V

SUMMARY AND CONCLUSIONS

SUMMARY AND CONCLUSIONS

The present work on synthesis of $SrTiO_3$ substrate for superconducting films is carried out with a view to consider the possibility of using $SrTiO_3$ thin film layers on metal substrates as the substrates for superconducting and substrate materials.

Single crystals of $SrTiO_3$ have been used as successful substrates for development of superconducting materials. Due to higher cost of single crystal samples researchers are looking forward to prepare same material substrates by other technique. We have made an attempt to synthesis $SrTiO_3$ substrate by electrodeposition technique and followed by oxidation.

Sr-Ti alloys are electrodeposited and subsequent oxidation produces the $SrTiO_3$ thin films. In the present chapter the highlights of our work are summarized with some conclusions.

Chapter I opens with the substrate characteristics giving a brief account of different types of substrate materials. A review of superconducting materials developed on different type of substrate materials has revealed the importance of substrate materials on the superconducting characteristics, particularly on the HTc temperature. From the review it is revealed that, there is no report on electrodeposition of strontitum titanate substrate. We have planned to synthesis Sr-Ti alloy by electrodeposition technique from aqueous and non-aqueous baths and subsequent oxidation to produce $SrTiO_3$ films.

Various techniques used to produce substrate materials are discussed in brief in chapter II. Electrodeposition technique is planned to introduce as a successful technique to deposit HTc superconducting substrate materials on metallic strips. Brief theoretical background for electrodeposition technique is introduced with mechanism of electrodeposition. Experimental setup for electrolytic deposition with characteristics of various units used are discussed. Brief account for characterization of electrodeposited films and oxidised films, by studing their microstructural properties and X-Ray diffraction patterns is given.

Chapter III reports the systematic studies of preparative parameters for electrodeposition of strontium, titanate and strontium titanate alloy from aqueous bath. For electrodeposition bath the basic ingradients used were strontium nitrate of 100 mM and titanium chloride of 60mM. For alloy deposition the composition of two constituent ingradients are varied to optimise the parameters and to obtain the uniform, adhesive, dense and smooth films. Deposition potentials were optimised and tabulated in Table 3.1 onto various substrate. Due to higher charge to atomic radius ratio of titanium, it has higher affinity towards formation of its oxides in aqueous bath, which restricts to use fresh solutions. On conducting glass substrates (FTO), smooth, uniform, dense and adhesive titanium deposition of thickness 2μm to 3μm was obtained. The depositions were grey of metal colour.

Strontium as it belongs to rare alkaline group which are all electroinactive have higher E reduction potentials. We have obtained the electrodeposition of strontium from its nitrate bath onto various substrates, but due to higher Ered potential hydrogen evolution is observed at cathode which effects the adhesiveness of the deposits onto the substrate. The deposits are white in colour. Our main goal was to synthesis strontium titanate alloy from the complex bath of strontium nitrate and titanium chloride. The composition was varied from 10:90 to 50:50 of strontium nitrate and titanium chloride bath. At 20:80 percent composition we have obtained smooth, uniform dense, adhesive and greyish white films onto various substrate. Due to higher affinity of titanium towards oxygen in aqueous bath,

$$TiCl_3 + 2H_2O \longrightarrow TiO_2 + 3HCl + 1/2\ H_2 \uparrow$$

reaction takes place while deposition. Even though we could get deposition due to evolution of hydrogen the uniformity of the films distructed with pin holes. To overcome these affecting factors, we have tried with various complexing agents. With sodium acetate we have obtained uniform films but they were not adherent to the substrate. Since the increasing acidicness of the bath due to formation of HCl we have tried with buffer solutions, which was also found uneffective.

We have also discussed the effect of temperature of bath solution on the deposits and aging effect of the bath solution. Over all conclusion, we found that fresh solutions of electrolytes of constituent gradients of optimised molarity and composition should be used to electrodeposit strontium titanate alloy of optimized potential -1V Vs (SCE). The microstructural studies showd that the deposits were smooth, dense uniform with compact arrangement of grains. The grain coalescence was observed with decreased intergrain boundary spacing for oxidised films. X-ray diffraction pattern of oxidised films shows increased peak intensity and a (100) peak of $SrTiO_3$ substrate material.

Chapter IV reports on the electrodeposition of strontium titanate alloy from Non-aqueous DMSO (Dimethyl Sulfoxide) bath. Both constituent ingradients of the alloy have special factors which restrict to use some aprotic solvents. DMSO is found as a successful electrolytic medim because it is stable at strongly reducing potentials and is a good solvent for nitrate salts of constituent ingradient. In chapter IV we have reported on the systematic studies of the preparative parameters for electrodeposition of strontium, titanium and strontium titanate alloy from non-aqueous bath. Concentrations of constituent ingradients were optimised. The electrodeposition of (100mM) strontium nitrate and 60mM titanium chloride from DMSO were carried out onto stainless steel, brass, copper and FTO coated glass substrate. For strontium titanate alloy deposition the composition of complex bath was varied from 10:90 to 50:50. Even though we have got the alloyed films with 10:90 composition of strontium titanate the resistance was around $1K\Omega/cm^2$. For 20:80 composition the resistance was around $1M\Omega/cm^2$. The depositions were uniform, smooth, adhesive and dense for all the compositions, but as the strontium composition was increased, the adhesiveness of the film to the substrate was affected. For 50:50 composition the films were found to be deattached from the substrate, when they were kept to dry up.

The variation of current density with time for different potentials were studied. Current was decreased suddenly within first five minutes afterwords the decrease in current with time was very slow. The variation of current density with time for first 140 secs shows the sudden decrease and then sinusoidal behaviour of current.

Thickness of the alloyed films for 20:80 composition of strontium titanate increases linearly within first 15 minutes for further deposition time the increase in film thickness was very slow 2μm to 3μm thick films were obtained for 30 minutes of deposition.

Resistance measurements of the as deposited films were in the range of $0.1M\Omega/cm^2$ to $1M\Omega/cm^2$. The alloyed films were oxidised at $400^\circ C$ for different time periods, of these samples electrical properties, dielectric constant, optical micrography and x-ray diffraction patterns were studied. Resistance were found to increase from $1M\Omega/cm^2$ to $15M\Omega/cm^2$. Dielectric constants was 300 for as deposited film. Annealing time inceased the dielectric constant, for 1 hour of annealing the samples showed 1660 dielectric constant.

Optical micrography shows that increase in annealing time decreases the intergrain spacing. The grain sizes were found to decrease from 3μm to 5μm, 1μm to 2 μm with coalescence of grains as the annealing period is increased. For one hour of annealing at 400°C optical micrography shows very compact arrangement of grains with increased resistance and dielectric constants. From these studies it shows that the films obtained by electrodeposition technique can be used as insulating substrates or as dielectric barrier layers between the metal substrates and superconducting materials. The superconducting 123 HTc compounds have been deposited by screen printing, spray pyrolysis and laser ablation techniques on these electrodeposited dielectric thin films.