Laboratory Experiments on Ferroelectricity

Experiment 1

1. Task:

1. Set up a Sawyer-Tower circuit to measure ferroelectric hysteresis curves.
2. Check the D(E) curves for a capacitor, a resistor and an RC-circuit.
3. Measure the D(E) curves of a TGS crystal as a function of temperature from room temperature up to the Curie temperature in a run with rising and in a second run with falling temperature
4. Determine the coercitive field and the spontaneous polarisation in dependence on temperature.
5. Draw diagrammes of E_c, P_r and P_r^2 versus temperature.

2. Basics:

Sawyer-Tower Circuit for measurement of the hysteresis curve of the D(E)-dependence

For the measurement of the dielectric displacement D versus the electric field E of a ferroelectric sample a so called Sawyer-Tower circuit is used. The electric circuit is shown in figure 1.

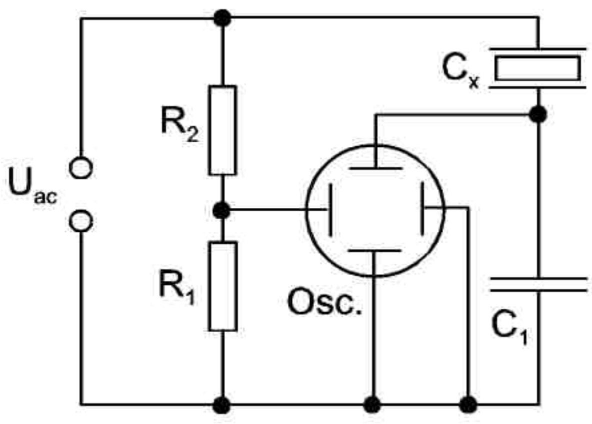

Figure 1: Sawyer-Tower circuit for measurement of the D(E)-dependence

The capacity C_x is the ferroelectric sample to be measured and C_1 is a reference capacitor of a well known capacity that is constant and independent of the applied voltage.
The measuring circuit is supplied with a sinusoidal voltage U_{ac}.
To display the D(E)-curves at the oscilloscope one needs at the x-channel a voltage that is proportional to the electric field inside of the sample C_x and at the y-channel a voltage proportional to the electric polarisation P or the dielectric displacement D of the sample (for

high values of P and D is D≅P). Both voltages have to be referred to the same potential, the mass potential.

The electric circuit from figure 1 can be explained as follows:

The input impedance of the oscilloscope is high enough in comparison to all resistances and impedances of the circuit so that we do not have to take it into account.

The x-channel of the oscilloscope gets a voltage that is proportional to the supply voltage U_0 via the voltage divider R1,R2. In the case of $C_1 >> C_x$ the voltage drop at C_1 can be neglected and therefore the voltage U_x at C_x is nearly equal to the supply voltage U_{ac}. Because of the proportionallity between voltage and electrical field strength it means that the voltage at the x-channel can be taken as a measure for the electric field inside of the sample C_x.

The y-channel gets a voltage that is proportional to the polarisation of the sample C_x. This can be explained as follows: The two capacities C_x and C_1 are connected in series and therefore always flows the same current through both elements. That means that both C_x and C_1, respectively carry the same charge Q at any arbitrary time. The capacity C_1 is independent of the applied voltage. Because of the proportionallity between voltage and charge the voltage at C_1 can be used as a measure for the charge of the capacity C_x.

Figure 2 shows the principle inner circuit of the Sawyer-Tower measuring box used for the experiment and its connection with the sample, the generator and the oscilloscope. The capacities can be switched with the help of Sw to get a compromise between a neglectible voltage drop at the capacity for exact measurement (high capacity) and a voltage drop that is as high as possible for easy measurement of the hysteresis curve (low capacity).

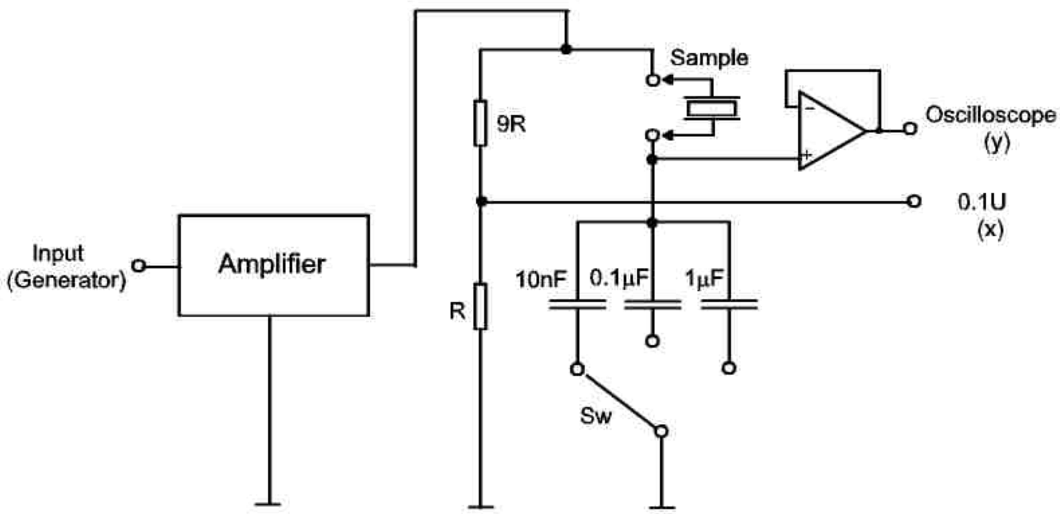

Figure 2: Principle inner circuit of the Sawyer-Tower measuring box and its connection with the sample, the generator and the oscilloscope.

Measurement of the sample temperature

The forward voltage of a common silicon diode flown by a constant current is linear dependent on temperature and has a coefficient of about –2mV/K. This fact can easily be used for temperature measurements. Figure 3 shows the principle:
A constant current of 1mA flows through the diode. The forward voltage is amplified by a factor of 50 to get a temperature coefficient of 0.1V/°C. The compensating input of the differential amplifier is connected with a voltage that is equal to the forward voltage at a temperature of 0°C to get a zero voltage at 0°C.
The temperature measuring diode is fixed on the copper sheet that carries the sample to be in good heat contact.
The amplified voltage can be measured at the output connectors of the temperature measurement box used in the experiment.

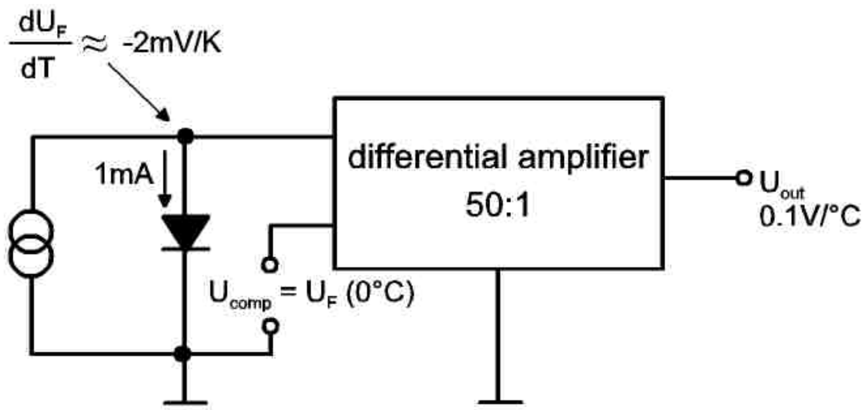

Figure 3: Principle of the temperature measurement with a silicon diode

3. Experimental Procedure

- Connect the sample in the small aluminium box, the generator and the oscilloscope with the Sawyer-Tower measuring box via BNC-cable as visible in figure 2.
  Be careful with the sample box because the sample is clamped with an spring bronze sheet and may be lost out of contact by mechanical shock.

- Connect the heater inside of the aluminium sample box (black and red connector) with the current supply. At the beginning keep the heating current swiched off.

- Connect the DIN female connector of the aluminium sample box with the DIN female connector of the temperature measuring box. Use the brown cable with the two DIN male connectors.

- Connect a digital hand held multimeter with the output of the temperature measuring box (black and red connectors). Use the 20V-DC-range of the multimeter.

- Connect the temperature measuring box with two 12V power supplies via the rear panel.

- Switch the multimeter on. The displayed voltage corresponds to the temperature of the sample. 0.1V correspond to 1°C.

- Switch the Sawyer-Tower measuring box and the generator on. Choose a frequency of about 40Hz and at first an amplitude of 0.1V (peak-peak).

- Increase the amplitude of the generator voltage step by step until you see a hysteresis curve at the oscilloscope that is well measurable.

- Now begin with the measurement of the temperature dependence of the hysteresis curve. Switch the heating current on and save the hysteresis curve on a storage stick in suitable temperature intervals to evaluate it later with a computer. The heating voltage should be not too high. Otherwise you get temperature measuring errors. Between room temperature and 47°C you should use a temperature increasing velocity of about 1°C/minute. As a recommended value use about 1.4 W for the heating power. From 47°C until crossing the Curie temperature the heating velocity should be very slow. Try to use about 0.2°C/minute. Be careful! Do not exceed a sample temperature of 75°C.

- After crossing the Curie temperature reduce the heating current and save the hysteresis curves for falling temperature in suitable intervals. Try to use again a cooling velocity of 0.2°C until you have crossed the Curie temperature downwards and after this use about 1°C/minute.

4. Evaluation

- Determine the values of the coercitive field and the spontaneous polarisation in dependence on temperature from the hysteresis curves saved on the memory stick. Use the sample dimensions given in table 1.

- Draw the diagrammes of E_c, P_r and P_r^2 versus temperature.

- Compare the experimental results with the prediction of the Landau theory. Is the ferroelectric phase transition of a TGS sample a first or second order transition?

Sample Number	Thickness (mm)	Area (mm^2)
1	1.40	31.33
2	1.25	30.34
3	1.10	31.51
4	1.10	29.50

Table 1: Sample dimensions

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Experiment 2

1. Task:

Measure the dielectric constant ε of a TGS crystal as a function of temperature from room temperature up to 70°C at a frequency of about 2 kHz in a run with rising and in a second run with falling temperature.

2. Basics

Determination of the Permittivity by Capacity Measurement

The dielectric constant (also called permittivity) ε of a dielectric material is measured mostly by determining the capacity of a parallel plate capacitor containing the material to be investigated.

The capacity of a parallel plate capacitor is given by

$$C = \varepsilon \cdot \frac{A}{d}$$

C – capacity of the parallel plate capacitor
A – area of the plates facing each other
d – distance of the two plates
ε – dielectric constant, also called permittivity

$$\varepsilon = \varepsilon_0 \cdot \varepsilon_r$$

ε_0 – dielectric constant or permittivity of the vaccum
ε_r – relative dielectric constant or relative permittivity of a special material

The principle of the capacity measurement used in the experiment is shown in figure 1 .

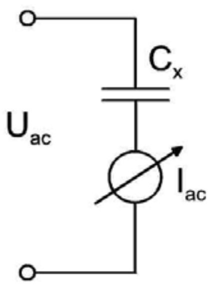

Figure 1: Principle of the capacity measurement

An AC voltage U_{ac} is connected with the capacity C_x to be measured. The resulting AC current I_{ac} is used as a measure for the unknown capacity. The capacity C_x is considered to be ideally. That means that the lost angle tanδ should be nearly zero. We take the absolute value of the complex current I_{ac} as a measure for the unknown capacity C_x according to $|I_{ac}| = \omega C_x |U_{ac}|$.

The AC current I_{ac} in figure 1 is measured with a current voltage converter shown in figure 2 .

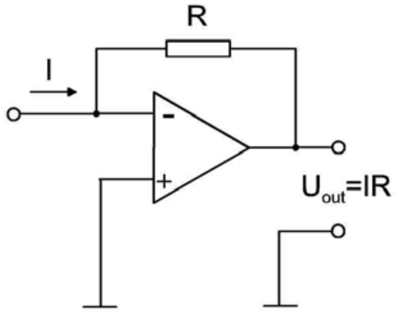

Figure 2: Electric circuit of a current voltage converter

The output voltage is linear dependent on the input current described by U_{out}=IR.

Using a current voltage converter instead of the measuring instrument in figure 1 we get to the electric circuit shown in figure 3. The absolute values of the complex input and output voltages U_{ac} and U_{out} obeye the following equation: $|U_{out}| = |U_{ac}| \omega R C_x$. Measuring the rms value of the AC voltage U_{out}, the resistance R can easily be calibratet in a way that the digits at a digital multimeter correspond to the value of the measured capacity C_x.

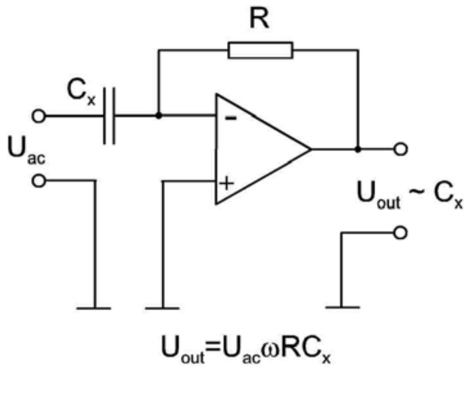

Figure 3: Current voltage converter for measurement of the capacity C_x.

Measurement of the sample temperature

The forward voltage of a common silicon diode flown by a constant current is linear dependent on temperature and has a coefficient of about –2mV/K. This fact can easily be used for temperature measurements. Figure 4 shows the principle:
A constant current of 1mA flows through the diode. The forward voltage is amplified by a factor of 50 to get a temperature coefficient of 0.1V/°C. The compensating input of the differential amplifier is connected with a voltage that is equal to the forward voltage at a temperature of 0°C to get a zero voltage at a temperature of 0°C.
The temperature measuring diode is fixed on the copper sheet that carries the sample to be in good heat contact.
The amplified voltage can be measured at the output connectors of the temperature measurement box.

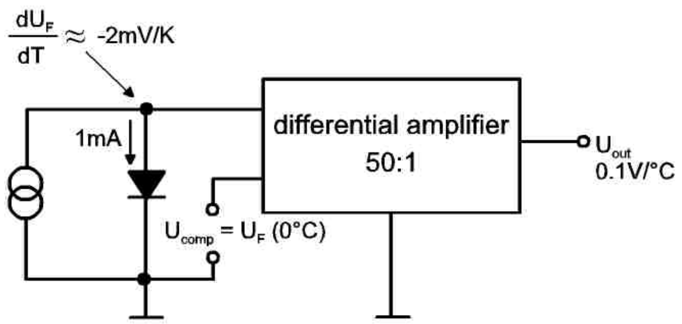

Figure 4: Principle of the temperature measurement with a silicon diode

3. Experimental Procedure

- Connect the two BNC connectors of the small aluminium sample box via BNC cables with the BNC connectors of the capacity measuring box. The two contacts of the sample are connected with the middle wire. The outer shell of the BNC connectors is only used for shielding.

- Connect a digital hand held multimeter with the output connectors of the capacity measuring box (black and red connectors). Use at first the 2V-AC-range of the multimeter.

- Connect the capacity measuring box with two 12V power supplies via the connectors at the rear panel.

- Switch the multimeter on. Now you should see on the multimeter a voltage that corresponds to the capacity of the sample. 1mV corresponds to 1pF.

- Connect the heater inside of the aluminium sample box (black and red connector) with the current supply. At the beginning the current supply is swiched off.

- Connect the DIN female connector of the aluminium sample box with the DIN female connector of the temperature measuring box. Use the brown cable with the two DIN male connectors.

- Connect a digital hand held multimeter with the output of the temperature measuring box (black and red connectors). Use the 20V-DC-range of the multimeter.

- Connect the temperature measuring box with two 12V power supplies via the connectors at the rear panel.

- Switch the multimeter on. Now you should see on the multimeter a voltage that corresponds to the temperature of the sample. 0.1V corresponds to 1°C.

- Now begin with the measurement of the temperature dependence of the sample capacity. Switch the heating current on and measure the capacity in intervals of about 1°C. The heating voltage should be not too high. Otherwise you get temperature measuring errors. Between room temperature and 47°C you should use a temperature increasing velocity of about 1°C/minute. As a recommended value use a heating voltage of about 5V. In the region between 47°C and about 50°C (until you notice that the Curie temperature is passed) the heating velocity should be very slow. Try to use about 0.2°C/minute and to measure in intervals of about 0.2°C. If you have passed the Curie temperature you can increase the heating velocity again to about 1°C/minute. Be careful! Do not exceed a sample temperature of 75°C.

- After reaching 70°C reduce the heating current and measure the sample capacity for falling temperature again in intervals of 1°C. Try to use again a cooling velocity of 1°C/minute far away from the Curie temperature and 0.2°C/minute between 50°C and about 47°C until you have passed the Curie temperature. After passing the Curie temperatur you can further decrease the heating current and increase the cooling velocity again to about 1°C/minute.

4. Evaluation

- Draw a diagramm of the measured permittivity ε in dependence on increasing and decreasing temperature. Use the sample dimensions given in table 1.

- Check the Curie-Weiss law for increasing and decreasing temperature by drawing regression lines of the inverse permittivity $1/\varepsilon$ versus temperature.

- Compare the experimental results with the predictions of the Landau theory. Is the ferroelectric phase transition of a TGS sample a first or second order transition?

Sample Number	Thickness (mm)	Area (mm^2)
1	1.40	31.33
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Table 1: Sample dimensions