Current measurement using Ferroelectrics like Lead Bismuth Niobate and PZT

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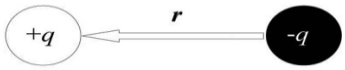
Abstract— This paper states the application of a ferroelectric substances in measurement of current using the property that their dielectric constant increases with temperature. Since the change in dielectric constant with temperature is very steep, the resolution of the current measuring device will be very high. Binary and ternary oxides have been used based on perovskite systems. The compounds have been synthesised, characterised with respect to their crystal structure and their dielectric constants have been measured

Index Terms— Ferroelectric substances, Current measurement, PZT, Lead Bismuth Niobate, Dielectric strength, Temperature, perovskites

1. FERROELECTRIC MATERIAL

To be ferroelectric, a material must possess a spontaneous dipole moment that can be generated by an applied electric field, i.e. spontaneous switchable polarisation. This is found when two particles of charge q are separated by some distance $\boldsymbol{r}$, i.e.:



The dipole moment, $\boldsymbol{\mu}$ is:

$$\boldsymbol{\mu} = q.\boldsymbol{r}$$

In a ferroelectric material, there is a net permanent dipole moment, which comes from the vector sum of dipole moments in each unit cell, $\Sigma\boldsymbol{\mu}$. This means that it cannot exist in a structure that has a centre of symmetry, as any dipole moment generated in one direction would be forced by symmetry to be zero. Therefore, ferroelectrics must be non-centrosymmetric. This is not the only requirement however. There must also be a spontaneous local dipole moment (which typically leads to a macroscopic polarisation, but not necessarily if there are domains that cancel completely). This means that the central atom must be in a non-equilibrium position.

1.1 Ferroelectricity of perovskites

Quite a lot of Ferroelectric materials are based on perovskite structure and hence at this point, let us highlight some aspects. The general chemical formula for perovskite compounds is ABX_3, where 'A' and 'B' are two cations of very different sizes built up of a Face centred cube and body centred cube. The 'B' cation is at the centre of the cube 'A' ions are larger than the 'B' ions. The ideal cubic symmetric structure has A cation in 12-fold cuboctahedral coordination. The relative ion size requirements for stability of the cubic structure are quite stringent; so slight buckling and distortion can produce several lower-symmetry distorted versions, in which the coordination numbers of A cations, B cations or both are changed.

Tilting of the BO_6 octahedra reduces the coordination of an undersized A cation from 12 to as low as 8. Conversely, off-centering of an undersized B cation within its octahedron allows it to attain a stable bonding pattern. The resulting electric dipole is responsible for the property of ferroelectricity and shown by perovskites such as $BaTiO_3$ that distort in this fashion.

1.2 Dielectric Constant versus Temperature

Another important property of these ferroelectric substances is that their dielectric constant increases as their temperature is increased. The dielectric constant of a material provides a measure of its effect on a capacitor. It is the ratio of the capacitance of a capacitor containing the dielectric to that of an identical but empty capacitor.

An alternative definition of the dielectric constant relates to the permittivity of the material. Permittivity is a quantity that describes the effect of a material on an electric field: the higher the permittivity, the more the material tends to reduce any field set up in it. Since the dielectric material reduces the field by becoming polarised, an entirely equivalent definition is that the permittivity expresses the

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ability of a material to polarise in response to an applied field. The dielectric constant (sometimes called the 'relative permittivity') is the ratio of the permittivity of the dielectric to the permittivity of a vacuum, so the greater the polarisation developed by a material in an applied field of given strength, the greater the dielectric constant will be.

These ferroelectric materials are generally semi conductor in nature. In case of semiconductors as temperature increases resistance decreases. This is because at higher temperatures more electrons and holes are generated. Therefore at higher temperatures more dipoles are created, so permittivity increases and hence the dielectric constant as described above.

2. EXPERIMENT

2.1 Aim

The aim of the experiment is to measure current in a circuit using the property of ferroelectric materials that dielectric constant of some such substances change very steeply with temperature. The various characteristics of the ferroelectric material used have also been studied.

2.2 Principle

The ferroelectric compound is placed in a circuit. As current flows in the circuit the compound gets heated up due to is high resistance (being of semiconductor nature). This in turn leads to an increase in temperature of the compound. Now, as temperature increases its dielectric constant also increases. And the dielectric versus temperature graph is quite steep for most ferroelectrics i.e. for a very small change in temperature the change in dielectric constant is very high. (this makes the measurements sensitive). Also **resolution is very high**. Thus by noting the change in dielectric constant the amount of current in the circuit can be predicted.

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2.3 Equations involved

- **$D \propto T$** **D- dielectric constant**

or, **$\Delta D \propto \Delta T$** **T- temperature**

- **$I^2Rt = ms\Delta T$** **I- current**
  R- resistance
- **$\Delta D \propto I^2Rt/ms$** **m- mass**
  s- specific heat capacity
- **$\sqrt{\Delta D} \propto I$** **t- time**

Here dielectric constant can be considered proportional to temperature within a certain range of temperature which is different for each ferroelectric. Hence change in dielectric constant is also proportional to the change in temperature over that range. **I^2Rt** is the heat generated in the ferroelectric substance used due to the flow of current in it. Using **$ms\Delta T$** we see the rise in temperature due to this heat. Thus we see that square root of the change in dielectric constant is proportional to the amount of current flowing in the circuit. And the proportionality constant is **Rt/ ms.**

The ferroelectrics used for the experiment are lead Bismuth Niobate ($PbBi_2Nb_2O_9$) and Lead Zirconium Titanate ($PbZr_xTi_{1-x}O_3$) or PZT based on perovskite structure.

2.4 Preparation of $PbBi_2Nb_2O_9$

The preparation of lead bismuth niobate follows the equation given below:-

$$PbO + Bi_2O_3 + Nb_2O_5 \rightarrow PbBi_2Nb_2O_9$$

For 4 gms. of the material, 0.982g of PbO, 2.049g of Bi_2O_3 and 1.113g of Nb_2O_5 are taken in an agate mortar after weighing in a Mettler electronic balance (Model AE-240) weighing accurately up to 0.1 mg.

The mixture is ground in a mortar pestle for two purposes

- To convert the mixture into fine powder
- To make a uniform mixture

During grinding, isopropyl alcohol was added to the mixture for more uniform mixing.

Now this mixture is placed in an alumina(Al_2O_3) container, with the top covered and then placed in a tubular furnace. Heating and cooling were done in four steps which are as follows :

Temperature range (in degree C)	Time(in minutes)	Rate(in degrees per min)
RT-800	80	10
At 800	360	0
800-500	120	2.5
500-100	120	3.33

After this, the compound was taken out of the furnace. Due to cooling, it formed a lump. Now again it was powdered by grinding using a mortar and pestle.

Making compact pellets using a hydraulic press.

Pellets have a diameter of 8mm and are about 2mm thick. Four tonnes of pressure was applied on them using the hydraulic press for about 5 to 7 minutes. A little poly vinyl alcohol (PVA) known as binder was added to the powder before putting it under the hydraulic press so that compact pellets are formed and they don't stick to the container.

Sintering

For sintering the pellets are kept on an alumina crucible placed on a brick which is made of refractory material (inert and has high structural integrity).then the entire setup is covered with a crucible made of alumina (which is also inert and has high thermal stability) to avoid escape of lead vapours. Then the edges of the crucible are sealed with heavy MgO and then placed in a programmable temperature controlled box furnace with the following temperature programme:

Temperature range (in degree C)	Time(in minutes)	Rate(in degrees per min)
RT – 800	100	8
800 – 1100	120	2.5
At 1100	360	0
1100 – 800	100	2.5
800 – 500	60	5
500 – 100	60	6.66

It is mainly done for the completion of the reaction. It also provides time for grain growth which increases the breakdown voltage.

The cooled sintered pellets were taken out of the furnace and one of them was polished using emery paper.(only the two faces). The two polished faces were cleaned using iso-propanol and tissue paper. Then the two faces were silvered using a silver sol(silver dispersed in polymer). This was done to make the two surfaces conducting which is required for the dielectric constant measurements.

The silvered pellet was placed in the box furnace and kept at 500^0C for 5 minutes for annealing and diffusion of silver in the matrix of the compound synthesised.

2.5 Preparation of lead zirconium titanate(PZT)

The formula of PZT is $Pb[Zr_xTi_{1-x}]O_3$. It was synthesised from PbO, ZrO_2 and TiO_2 by the usaula solid state reaction as before.

$$\mathbf{PbO + xZrO_2 + (1\text{-}x)TiO_2 \rightarrow Pb[Zr_xTi_{1-x}]O_3}$$

The sample used in this experiment has x=0.9

In this case it is calcined in a furnace at **1000^0C** for **three hours.** And the sintering temperature is **1150^0C** for **six hours.**

After synthesis, the compounds in powder form were characterised with respect to crystal structure by powder X-Ray Diffraction (XRD).

2.6 X-Ray Diffraction

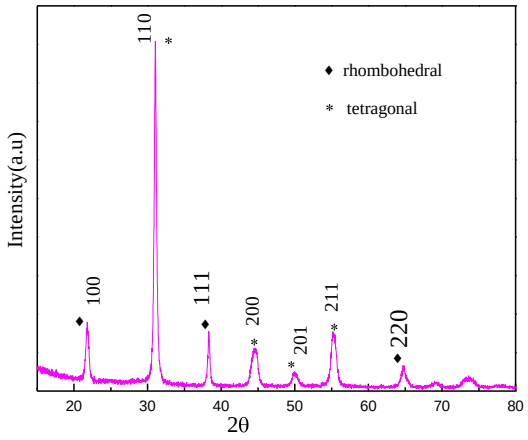

Figure 1

Figure 1 demonstrates the XRD pattern of PZT. It shows the coexistence of rhombohedral and tetragonal phases. The rhombohedral phase less symmetric compared to the tetragonal is expected to be ferroelectric.

2.7 DSC Measurements

The sample of $PbBi_2Nb_2O_9$ was characterised by Differential Scanning Calorimetry (DSC) to get an indication of the phase changes. In DSC there is one reference crucible and one sample crucible both made of platinum (inert and high melting point) placed on a block which is heated at a programmable rate. There is a differential thermocouple one end of which is connected to the reference crucible and the other end to the sample crucible. The temperature difference at this junction gives rise to a voltage at the junction due to seebeck effect. This voltage signal generated gives us an idea of the difference in power consumed by the reference crucible and the sample crucible. We get a power versus time graph. This power is, as stated not the absolute power consumption of any crucible but the difference in the power consumed by the reference crucible and the sample crucible. If the sample undergoes an exothermic process the graph shows us a peak and the opposite in case of an endothermic process. If we integrate the area covered by the peak with the time axis we can get the heat generated in the process. In our case we heated the sample from 30^0C to 700^0C at the rate of 5^0/min. Any phase transition is associated with evolution or

absorption of heat which is indicated in the power versus time graph of DSC.

In the DSC measurement of $PbBi_2Nb_2O_9$, we did not observe any significant phase transition from room temperature till 700^{0}C.

2.8 Dielectric vs Temperature Measurement

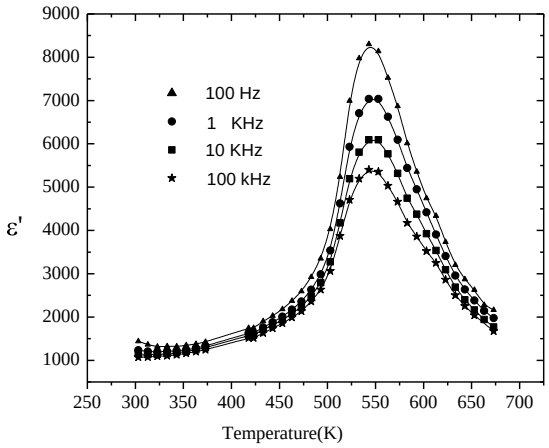

Figure 2

The dielectric constants of PZT were measured from room temperature to 650^{0}C at different frequencies from 100Hz to 100KHz and shown in Figure 2. It is observed that there is a sharp rise in dielectric constant from 450-550^{0}C.

G. Details of the PZT sample used

WEIGHT OF THE SAMPLE: **0.4620g**

AVERAGE THICKNESS: **1.53X10^-3 m**

AVERAGE DIAMETE**: 7.335X10^-3 m**

RADIUS: **3.6675X10^-3 m**

AREA**: 4.225653X10^-5 m^2**

Dielectric constant D = 66.67 T (where T is the temperature) approximately

Calculated Data:

ΔT	I(calculated)	V(required)	ΔD
	AMPERES	VOLTS	
50	8.96X10^-5	320	3333.5
70	1.060X10^-4	379.35	4666.9
90	1.202 X10^-4	430.14	6000.3
100	1.267 X10^-4	453.38	6666.6
110	1.329 X10^-4	475.5	7333.7
120	1.388 X10^-4	496.68	8000.4
130	1.445 X10^-4	516.9	8667.1
140	1.499 X10^-4	536.45	9333.8
150	1.552 X10^-4	555.28	10000.5
160	1.603 X10^-4	573.49	10667.2
170	1.652 X10^-4	591.14	11333.9
180	1.700 X10^-4	608.28	12000.6
190	1.747 X10^-4	624.9	12667.3
200	1.793 X10^-4	644.2	13334.0

For the applied voltage V the change in dielectric constant of the ferroelectric material and the corresponding current flowing in the circuit I is as shown in the table above.

3. CONCLUSION

3.1 Reasons for using PZT or $PbBi_2Nb_2O_9$

The main reason for using $PbBi_2Nb_2O_9$ is that its dielectric constant versus temperature graph has a very steep slope i.e. for a very small change in temperature the change in dielectric constant is very high. Therefore the resolution will be very high. According to calculation the slope is approximately 82 degrees.

For PZT the difference between two readings of current can be of the order of 10^{-6} and it is similar for $PbBi_2Nb_2O_9$. Both these compounds have very high breakdown voltages. Also the range of temperature over which the steep change takes place is pretty high, about 400.

The main reasons for using PZT are that firstly, the temperature values over which the dielectric constant change takes place is low comparatively. Hence it gets heated up at low current. Secondly according to the XRD analysis it has a mixed rhombohedral and tetragonal phase. While titanium rich compositions are in tetragonal phase, the zirconium rich are in rhombohedral phase. Therefore it has asymmetry in structure which gives better ferroelectric and piezoelectric properties.

3.2 Advantages of using this method of current measurement and its Application.

This technique of current measurement can be used to measure leakage current in insulators when placed in series with the insulators in the circuit. Also current through ferroelectric materials which is a semiconductor can be easily measured. The main advantage of using this method is the fact that the resolution is very high. The least count can be as low as 10^-6. Sensitivity is also high.

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