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

- Helium gas
- Piston pressure cells
- (modified) Bridgman anvil cells
- Diamond anvil pressure cells

hydrostaticity, sample space
CeRh$_2$Si$_2$ (piston pressure cell + thermal relaxation)

- A piston-cylinder type pressure cell made of Be–Cu alloy
- Pressure medium: Fluorinert FC70 : FC77 = 1:1 mixture
- 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.

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 backup, 5: CuBe cylinder, 6: piston, 7: Sn manometer, 8: sample, 9: Cu cell, 10: Cu cap, 12: mount screw.

Physica B, 2005, 359-361, 172
CeRh$_2$Si$_2$ (piston pressure cell + thermal relaxation)

$\Delta T_{av}$ (temperature variation) was fitted by the thermal relaxation equation,

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

$A = \text{temperature difference between the reservoir and new-equilibrium sample-temperatures}$

$\tau = \text{relaxation time}$.

$$c = \tau P/A$$

C = 50.39 J/Kmol for 35 K
CeRh$_2$Si$_2$ (piston pressure cell + thermal relaxation)

Heat capacity of Fluorinert

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

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

a, b and c = constants
p = applied pressure

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

C = specific heat
T = absolute temperature
$\alpha$ and $\beta$ = constants

Fig. 2. Pressure dependencies of constant values of $\alpha$, $\beta$.
CePd$_2$Si$_2$ (piston pressure cell + AC modulation)

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.

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.
CePd$_2$Si$_2$ (piston pressure cell + AC modulation)

- AC current $\rightarrow$ sample through 20 $\mu$m Au wire.

- AC Joule heating power $P$ generated into sample.

- Frequency of $P$ is proportion to $2\omega$. ($\omega$ is that of the current.)

- $P$ modulates the temperature of sample with same frequency $2\omega$.

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

$$T_{AC} \sim 1/C_S$$
CePd$_2$Si$_2$ (piston pressure cell + AC modulation)

- 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.

- Neel temperature shift to lower temperatures and broadened.
- Anomaly almost disappears at 1.56 GPa.
EuFe$_2$As$_2$ (cubic anvil cell + AC modulation)

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.
EuFe$_2$As$_2$ (cubic anvil cell + AC modulation)

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

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

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

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

Figure 2. Temperature dependence of ac specific heat and magnetization of EuFe$_2$As$_2$ at 1.2 GPa.

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

Anomaly shifts to higher temperatures with increasing pressure
Thank you.