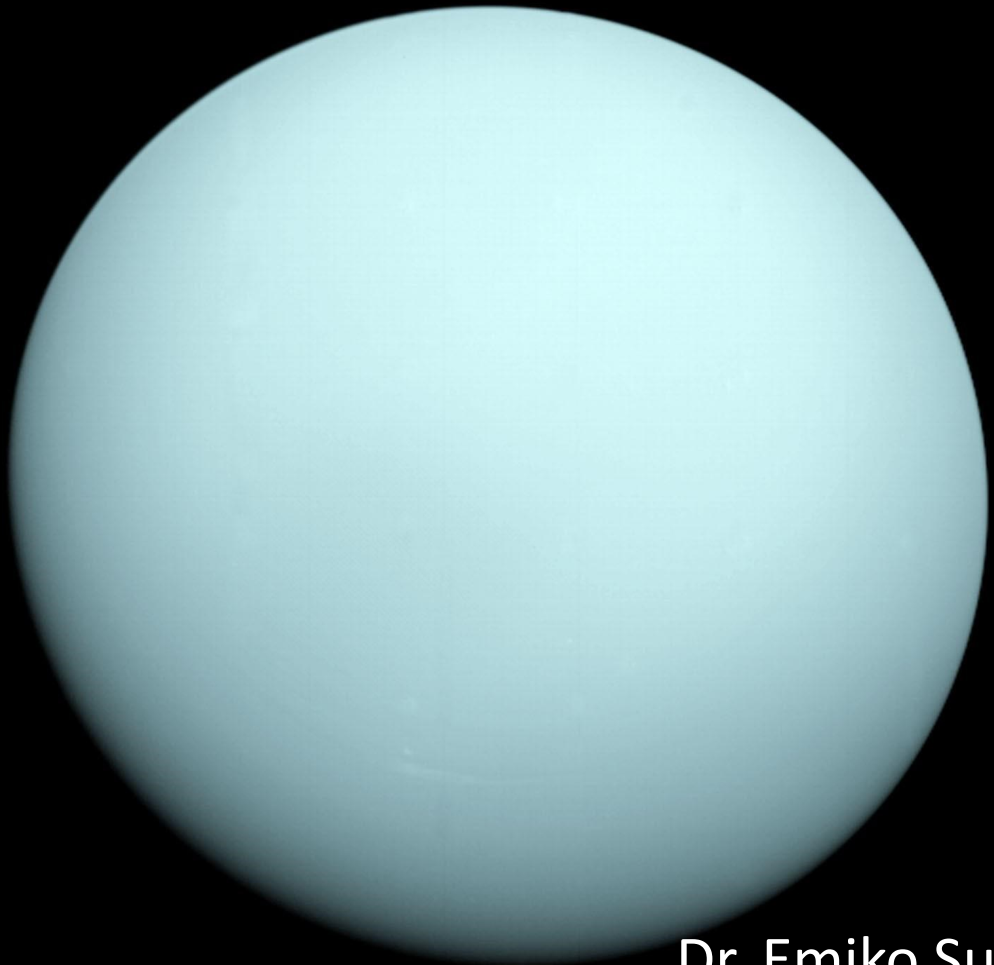
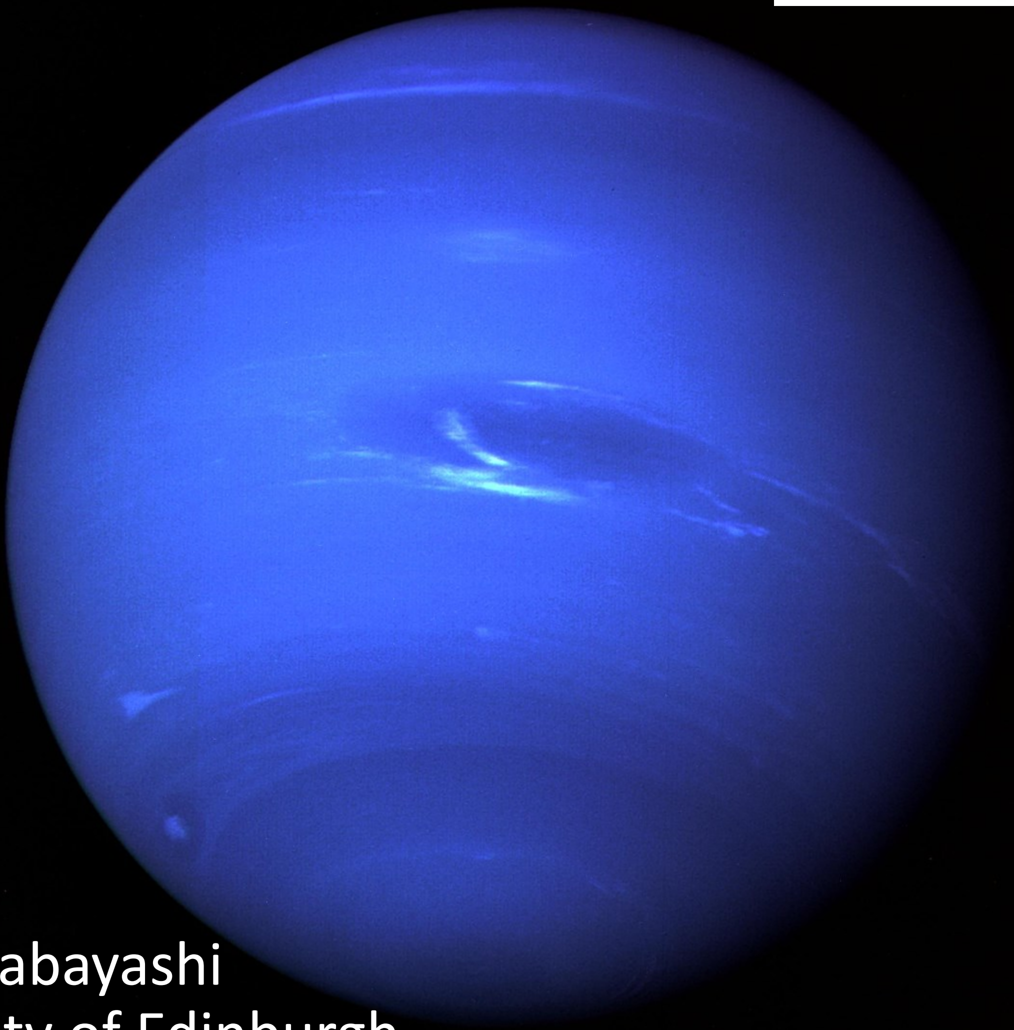
Development of the new internally-heated diamond-anvil cell for planetary mineral physics: Application to high-pressure melting of H_2O ice

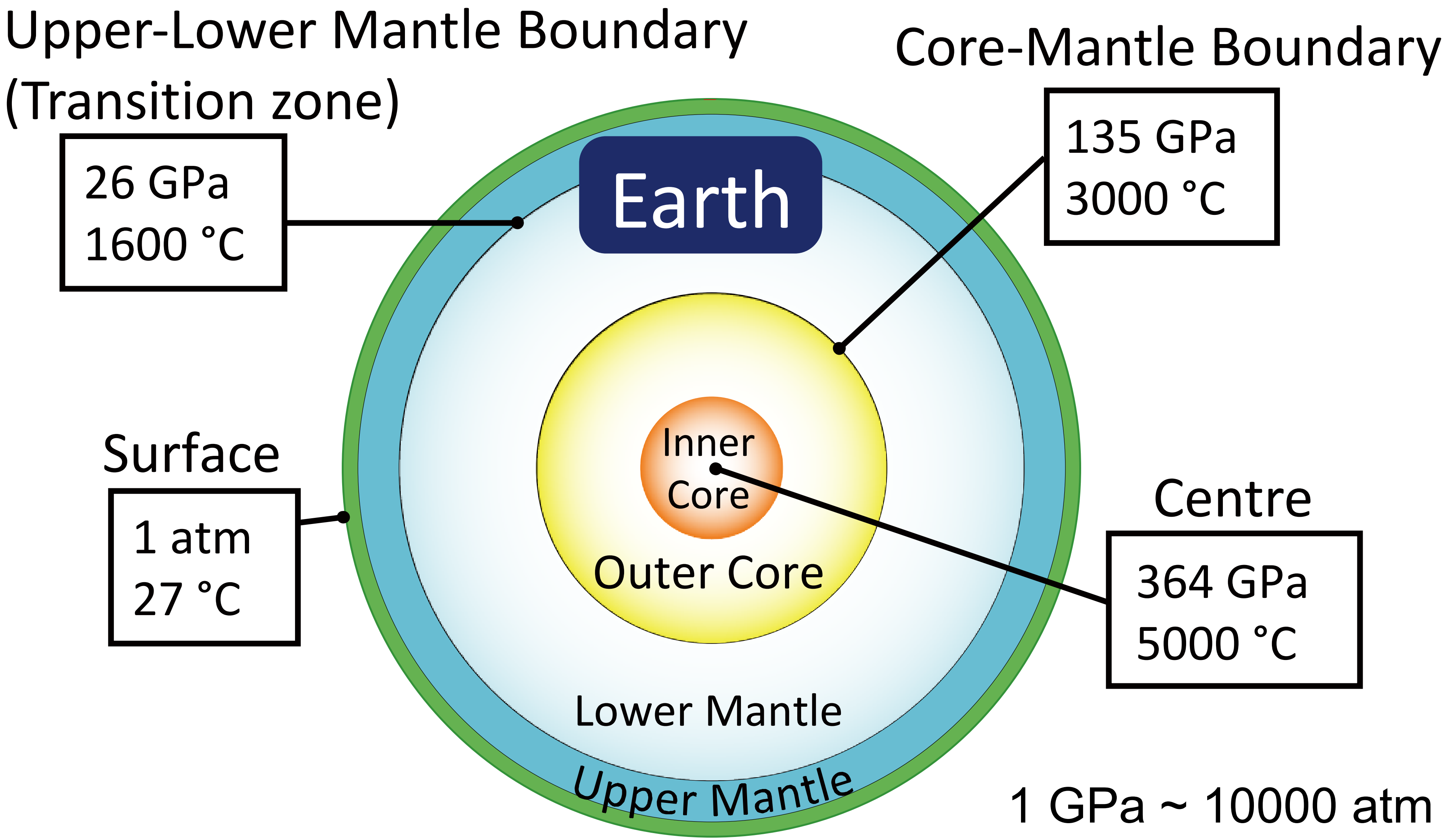




Dr. Emiko Sugimura-Komabayashi
School of GeoSciences, University of Edinburgh

Photo by NASA

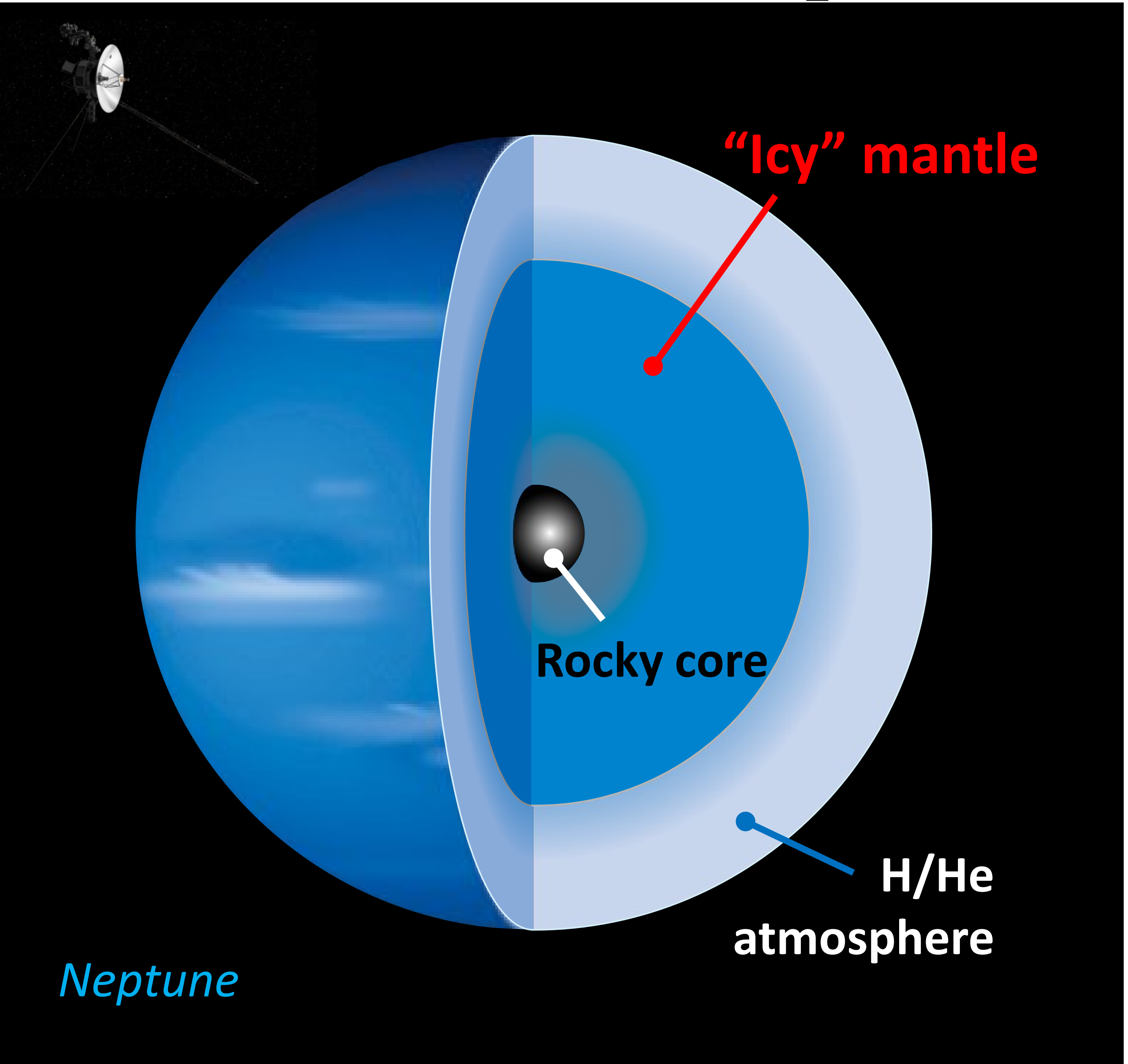
Planetary interior



1 GPa ~ 10000 atm

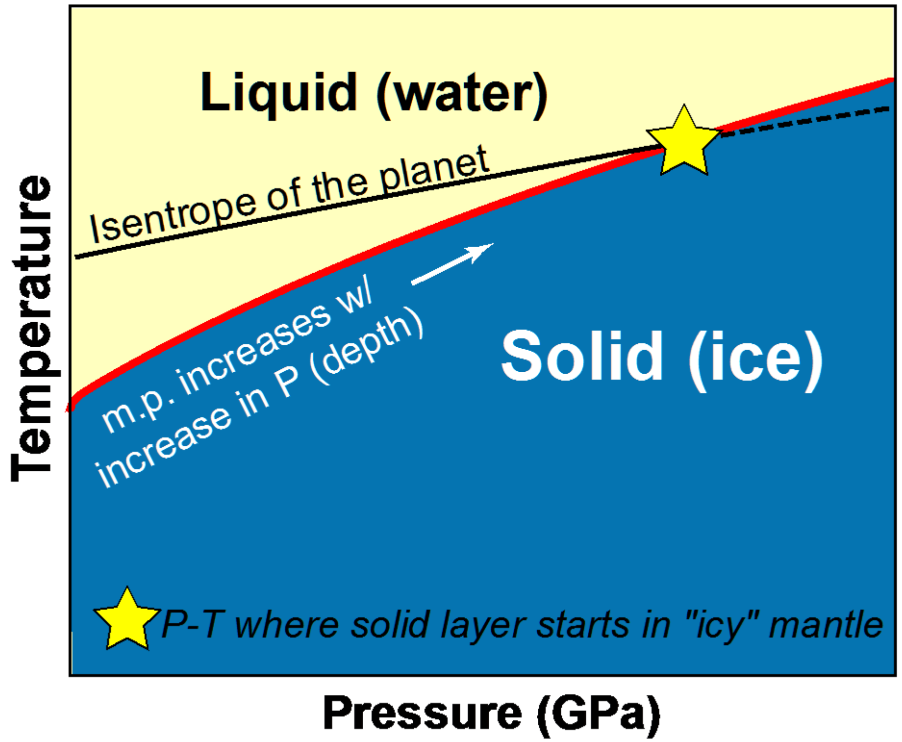
High-pressure (P) & High-temperature (T) conditions

H_2O in Ice Giants



Neptune & Uranus

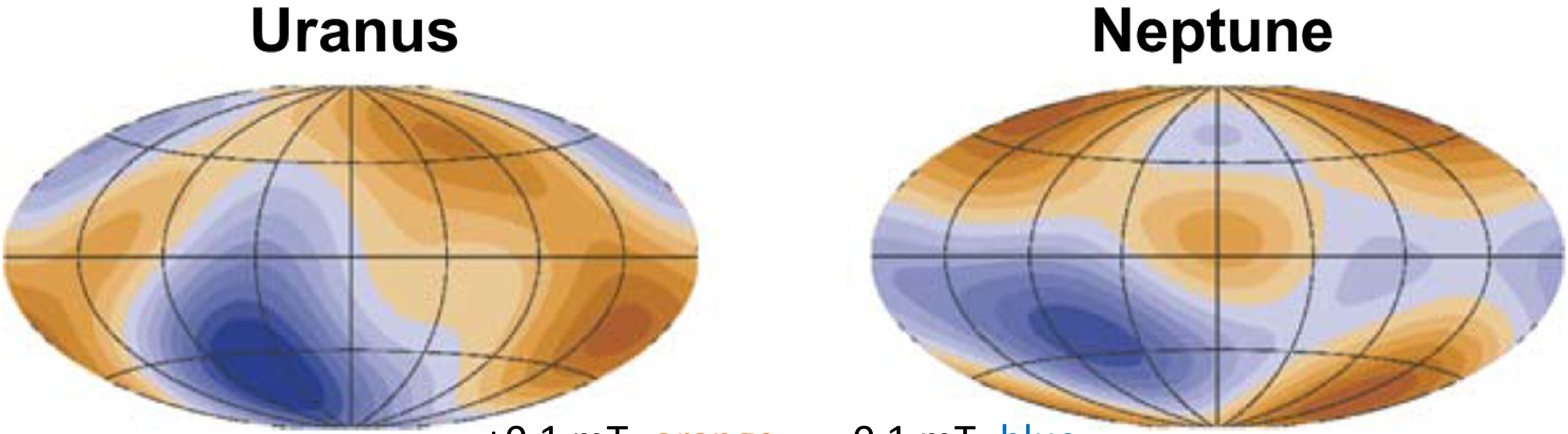
- ~50 wt%H_2O + CH_3, NH_3 etc
- "Icy" mantle: P ~ 20-600 GPa
  T ~ 2000-7000 K
  (e.g. Hubbard, 1990)



H_2O ice in Ice Giants?

Magnetic fields of Neptune & Uranus

Surface radial magnetic fields

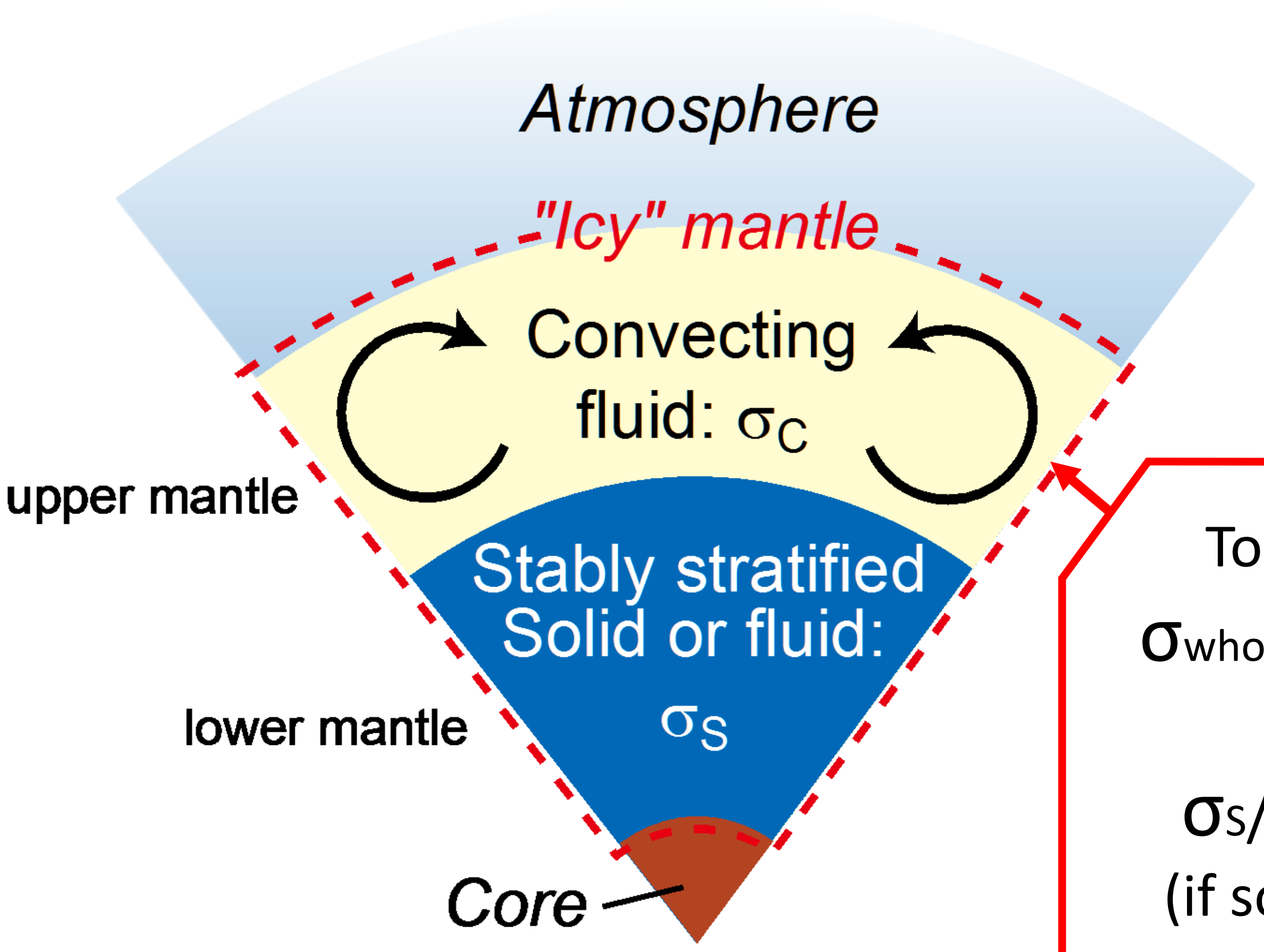


+0.1 mT orange ~ -0.1 mT blue

Stanley & Bloxham, 2004, Nature

① Non-axisymmetric
② Non-dipolar

Possible dynamo model generated by "Icy" mantle



Modified after Stanley and Bloxham, 2004

To sustain dynamo

$\sigma_{\text{whole mantle}}$ > ~10 S/cm

and

σ_S / σ_C = 0.1 – 0.001

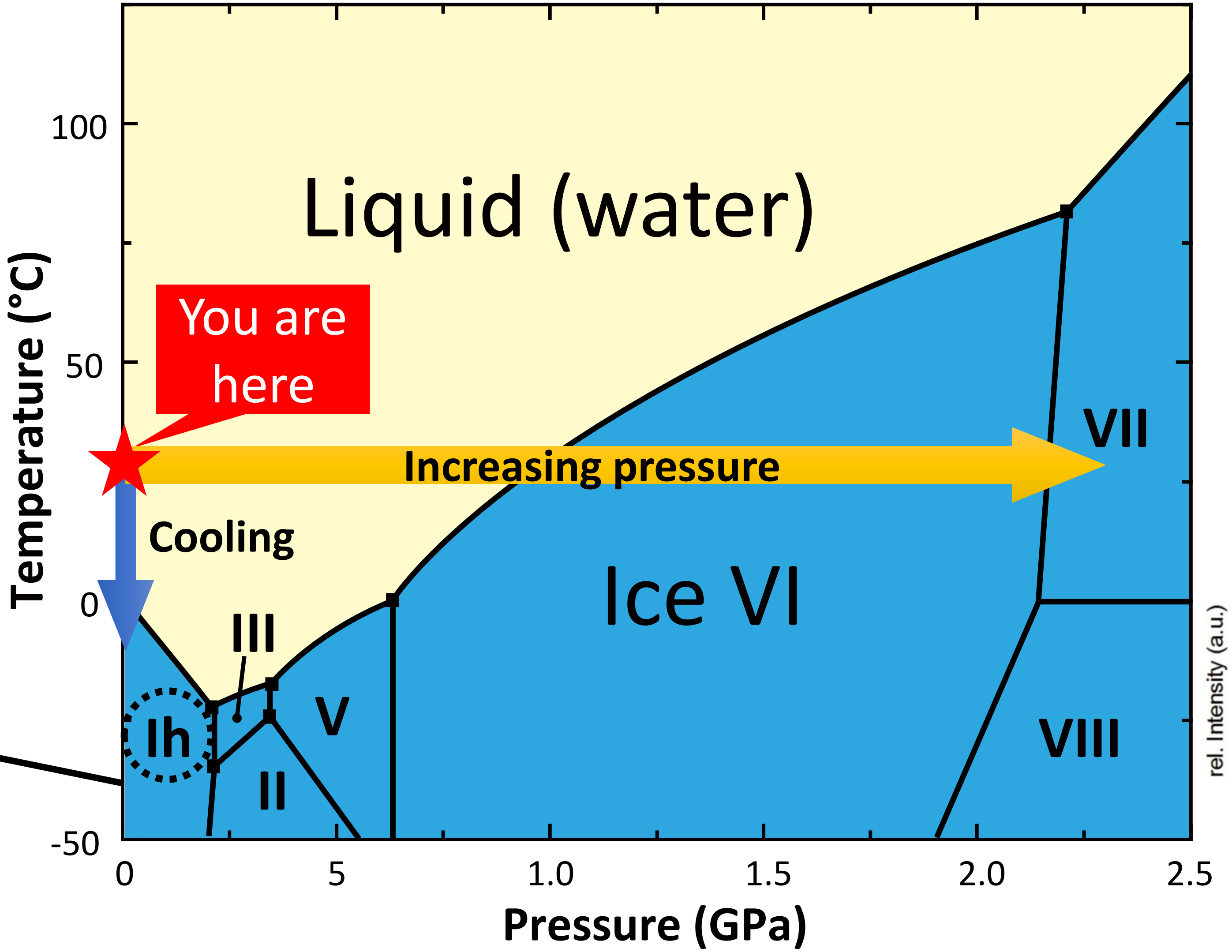
(if solid lower mantle)

Solid H_2O (ice) should have high ionic conductivity

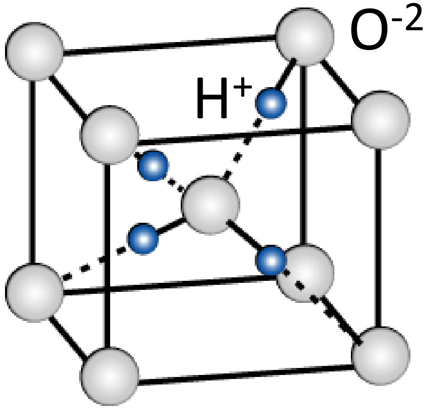
Phase diagram of H_2O

Ordinary ice
II
Hexagonal Ice (Ih)

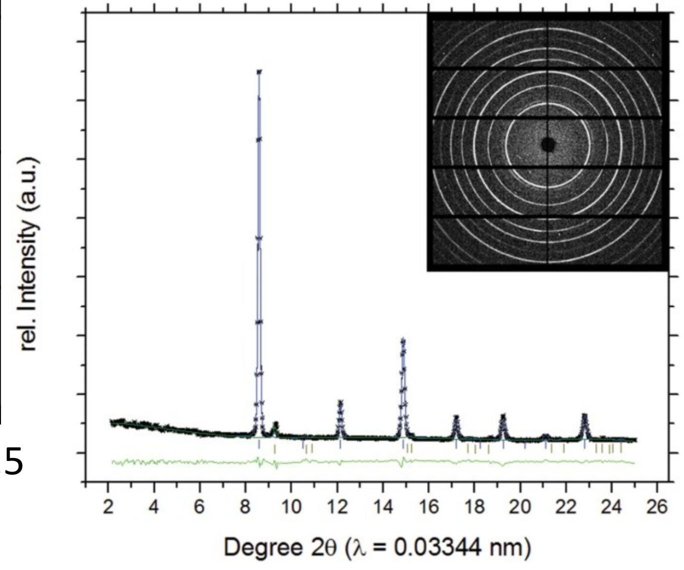
https://physicsgeekblog.wordpress.com/2016/02/13/why-ice-cubes-dont-sink/



Ice VII

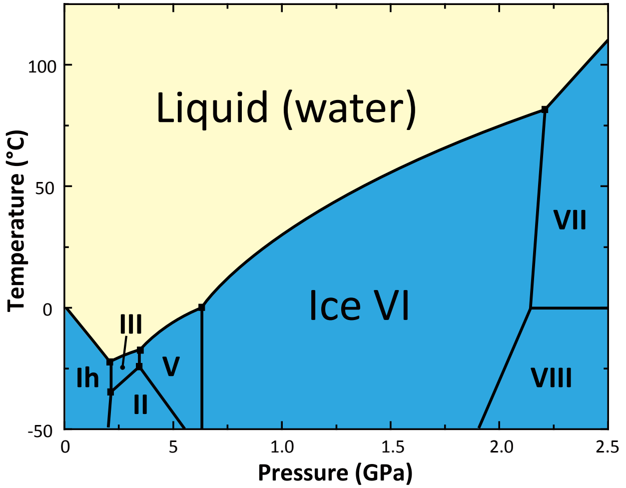


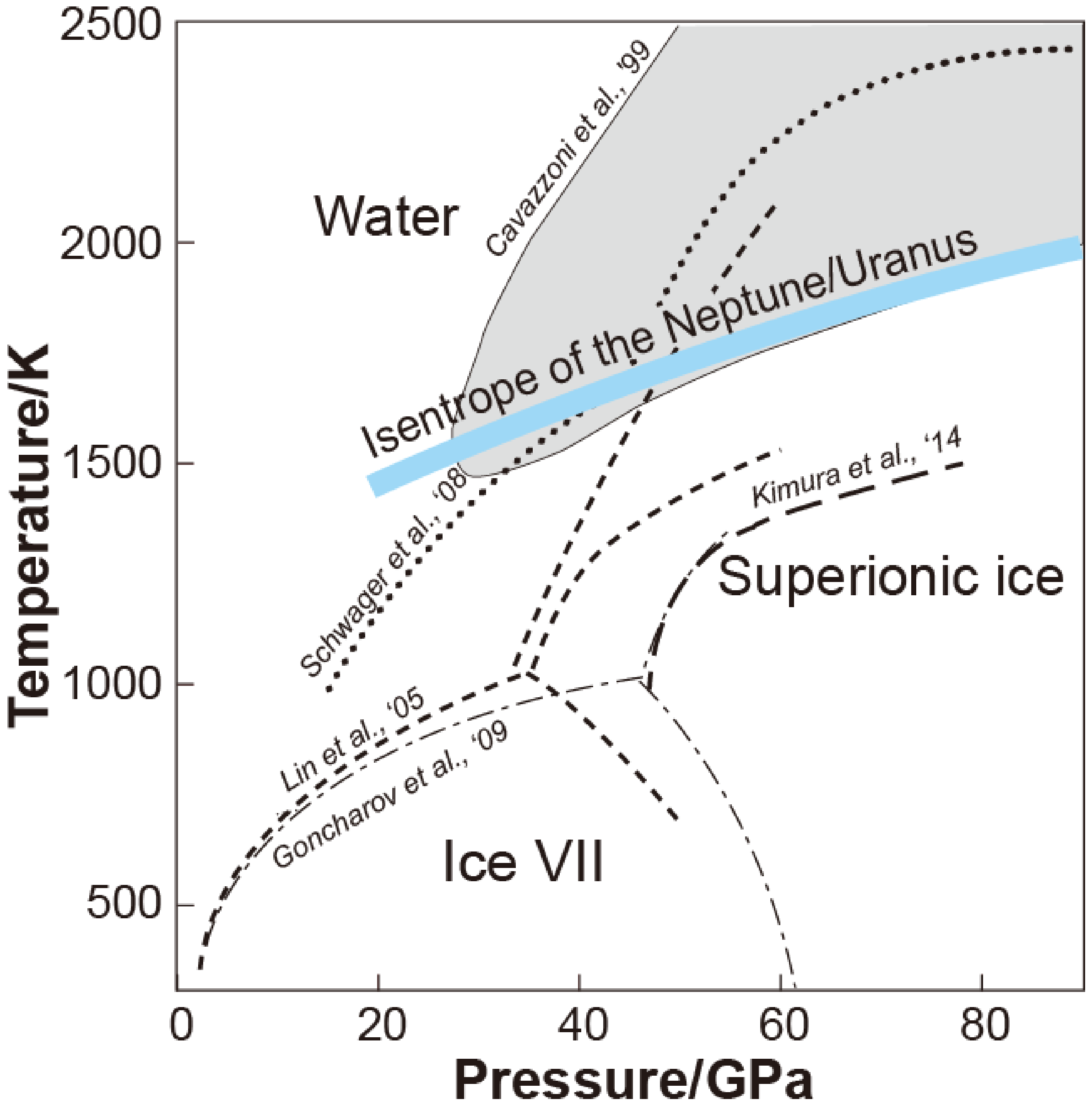
Body-centred cubic
(bcc)
oxygen sublattice

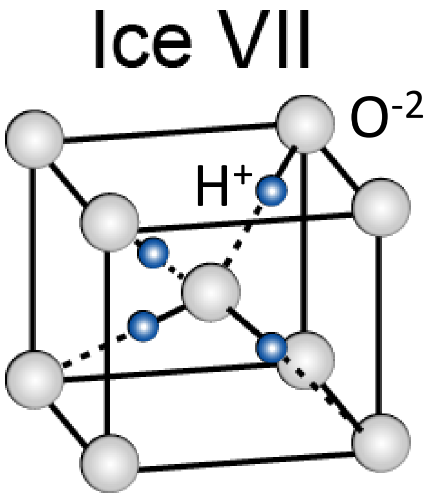


Tschauner et al., 2018

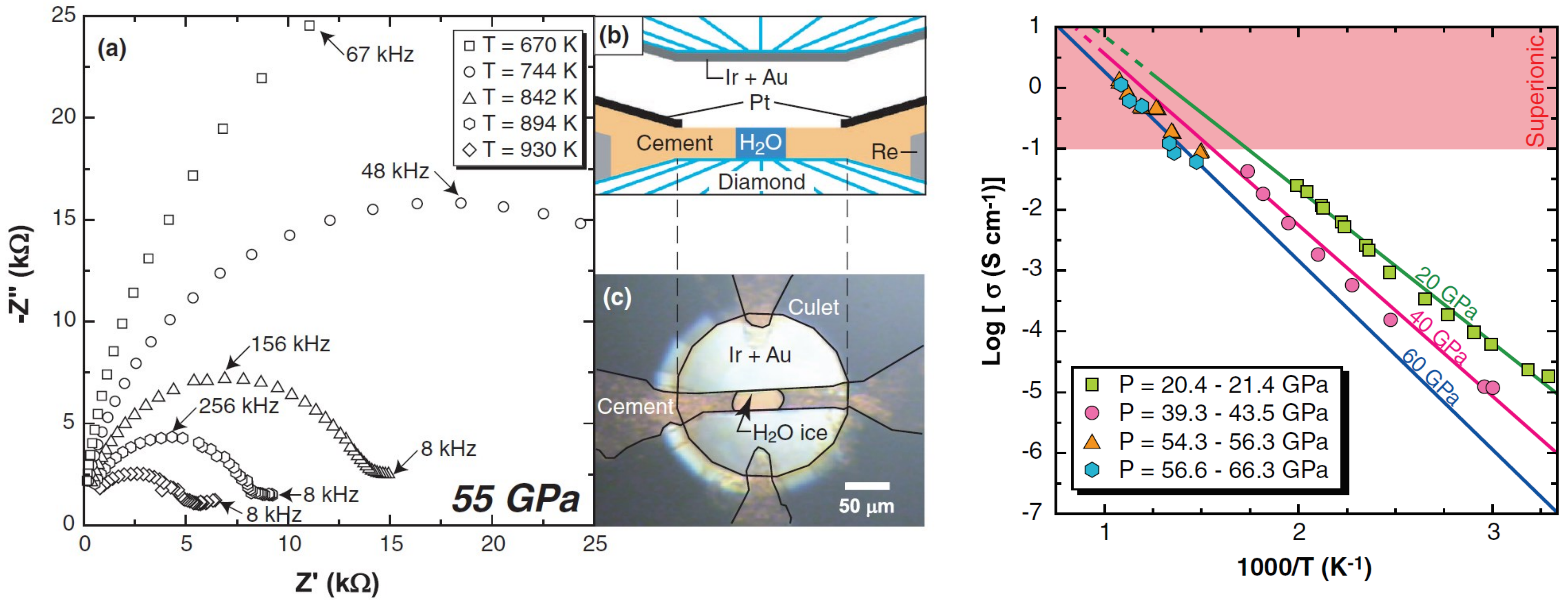
Superionic ice in Ice Giants?







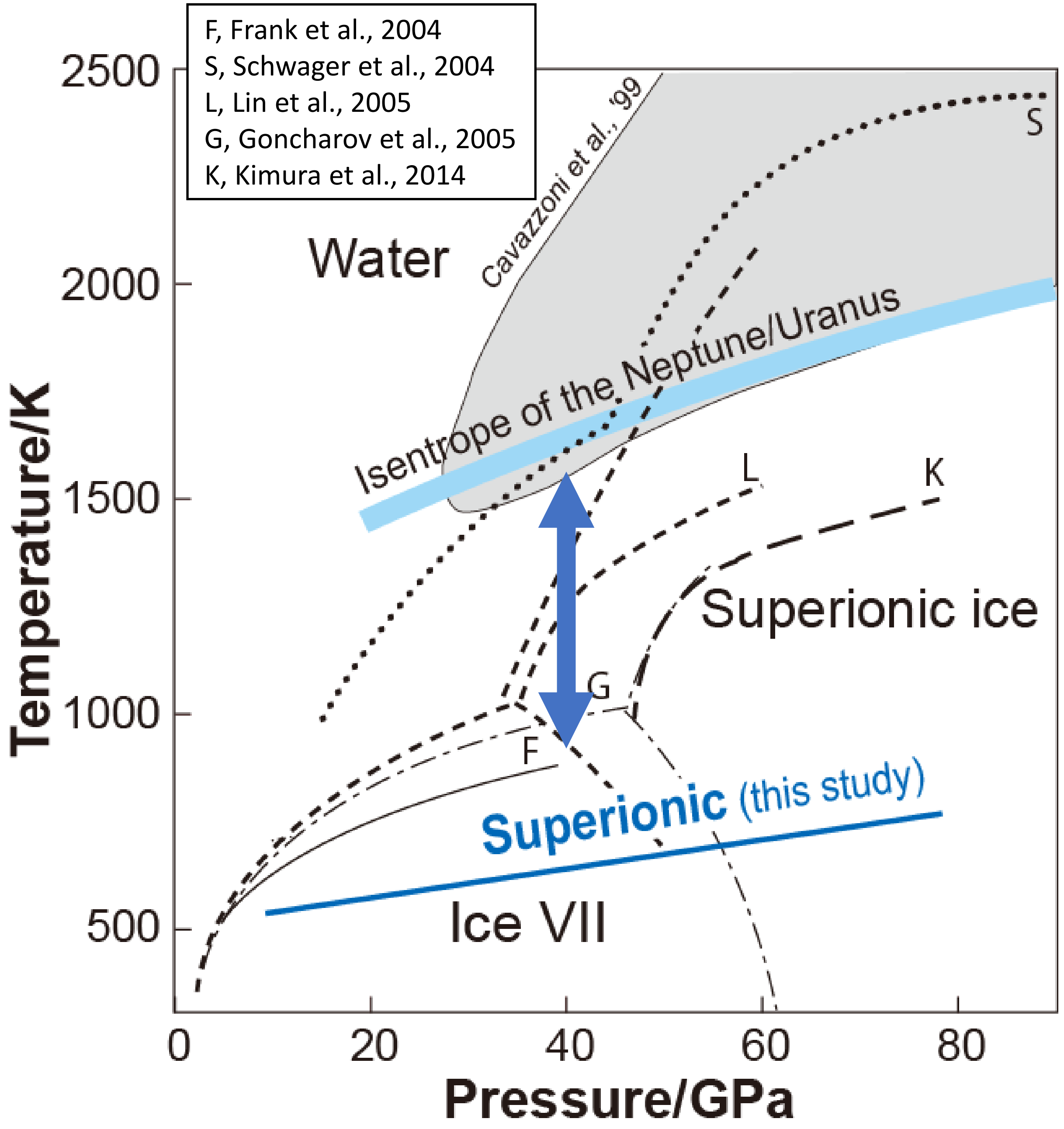
Ionic conductivity of ice at high-P-T



Sugimura et al., 2012 *J. Chem. Phys.*

Existence of superionic ice was proven directly by high-*P*-*T* experiments

Controversial melting curves



Melting temperature varies between each experimental study

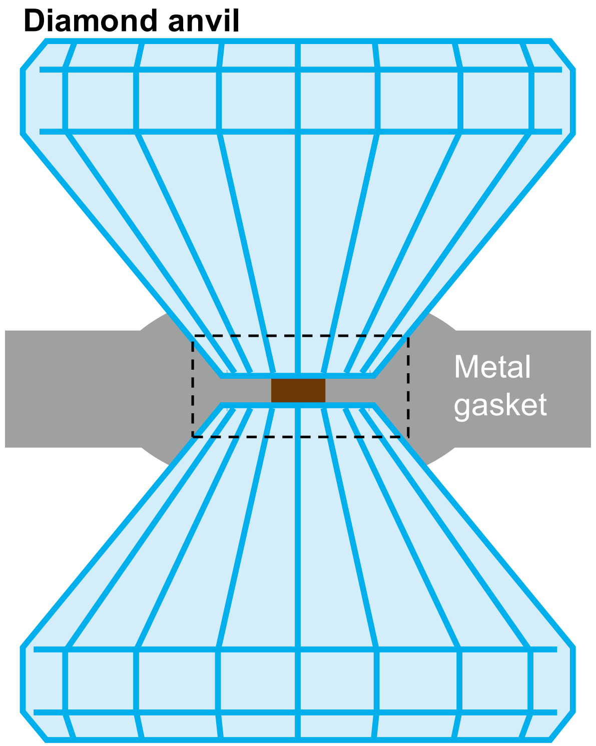
e.g. 800 – 1500 K@ 40 GPa

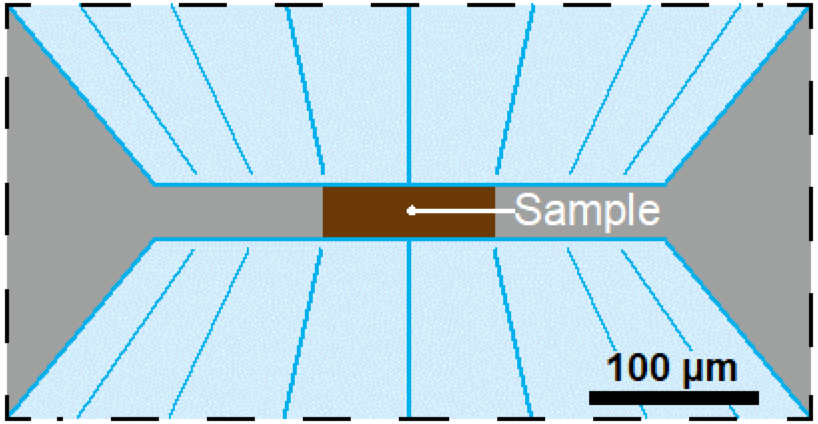
Possible factors

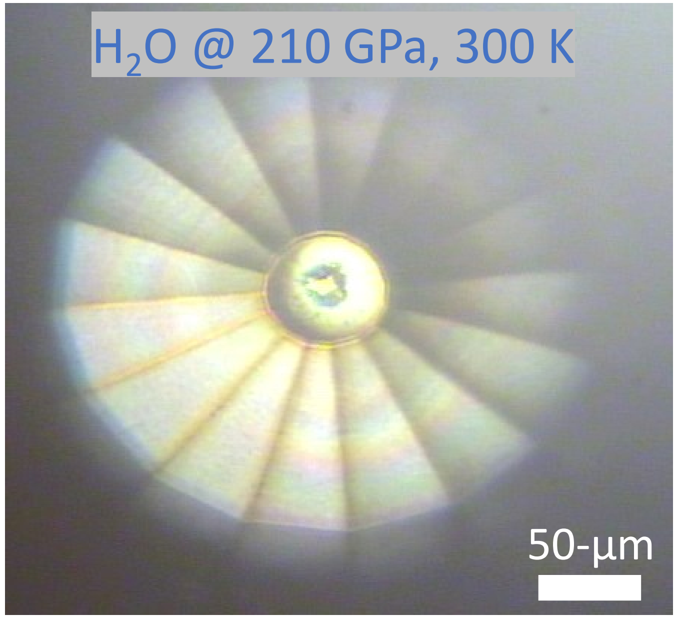
- Difference in melting/solidification criteria i.e. XRD, Raman, texture change
- Large temperature uncertainties in laser-heated DAC experiments
  - Large temperature gradients
  - Laser fluctuation

Better heating method must be used

Recreating high-pressure condition: Diamond Anvil Cell (DAC)



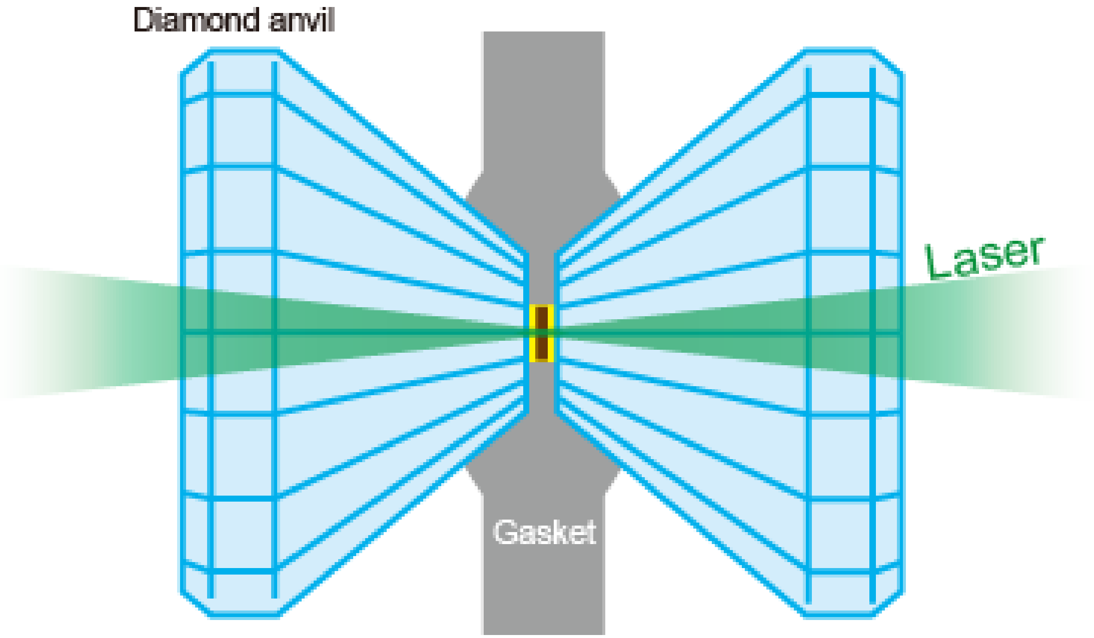




<u>Why using diamonds?</u>

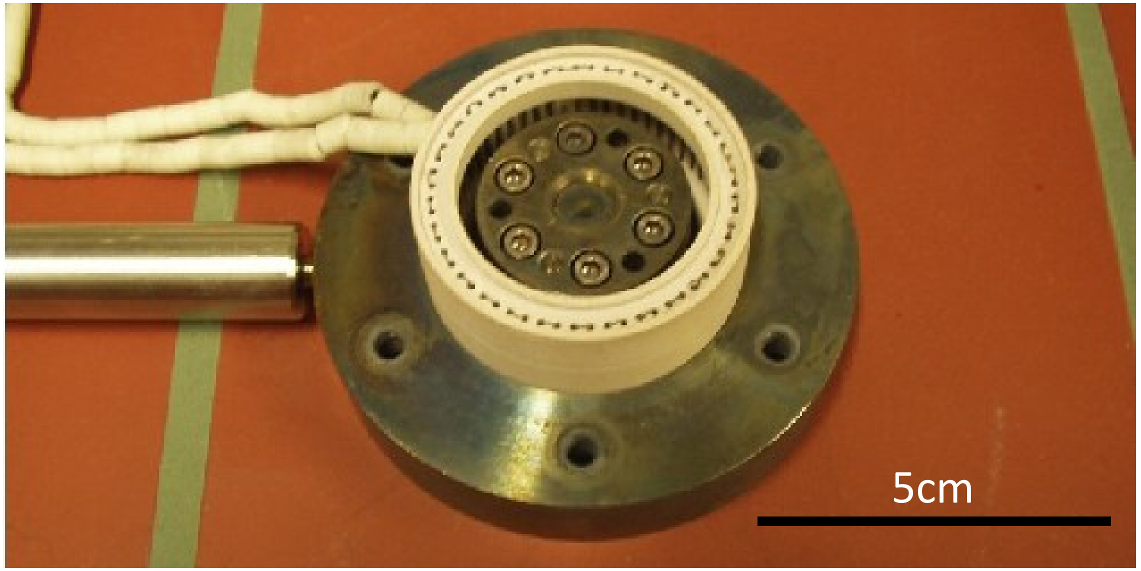
- ✓ The hardest material
- ✓ Transparency to a wide range of light source
  - Sample observation under visible light
  - Laser heating
  - X-ray & neutron diffraction
  - Spectroscopy (Raman, IR, Mössbauer, NMR etc)
- ✓ Small and portable– easy to transport between facilities

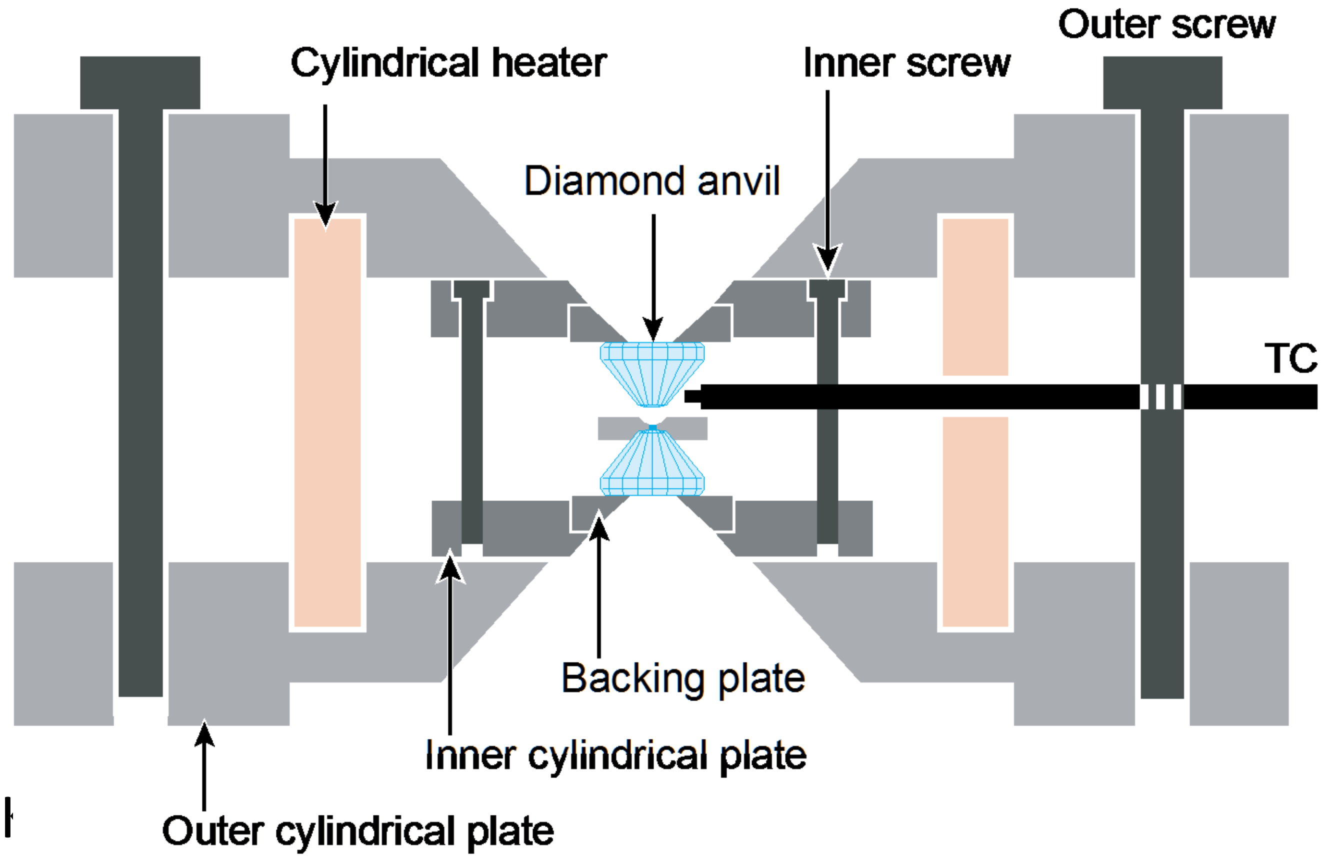
Laser-heated diamond-anvil cell



- ✓ T > 5000 K & P > 600 GPa ~ Bottom of the "icy" mantle
- ■ Large temperature (& pressure) error ±10%
  - -Large temperature gradient within the sample
  - -Laser instability

Externally resistive-heated diamond-anvil cell





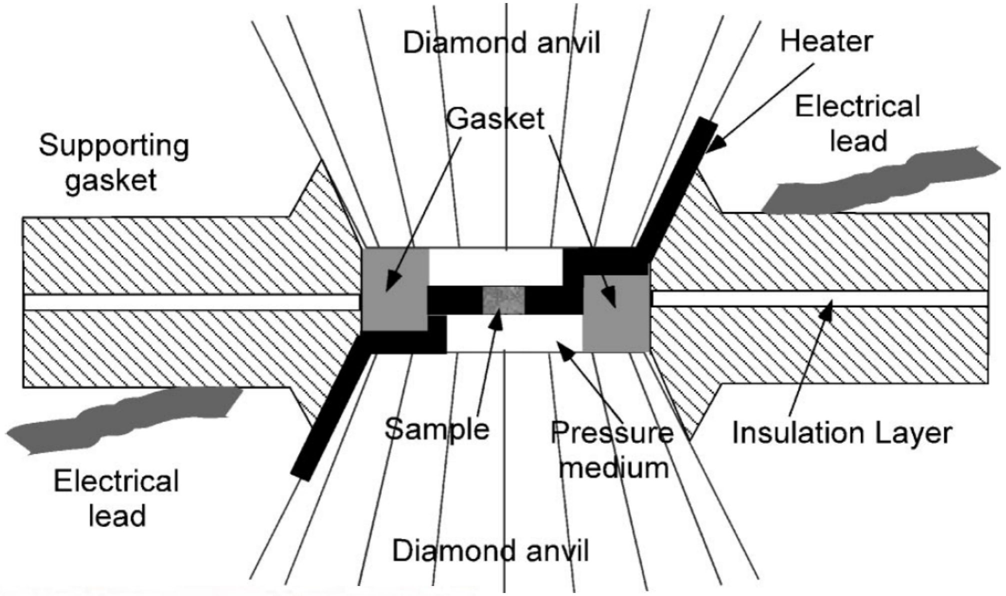
✓ Small temperature errors ±1-10 K

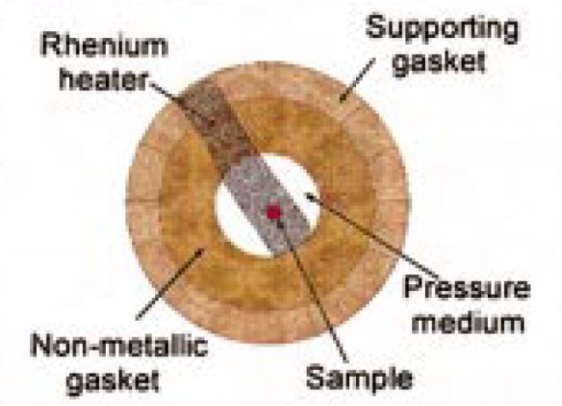
- -Homogeneous heating
- -Thermocouple placed near the sample

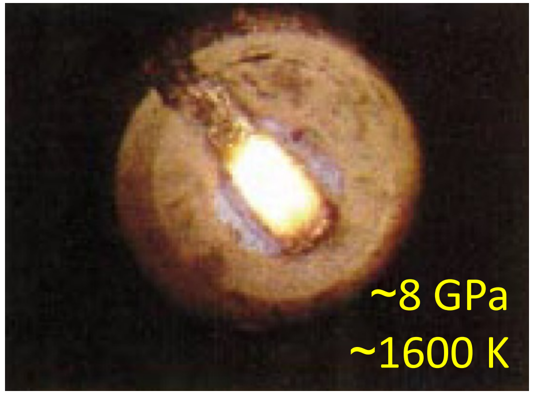
■ Temperature (& pressure) limitation

- -Diamond anvils graphitise at T > 1500 K
- -Sample, gasket, and mechanical parts deform at high temperatures

Existing internally resistive-heated diamond anvil cells (IHDAC)

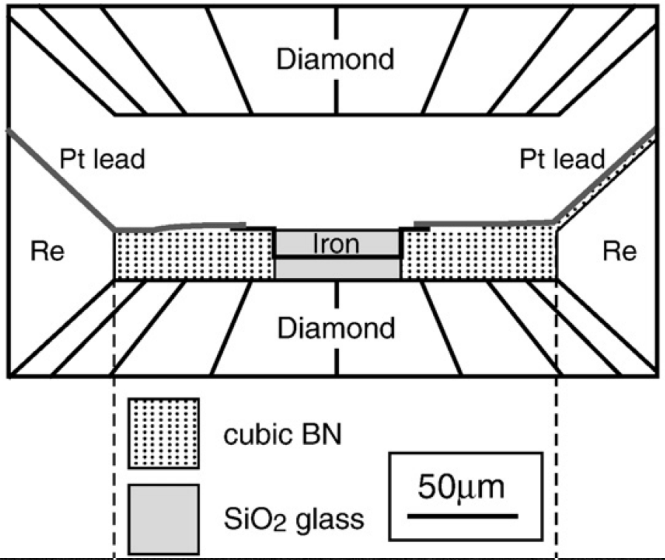


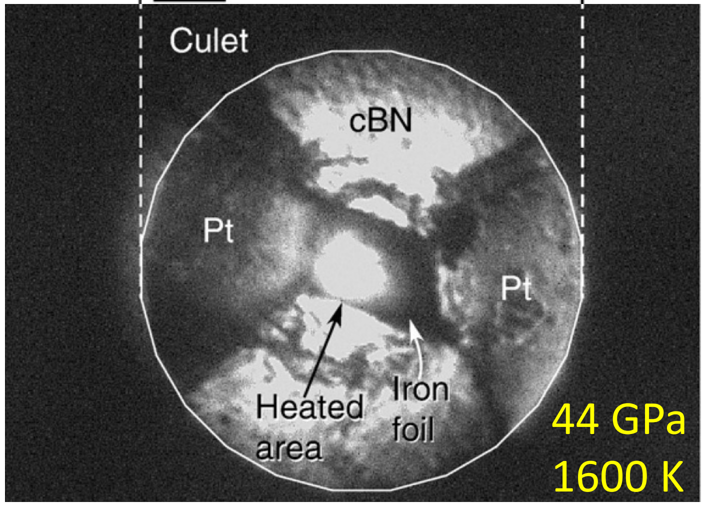




Zha & Bassett, 2003

Sample volume is very small





Komabayashi et al., 2009

Heater = Sample (e.g. Fe)
Only metals can be heated

New internally resistive-heated diamond anvil cell

- Double-sided heating enables homogenous heating

- Combination of
  - Water resistant composite gasket
  - Water resistant heaters

  makes it possible to heat various types of samples other than water, including silicates and oxides

- Aiming to generate up to 200 GPa, 4000 K (current record P = 100 GPa, T = 4000 K) using thin-film heaters

Candidate metals for the heater

Metal	Resistivity at 1 atm, 293 K (10^{-6} Ω*cm)	T_{melt} at 1 atm (K)	Bulk modulus at 1atm, 300 K (GPa)	Note
e.g. Fe	9.7	1810 (bcc)	179.3 (hcp) (Sakai et al., 2014)	—
Pt (fcc)	10.6	2042	273.5 (Zha et al., 2008)	Ductile, stable against air and water
Ir (fcc)	5.3	2727	306 (Cerenius & Dubrovinsky, 2000)	Brittle, stable against air and water. Very expensive.
Re (hcp)	19.3	3453	352.6 (Anzellini et al., 2014)	Ductile, resist oxidation, tarnish slightly in wet condition
W (bcc)	5.65 (300 K)	3683	298 (Dewaele et al., 2004)	Very brittle, easily oxidised in wet conditions

Experimental procedures

Gasket preparation

↓

Heater preparation

↓

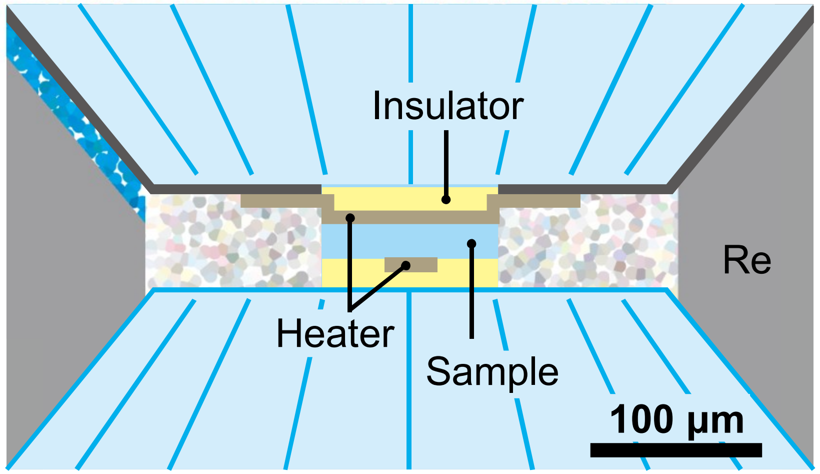
Sample loading

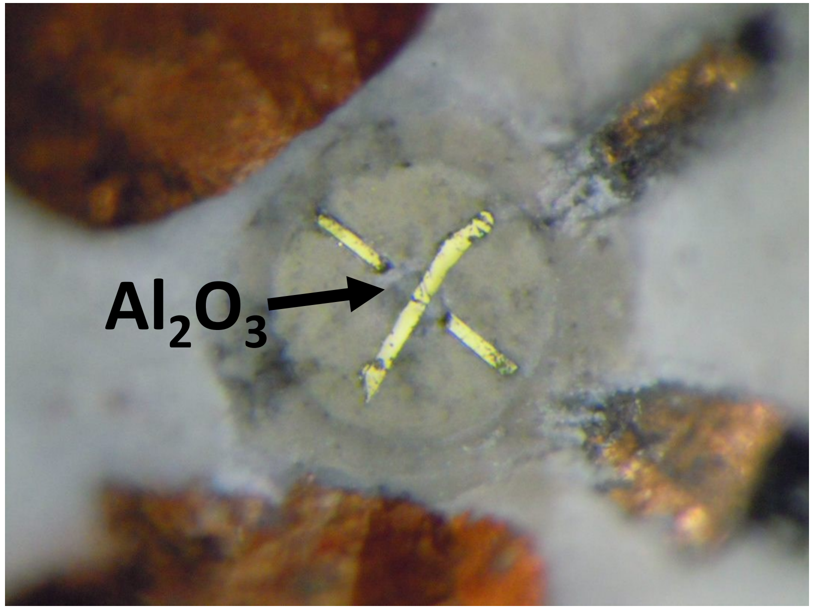
↓

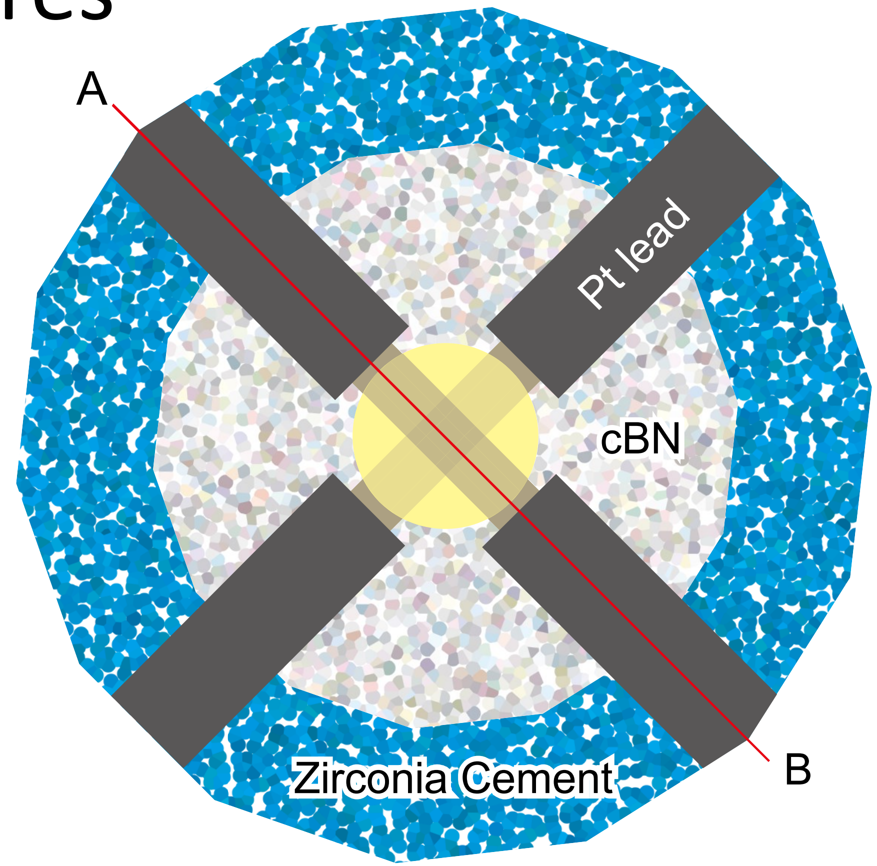
Applying pressure

↓

Heating + Measurements







<u>Heater properties</u>

Pt80wt%-Ir20wt% alloy / Re (Goodfellow™)

Heater width 20-30 μm

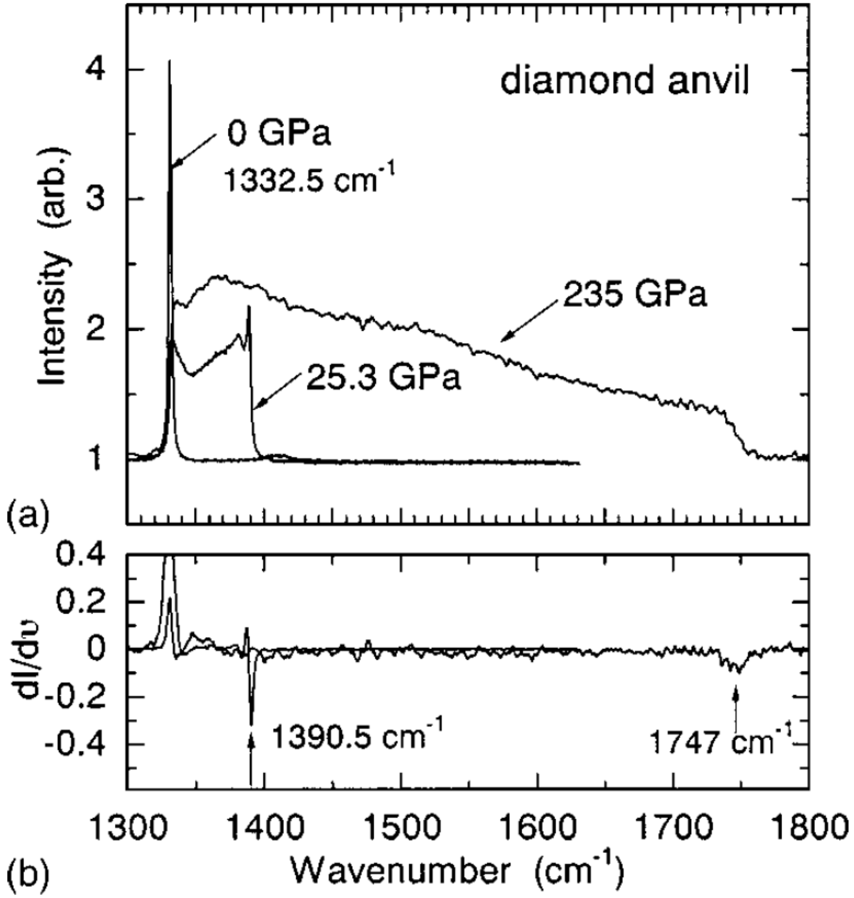
Heater thickness 5-6 μm

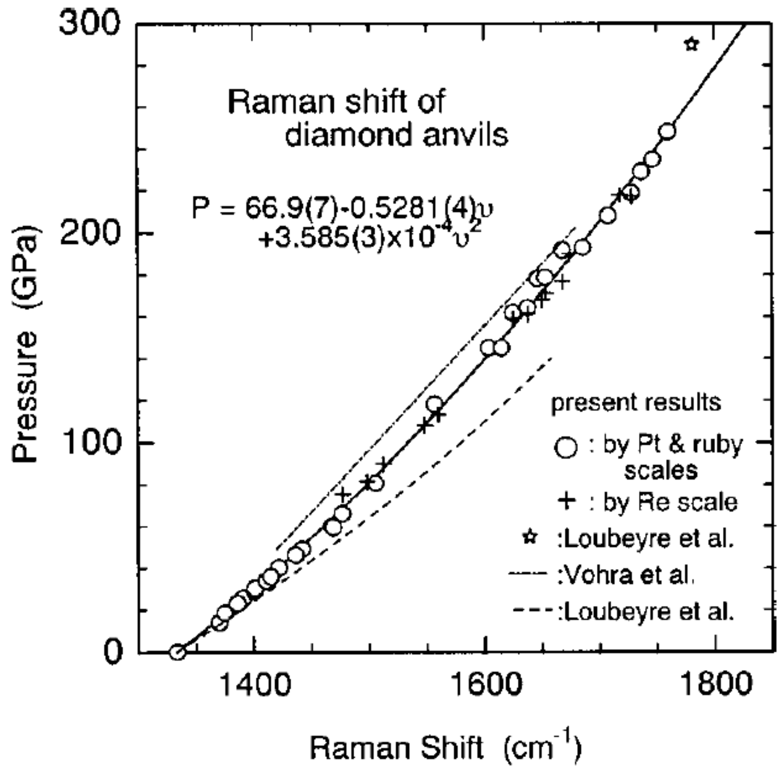
Experimental procedures

- Gasket preparation
- Heater preparation
- Sample loading
- **Applying pressure**
- Heating + Measurements

Pressure calibrants

- Ruby (Fluorescence)
- Volume of pressure calibrants (X-ray diffraction)
- Diamond (Raman spectroscopy)





Akahama & Kawamura, 2004

Experimental procedures

Gasket preparation

↓

Heater preparation

↓

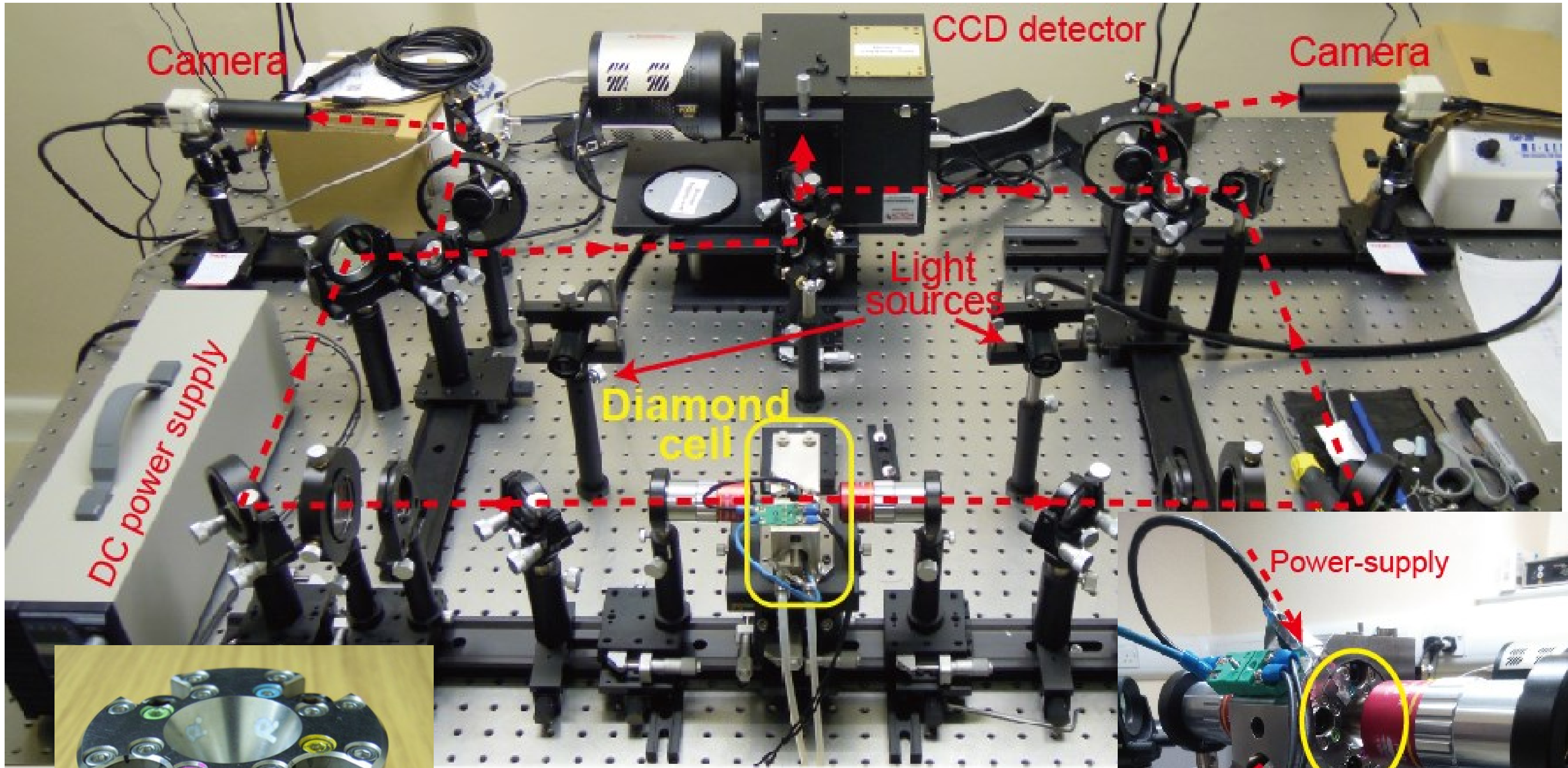
Sample loading

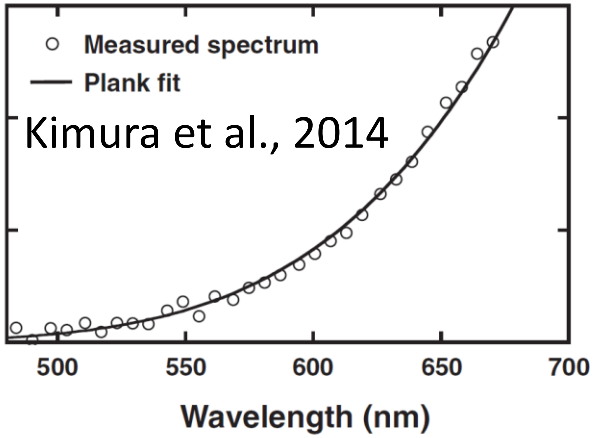
↓

Applying pressure

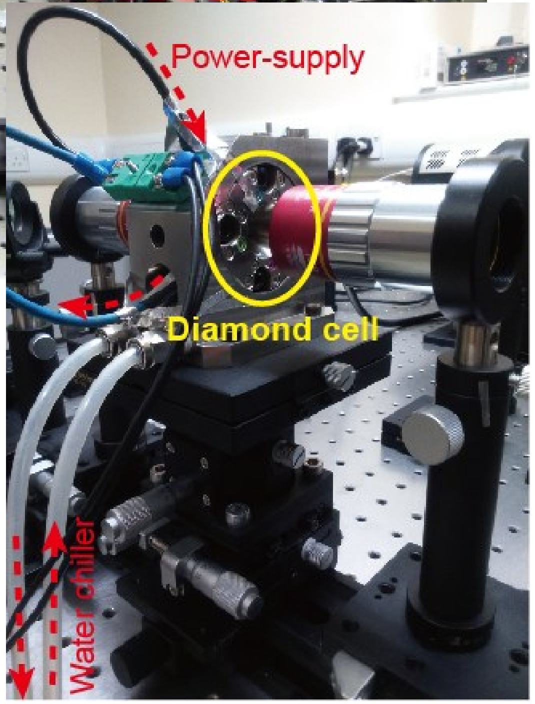
↓

Heating + Measurements



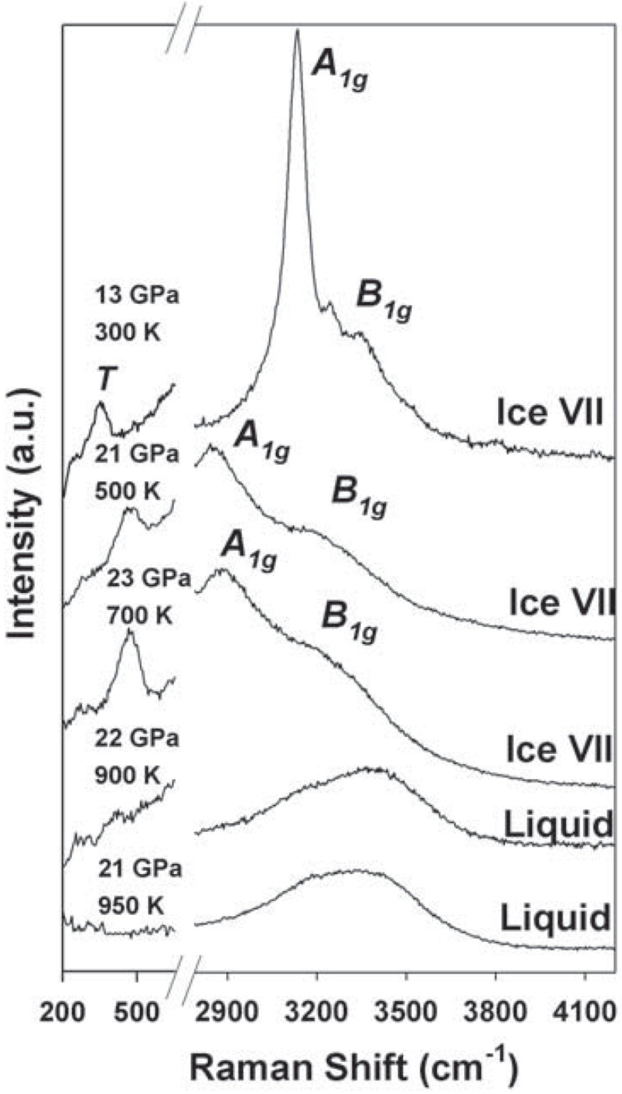


Kimura et al., 2014

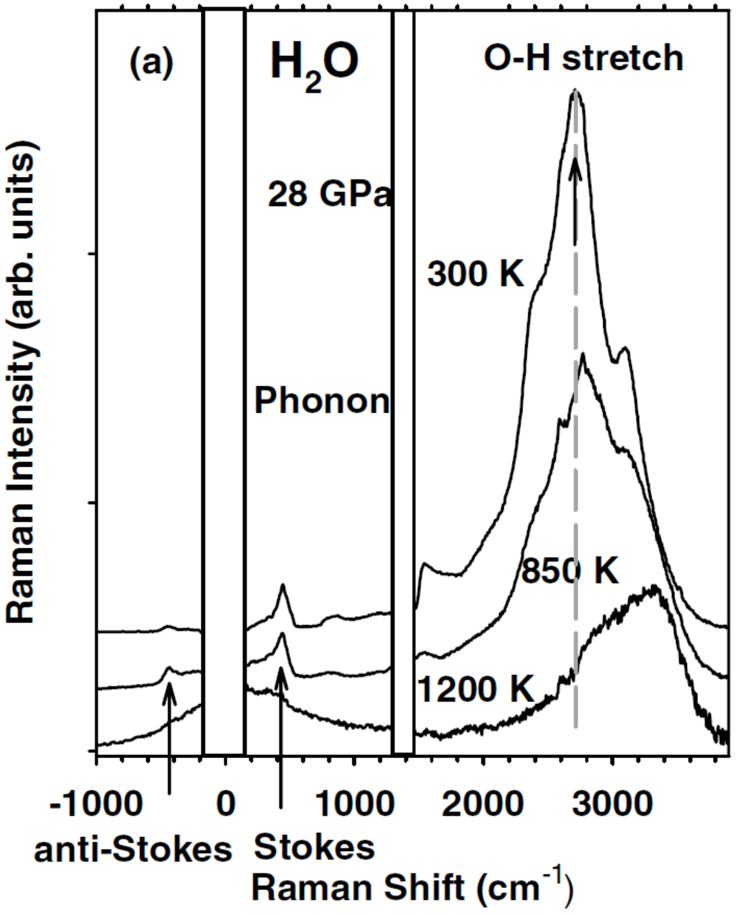


Melting criteria

Raman spectroscopy

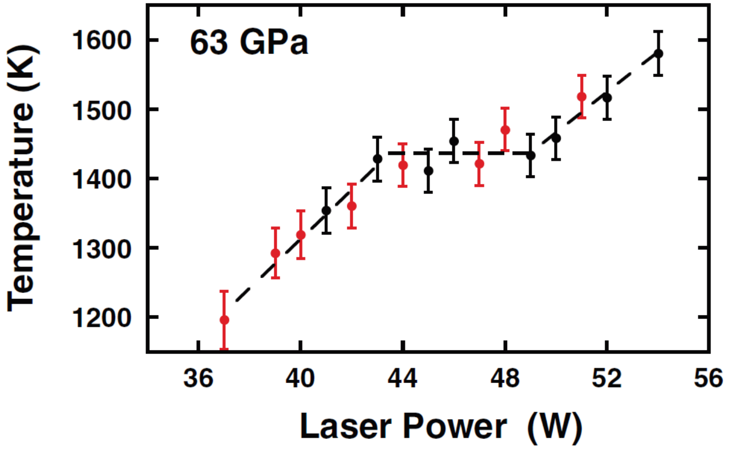


Lin et al., 2005



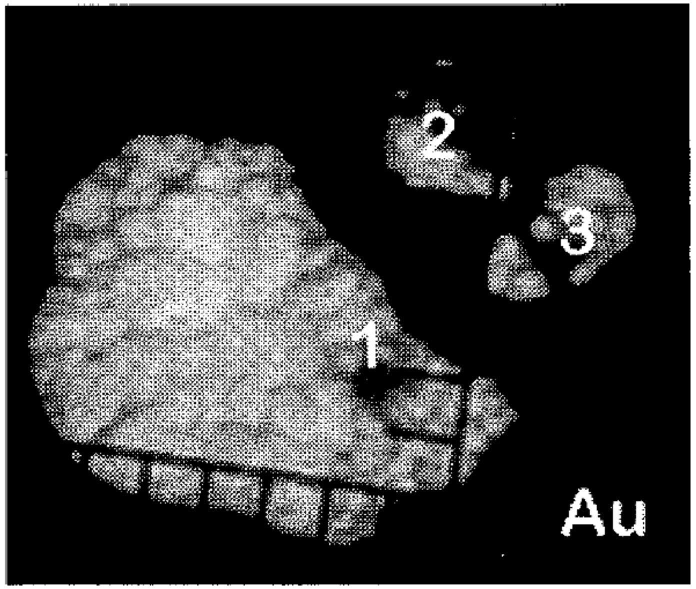
Goncharov et al., 2005

Laser power VS Temperature



Kimura et al., 2014

Visual observation



Datchi et al., 2000

Testing Pt-Ir alloy & Re heaters

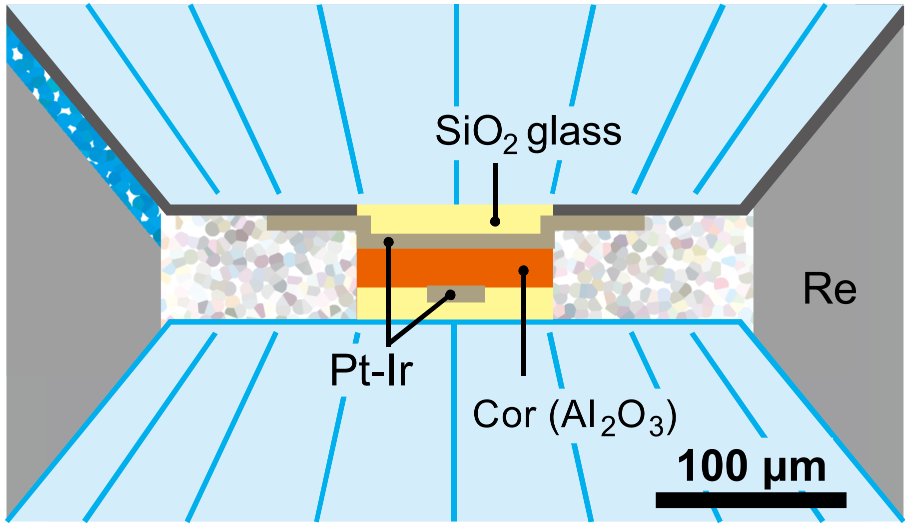
Starting materials (samples)

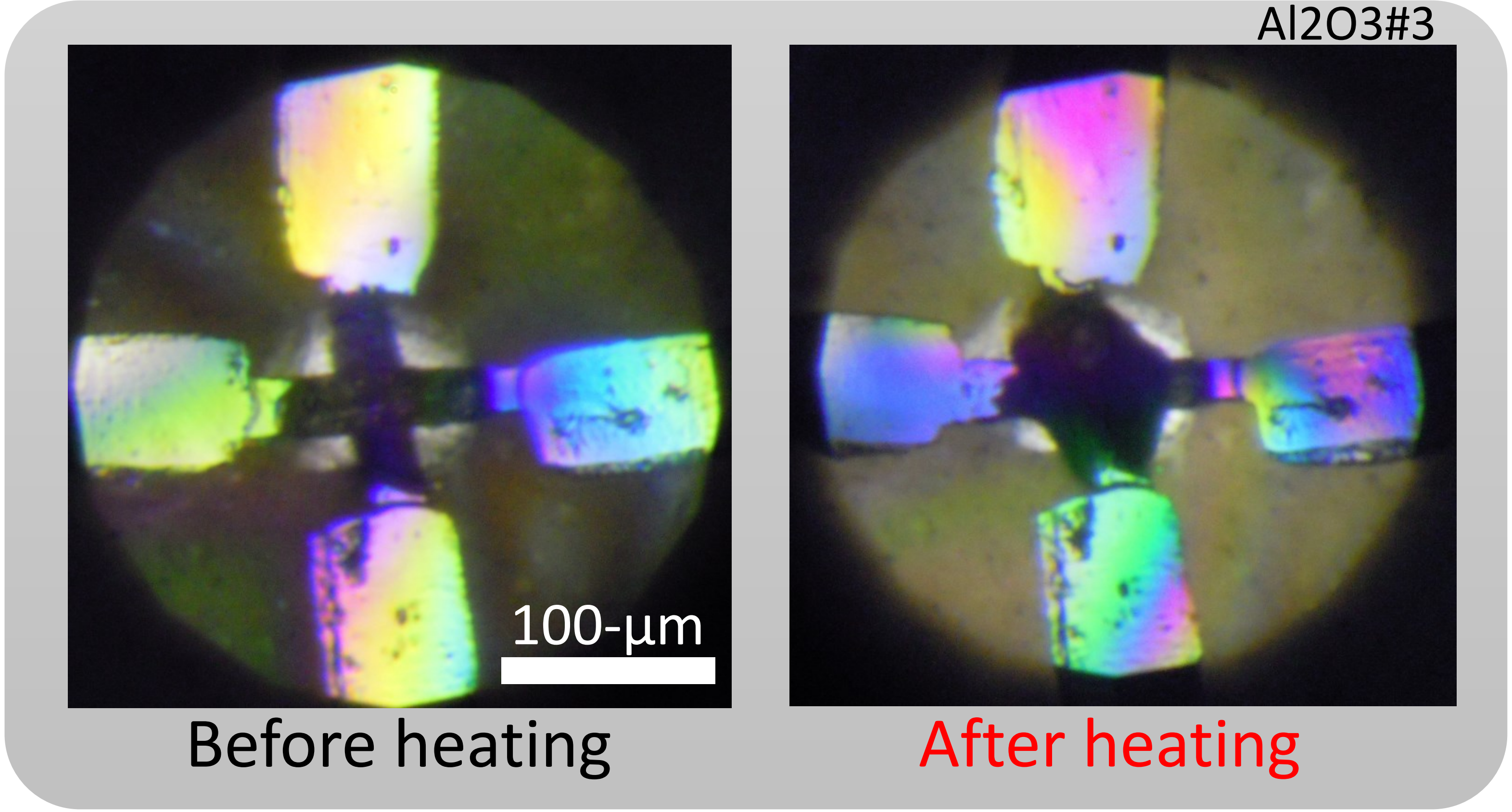
Corundum (Al_2O_3)

Forsterite (Fe_2SiO_4)

Deionised water (H_2O)

Pt-Ir alloy foil heater experiment with Corundum (Al_2O_3): Method





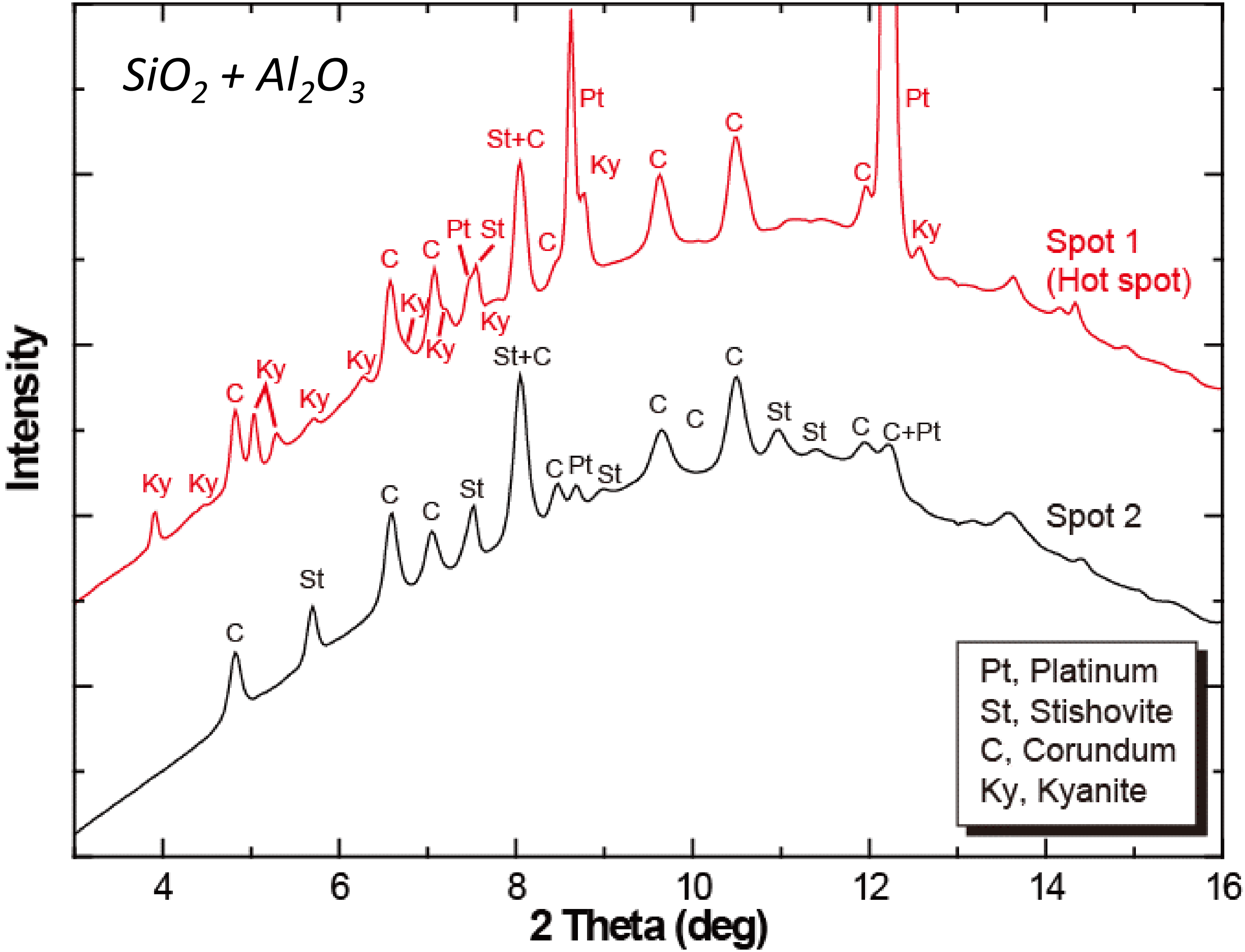
Experimental condition

Pressure: 13.4 GPa

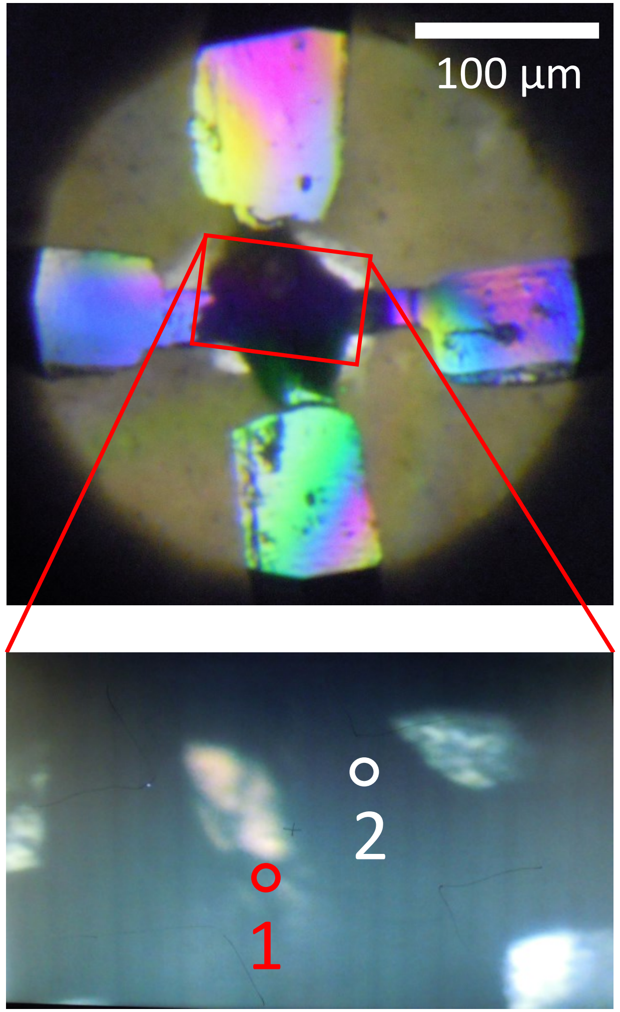
Max. temperature: 2025 K

Heating duration: 60 mins

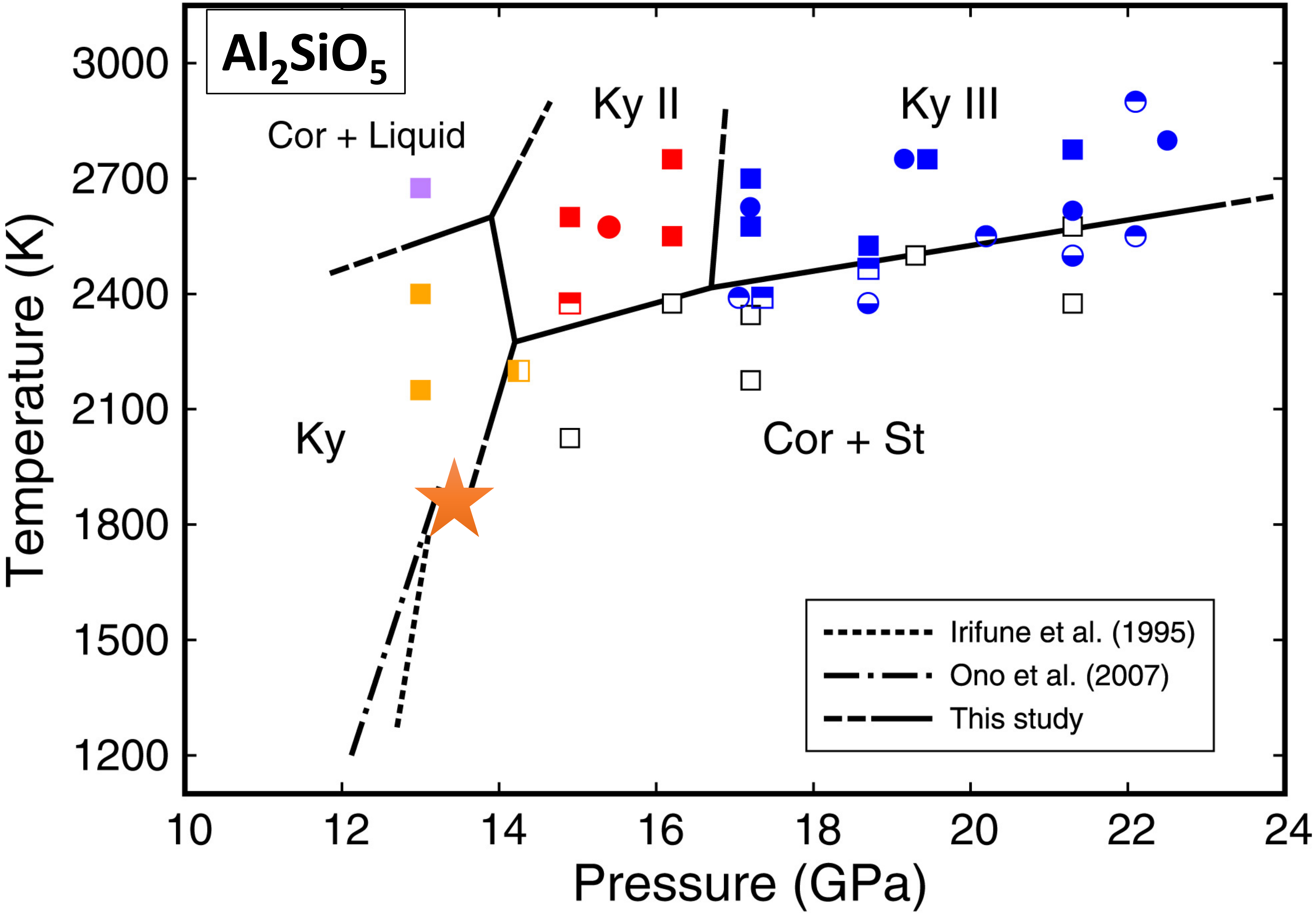
Pt-Ir alloy foil heater experiment with Corundum: X-ray diffraction



Sample: Corundum
Insulator: SiO_2 glass



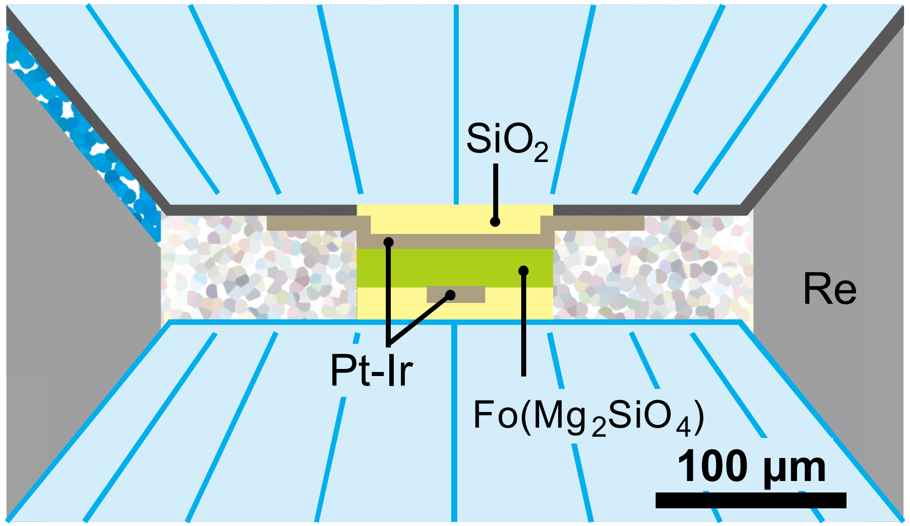
Pt-Ir alloy foil heater experiment : Ky = St + Cor

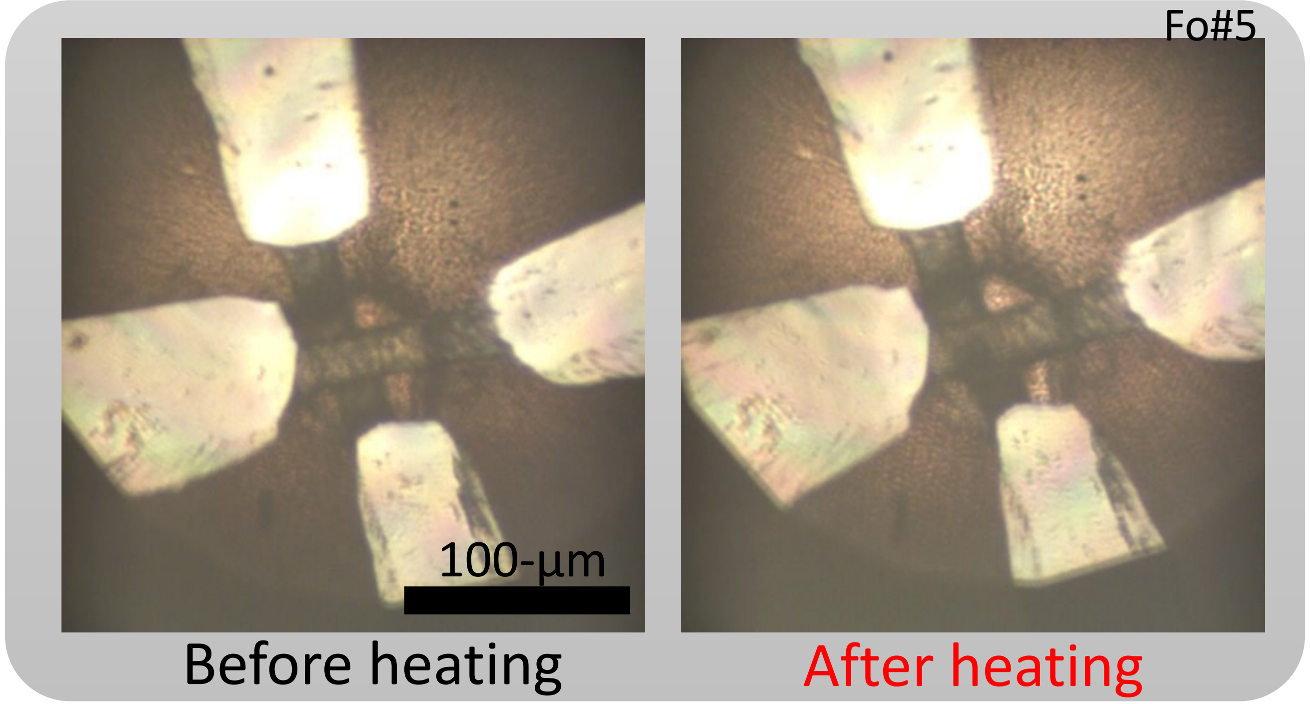


Zhou et al., 2018

- Kyanite was formed at 13.4 GPa, 1833K
- Measured temperature is reliable

Pt-Ir alloy foil heater experiment with Forsterite (Mg_2SiO_4)





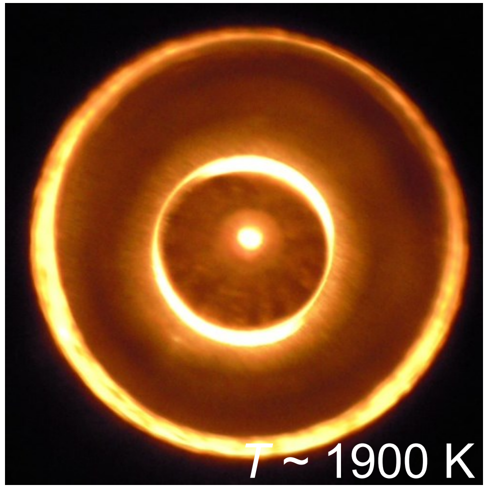
<u>Experimental condition</u>

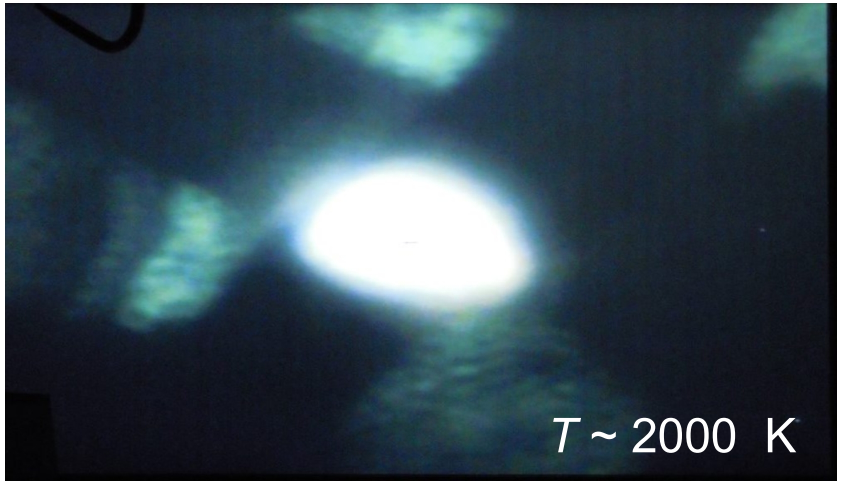
Pressure: 42 GPa

Max temperature: 2350 K

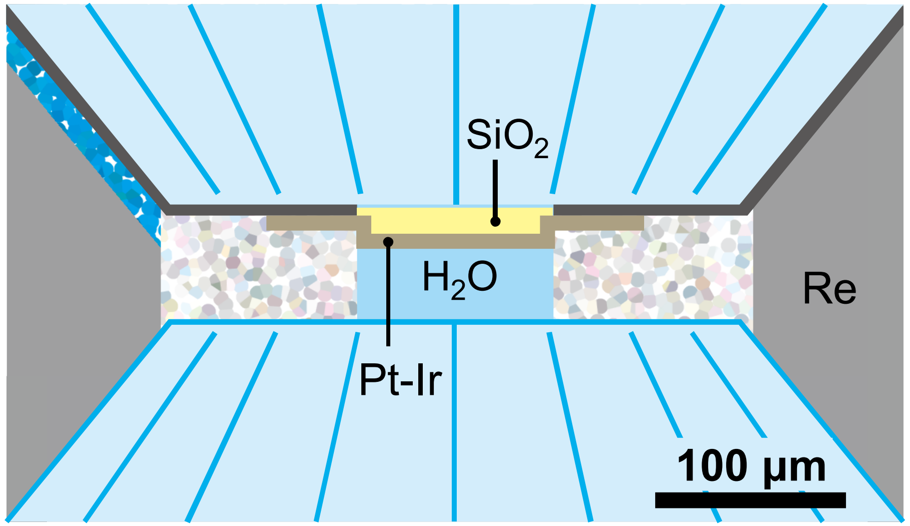
Heating duration: 1h 15 mins

No Raman signal from recovered sample





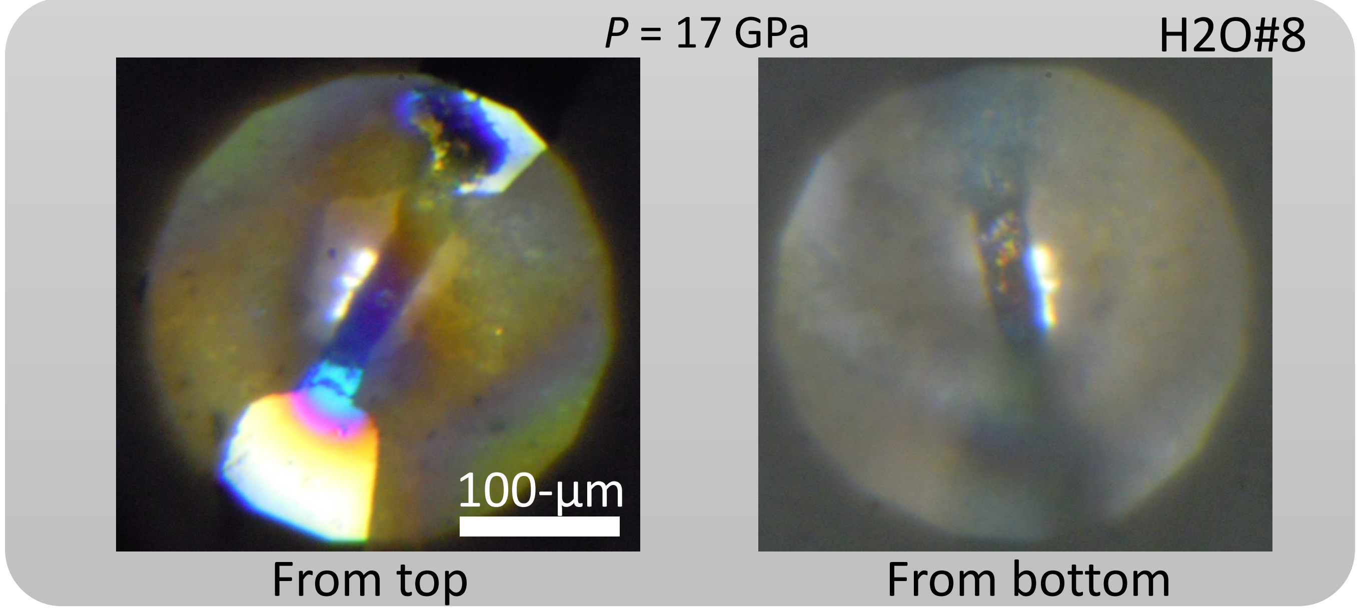
Pt-Ir alloy foil heater experiments with H_2O

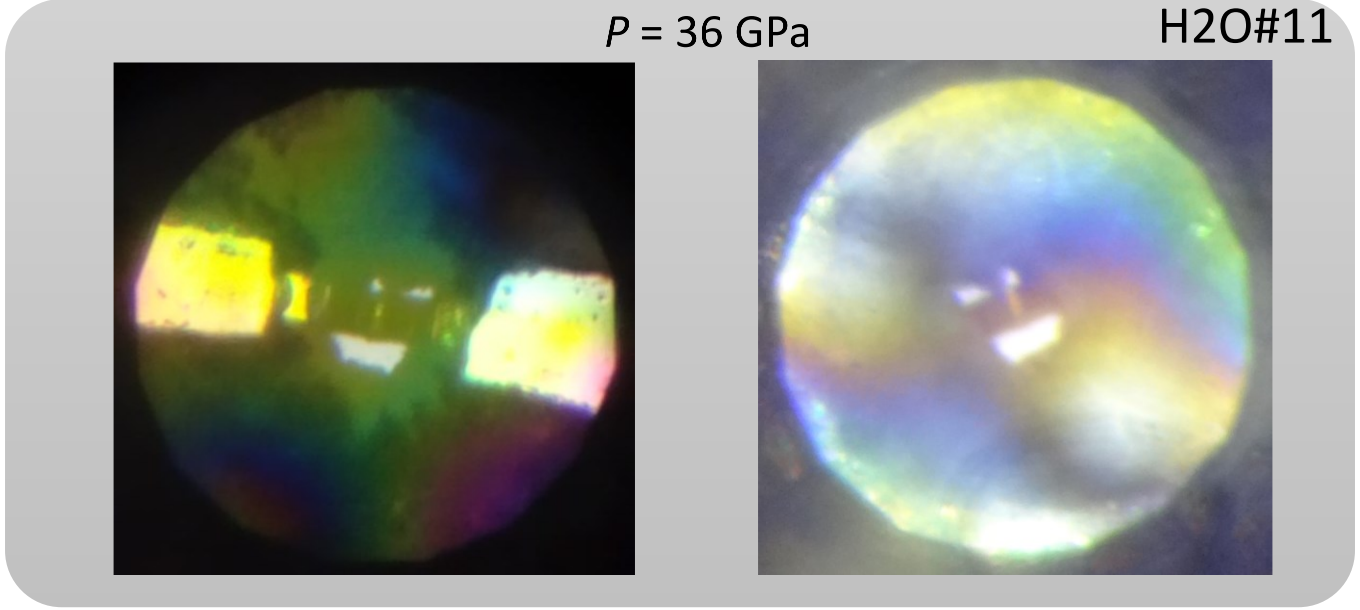


<u>Experimental condition</u>

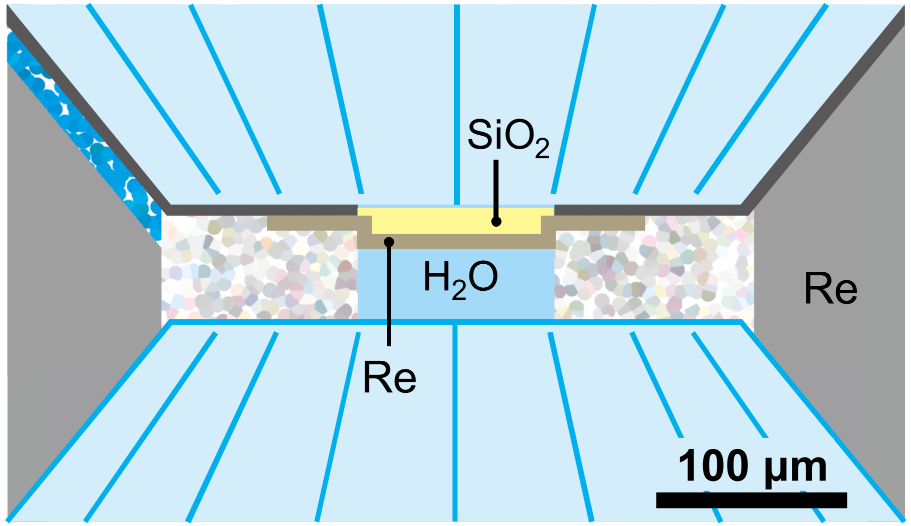
Pressure: 17 & 36 GPa

Heater gets too close to the anvil
↓
Heat escaped through the anvil
↓
No heating

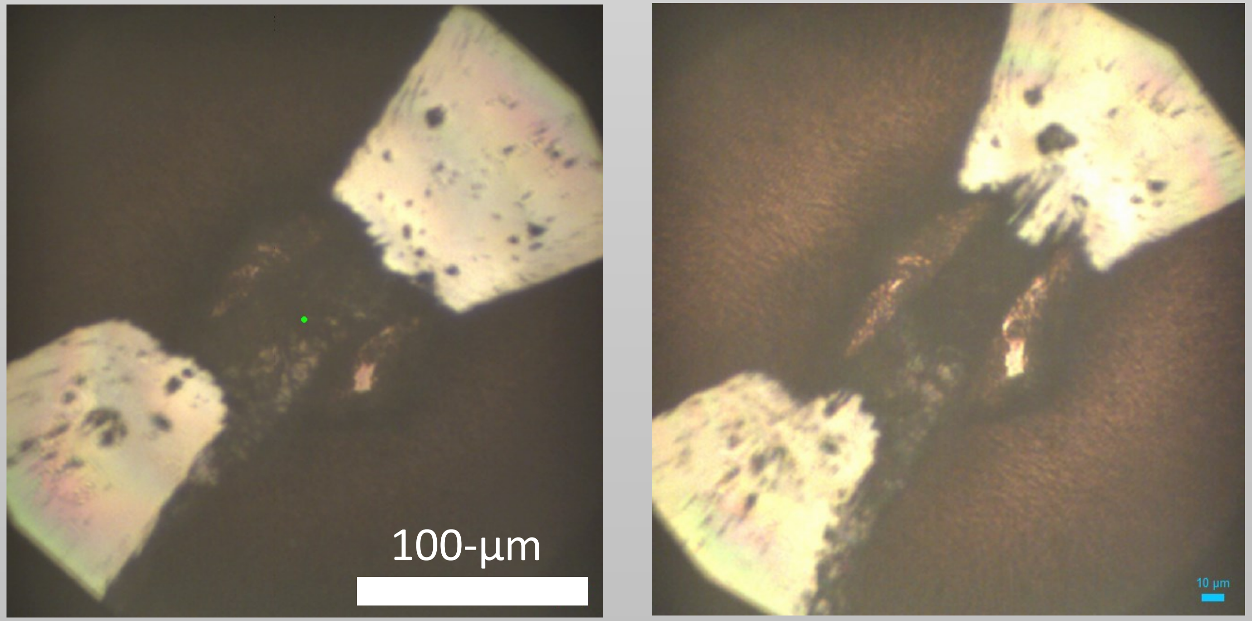




Re foil heater experiment with water: Method



H2O#12



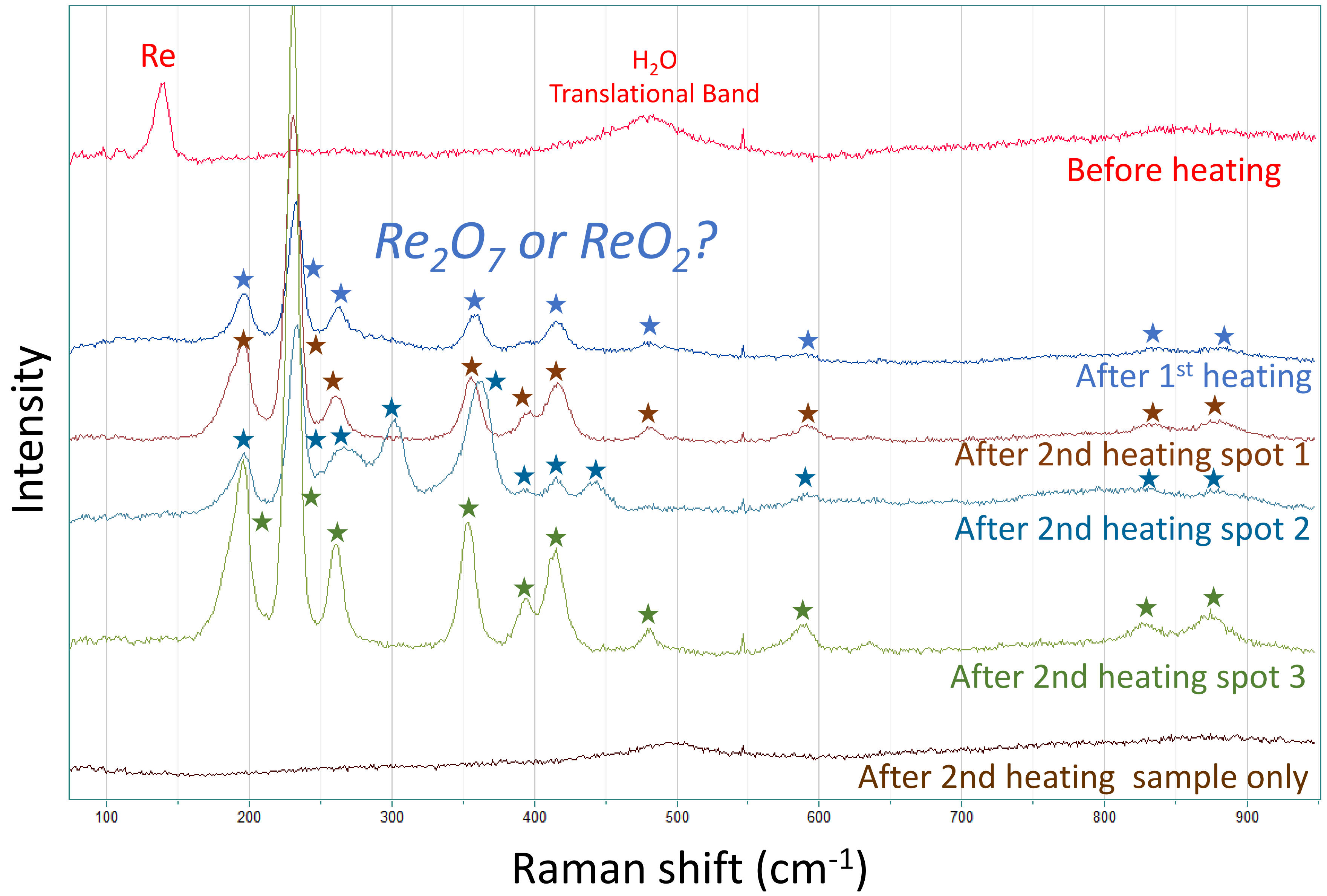
Before heating After heating

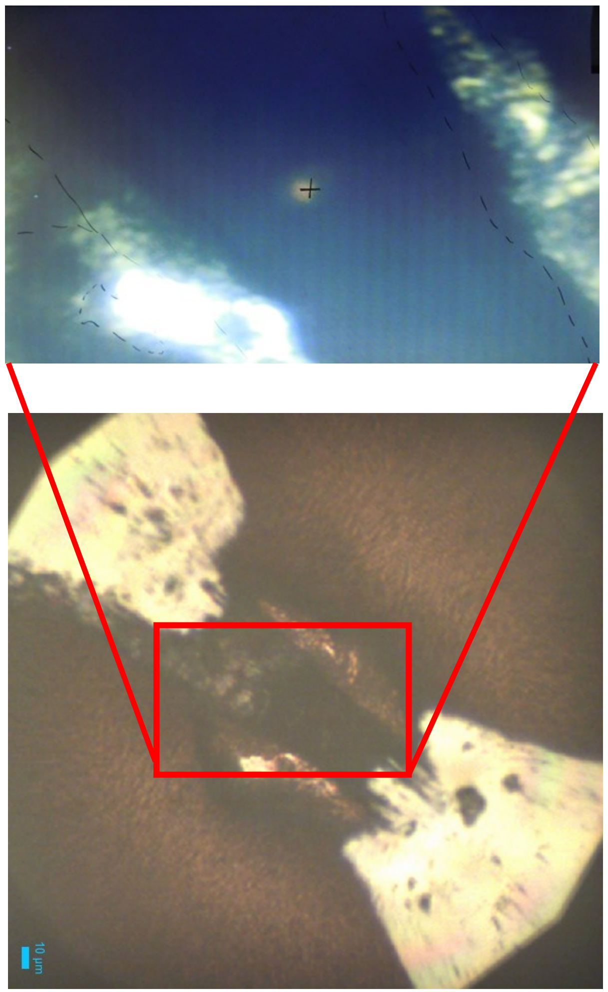
<u>Experimental *P*-*T* condition</u>

P after 1st heating: 40 GPa
Temperature: 1528 K
Heating duration: 1 mins

P after 2nd heating: 42 GPa
Temperature: 1266 K
Heating duration: 40 mins

Re foil heater experiment with water: Raman spectroscopy





Chemical reaction between H_2O and Re

Summary for testing Pt-Ir alloy & Re heaters

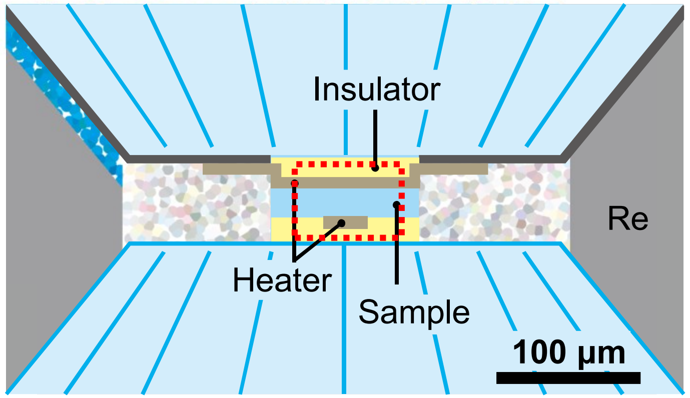
Pt-Ir

✓ No damage to the heater for up to 75 mins.

✓ Temperature generation up to 2350 K at 41 GPa

X The heater gets too close to the anvil

- Bent very easily – uneven surface, too ductile?
- Too thick

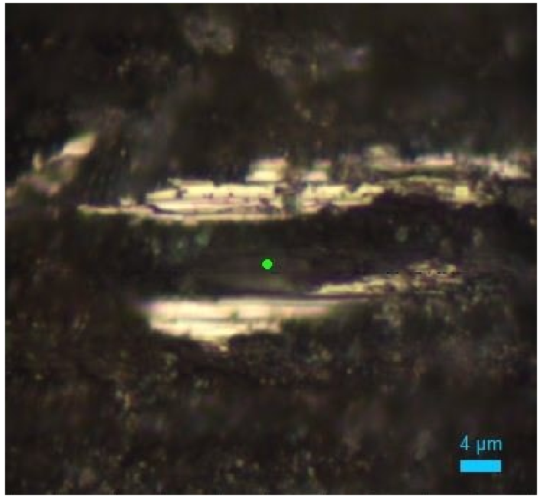


Re

✓ No significant deformation upon loading water

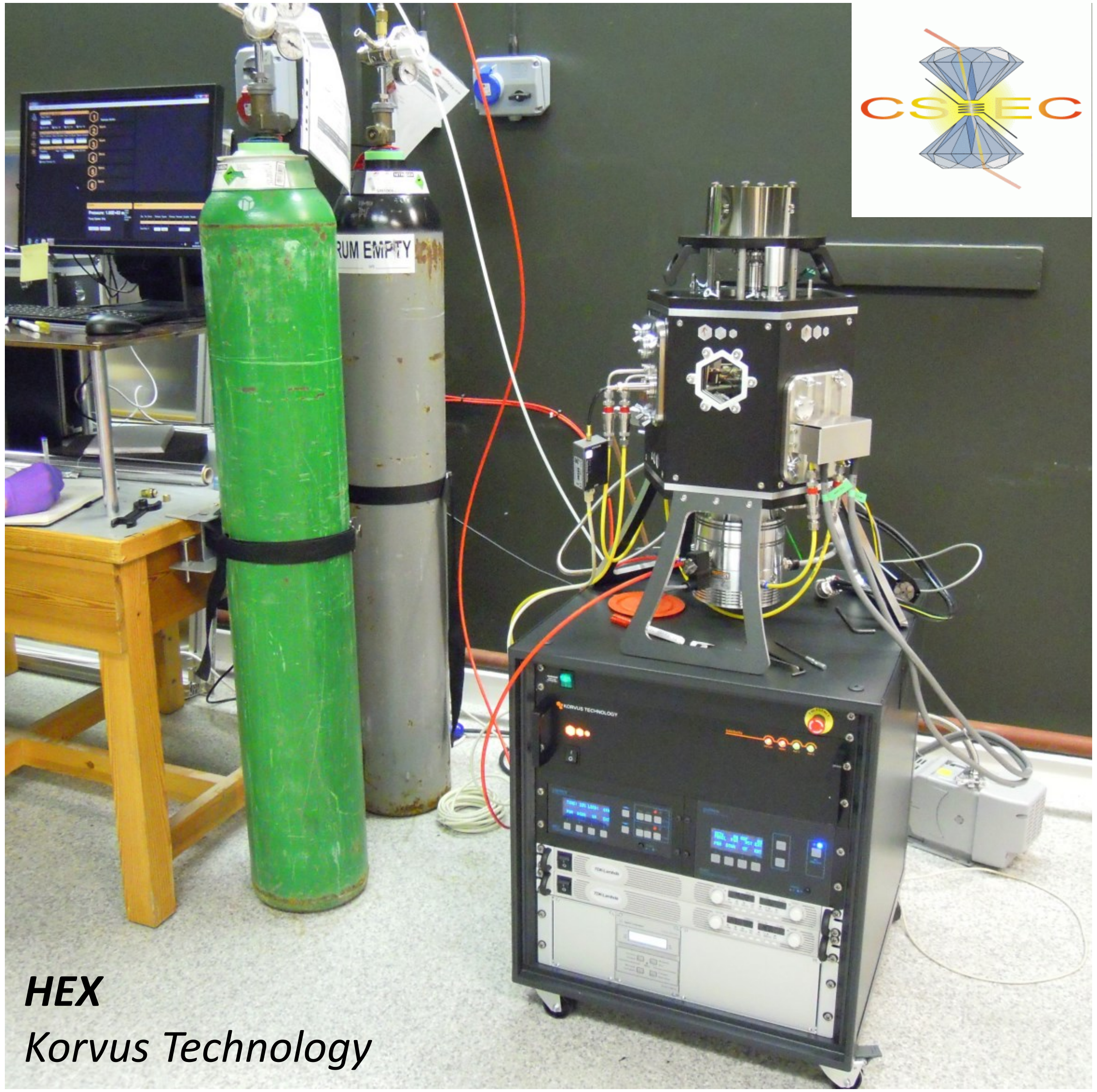
✓ Temperature generation up to 1528 K at 42 GPa

X Chemical reaction between water/ice and the heater

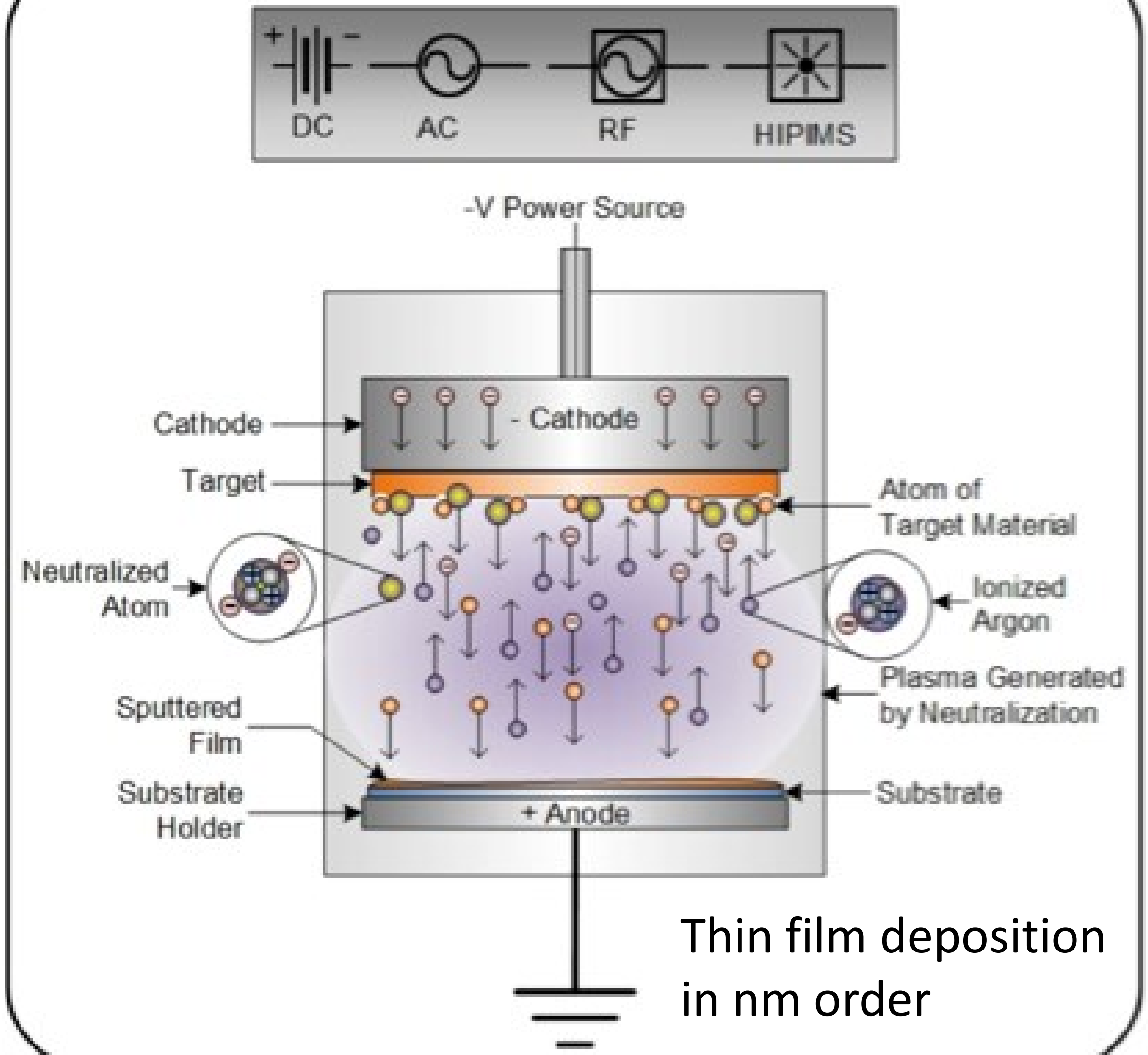


Pt-Ir alloy needs to be tested further as a thin-film heater

Thin film deposition by sputtering



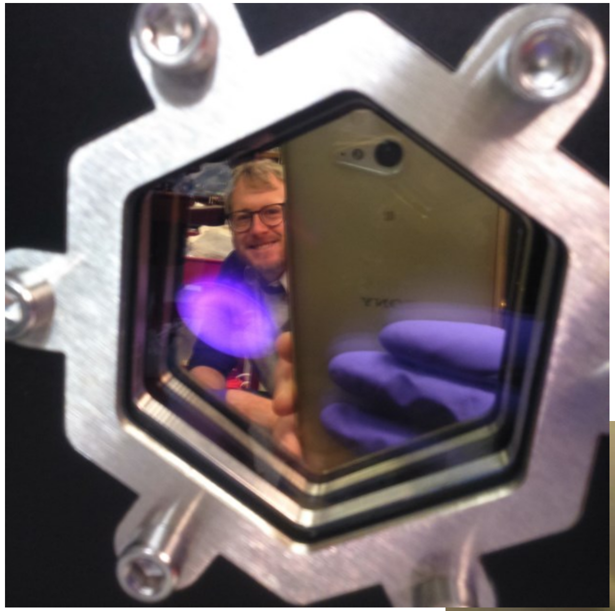
HEX
Korvus Technology

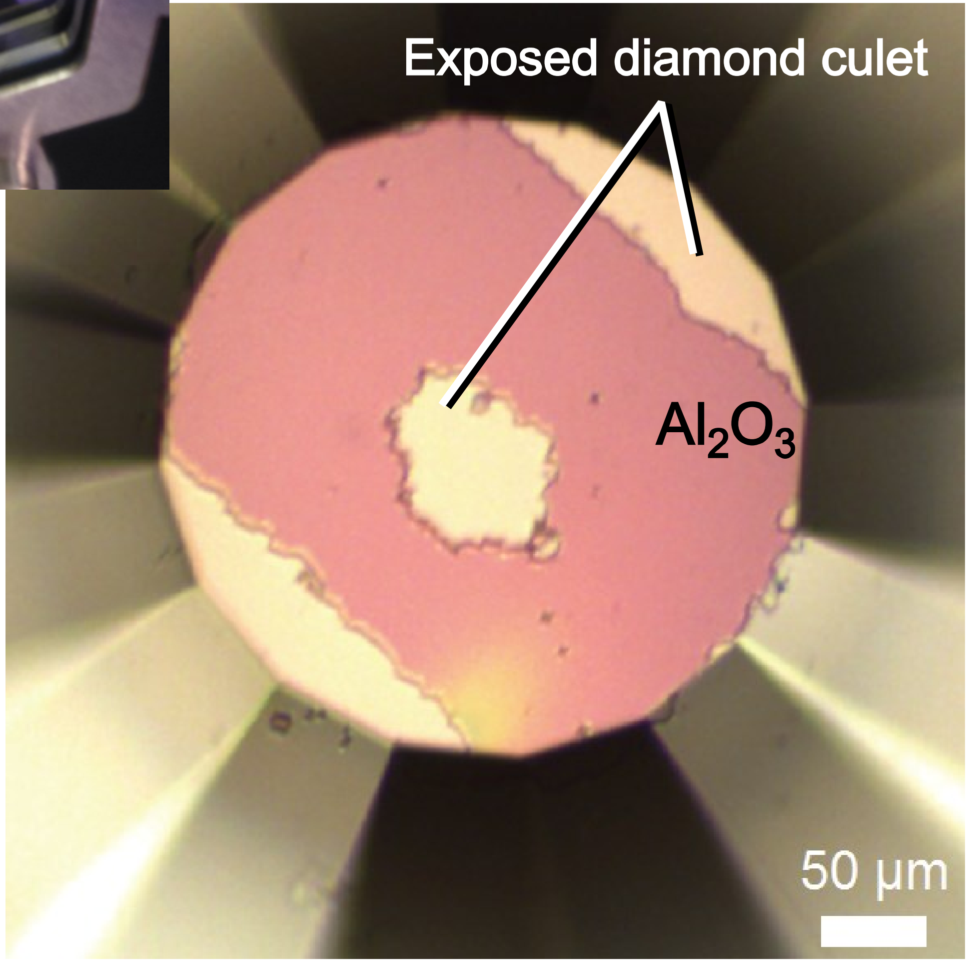


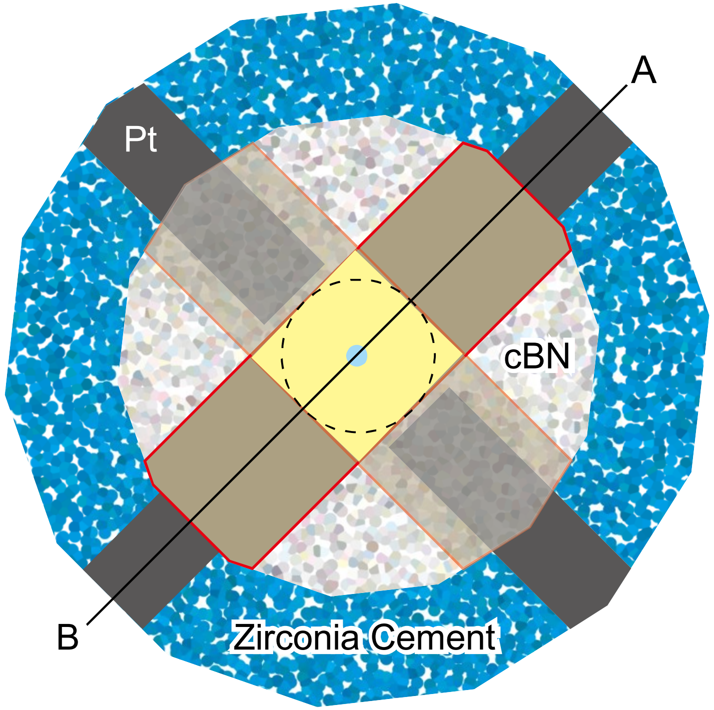
Thin film deposition in nm order

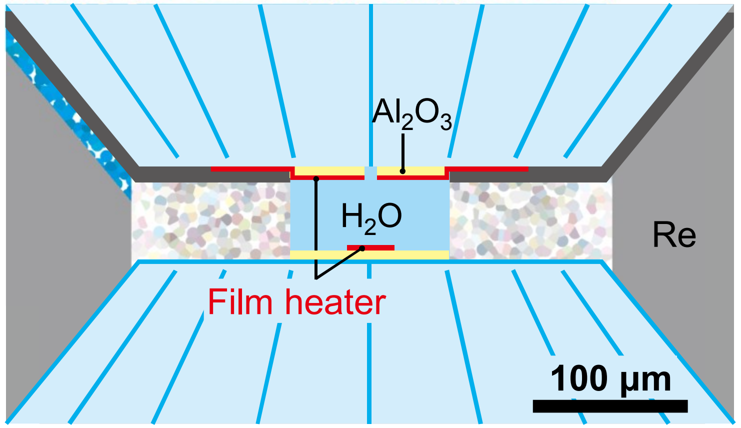
http://www.semicore.com/what-is-sputtering

Deposition of an insulation layer



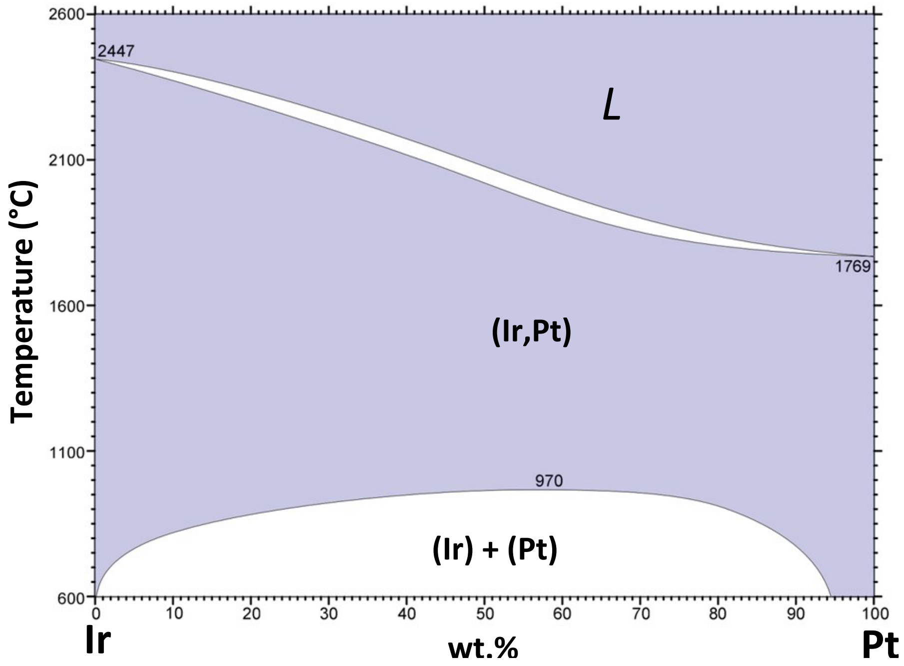






Next steps

- Pt-Ir alloy deposition
- Test Pt-Ir film heater and check if it reacts with ice/water at high P-T
- Check if Pt-Ir miscibility gap persists/disappears at high-P
- Test if Raman spectrum of water can be measured with XploRa



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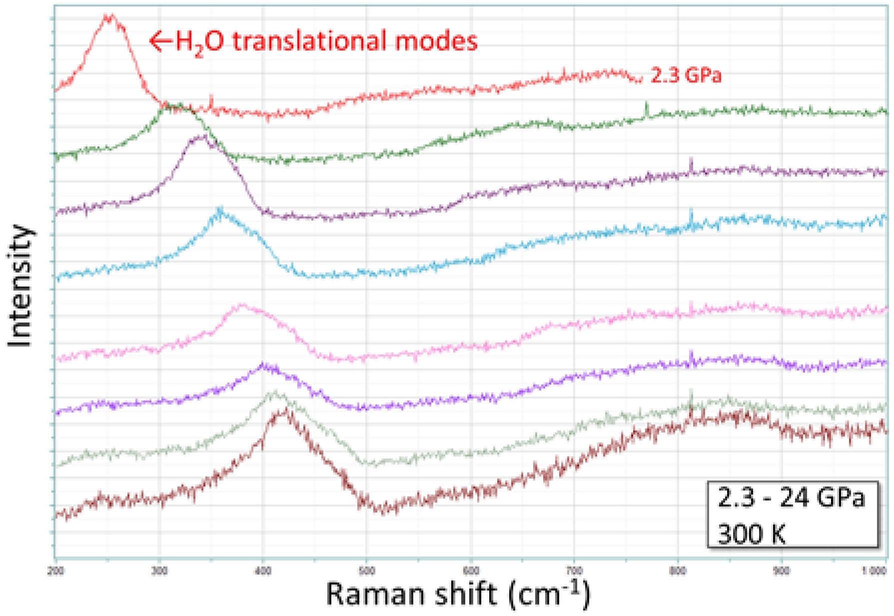

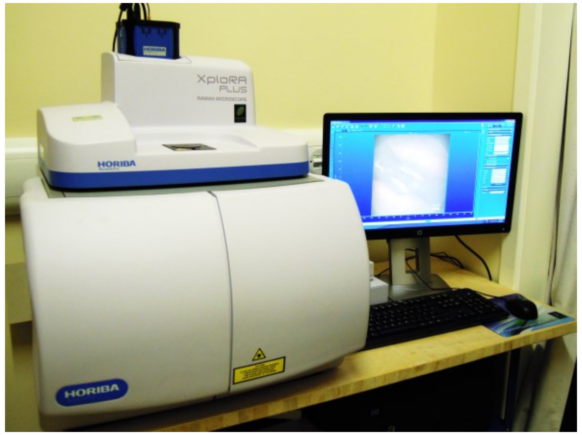

Raman microscope
XploRa (HORIBA)