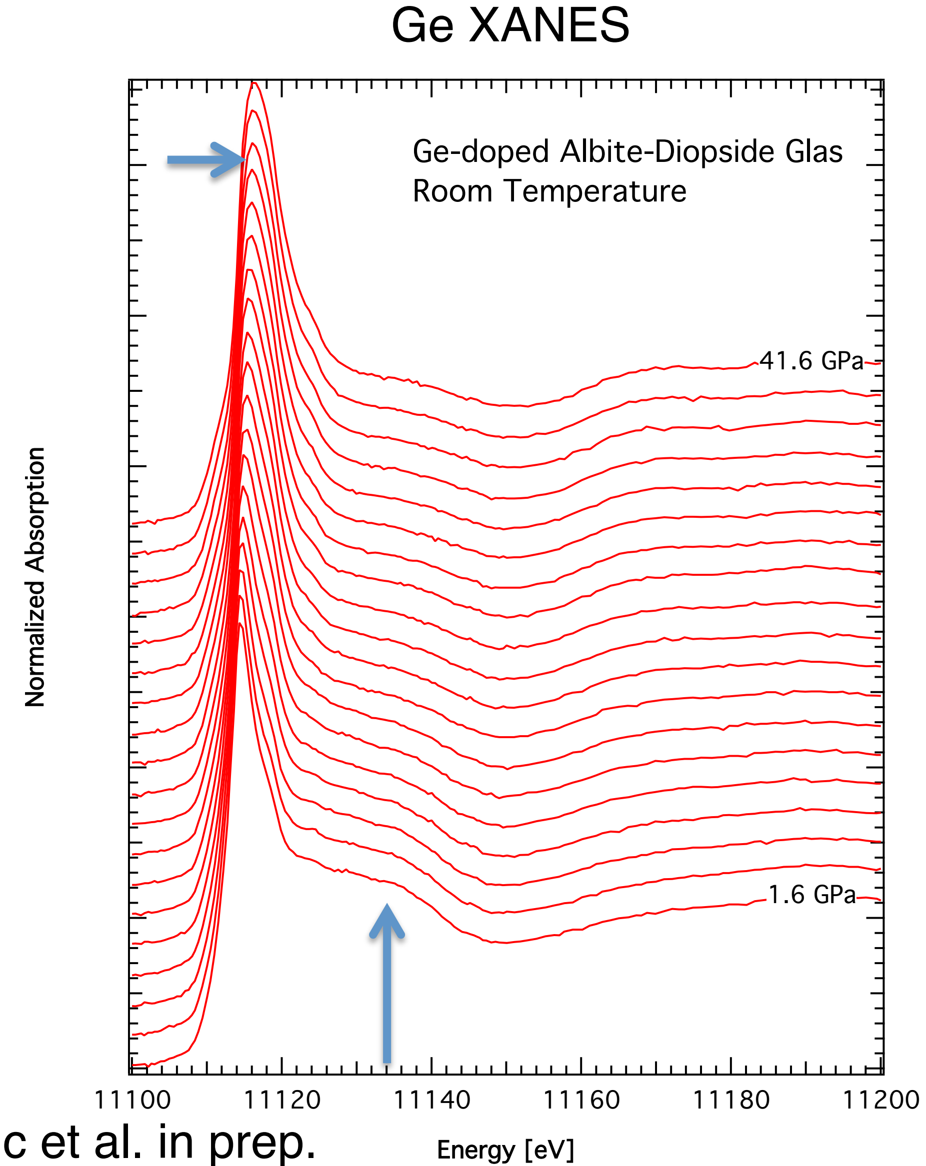
Cross Section Earth



Ge-doped Silicate Glass/Melt at extreme pressures

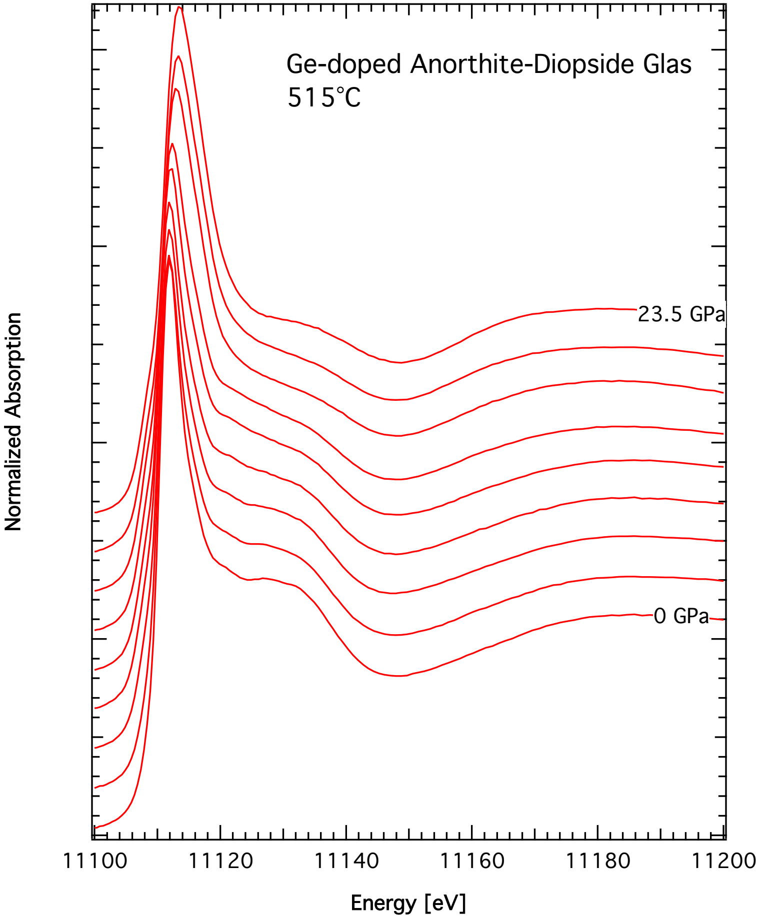


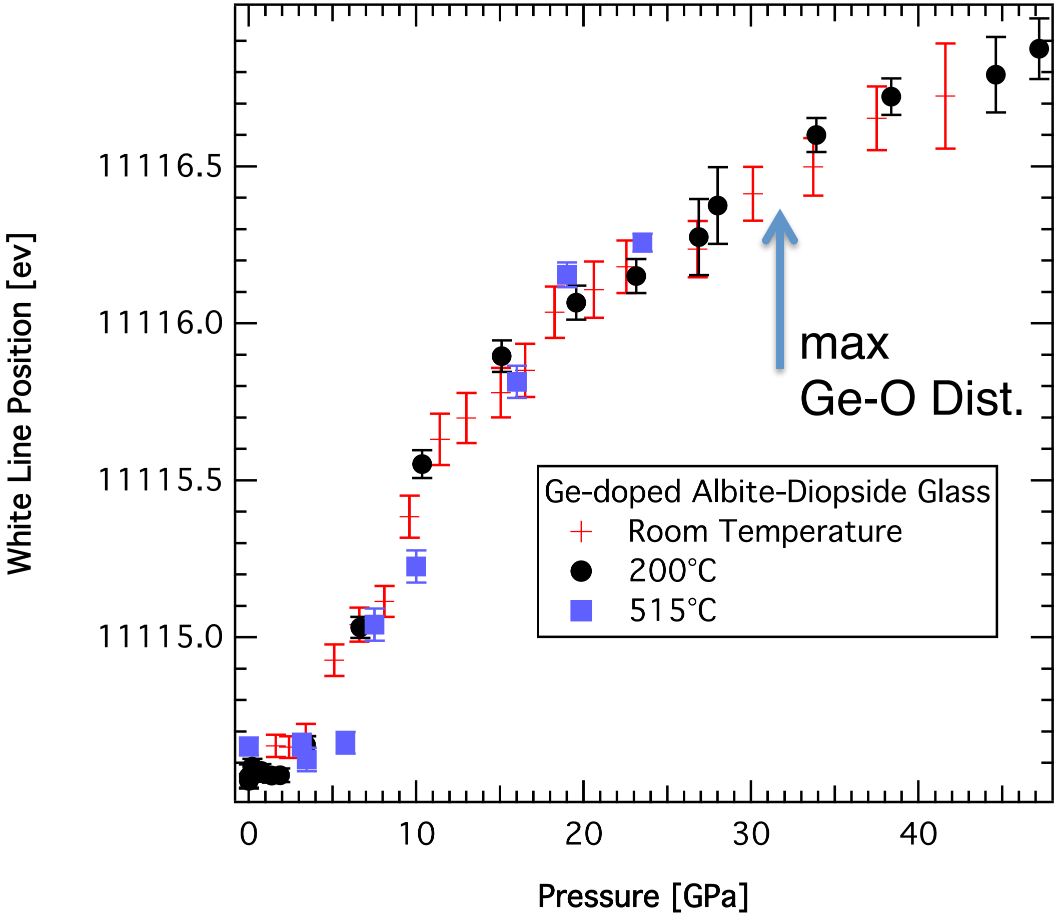




Krstulovic et al. in prep.

Ge-doped Silicate Glass/Melt at extreme pressures





Krstulovic et al. in prep.

Conclusion

- Resistively Heated DAC usable up to ca. 1500 K
- Precise Temperature Measurements
- HDAC for work with aqueous fluids
- RH-DAC for Megabars are complementary to Laser-heated DAC
- XRD, XRF, XAS, Raman spectr. etc.

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