Basic Training in Diamond Anvil Cell Operation

Basic skills you need to master:
Mounting anvils
DAC alignment
Gasket preparation
Sample Loading
- Powder sample
- Single crystal sample
- Sample in fluid transmitting medium
- Sample in gas transmitting medium

Other skills:
- Operation of Ruby/Raman system
- Operation of cryogenic loading system
- Fitting spectral peaks

I. Pressure measurement using ruby fluorescence

Steps:
(i) Prepare a DAC with 300 or 500 um culets

(ii) Load cell with NaCl powder and distribute a few ruby spheres across the sample

(iii) Tighten cell slowly while recording angular screw rotation in ~15 degree intervals

(iv) Under microscope, meas. the distancve of the rubies from the center of the culet. Recheck these positions as pressure increases to determine whether they move.

(v) Meas. ruby fluorescence across the sample at ~2-3 GPa intervals. Measure fluorescence for several rubies across the sample chamber at each step.

(vi) After reaching the maximum pressure, decrease the pressure and make ruby measurements on unlaoding.

(vii) Perform peak fitting on the ruby peaks and make the following plots:

a. Pressure vs # of screw turns. Are the loading and unloading curves similar or not?
b. Pressure profiles across the sample as a function of P (or screw turns)
c. Full width at half maximum (FWHM) for ruby R1 peak with pressure

Questions:
What is the appropriate ruby scale to use for this exercise?
What is the accuracy of this ruby scale?

What is the precision with which you can measure the ruby pressure shift?

What is the total shift in ruby wavelength over your pressure measurement range?

Can you distinguish the R1 and R2 peaks at high pressures?
Do you require 1 peak or 2 peak for peak fitting?

Do the following: Increase pressure by a few GPa and measure it. Then remeasurehe pressure at 1 hour intervals for several hours and then leave the cell overnight and measure it again. How does the pressure change over time? How long is required before the pressure stabilizes? Did the gradient across the sample change?

How does temperature affect ruby measurements?
What error in pressure is introduced if your measurements are made at 5 K higher or lower temperature?

II. Testing DAC alignment and visualizing phase transitions: AgI

(i) Using a DAC with 500 um culets, align the diamonds but do not prepare a gasket.
(ii) Spread AgI powder across the culet
(iii) Gently compress the anvils together without a gasket. At ~10 GPa, AgI undergoes a polymorphic phase transition (NaCl→CsCl-type) that produces a change in refractive index that is optically visible. As the pressure increases, the color change should be first observed near the center of the sample and then move outward following the 10 GPa isobar as the peak pressure increases above 10 GPa. (There is also a narrow phase transition at 0.3 GPa which can be seen as a ring at the edge of the sample)
(iv) Observe the shape of the isobar. If the diamonds are well aligned, the phase transition should be distributed symmetrically about the loading axis of the cell.
(v) **Note: Use caution.** The number of screw turns required to produce a given pressure without a gasket is less than it is with a gasket as the gasket absorbs some of the load. Furthermore, the diamonds will break if used without a gasket above about 30 GPa. To avoid this, make sure the phase transition region does not extend out from the center of the culet by more than 2/3 of the radius of the culet.

Questions: Describe the color change you observe? Look up in the literature to find a physical explanation for this color change.

Ref: Jephcoat et al., in Hydrothermal Experimental Techniques. See page 484.
Bassett and Takahashi, Silver iodide polymorphs, Am. Mineral., 50, 1576, 1965

III Magic Trick: Make CaF2 disappear

(i) Prepare a cell with 500 um culets.
(ii) Load a small single crystal of CaF_2 into the sample chamber. This is good practice for learning to handle small (and sometimes very rare and precious) samples.
(iii) Load an alcohol (methanol-ethanol) pressure medium
(iv) When increasing the pressure, the CaF_2 crystal gradually gets smeared out and becomes invisible. At what pressure does this occur?

Questions:
What is happening here?

IV Making warm ice

(i) Prepare a cell with 500 um culets. Fill the sample chamber with H_2O but leave a small air bubble.
(ii) Increase pressure slowly. Upon compression, the air buble will shrink and then disappear. At 1 GPa, the water freezes to form ice VI.
(iii) Decrease pressure to get ice crystals coexisting with H_2O.
(iv) Experiment with growing single crystals of ice by slowly increasing the pressure. Experiment with taking photos of the single crystals that you grow.
(v) By further increasing pressure to 2.0 GPa, ice VI will transform to ice VII

V Measuring Diamond Fluorescence

(i) Select one of the synthetic anvils and measure its fluorescence. Determine the fluorescence ratio. You should be able to reproduce the value recorded in the diamond inventory.
(ii) SNow slect an anvil from our collection for which there is no fluorescence record. Record the diamond fluorescence spectrum of this anvil.
(iii) Determine the fluorescence ratio; make a nice plot with the ratio labeled on the plot, and add it to the diamond inventory for this anvil.
(iv) Repeat for as many diamonds as you can find.

Questions
Is your diamond useful for Raman spectroscopy?

VI Gem identification with Raman Spectroscopy

(i) Obtain an unknown gem from the Princeton gem collection.
(ii) Measure the Raman spectrum
(iii) Using the Rruff database, identify the gem
(iv) What other properties of the gem can you determine from a comparison to the Raman database?

(v) Make one of more well-labeled plots that demonstrate your results, and add your findings to the gem database in the lab.

***VII Pressure measurement using ruby fluorescence (Advanced)**

Steps:
(i) Prepare a DAC with 300 or 500 um culets
(ii) This exercise will require you to prepare and measure 3 separate samples:
    a. ruby spheres distributed in NaCl powder
    b. ruby spheres distributed in Meth-eth liquid
    c. ruby spheres distributed in Ar medium
(iii) For each sample, measure ruby signals up to the pressure limit as in the previous exercise.
(iv) For each sample, also measure the diamond Raman signal and estimate pressure from diamond Raman. (Be sure to use highest resolution grating for accurate P determination)
(v) What is the effect of heating the sample? For a sample showing considerable differential stress (e.g. meth-eth medium), heat the sample in an oven to ~100 C. Then measure the pressure – by how much did the cell relax? Compare ruby linewidths before and afterheating.
(vi) Analyze the data addressing the following:
    a. How do the load/unload curves depend on pressure medium?
    b. How does R1 peak with vary with P media upon loading?
    c. How does the P gradient across the sample depend on the medium?
    d. To what P can the R1-R2 peak widths be clearly resolved for each media?
    e. How accurate is the pressure determined from diamond Raman compared to ruby for diffferent media?

Questions:

What is the appropriate ruby scale for each pressure medium?
How was the ruby scale calibrated?

Can you use your data to determine the freezing pressure of your meth-eth mixture?