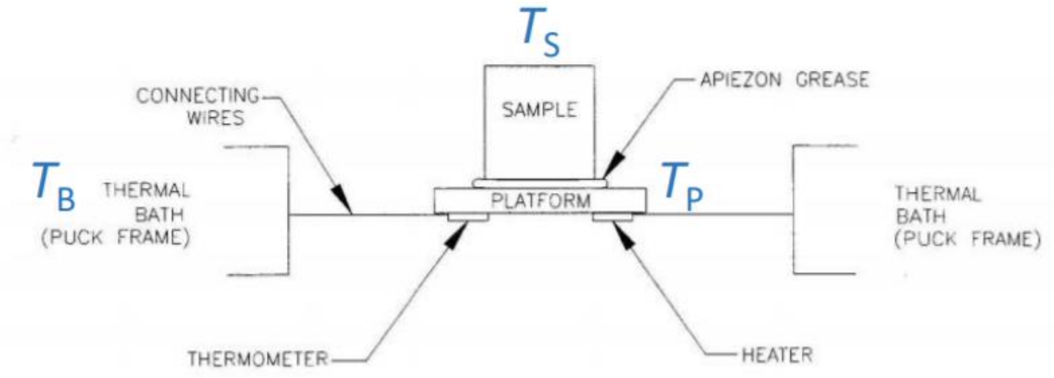
Experimental Implementation of Measurement of Specific Heat under Pressure

Phys 590B
Sangki Hong

Methods: Specific Heat Measurement

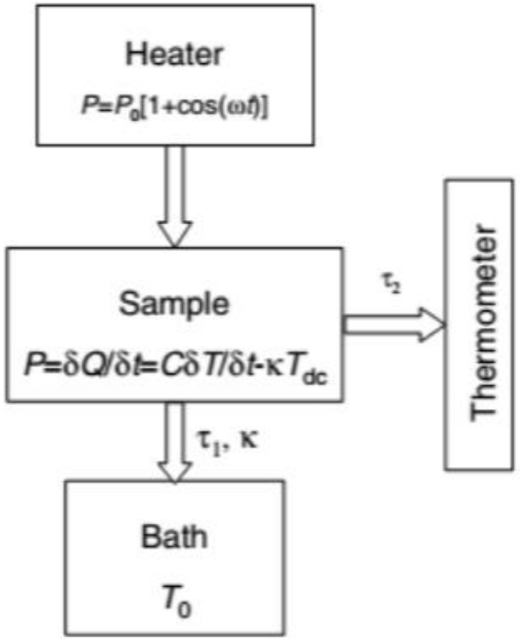
Relaxation



+ sophisticated temperature control and fitting software
+ good for flat and thin samples with reasonable thermal conductivity

- need to measure addenda (platform + grease) every time
- need to calibrate heater and thermometer in magnetic field
- need to shape your sample
- vertical ^{3}He platform may oscillate in magnetic field
- assembly is fragile
- measurements take long time

AC modulation

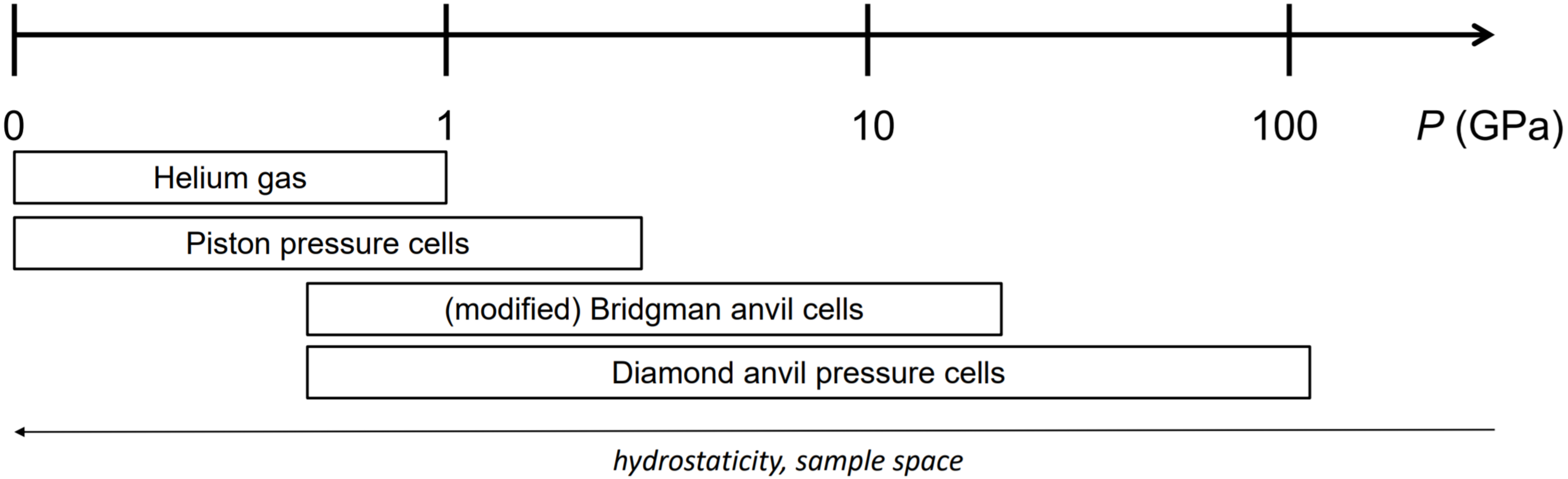


+ Fast and accurate (relative measurement)
+ Good for small samples
+ Easy to put on rotator

- Hard to get absolute values

Presentation from Elena Gati (Phys 590B)
Presentation from Sergey L. Bud'ko (Phys 590B)

Methods: High pressure



$CeRh_2Si_2$ (piston pressure cell + thermal relaxation)

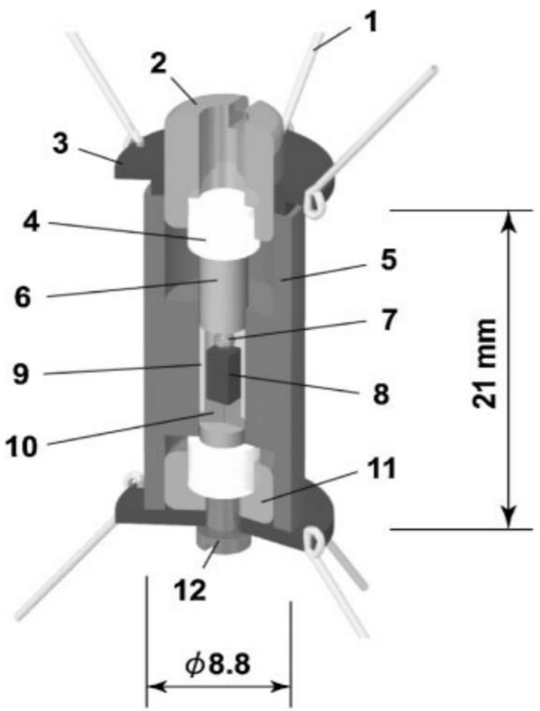


Fig. 1. Schematics of the miniature piston–cylinder pressure-cell with outer diameter 8.8 mm and length 21 mm; 1: lifting thread, 2,11: upper and lower lock nut, 3: swivel, 4: piston buckup, 5: CuBe cylinder, 6: piston, 7: Sn manometer, 8: sample, 9: Cu cell, 10: Cu cap, 12: mount screw.

- A piston-cylinder type pressure cell made of Be–Cu alloy

- Pressure medium: Fluorinert FC70 : FC77 = 1:1 mixture

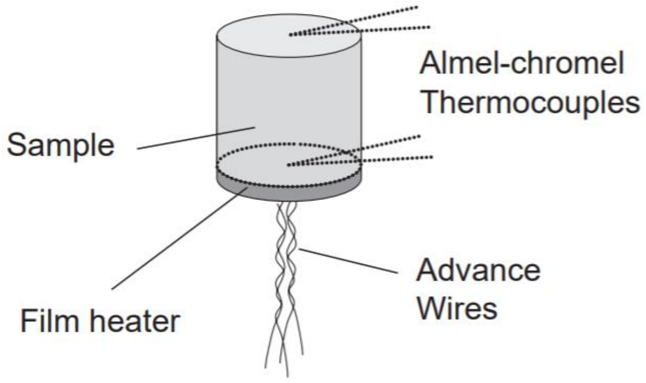


Fig. 1. Experimental arrangement of the sample, heater and thermocouples in the pressure cell.

- Two pairs of Alumel-chromel thermocouples (25 μmØ) were spot-welded on each bases of the cylindrical polycrystals of the sample (3 mmØ X 1.5 mm)

- A film heater of 350Ω was attached to the base.

- The sample temperature variation from a reservoir (pressure cell) temperature was directly measured by the thermocouples.

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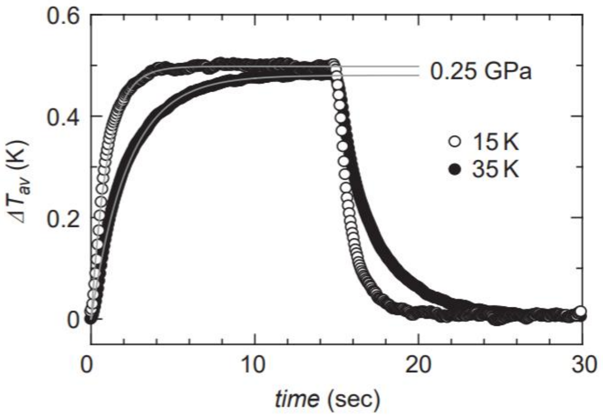

Fig. 2. Temperature variation ΔT_{av} versus time for the reservoir temperature of 15 and 35 K measured at 0.25 GPa. Dotted lines are the results of fitting (see text).

ΔT_{av} (temperature variation) was fitted by the thermal relaxation equation,

$$\boxed{\Delta T_{av} = A(1 - exp(-t/\tau))}$$

A = temperature difference between the reservoir and new-equilibrium sample-temperatures

τ = relaxation time.

$$\boxed{c = \tau P / A}$$

C = 50.39 J/Kmol for 35 K

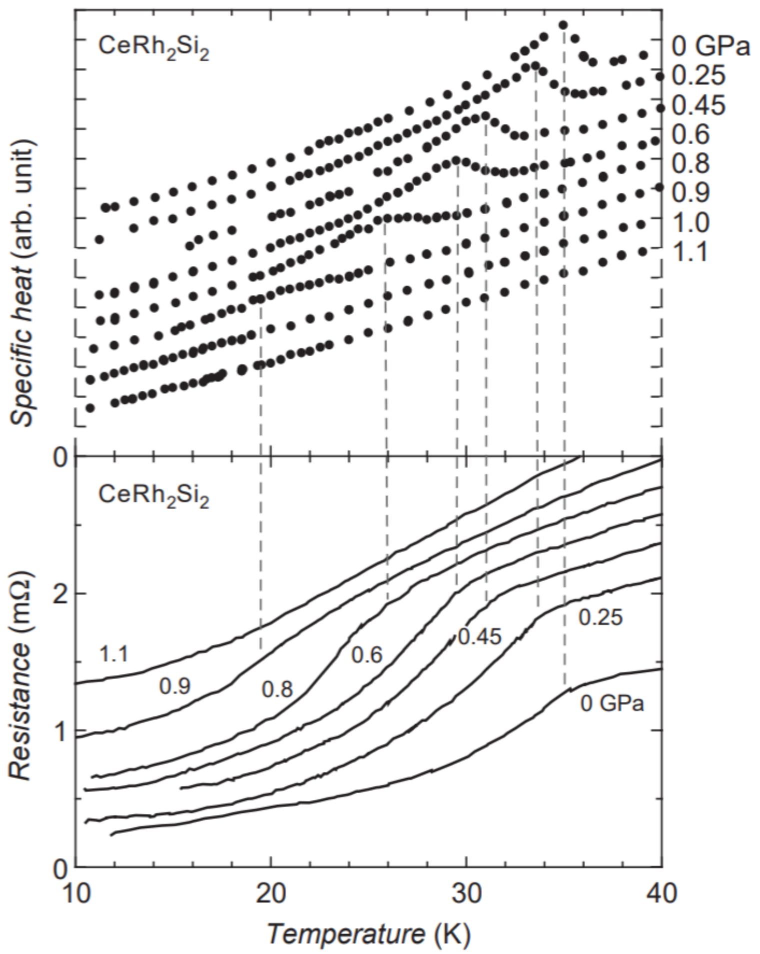

Fig. 3. Pressure dependence of specific heat and electric resistance for $CeRh_2Si_2$. The dashed lines indicate the correspondence between the temperatures of the specific heat peak and the resistance kink.

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$CeRh_2Si_2$ (piston pressure cell + thermal relaxation)

Heat capacity of Fluorinert

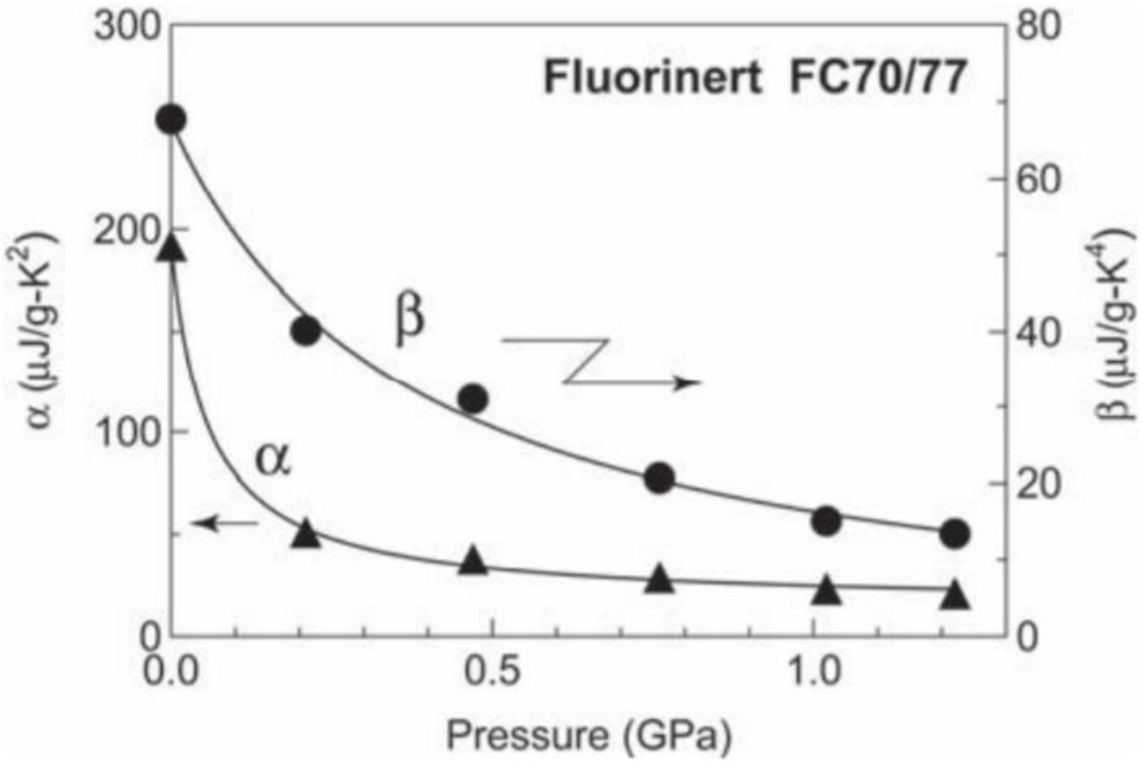

Fig. 2. Pressure dependencies of constant values of α, β.

Heat capacity of Fluorinert under any pressures, the curves fitted by an asymptote equation below.

$$\boxed{f(p) = \frac{a}{p+b} + c}$$

a, b and c = constants
p = applied pressure

$$C = \alpha T + \beta T^3$$

C = specific heat
T = absolute temperature
α and β = constants

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$CePd_2Si_2$ (piston pressure cell + AC modulation)

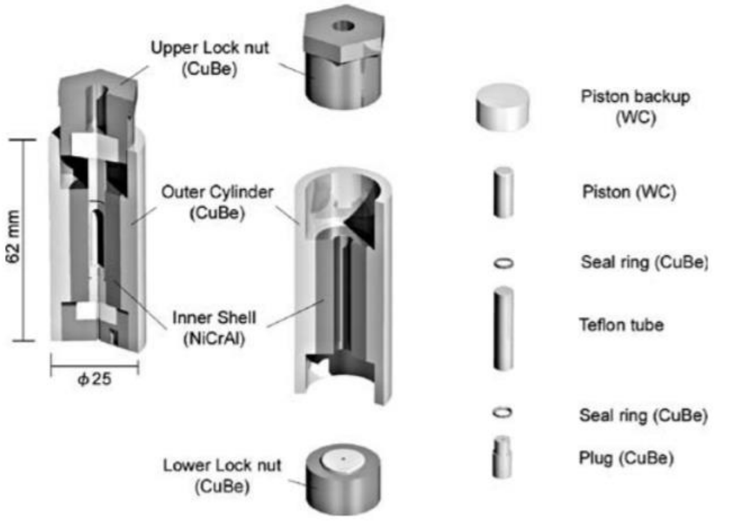

Fig. 1. Cross section view of the hybrid high pressure cell.

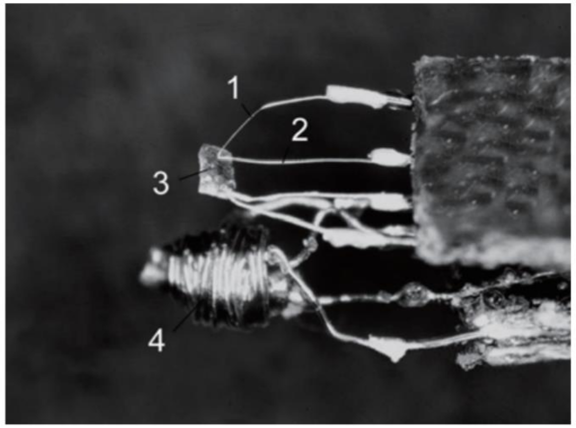

Fig. 4. Setting for AC calorimetric measurement.
1-AuFe(0.07)25 μm-wire, 2-Au20 μm-wire, 3-sample and 4-coil for manometer.

High pressure by piston pressure cell
- Non-magnetic WC piston
- Ni-Cr-Al inner cylinder inserted into the Cu-Be outer sleeve.
- Dimensions of cylinder: 62 mm (length), 25 mm (O.D), and 5 mm (I.D)
- Pressure medium: Daphne oil 7373.

AC calorimetric measurement
- Sample size: 0.3 x 0.5 x 0.15 mm^3 .
- Heater: 20 μm Au wire
- Thermocouple: 25μm Au/Au:Fe(0.07%)
- Spot-welded directly to the sample.

The pressures determined by measuring the temperature dependence of AC susceptibility for the superconducting transition of Sn.

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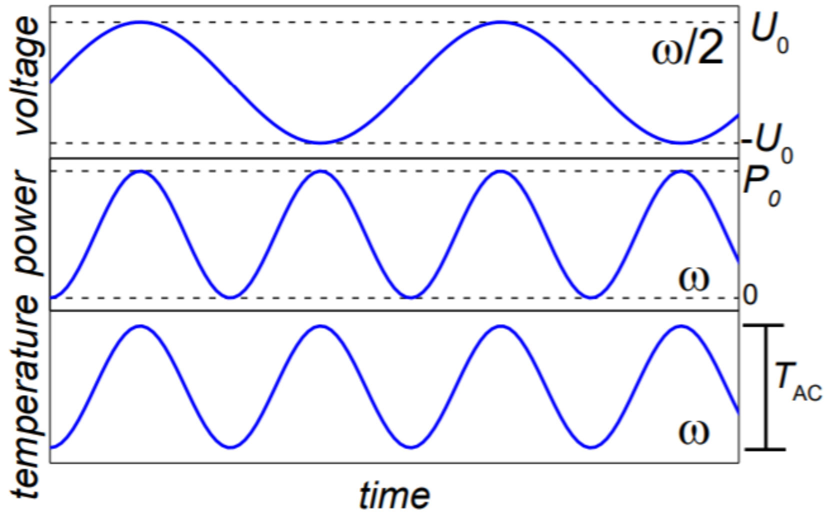
$CePd_2Si_2$ (piston pressure cell + AC modulation)



$$\boxed{T_{AC} \sim 1/C_S}$$

- AC current → sample through 20 μm Au wire.

- AC Joule heating power P generated into sample.

- Frequency of P is proportion to 2ω. (ω is that of the current.)

- P modulates the temperature of sample with same frequency 2ω.

- Measured the temperature modulation with 25μm Au/Au:Fe(0.07%) thermocouple by Lock-in Amp.

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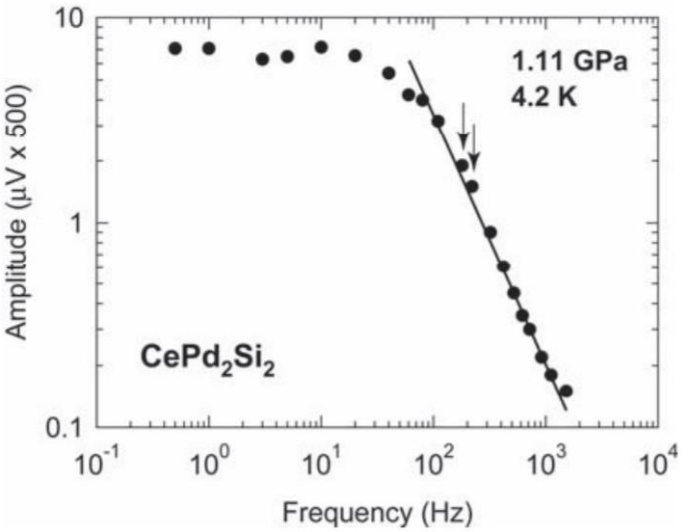
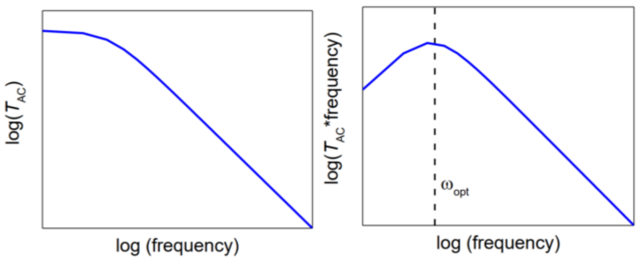


Fig. 5. Frequency dependence of Lock-in-voltages at 4.2 K under pressure of 1.11 GPa.

$$T_{AC} = P_0/(2\omega C) * (1+\omega^2\tau_2^2 + 1/(\omega^2\tau_1^2)+\text{const.})^{-1/2}$$

$$C = C_S + C_H + C_\Theta$$

If $\omega\tau_2 << 1$ and $\omega\tau_1 >> 1$: $T_{AC} = P_0/(2\omega C)$



$$\boxed{\tau_1 = C_S/\lambda_S}$$

Need to choose measurement frequency, then T_{AC} independent of λ's!

- If frequency too high, Joule heating power cannot not pass through the sample due to decoupling of sample from the thermocouple.

- If frequency too low, Joule heating power fades away to the circumstance through wires and pressure medium.

- suitable frequency range : 100 Hz to 1000 Hz.

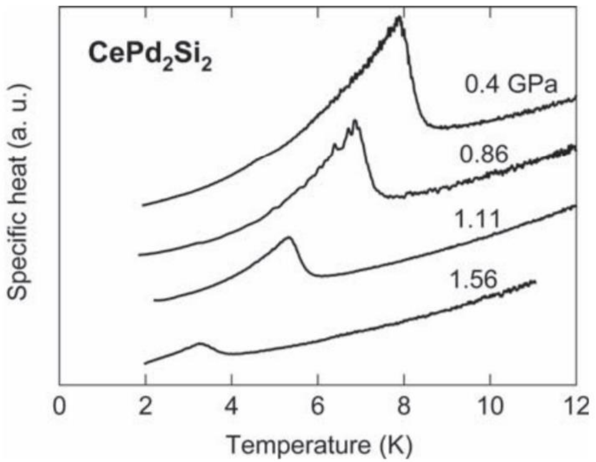


Fig. 8. Estimated heat capacity of $CePd_2Si_2$ as a function of temperature under several pressures.

- Neel temperature shift to lower temperatures and broadened.
- Anomaly almost disappears at 1.56 Gpa.

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$EuFe_2As_2$ (cubic anvil cell + AC modulation)

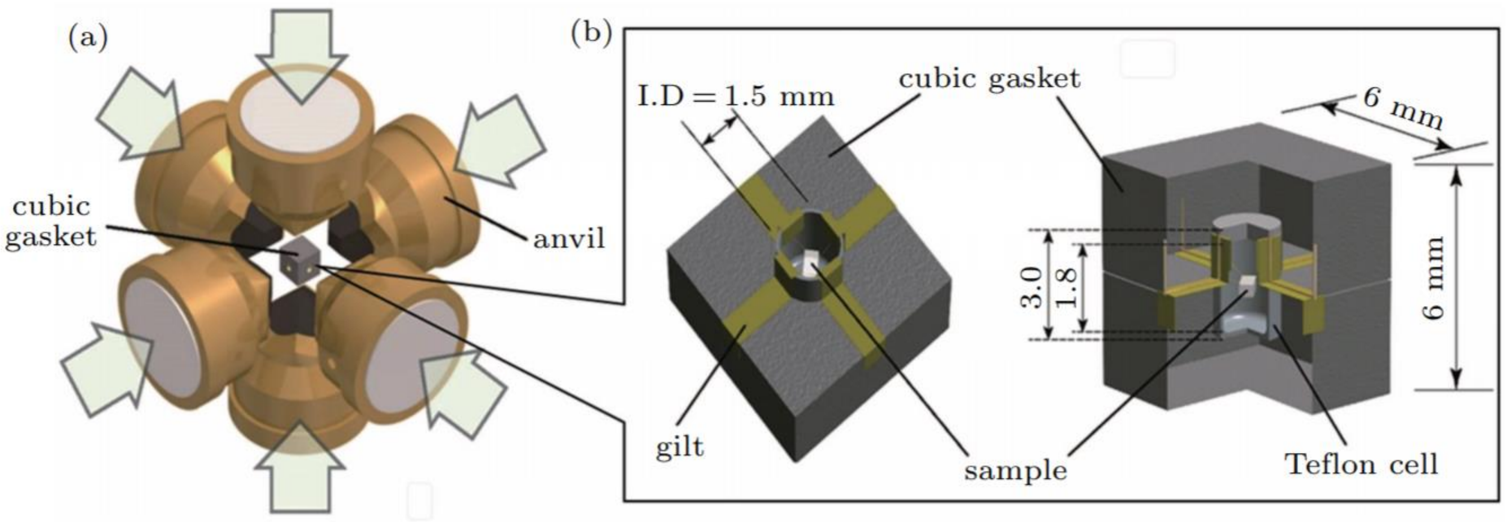

Fig. 1. (color online) (a) Illustration of the operation principle of the CAC in which six anvils converge onto the cubic gasket from three orthogonal directions. (b) Sample configuration in the cubic gasket with the typical dimensions of a gasket and Teflon capsule used for the 4 mm anvils.

High pressure by a cubic anvil cell
- Six tungsten carbide anvils.
- Pressure calibration: measurements of resistive change of Bi and Te structural phase transitions at room temperature.
- Pressure medium: Daphne7373
- Gasket: pyrophyllite
- Pressure up to 8 GPa.

AC calorimetric measurement
- Sample size: 0.3 x 0.5 x 0.05 mm^3 .
- Heater: 20 μm Au wire
- Thermocouple: 25μm Au/Au:Fe(0.07%)
- Spot-welded directly to the sample.

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$EuFe_2As_2$ (cubic anvil cell + AC modulation)

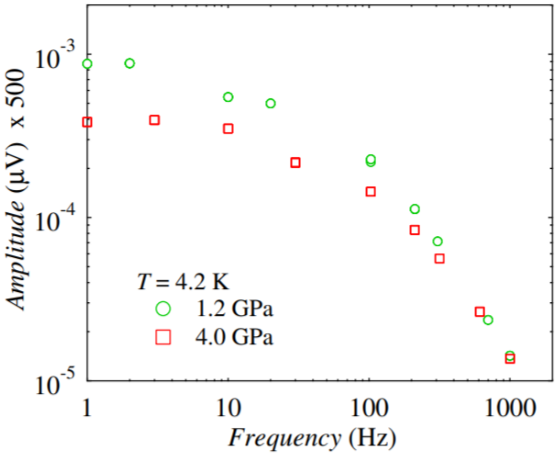

Figure 1. Frequency dependence of Lock-in-voltage measured at 4.2 K at selected pressures.

- Heater power P: frequency ω
- Temperature modulation of sample at frequency 2ω by lock-in amplifier.
- 200 Hz and 800 Hz

$$C_{ac} = P/\omega T_{ac}$$

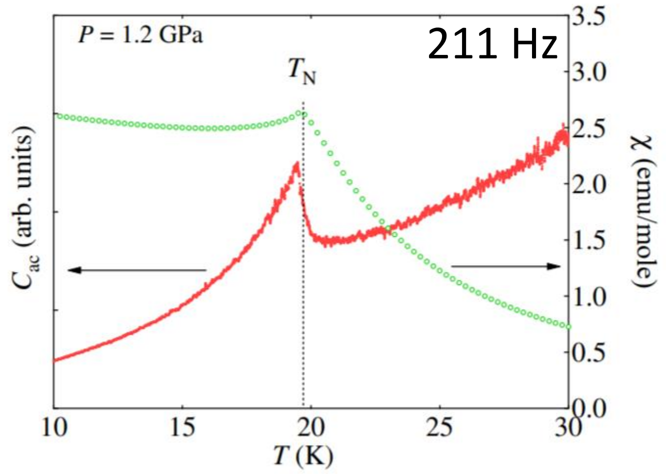

Figure 2. Temperature dependence of ac specific heat and magnetization of $EuFe_2As_2$ at 1.2 GPa.

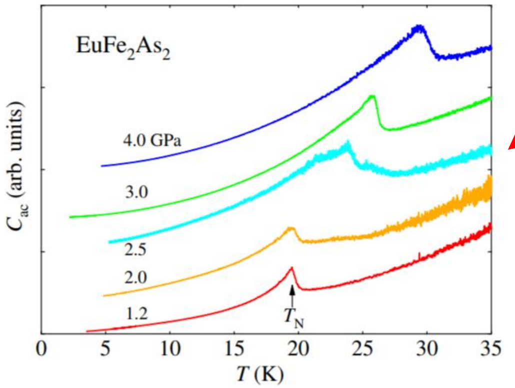

Figure 3. Temperature dependence of C_{ac} for $EuFe_2As_2$ at selected pressures. The curves are shifted vertically for clarity.

- critical pressure of superconductivity (2.5 Gpa)
- C_{ac} exhibits double transition.
- AF ordering of Eu^{2+} moments still occurs in the superconducting phase.

Anomaly shifts to higher temperatures with increasing pressure

- The transition temperature corresponds to a jump in magnetization measurement.
- Anomaly in C_{ac} at ~ 20 K: antiferromagnetic (AF) ordering of Eu^{2+} moments.

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Thank you.