CHAPTER IX

Dielectric Properties of SrTiO$_3$
(Strontium Titanate)

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9.2 Synthesis by SSR
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  9.22 Preparation of ST
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9.3 X-RD characterization
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References
Dielectric Properties of SrTiO$_3$
(Strontium Titanate)
9.1 INTRODUCTION

SrTiO$_3$ belongs to the material class of the perovskites, ternary oxides of a structure ABO$_3$, where ‘A’ is mostly a group I, II element and ‘B’ is mostly a transition metal. The occupation of the ‘A’ and ‘B’ sites is very variable; Goodenough has reviewed more than 50 different perovskites. The cubic unit cell of SrTiO$_3$ with a lattice constant of 3.905. The octahedral corner-shared TiO$_6$ units form a tightly bonded network, which makes up the structural backbone of the lattice [1-5].

The chemical bonds in SrTiO$_3$ have a highly ionic character, but the covalent contribution to the titanium-oxygen bonds is not negligible. The perovskite structure is ideal for investigating the electronic structure of linear metal-oxygen-metal bonds of octahedrally coordinated metal ions. Direct metal-metal interactions are not possible due to an oxygen atom lying midway in between two titanium neighbors. Oxygen-oxygen interactions are comparatively weak, since they have to get over distances twice as large as the titanium-oxygen bond lengths. In tight-binding descriptions of the perovskite electronic structure, interactions between atoms of the same element have often been considered weak enough to be treated as perturbations [6-8]. In SrTiO$_3$, the influence of the highly ionized and electronically rather passive strontium ion on the titanium-oxygen covalence is small [9-11].

Relating the valence electronic structure to the atomic structure of a solid requires a physical description of the valence charge, which connects the atoms in the solid. The molecular orbital model is a description between as far as the strength of the interatomic interaction is concerned, the ligand field model, which applies to strictly localized electrons of weakly interacting atoms, and the band model, which applies
to valence wave functions extended over many atoms [12-15]. Considering a basic molecular orbital model for an isolated octahedral TiO$_6$ structural unit is instructive for interpreting the valence electronic structure of SrTiO$_3$.

The unit cell of SrTiO$_3$ is depicted with the titanium atoms at the corners. Alternatively, by shifting the origin by half a space diagonal, the strontium atoms will be found at the corners and the oxygen atoms face-centered. Each titanium atom is surrounded by an octahedron of oxygen atoms.

The 5-atom unit cell structure is shown in Figure 9.2.
Recent studies on strontium titanate have shown its great potential for practical applications, such as: the usage in fuel cells, steam electrolysis and hydrogen gas sensors [16-19] because of its high photonic conductivity at high temperature; the usage as substrates for the fabrication of high-$T_c$ superconducting films and the good lattice match; and employment in power generation through recovered waste heat or electronic refrigeration for electronic devices [20] due to its good thermoelectric properties.

SrTiO$_3$ crystallizes in a cubic perovskite lattice; titanium is in the center of the unit cell octahedrally surrounded by six oxygen ions and a strontium ion in each corner. It has been suggested that this phase transition corresponds to a rotation of one rigid TiO$_6$ octahedron and a mirrored rotation of the next neigh-boring TiO$_6$ octahedron, thereby doubling the unit cell in all three crystallographic directions [21-22]. SrTiO$_3$ remains paraelectric when lowering the temperature, with a sharp increase of the dielectric constant.

**Fig. 9.2:** Structure of the 5-atom unit cell of SrTiO$_3$. Blue: Strontium; Black: Titanium; Red: Oxygen.
9.2 Synthesis by SSR

Fig. 9.3: Flow Chart for Preparation of ST

9.21 Starting / Raw materials

For preparation of ST ferroelectric ceramic material required following starting materials:

(i) Strontium Carbonate (SrCO₃) – (Loaba –Chem).
(ii) Titanium dioxide (TiO₂) – (Loba-Chem).

9.22 Preparation of ST (SrTiO₃)

Calculation of molecular weight:

1) Molecular weight of SrCO₃ = 147.63 x 1 = 147.63
   Then M.W. of Sr = 147.63
2) Molecular weight of TiO$_2$ = 79.89 x 1 = 79.89

Then, M.W. of Ti = 79.89

Total molecular weight = M.W. of SrCO$_3$ + M.W. of TiO$_2$

= 147.63 + 79.89

= 227.52

**Calculation for 50 gm:**

1) SrCO$_3$

\[
X_1 = \frac{\text{M.W. of SrCO}_3}{\text{total molecular weight}} \times 50
\]

\[
X_1 = \frac{147.63 \times 50}{227.52}
\]

\[
X_1 = 32.44 \text{ gm}
\]

2) TiO$_2$

\[
X_2 = \frac{\text{M.W. of TiO}_2}{\text{total molecular weight}} \times 50
\]

\[
X_2 = \frac{79.89 \times 50}{227.52}
\]

\[
X_2 = 17.56 \text{ gm}
\]

Total weight of ST sample = $X_1 + X_2 + X_3$

= 32.44 + 17.56

= 50 gm
9.23 Calcinations of SrTiO$_3$ (ST):

First grind raw material for 3½ hrs by using agate mortar and dried property and then calcined in alumina crucible at 1000°C for 5 hrs. The calcinations help in driving out all volatile and gaseous material from powder. After first calcinations it again grinds 45 min for final calcinations at temperature 1300°C for 5 hrs.

9.3 X-Ray Diffraction Study:

Table: 9.1

Values of Bragg’s angle ($2\theta$), Interplanar spacing (d), Intensity (I) and miller indices (h k l) from JCPDF (No: 01-070-8508)

<table>
<thead>
<tr>
<th>$2\theta$</th>
<th>d(Å)</th>
<th>Intensity</th>
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<th>k</th>
<th>L</th>
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Fig. 9.4: XRD pattern of ST from JCPDF (No. 01-070-8508)
Table 9.2
Values of Bragg’s angle ($2\theta$), Interplanar spacing (d), Intensity (I) and miller indices (h k l)

<table>
<thead>
<tr>
<th>$2\theta$</th>
<th>d(Å)</th>
<th>Intensity</th>
<th>h</th>
<th>k</th>
<th>L</th>
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Fig. 9.5: XRD pattern of SrTiO$_3$
X-Ray Diffraction:

X-ray diffraction patterns of SrTiO$_3$ (ST) were recorded using X-ray diffractometer with Cu.ka ($\lambda = 1.5418$ Å) radiation. (Make: Bruker AXS, Analytical instrument Pvt. Ltd. Germany, Model No. D2PHASER).

The Sharpness of ST diffraction peaks suggests better homogeneity and crystallization of the samples. The x-ray analysis indicates that the ST has single phase with cubic structure. All the reflection peaks were indexed using observed interplaner spacing $d$ and lattice parameters with the JCPDS data of ST (Card no. 01-070-8508) were determined using a least squares refinement method. A good agreement between calculated and observed values of all diffraction lines of ST suggests that (there is no change in the basic crystal structure) prepared sample is pervoskite ferroelectric material and it useful for electro optic waveguide.

From above table 9.1 and Fig. 9.4 shows, the data of ST and XRD from JCPDF card (No.: Card no. 01-070-8508). Table 9.2 and Fig. 9.5 shows that the observed data of ST and XRD.

It was observed that high intensity reflection peaks of $2\theta = 22.244^0$ (100) plane, $2\theta = 31.590^0$ (110) plane,

$2\theta = 38.944^0$ (111) plane, $2\theta = 45.433^0$ (200) plane,

$2\theta = 51.069^0$ (210) plane, $2\theta = 56.299^0$ (220) plane,

$2\theta = 78.822^0$ (310) plane, $2\theta = 83.522^0$ (311) plane,
The corresponding ‘d’ and intensity values are mentioned in the table 4.2. The observed values of $2\theta$ & d are found to be nearly matched with the values from JCPDF.

The strongest reflection peak is observed at $2\theta = 31.590^0$ (110) plane which is matched with the value form JCPDF as $2\theta = 32.4377^0$ (110). It is observed the ‘d’ spacing is at $\lambda= 1.54184$. The lattice parameters are as $a = 3.903$, $b = 3.903$, $c = 3.903$. The cell to axial ratio i.e. $a/b = 1.000$ and $c/b = 1.000$. The crystal symmetry of ST is found to be centrosymmetric and cell volume is 59.47.

### 9.4 Material preparation for Dielectric studies:

Similar material preparation for dielectric studies is as mentioned in Chapter Three (3).

### 9.5 Dielectric measurements by using wave guide reflectometer techniques

For accurate measurement of $\lambda_d$ by using x-band relflectometric technique the same procedure has been followed as mentioned in the Chapter Three.
9.6 Dielectric Parameter:
Table 9.3: Values of $\varepsilon'_p$, $\varepsilon''_p$, Q x f, $\tau_p$ and $\sigma_p$ of SrTiO$_3$ at different temperatures and particle sizes.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Relative packing factor ($\delta r$)</th>
<th>$\varepsilon'_p$</th>
<th>$\varepsilon''_p$</th>
<th>tan$\delta$</th>
<th>$Q = 1/tan\delta$</th>
<th>Q x F GHz (9.85)</th>
<th>$\tau_p$ (P.S.)</th>
<th>$\sigma_p$</th>
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Table 9.4: Measured and calculated Values of $\varepsilon'_s$ and $\varepsilon''_s$, for solid bulk from SrTiO$_3$ at different temperatures and packing fractions.

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<th>Temperature (°C)</th>
<th>Relative packing factor ($\delta r$)</th>
<th>Measured value $\varepsilon'_p$</th>
<th>Calculated from Bottcher’s $\varepsilon'_s$</th>
<th>Calculated from L-L-L $\varepsilon'_s$</th>
<th>Measured value $\varepsilon''_p$</th>
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<th>Calculated from L-L-L $\varepsilon''_s$</th>
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</table>
Chapter IX

Dielectric Properties of SrTiO$_3$ (Strontium Titanate)

![Graph 1: Fig 9.6 - $\delta_r$ Vs $\varepsilon'$](image1)

![Graph 2: Fig 9.7 - $\delta_r$ Vs $\varepsilon''$](image2)

-10°C, 10°C, 30°C, 50°C
Fig: 9.8- $\delta r$ Vs $Q = 1/\tan \delta$

Fig: 9.9- $\delta r$ Vs $Q \times F$
Dielectric Properties of SrTiO$_3$ (Strontium Titanate)

**Fig: 9.10- $\delta r$ Vs $\tau_p$**

**Fig: 9.11- $\delta r$ Vs $\sigma_p$**
9.7 Verification of result by Bottchers and Landau – Litshitz – Looyenga theoretical equations

Table 9.4, indicates the measured and computed values of $\varepsilon'_s$ and $\varepsilon''_s$ for bulk from powder measurements. The results reported at $\delta r = 1$ are those measured on the finest crushed powder sample packed at very closely in a sample holding dielectric cell at 98N force, so minimum voids between the particles. The smallest particle size 125 micron or less assumed this system as solid bulk for getting correlation between powder and solid bulk. The correlation formulae of Landau – Lifshitz – Looyenga and Bottcher were used. The bulk values $\varepsilon'_s$ and $\varepsilon''_s$, are very much closer to measured values.

![Graph: δr Vs ε (Exp, Bott., L.L.L)](image-url)

**Fig: 9.12 - δr Vs ε' (Exp, Bott., L.L.L)**
9.8 RESULTS AND DISCUSSION

- The SrTiO$_3$ pervoskite material synthesized by solid state reaction method is confirmed from the XRD which is matched with the JCPDF file no. (01-070-8508).
- ST material is obtained in the form of powder which is then separated in different particles size.
- The dielectric and physical parameter are determined by using x – band microwave bench having special designed solid cell. The reflectometric technique is used to determine accurate dielectric wavelength ($\lambda_d$).

1. **Effect of density (i.e. $\delta_r$) and Temperature on $\varepsilon'$ and $\varepsilon''$**

   By increasing $\delta_r$ i.e. density of material there is systematic increase in values of $\varepsilon'$ and $\varepsilon''$. The $\varepsilon'$ and $\varepsilon''$ values decreased by increasing the temperature. This expected
because at higher values of $\delta_r$. The interparticle hindrance offered to the dipolar motion for a compact medium is much higher than less bounded particles.

2. **Effect of $\delta_r$ & Temperature on $(Q \times F)$ values**

   As $Q = 1/\tan \delta$ and $F$ is the resonant microwave frequency by decreasing $\delta_r$, the $(Q \times F)$ values decreases and by increasing $\delta_r$, the $(Q \times F)$ values increases.

3. **Effect of $\delta_r$ & $T$ on $\tau_p$ & $\sigma_p$**

   There is increase in $\tau_p$ and $\sigma_p$ by increasing $\delta_r$ and the values of $\tau_p$ and $\sigma_p$ decreases by increasing temperature. This is due to at higher compaction; no microcracks develop in sample at high mechanical pressure. As $\delta_r$ increases means number of particle increases. Hence, there is increasing hindrance to the process of polarization.

9.9 Conclusion

1. In the present study of SrTiO$_3$ materials, the highest values of $\varepsilon'= 29.26$ at $t= -10^\circ C$ and $\delta_r = 1$ and lowest value of $\varepsilon'= 13$ at $t= 50^\circ C$ and $\delta_r = 0.90$. The dielectric constant value varies from 13 to 29.26.

2. The value of $\varepsilon'$ and $\varepsilon''$ are verified for solid bulk by using theoretical formulae of Bottchers and Landu-Lifshitz-Looyenga. Then it is found at $\delta_r = 1$, the value of $\varepsilon'$ and $\varepsilon''$ are nearly same.

3. There is fair agreement between values obtained experimentally and theoretically.
4. Hence it is proved that our assumption by taking lowest particle size powder of ST as solid bulk.
5. From XRD of ST material, it is confirmed that the it is matched with JCPDF file no. 01-070-8508
6. From the crystal data of PDF card, the lattice parameters a=3.903, b=3.903, c=3.903 and crystal data axial ratio c/a = 1, a/b = 1, c/b = 1.
7. The crystal symmetry of ST is found to be centro-symmetric.
8. The sharp peak observed suggests the better homogenizing.
9. The high intensity reflection peak is observed at an angle 2θ=31.590 (110) plane which is matched with angle 2θ=32.4377 (110) plane.
REFERENCES:


11. P. G. Gawali and B.K Bongane, Microwave dielectric properties of (Ba$_{0.5}$Pb$_{0.5}$)TiO$_3$ with wave various temperature and particle sizes, Bio-nano Frontier (Communicated).


