CHAPTER IV

EXPERIMENTAL METHODS AND SET UP

This chapter presents experimental method and setup. Barium titanate, Fe-doped barium titanate, calcium copper titanate and Fe-doped calcium copper titanate thin films were grown by thermal decomposition of the precursors deposited from a sol-gel system onto quartz substrates. A ^{60}Co gamma radiation source was used to irradiate our films. The optical transmission spectra of the barium titanate, Fe-doped barium titanate and Fe-doped calcium copper titanate thin films were measured before and after gamma irradiation with different doses using a Perkin-Elmer Lambda 750 UV-Vis-NIR spectrophotometer. The capacitance of a coplanar capacitor made of calcium copper titanate film was measured before and after gamma ray irradiation.

4.1 Sample preparation

In this section, we will explain sample preparation of barium titanate, Fe-doped barium titanate, calcium copper titanate and Fe-doped calcium copper titanate precursors and how to deposit the films by a spin-coating sol-gel method.

4.1.1 Preparation of BTO and Fe-doped BTO precursors.

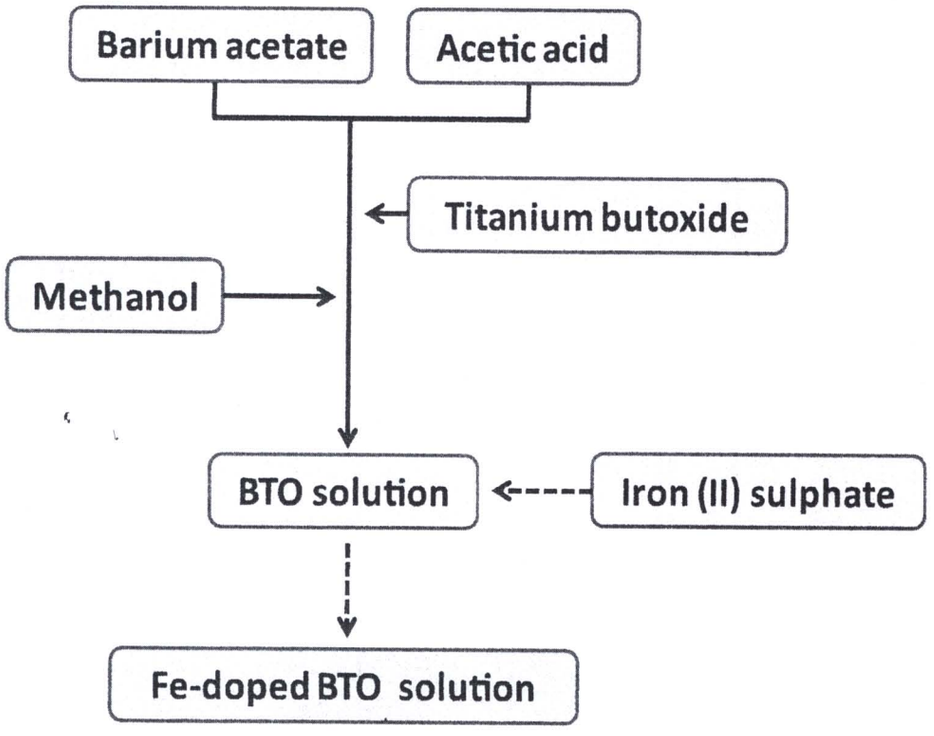

Figure 4.1: Flow chart of preparation of BTO and Fe-doped BTO.

Figure 4.1 shows the flow chart of preparation of BTO and Fe-doped BTO. Firstly, barium acetate ($Ba(CH_3COO)_2$) was dissolved in acetic acid. Then, pure titanium n-butoxide and methanol were added to the solution. In this thesis, 10% by weight of iron (see Appendix B) was done by dissolving iron (II) sulphate ($FeSO_4$) in the $BaTiO_3$ solution. The solution was mixed and stirred in a beaker with the aid of magnetic stirrer. This process was done on a hot plat at 60 °C.

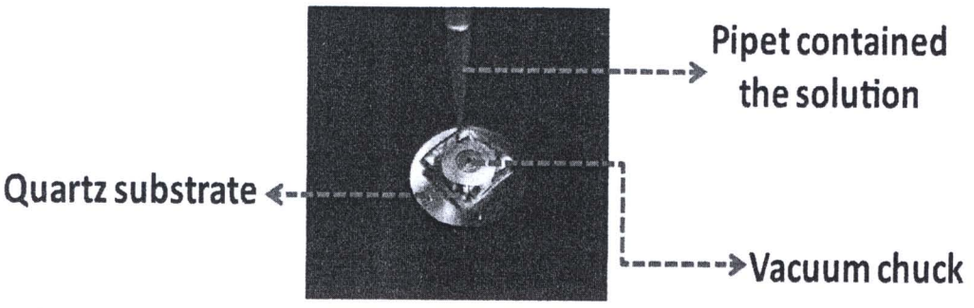

Figure 4.2: Spin coating process on a clean quartz substrate.

BTO or Fe-doped BTO precursor solutions were dropped on the clean quartz substrates as shown in Fig. 4.2 with the spinning speed of 1500 rpm to provide the first layer of each film. We have chosen quartz to be the substrate for our films. Quartz has been suitable substrates for studying the transmission of the materials being supported due to its transparency and high temperature melting point [45].

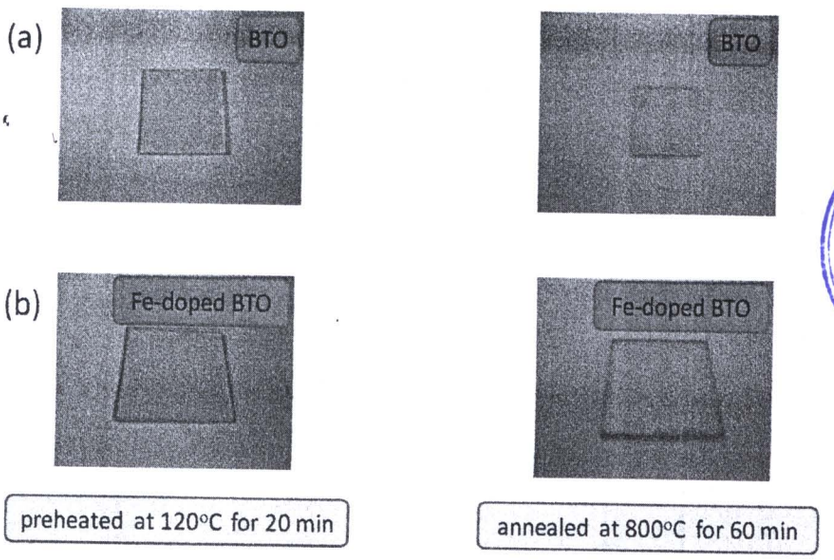

Figure 4.3: (a) BTO thin film after preheated and annealed (b) Fe-doped BTO film after preheated and annealed.

The BTO and Fe-doped BTO films were preheated at 120 °C for 20 min before annealing in an atmosphere of air at 800 °C for 60 min in order to form the crystalline structure. Figure 4.3(a) shows BTO thin film after preheating and annealing (b). Figure 4.3 shows Fe-doped BTO thin film after preheating and annealing. Different film thicknesses can be obtained by varying the number of deposition cycles. This process was repeated until the desired thickness was obtained. In this thesis, denoted each film by the material formula followed by the number of layers (L). Here, we have the BTO film with 2L, Fe-doped BTO with 4L, 6L and 8L, respectively.

4.1.2 Preparation of CCTO and Fe-doped CCTO precursors.

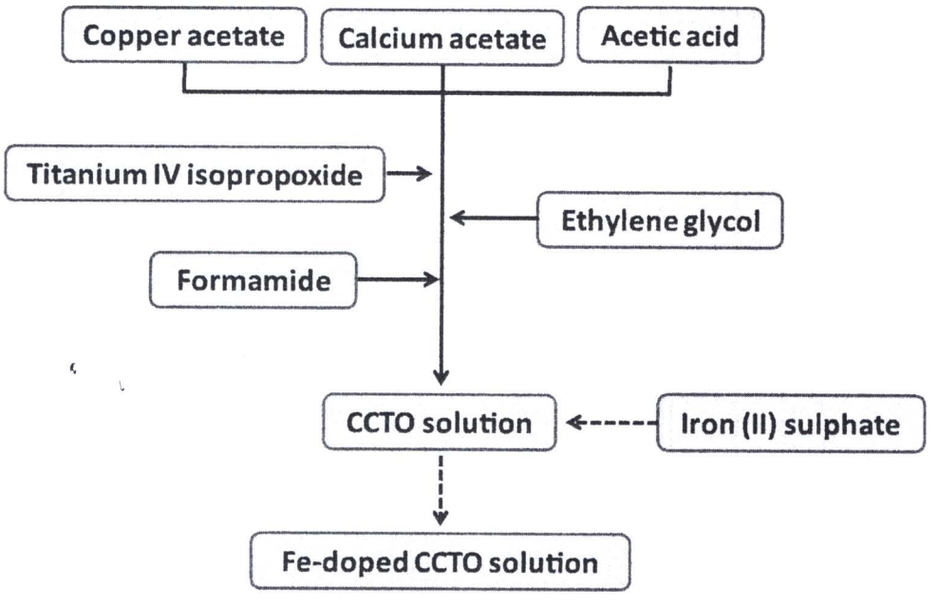

Figure 4.4: Flow chart of preparation of CCTO and Fe-doped CCTO.

Figure 4.4 shows the flow chart of preparation of CCTO and Fe-doped CCTO. Copper acetate ($Cu(CO_2CH_3)_2$) and calcium acetate ($Ca(C_2H_3O_2)_2.H_2O$) were firstly dissolved in acetic acid, and then titanium IV isopropoxide was slowly added. Ethylene glycol and formamide were added into the solution in order to increase solution stability. In this step, the solution viscosity can be also controlled to prevent the film cracking during the baking and annealing. In this thesis, 2% by weight of iron (see Appendix B) was done by dissolving iron (II) sulphate ($FeSO_4$) in the solution. The solution was stirred with a magnetic stirrer on a hot plat at 120 °C. CCTO and Fe-doped CCTO precursor solutions were doped on the quartz substrates with the spinning speed of 1500 rpm to provide the first layer of the film (see Fig. 4.2). The CCTO and Fe-doped CCTO films preheated at 120°C for 20 min and annealed at 800 °C for 60 min, respectively. Fig. 4.5(a) shows CCTO thin film after preheating and annealing and Fig. 4.5(b) shows Fe-doped CCTO thin film after preheating and annealing. The film with different thicknesses can

be obtained by repeating this process. In this thesis, we have prepared the CCTO thin film on Al_2O_3 with 6L and Fe-doped CCTO on quartz substrate with 2L, respectively.

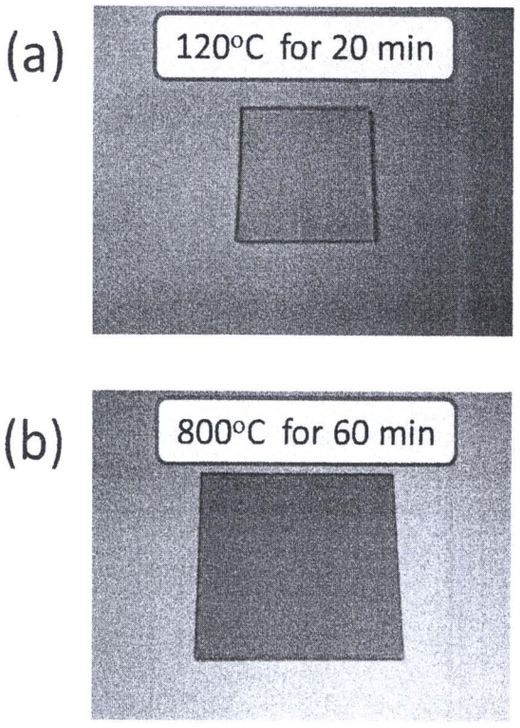

Figure 4.5: (a) Fe-doped CCTO film after preheated at 120°C (b) Fe-doped CCTO film after annealed at 800°C.

4.2 Gamma ray irradiation

A ^{60}Co put gamma radiation with an activity of 10 kCi (Gammacel 220 Excell) at a rate of 10 kGy/hr was used to irradiate our BTO, Fe-doped BTO, CCTO and Fe-doped CCTO thin films. Gammacel 220 Excell has many ^{60}Co cylinder sources around the aluminium cylinder which we used to hold our samples. Consequently, the positions in Gammacel 220 Excell are not equal doses. A Red Perspex dosimeter was used to calibrate the gamma rays doses at the positions of Gammacel 220 Excell. Figure 4.6 shows the different positions of dosimeter in a aluminium cylinder. The measured radiation doses at several positions are described in Fig. 4.6.

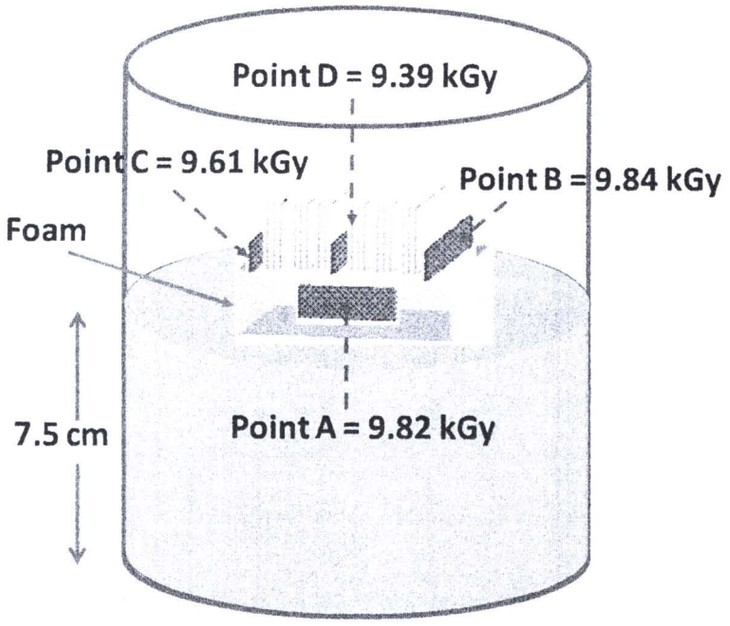

Figure 4.6: The position of dosimeter for dose mapping.

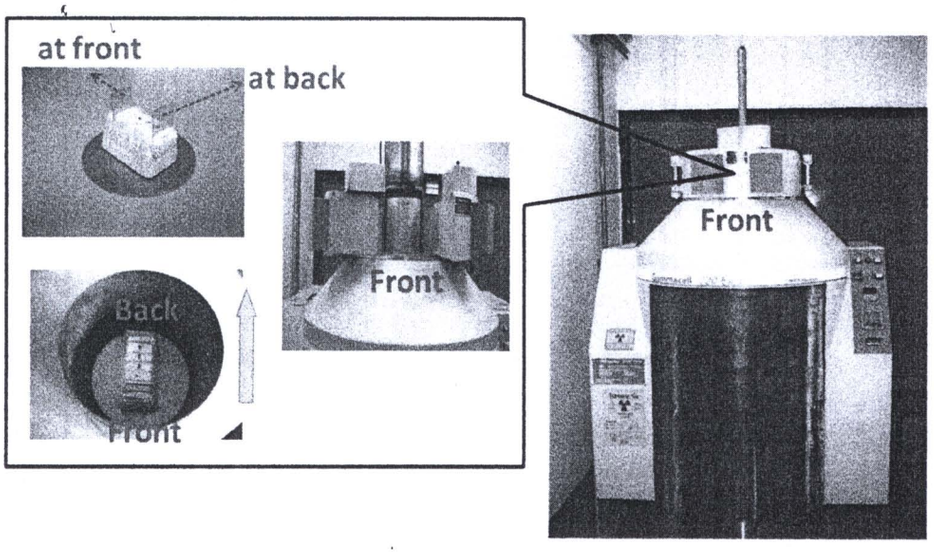

Figure 4.7: The ^{60}Co gamma radiation source (Gammacel 220 Excell).

Note that the dose of gamma ray at the Point D was minimum because at this position the sample was blocked by other samples which were placed at the and the front positions. We avoided putting the samples in the middle position. The table 4.1 shows the doses of gamma ray irradiation at different position for 1 hr. We average all three values except the dose value in the middle. The calculated average radiation doses is 9.760 ± 0.161 kGy. For the ease, we used the round number which is approximately 10 kGy. In this thesis, we used approximate doses as shown in the right column of the table 4.1. The radiation doses were varied via the exposure time up to 14.640 ± 0.161 or approximately 15 kGy for about 90 min. We avoided putting the samples in the middle position.

Table 4.1: The doses of gamma ray used in this thesis.

times (min)	doses (kGy)	approximately doses (kGy)
6	0.976 ± 0.161	1
18	2.928 ± 0.161	3
30	4.880 ± 0.161	5
60	9.760 ± 0.161	10
90	14.640 ± 0.161	15

Figure 4.7 shows the process of gamma ray irradiation. Foam was used to hold the film at different positions. The reason for choosing foam is because it is a cheap insulator and it is not difficult to machine. The foam which was used to hold the films was put in the middle of the aluminium cylinder and send it in Gammacel 220 Excell.