

STUDY OF DIELECTRIC CONSTANT

Model: DEC-01



DIELECTRIC AND CURIE TEMPERATURE MEASUREMENT OF FERROELECTRIC CERAMICS

INTRODUCTION

Research in the area of Ferroelectrics is driven by the market potential of next generation memories and transducers. Thin films of ferroelectrics and dielectrics are rapidly emerging in the field of MEMS applications. Ultrasonic micro-motors utilizing PZT thin films and pyroelectric sensors using micro-machined structures have been fabricated. MEMS are finding growing application in accelerometers for air bag deployment in cars, micro-motors and pumps, micro heart valves, which have reached the commercial level of exploitation in compact medical, automotive, and space applications. Extremely sensitive sensors and actuators based on thin film and bulk will revolutionize every walk of our life with Hi-Tech gadgets based on ferroelectrics. Wide spread use of such sensors and actuators have made Hubble telescope a great success story. New bulk ferroelectric and their composites are the key components for the defence of our air space, the long coastline and deep oceans.

The quest of human beings for developing better and more efficient materials is never ending. Material Science has played a vital role in the development of society. Characterization is an important step in the development of different types of new materials. This experiment is aimed to expose the young students to Dielectric and Curie Temperature Measurement technique for Ferroelectric Ceramics.

Dielectric or electrical insulating materials are understood as the materials in which electrostatic fields can persist for a long time. These materials offer a very high resistance to the passage of electric current under the action of the applied *direct-current* voltage and therefore sharply differ in their basic electrical properties from conductive materials. Layers of such substances are commonly inserted into capacitors to improve their performance, and the term *dielectric* refers specifically to this application.

The use of a dielectric in a capacitor presents several advantages. The simplest of these is that the conducting plates can be placed very close to one another without risk of contact. Also, if subjected to a very high electric field, any substance will ionize and become a conductor. Dielectrics are more resistant to ionization than air, so a capacitor containing a dielectric can be subjected to a higher voltage. Also, dielectrics increase the capacitance of the capacitor. An electric field polarizes the molecules of the dielectric (Figure-1), producing concentrations of charge on its surfaces that create an electric field opposed (antiparallel) to that of the capacitor. Thus, a given amount of charge produces a weaker field between the plates than it would without the dielectric, which reduces the electric potential. Considered in reverse, this argument means that, with a dielectric, a given electric potential causes the capacitor to accumulate a larger charge.

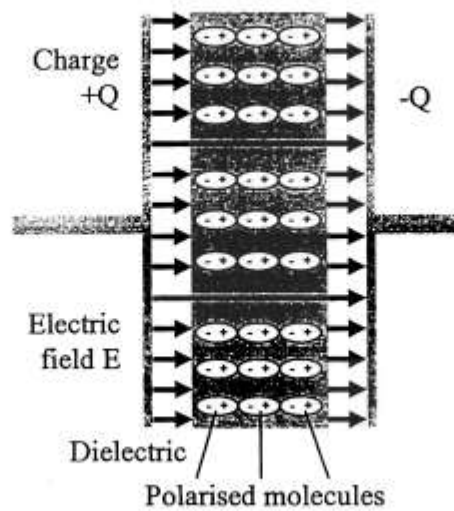


Figure-1

The electrons in the molecules shift toward the positively charged left plate. The molecules then create a leftward electric field that partially annuls the field created by the plates. (The air gap is shown for clarity; in a real capacitor, the dielectric is in direct contact with the plates.)

Perovskite Structure

Perovskite is a family name of a group of materials and the mineral name of calcium titanate (CaTiO_3) having a structure of the type ABO_3 . Many piezoelectric (including ferroelectric) ceramics such as Barium Titanate (BaTiO_3), Lead Titanate (PbTiO_3), Lead Zirconate Titanate (PZT), Lead Lanthanum Zirconate Titanate (PLZT), Lead Magnesium Niobate (PMN), Potassium Niobate (KNbO_3) etc. have a cubic perovskite type structure (in the paraelectric state) with chemical formula ABO_3 (figure 2 a, b).

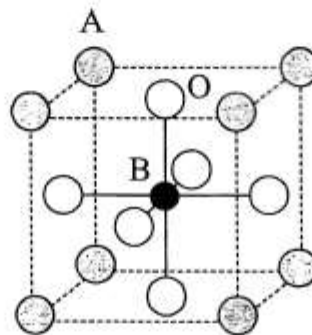


Figure 2 (a). Perovskite ABO_3 structure with the A and B cations on the corner and body center positions, respectively. Three oxygen anions per unit cell occupy the faces and form octahedra surrounding the B-site.

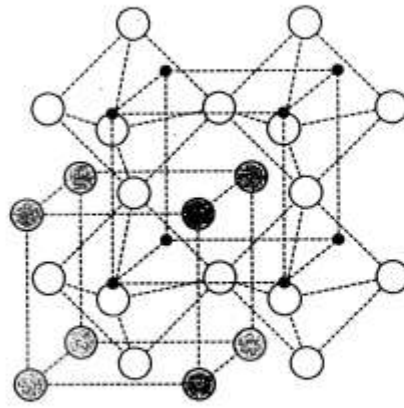


Figure 2 (b) Perovskite structure (Ba: Grey; Ti: Black; O: White)

As conventionally drawn, A-site cations occupy the corners of a cube, while B-site cations sit at the body center. Three oxygen atoms per unit cell rest on the faces. The lattice constant of these perovskite is always close to the 4 Å due to rigidity of the oxygen octahedral network and the well-defined oxygen ionic radius of 1.35 Å.

A practical advantage of the perovskites structure is that many different cations can be substituted on both the A and B sites without drastically changing the overall structure. Complete solid solutions are easily formed between many cations, often across the entire range of composition. Even though two cations are compatible in solution, their behavior can be radically different when apart from each other. Thus, it is possible to manipulate a material's properties such as Curie Temperature or dielectric constant with only a small substitution of a given cation.

All ferroelectric materials have a transition temperature called the Curie point (T_c). At a temperature $T > T_c$ the crystal does not exhibit ferroelectricity, while for $T < T_c$ it is ferroelectric. On decreasing the temperature through the Curie point, a ferroelectric crystal undergoes a phase transition from a non-ferroelectric (paraelectric) phase to a ferroelectric phase.

Barium Titanate (BaTiO_3 , BT)

Barium Titanate (BaTiO_3) has a ferroelectric tetragonal phase (Fig-3(a)) below its curie point of about 120°C and paraelectric cubic phase (Fig-3(b)) above Curie point. The temperature of the curie point appreciably depends on the impurities present in the sample and the synthesis process.

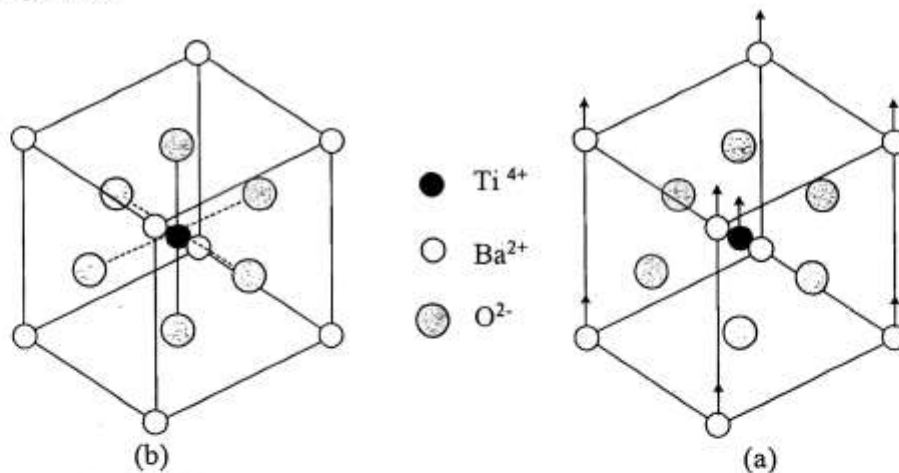


Fig 3 A Perovskite unit cell and the displacements in its ions on the application of an electric field.

In the paraelectric cubic phase the center of positive charges (Ba^{2+} , Ti^{4+}) coincides with the center of negative charges (O^{2-} ion) and on cooling below T_c , a tetragonal phase develops where the center of Ba^{2+} and Ti^{4+} ions are displaced relative to the O^{2-} ions, leading to the formation of electric dipoles.

As the BT ceramics have a very large room temperature dielectric constant, they are mainly used in multilayer capacitor applications. The grain size control is very important for these applications.

Dielectric Constant

The dielectric constant (ϵ) of a dielectric material can be defined as the ratio of the capacitance using that material as the dielectric in a capacitor to the capacitance using a vacuum as the dielectric. Typical values of ϵ for dielectrics are:

Material	DIELECTRIC CONSTANT (ϵ)
Vacuum	1.000
Dry Air	1.0059
Barium Titanate	100-1250
Glass	3.8-14.5
Quartz	5
Mica	4-9
Water distilled	34-78
Soil dry	2.4-2.9
Titanium dioxide	100

Dielectric constant (ϵ) is given by

$$\epsilon = \frac{C}{C_0}, \quad C_0 = \frac{\epsilon_0 A}{t}$$

Where

C = capacitance using the material as the dielectric in the capacitor,

C_0 = capacitance using vacuum as the dielectric

ϵ_0 = Permittivity of free space (8.85×10^{-12} F/m)

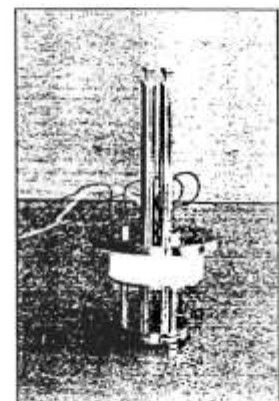
A = Area of the plate/ sample cross section area

t = Thickness of the sample

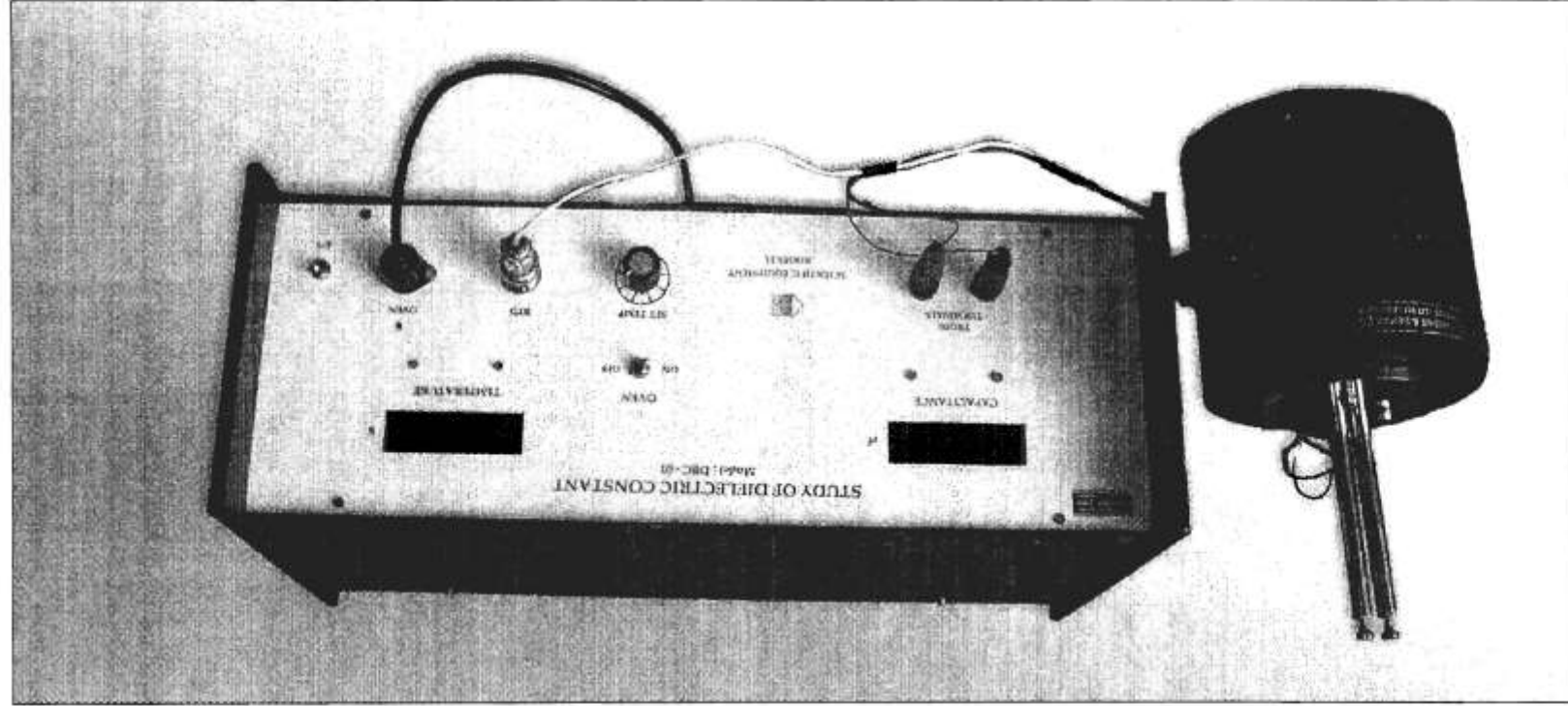
Brief Description of the Apparatus

1. Probe Arrangement

It has two spring loaded probes. These probes move in pipes and are insulated by teflon bush, which ensure a good electrical insulation. The probe arrangement is mounted in suitable stand, which also holds the sample plate and RTD sensor. The RTD is mounted in the sample plates such that it is just below the sample, separated by a very thin sheet of mica. This ensures the correct measurement of sample temperature. This stand also serves as a lid of the oven. The leads are provided for the connection to RTD and capacitance meter.



STUDY OF DIELECTRIC CONSTANT SETUP



2. Sample

Barium Titanate (BaTiO_3) plate with top and bottom conducting surface.

3. Oven

This is a high quality temperature controlled oven. The oven has been designed for fast heating and cooling rates, which enhance the effectiveness of the controller.

4. Main Units

The Set-up consists of two units housed in the same cabinet.

(i) Oven Controller

Platinum RTD (A class) has been used for sensing the temperature. A Wheatstone bridge and an instrumentation amplifier are used for signal conditioning. Feedback circuit ensures offset and linearity trimming and a fast accurate control of the oven temperature.

Specifications of the Oven	
Temperature Range	: Ambient to 200 °C
Resolution	: 0.1 °C
Stability	: ± 0.1 °C
Measurement Accuracy	: ± 0.5 °C
Oven	: Specially designed for Dielectric measurement
Sensor	: RTD (A class)
Display	: 3½ digit, 7 segment LED with autopolarity and decimal indication
Power	: 150W

(ii) Digital Capacitance Meter

This a compact direct reading instrument for the measurement of capacitance of the sample.

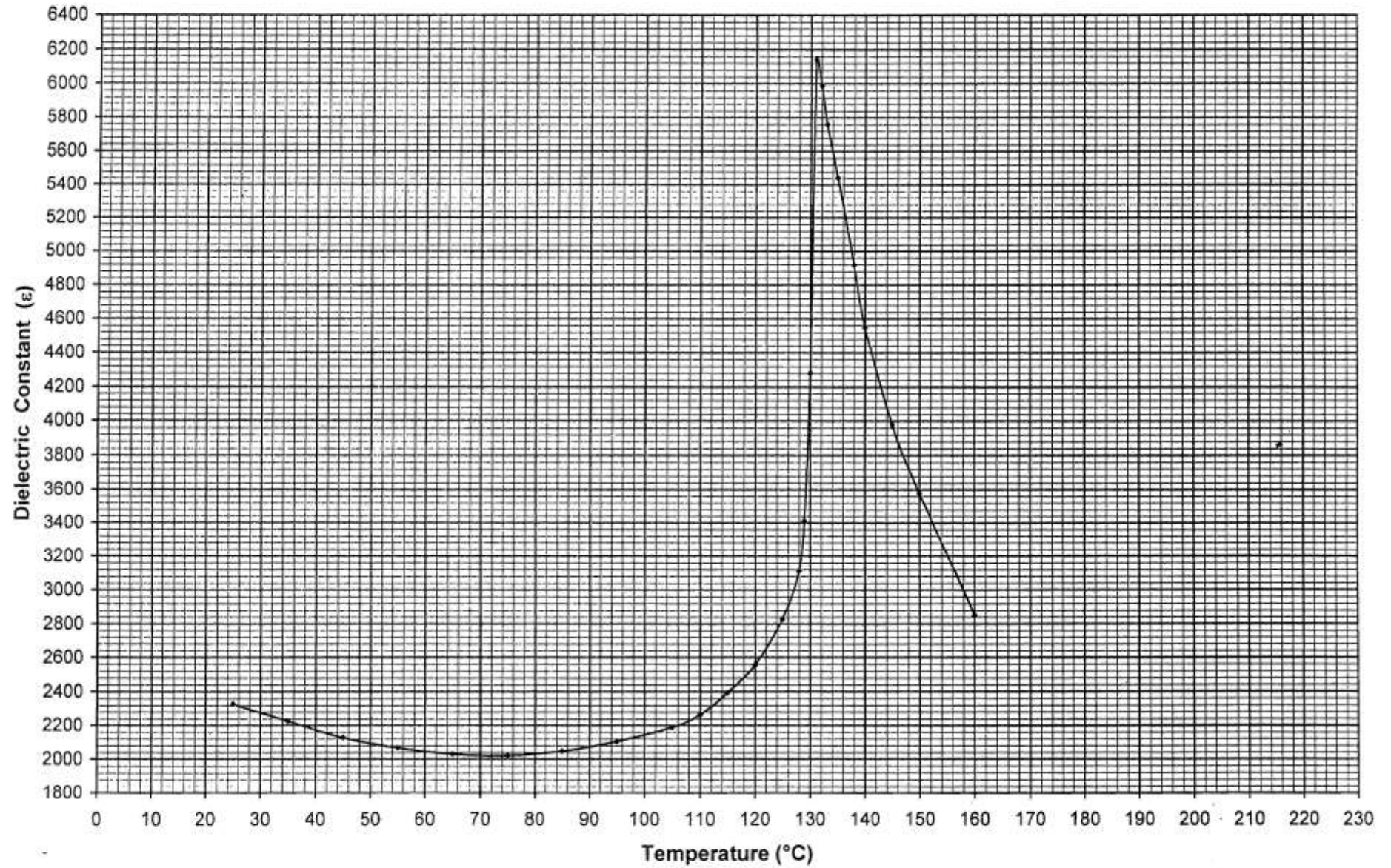
Specifications of the Oven	
Range	: 50 to 6000 pf
Resolution	: 1pf
Display	: 3½ digit, 7 segment LED

Experimental Procedure

1. Put a small piece of aluminum foil on the base plate. Pull the spring loaded probes upward, insert the aluminum foil and let them rest on it. Put the sample (BaTiO_3) on the foil. Again pull the top of one of the probe and insert the sample below it and let it rest on it gently. Now one of the probes would be in contact with the upper surface of the sample, while the other would be in contact with the lower surface through aluminum foil.
2. Connect the probe leads to the capacitance meter.
3. Connect the oven to the main unit and put the oven in OFF position.
4. Switch on the main unit and note the value of capacitance. It should be a stable reading and is obtained directly in pf.
5. (i) Switch ON the temperature Controller and approx adjust the set-temperature. The green LED would light up indicating the oven is ON and temperature would start rising. The temperature of the oven in °C would be indicated by the DPM.

DIELECTRIC CONSTANT

SAMPLE : Barium Titanate



- (ii) The controller of the oven would switch ON/OFF power corresponding to set-temperature. In case it is less than the desired, the set-temperature may be increased or vice versa.
- (iii) Because of thermal inertia of oven, there would be some over shoot and under shoot before a steady set-temperature is attained and may take 10 minutes for each reading.
- (iv) To save time, it is recommended to under adjust the temperature. Example, it is desired to set at 50°C, adjust the temperature set knob so that LED is OFF at 45°C. The temperature would continue to rise. When it reaches 50°C adjust the temperature set knob so that oven is just ON/OFF. It may go up 1 & 2°C, but would settle down to 50°C. Since the change in temperature at this stage is very slow and response of RTD and sample is fast, the reading can also be taken corresponding to any temperature without waiting for a steady state.

Observations and Calculations

Sample : Barium Titanate (BaTiO₃)

Area (A) : 8 x 6 mm

Thickness (t) : 1.42 mm

Permittivity of Space (ϵ_0) : 8.85×10^{-12} F/m or 8.85×10^{-3} pf/mm

$$\epsilon = \frac{C}{C_0} ; \text{ where, } C_0 = \frac{\epsilon_0 A}{t} \Rightarrow \frac{8.85 \times 10^{-3} \times 48}{1.42} \Rightarrow 29.9 \times 10^{-3} \text{ pf}$$

S.No.	Temperature (°C)	Capacitance, C (pf)	Dielectric Constant, (ϵ)
1	25	696	2328
2	35	665	2224
3	45	636	2127
4	55	618	2067
5	65	607	2030
6	75	604	2020
7	85	612	2047
8	95	629	2104
9	105	654	2187
10	110	677	2264
11	115	715	2391
12	120	764	2555
13	125	846	2829
14	128	930	3110
15	129	1020	3411
16	130	1280	4281
17	131	1836	6140
18	132	1790	5987
19	133	1722	5759
20	135	1627	5441
21	138	1470	4916
22	140	1360	4548
23	145	1190	3980
24	150	1070	3579
25	160	853	2853

Typical Results

From the graph, Curie Temperature (T_c) = 131°C

Precautions

- (1) The spring loaded probe should be allowed to rest on the sample very gently, other wise it may damage the conducting surface of the sample or even break the sample.
- (2) The reading of capacitance meter should be taken when the oven is OFF. This would be indicated by the green LED. In ON position there may be some pick ups.
- (3) The reading near the Curie Temperature should be taken at closer intervals, say 1°C.

Reference

- (1) Introduction to Solid State Physics – C. Kittel, Wiley Eastern Limited (5th Edition).