# Additional apparatus notes for Ferroelectricity Lab

#### Cooling the sample chamber with ice water:

- (1) The experiment is done between roughly  $0^{\circ}$  C and  $60^{\circ}$  C, so ice water in the dewar is sufficient for cooling purpose. There is a mark on the inside wall of the dewar indicating the maximum fill level in order to avoid submerging the apparatus in the water;
- (2) It takes a few hours before the sample chamber is cooled down through the copper rod, so you may want to start the cooling in the morning on the day of the experiment.

#### Temperature control with a thermocouple and an OMEGA-CN7600 controller:

Once the sample chamber has been cooled for a few hours without any additional heating, you can use the front panel of the OMEGA-CN7600 Controller to set the temperature.

- (1) Normally, the display next to "**PV**" indicates the **present value**; the display next to "**SV**" indicates the **set value**;
- (2) To change the set value, press "INDEX" on the Controller and the display next to "PV" shows the current set value (or set point). You change this value by pressing "up" or "down" arrows. Once you reach the desired set value and would like to make it the new set value, press "ENTER";
- (3) then press "INDEX" to return to the normal display;
- (4) Turn the variac dial from 0 to 40 volts, and you should observe the temperature (the present value) begin to rise slowly until it reaches the new set point.

**Warning**: DO NOT set the temperature over  $60^{\circ}$  C. The ferroelectric crystal is damaged if left for a long period of time above room temperature. <u>BE</u> SURE THE HEATER IS OFF WHEN YOU LEAVE.

### **Spontaneous Polarization** (**P**<sub>s</sub>) measurement with **Keithley 610B Electrometer**

- (1) Connect the thermocouple (Type T) plug and the ac heating power plug to the sample assembly;
- (2) Cool the sample chamber with ice water and at the same time set the temperature (SV) to 54°C and turn on the heater on;
- (3) Once the sample reaches 54°C so that it is in normal state instead of ferroelectric state, connect a dc power supply across the sample (between "S" and "D");

Initially the applied voltage should be zero;

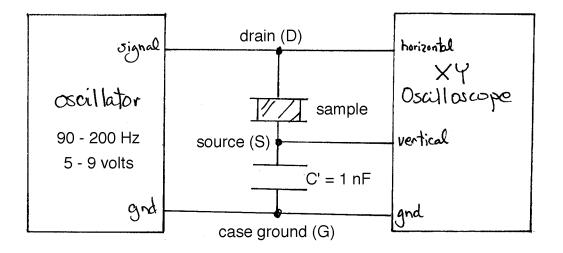
- (4) Slowly turn up the voltage from zero to 50 volts;
- (5) With the 50-volt power supply connected, cool the sample down to 5°C;
  (Notes: you may just turn off the heating and let the sample to be cooled to the lowest temperature possible);
- (6) Once 5°C or the lowest temperature is reached, turn down the voltage across the sample from 50 volts to zero, and disconnect the power supply;
- (7) Short out the sample by connecting "S" to "D" on the sample assembly;
- (8) Note:

Spontaneous polarization **P**, in the sample, aligned by the 50-volt applied voltage as the sample is cooled through the transition temperature, remains even when the applied voltage is reduced to zero. This is a property of a sample is in ferroelectric state. Since the potential drop across the sample is zero, the total charge (free and bound) on each side of the sample is also zero. However corresponding to **P**<sub>s</sub>, there is a bound surface charge with a density equal to the magnitude of  $\mathbf{P}_{s}$  on one side the sample and an equal and opposite bound charge on the other. They each attract an equal amount of free charge. Now if you heat the sample up from 5°C in ferroelectric state to 54  $^{\circ}$ C in normal state, **P**<sub>s</sub> slowly diminishes as driven by the thermal fluctuation. This causes the bound charge to diminish as well. The excess free charges held by the bound charges can now move about if the two sides of the sample is connected. By measuring the amount of free charge that flows from one side to the other with a serially connected electrometer, you can deduce how P<sub>s</sub> changes with the sample temperature. To do so,

- (9) Connect the two sides of the sample to Keithley 610B Electrometer, set the sensitivity on "10-7 Coul" scale;
- (10) Increase the sample temperature from 5°C to 54°C, and record the free charge reading on the electrometer as a function of the sample temperature. (Note: you may need to adjust the multiplier dial to change sensitivity).

## DO NOT CONNECT THE 50V POWER SUPPLY TO THE ELECTROMETER!

Hysteresis loop measurement: (Displacement versus Electric field)



- (1) When the sample with a capacitance  $C_s = C_{s0}$  is connected in series with another capacitor of much larger value, C' = 20 nF, the total capacitance between "D" and "G" is  $C_t = C_s C' / (C_s + C') - C_s$ . When a voltage  $V_t$  is applied between "D" and "G", it falls mostly across the sample so that  $V_t$  is proportional to the electric field (E) in the sample; (Note: this is the case only when the sample temperature is at least a few degrees from the transition temperature at around  $T_c \sim 49^{\circ} C$ );
- (2) The voltage across C' is given by  $V' = C_s V_t / (C_s + C') (C_s / C') V_t$ , or  $V' (C_{s0} / C') (V_t)$ .  $V_t$  is proportional to the displacement vector  $D = {}_0 E = {}_0 E + P$  in the sample. By plotting the voltage drop across C' (namely, between "S" and "G") versus the total voltage drop (namely between "D" and "G"), you have a measure of D versus E, or the hysteresis loop of the sample;
- (3) Connect the output of a function generator (HP-CFG280) across "D" and "G" to provide V<sub>t</sub>;
   Connect the same voltage to "Ch 1" of a Tektronix 2205 XY oscilloscope for the horizontal deflection;
- (4) Set the function generator output with Frequency: 90 Hz
   Peak-to-peak amplitude: 18 volts
   Waveform: Sawtooth
- (5) Connect V' between "S" and "G" to a **unit-gain isolation amplifier** so that the output impedance to C' is 66 mega-ohm instead of 1 mega-ohm for the oscilloscope. Then connect the output of the isolation amplifier to "Ch 2" of the oscilloscope for the vertical deflection;

(6) Set the "Sweep" to "XY", and you should see the hysteresis loop on the screen;

(**Question**: can you think of a way to determine the scaling factor between x and y axes so that the display of D vs. E is properly scaled ?)

- (7) You record and later analyze the hysteresis loop by using a PC-based data acquisition system:
  - Connect V<sub>t</sub> to "ACH0" on BNC-2090, and V' to "ACH1" on BNC-2090;
  - Connect the thermocouple output to "ACH2" on BNC-2090;
  - Run "Ferroelectricity\_2002\_TC.vi" on the C Drive in "Physics122Lab folder";
  - Set the "Sampling frequency" to "45000";
  - Set the "Number of points per waveform" to "500";
  - Set the "Number of data points shifted" to "0";
  - Set the "Number of waveforms to be averaged"  $N_{\rm w}$  to an integer larger than 0;
  - If you only want to look at the hysteresis loop, do not press "Save the data ?" button;
  - If you would like to save the data, press "Save the data ?" button before hit the run button;
  - At the end of each run, the panel display the heteresis loop and the averaged sample temperature during the measurement;
  - When a window pops up at the end of the run, save the data with a name and record the sample temperature for the run;

The saved data have three columns:

First column: Electric field

- Second column: Displacement
- Third column: Sample temperature (total of N<sub>w</sub> values)
- (Note: you need to perform the average of the N<sub>w</sub> values yourself);
- You may use the hysteresis loop data to compute the sample capacitance  $C_s$  as it is cooled down from 54°C, using  $V' = C_s V_t / (C_s + C') = V_t / (1 + C'/C_s)$ .
  - (Note: in this case,  $C_s$  may be as large as twice of C' = 1nF).