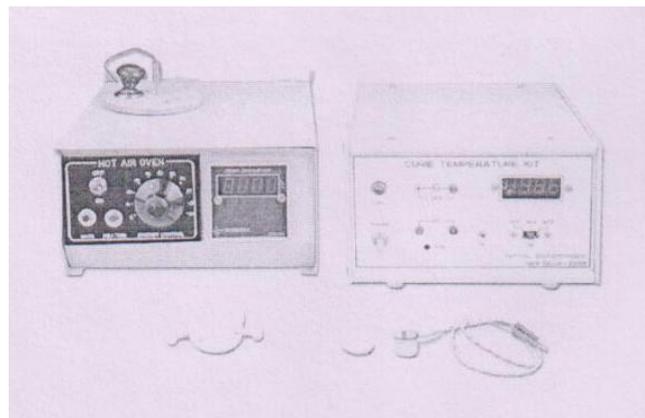


INSTRUCTIONAL MANUAL



CURIE TEMPERATURE MEASUREMENT (For Ferroelectric Materials)



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Curie Temperature Measurement

OBJECTIVE: Measurement of Curie Temperature of given Ferroelectric Material using Experimental kit.

INTRODUCTION

Ferroelectric materials exhibit electric dipole moment even in the absence of an external electric field. Ferroelectric materials are of theoretical and technical interest as they have unusually high and unusual temperature dependent values of the dielectric constant, the piezoelectric effects etc. Ferroelectricity usually disappears above a certain temperature called the transition (or Curie) temperature. Knowledge of the Curie temperature and the variation of the dielectric constant below and above the Curie temperature is of interest to the physicists and the engineers.

Probably the best-known ferroelectric material is barium titanate BaTiO_3 ; it is a representative of the so-called oxygen octahedron group of ferroelectric materials. The reason for this name is that above the Curie temperature ($\theta = 120^\circ\text{C}$), BaTiO_3 corresponds to the cubic structure presented in Fig.1. In this structure, the Ba^{2+} ions occupy the corners of a cube; the centers of the cube faces are occupied by O^{2-} ions. The oxygen ions form an octahedron, at the center of which the small Ti^{4+} ion is located. The Ti^{4+} ion is considerably smaller than the space, which is available inside the oxygen octahedron. It thus brings with it a high ionic polarizability for two reasons: (a) it has a charge of $4e$ and, (b) it can be displaced over a relatively large distance. This may be the explanation for the occurrence of spontaneous polarization in BaTiO_3 .

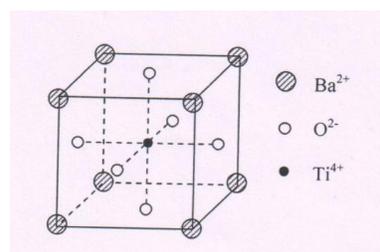


Fig.1

There is an intimate relationship between the ferroelectric properties and the atomic arrangement in ferroelectric materials. Above 120°C , BaTiO_3 has the cubic structure indicated in Fig.1. When the temperature is lowered through the critical temperature of 120°C , the material becomes spontaneously polarized and at the same time the structure changes. The direction of spontaneous polarization may lie along any of the

cube edges, giving total 6 possible directions for the spontaneous polarization. Along the direction of spontaneous polarization of a given domain, the material expands, whereas perpendicular to the polarization direction it contracts. Thus, the material is no longer cubic, but corresponds to a so-called tetragonal structure.

EXPERIMENTAL DESCRIPTION:

Front panel comprises of

- i) Digital Voltmeter (DVM) to measure the voltage across the dielectric cell (DC) or standard capacitor (SC).
- ii) Switch S_1 to select di-electric cell or standard capacitor.
- iii) Switch S_2 to select one of the standard capacitors SC_1 , SC_2 or SC_3 .

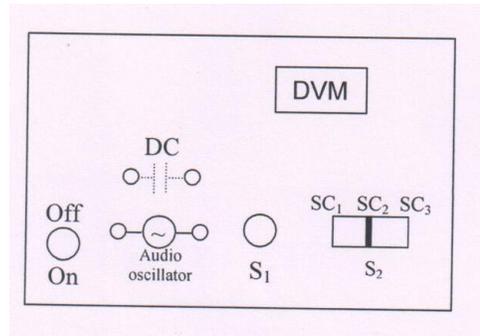


Fig.2.

Dielectric Cell:

Dielectric cell consists of two 1” dia. Gold plated brass discs fitted in between the cell holder (Teflon plates). Keep the ferroelectric sample in between the metal plates and tighten the three screws such that sample fits in between the metal plates without any air gap.

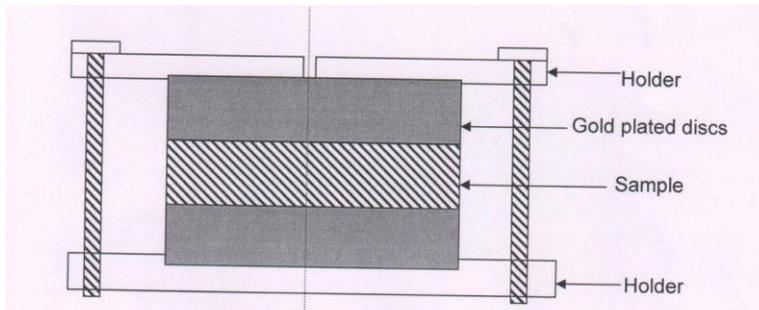


Fig.3

IMPORTANT: Dielectric cell (metal discs) and sample should be coaxial. Do not apply extra pressure on screws as that may damage the sample.

FORMULATION

In this experiment a LC circuit is used to determine the capacitance of the dielectric cell and hence the dielectric constant. The circuit details are shown below:

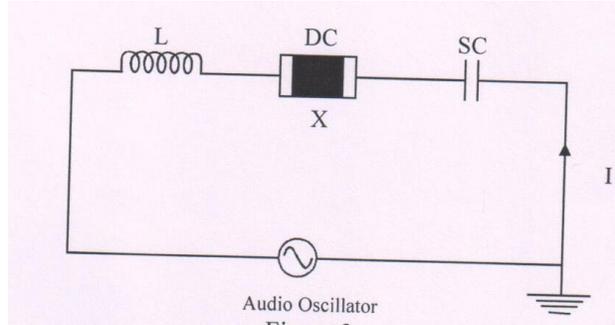


Fig. 4

DC: Dielectric cell, **SC:** Standard capacitor, **L:** Inductance, **X:** Ferroelectric sample

The dielectric cell DC is placed in a tubular furnace, which is fed by a variable ac power supply. By changing the voltage the voltage applied to the furnace, the temperature of the furnace can be varied. The temperature of the furnace can be measured by inserting a thermocouple in a hole (provided on one of the Teflon discs), so that it touches one of the capacitor (metal) plates.

The audio oscillator is incorporated inside the instrument. If C_{SC} and C_{DC} represents the capacitances of the standard capacitor and dielectric cell respectively and if V_{C1} and V_C are the voltages across SC and DC then,

$$\frac{V_{SC}}{I} = \frac{1}{\omega C_{SC}} \quad \text{----- (1)}$$

$$\Rightarrow I = \omega \cdot V_{SC} \cdot C_{SC} \quad \text{----- (2)}$$

The same current I passes through the dielectric cell.

$$\frac{V_{DC}}{I} = \frac{1}{\omega C_{DC}} \quad \text{----- (3)}$$

$$\Rightarrow C_{DC} = \frac{I}{\omega V_{DC}} = \frac{\omega C_{SC} V_{SC}}{\omega V_{DC}} = \frac{C_{SC} V_{SC}}{V_{DC}} \quad \text{-----(4)}$$

By measuring V_{SC} & V_{DC} and using the value of C_{SC} we can determine the capacitance of the dielectric cell containing the sample.

If C_0 represents the capacitance of the dielectric cell without the crystal and the plates separated by air gap whose thickness is the same as the thickness of the crystal then C_0 is given by

$$C_0 = \frac{\epsilon_0 A}{d} = \frac{r^2}{36d} \text{ nf} \quad \text{----- (5)}$$

where r represents the radius of the crystal and d represents its thickness.

The dielectric constant of the crystal at any given temperature is given by

$$\epsilon_0 = \frac{C}{C_0} \quad \text{----- (6)}$$

CALIBRATION

- 1) Connect C.R.O. to the terminals provided on the front panel.
- 2) Switch ON the unit and adjust CAL. such that sinusoidal waveform appears on C.R.O. Adjust the CAL. such that amplitude of the sine wave is just before the clipping.
- 3) Switch OFF the unit and disconnect C.R.O. from the main circuit.

PROCEDURE

- 1) Connect C.R.O. to the terminals provided on the front panel. If no sinusoidal waveform appears on C.R.O. then follow calibration procedure first.
- 2) Assemble the dielectric capacitor as shown in figure (3) and connect it to the main unit. (For convenience we have the assembled dielectric cell.)
- 3) Connect hot air oven to the mains.
- 4) Place the dielectric cell DC in hot air oven and place the lid on the top. Insert the thermocouple to the hole provided on top Teflon disc of DC via hole provided on the insulating disc. Make sure that thermocouple touches the top metal disc.
- 5) Switch ON the unit.
- 6) Select SC1 among standard capacitors.

7) Measure the voltage (using digital voltmeter provided on front panel) across the dielectric cell DC, say V_{DC} , by throwing switch S_1 towards SC, while heater is switched off (i.e. at room temperature).

8) Determine the dielectric constant of the crystal using the relation

$$\epsilon_r = \frac{C}{C_0} = \frac{C_{SC} V_{SC}}{V_{DC} C_0} \dots\dots\dots (7)$$

where C_0 is calculated using relation (5).

9) Switch ON the oven and set the desired temperature (Follow instructions to set the temperature of the oven). Measure voltages V_{DC} and V_{SC} (as explained in step 8) at different temperatures at 15°C interval in the range 40°C -100°C.

NOTE: Readings should be taken in ascending order only.

10) Measure V_{DC} and V_{SC} at 5-10 °C interval upto 170 °C and at intervals of 2°C until you reach the maximum value of the dielectric constant (or C). Thereafter take few points.

NOTE: Choose standard capacitor SC1 For temperature upto 130°C and SC2 for temperature range 130°C to 190°C and SC3 for 190°C onwards.

IMPORTANT: DO NOT INCREASE THE TEMPERATURE OF OVEN BEYOND 225°C AS IT MAY DAMAGE THE TEFLON DISCS/SCREWS.

11) Make the observation table as shown below:

Temperature (°C)	SC1		SC2		SC3		Calculated Capacitance	ϵ_r
	V_{DC}	V_{SC}	V_{DC}	V_{SC}	V_{DC}	V_{SC}		

12) Calculate the dielectric constant (as explained in step 10).

13) Draw a graph of ϵ_r Vs T. At the transition the dielectric constant sharply rises and falls suddenly after the transition temperature and then decreases slowly beyond the transition temperature.

Fig.5 shows the variations of dielectric constant of BaTiO₃ ceramic as a function of temperature (After W.B. Westphal, Laboratory for Insulation Research, M.I.T.).

An examination of Fig. 5 shows that it is difficult to make measurements at the Curie temperature due to sharp variation. Extension of the curves below and above the transition temperature allows one to estimate the Curie temperature.

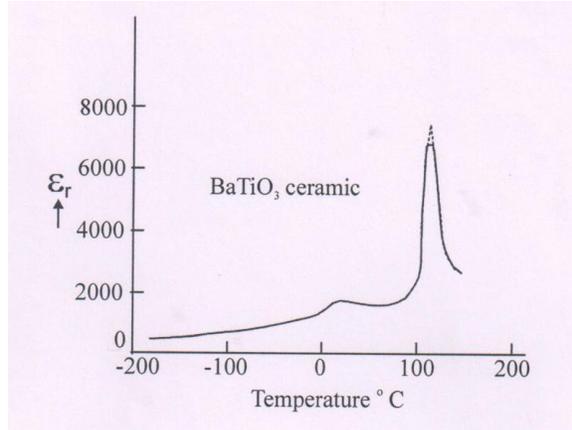


Fig. 5

14) Determine the transition temperature (Curie Temperature) from the graph.

NOTE: As the measurements are repeated the nature of the curve remains same but it might not be possible to get the same values.

Component Values (S.No.1112 855)

- L = 25 mH**
- SC₁ = 10.38 nf**
- SC₂ = 22.32nf**
- SC₃ = 47.80 nf**

INSTRUCTIONS TO SET THE DESIRED TEMPERATURE OF OVEN
(RT – 210 °C)

Note: Upper display (in red) shows current temperature reading and lower display (in green) shows set temperature.

1. Press up key (left most key on the controller cum indicator panel) or down key (middle key) to set the desired temperature.
2. Press enter (right most key) and hold for 1-2 sec.

Note: If after setting of desired temperature, enter key is not held for some time, it will go back to previous set value.

: If thermocouple is not connected to the unit, upper display will show “open”.

IMPORTANT: DO NOT CHANGE THE SETTING PARAMETERS OF CONTROLLER CUM INDICATOR

SAMPLE CALCULATIONS/READINGS:

Thickness of PZT sample : 1.08 mm.

Radius of PZT sample : 12 mm.

$$C_o = \frac{r^2}{36d} = \frac{(12 \times 10^{-3})^2}{36 \times 1.08 \times 10^{-3}} = 3.7 \times 10^{-3} \text{ nf}$$

Capacitance C(nf)	SC1=11nf		SC2=29nf		SC3=50nf		Capacitance C(nf)	Dielectric Constant ϵ_r
	V _{sc}	V _{DC}	V _{sc}	V _{DC}	V _{sc}	V _{DC}		
27	--	--					--	--
50	--	--					--	--
60	--	--					--	--
80	--	--					--	--
90	--	--					--	--
100	--	--					--	--
110	--	--					--	--
125	--	--					--	--
130			--	--			--	--
140			--	--			--	--
150			--	--			--	--
160			--	--			--	--
170			--	--			--	--
180					--	--	--	--
190					--	--	--	--
192					--	--	--	--
195					--	--	--	--
197					--	--	--	--
200					--	--	--	--
202					--	--	--	--
204					--	--	--	--
206					--	--	--	--
210					--	--	--	--
215					--	--	--	--
220					--	--	--	--

NOTE: Readings taken with similar sample and may differ from sample to sample but nature of the curve will remain same.
