

# GROWTH AND MICRO STRUCTURAL STUDIES ON YITTRIA STABILIZED ZIRCONIA (YSZ) AND STRONTIUM TITANATE(STO) BUFFER LAYERS

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## ABSTRACT

Microstructure of Yittria Stabilized Zirconia (YSZ) and Strontium Titanate (STO) of radio frequency magnetron sputtered buffer layers was studied at various sputtering conditions on Si<100>, Sapphire and  $\text{LaAlO}_3$ <100> substrates. The effect of substrate temperatures upto 800 C and sputtering gas pressures in the range of 50 mTorr. of growth conditions was studied. The buffer layers of YSZ and STO showed a strong tendency for columnar structure with variation growth conditions. The buffer layers of YSZ and STO showed <h00> orientation. The tendency for columnar growth was observed above 15 mTorr sputtering gas pressure and at high substrate temperatures. Post annealing of these films in oxygen atmosphere reduced the oxygen deficiency and strain generated during growth of the films. Strong c-axis oriented superconducting  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (YBCO) thin films were obtained on these buffer layers using pulsed laser ablation technique. YBCO films deposited on multilayers of YSZ and STO were shown to have better superconducting properties.

## INTRODUCTION

There have been many reports on the deposition of high quality  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (YBCO) thin films on Si and Sapphire substrates using buffer layers. It has been well established that the use of buffer layer makes it possible to deposit thin superconducting films on a variety of substrates which otherwise are not suited because of the chemical reactivity with the superconducting thin film [1-4]. The most desirable properties of a buffer layer are a) to act as an effective barrier against inter diffusion, b) to improve lattice match between superconducting film and substrate and c) to withstand for thermal cycling stresses. Yittria Stabilized Zirconia (YSZ) which satisfies the above criteria is widely used as buffer layer on Si and Sapphire. On the otherhand,  $\text{SrTiO}_3$   $\langle 100 \rangle$  (STO) which has a good lattice match with YBCO can also act as a buffer layer on the above mentioned substrates.

H.Schmidth etal [5] have discussed the insitu growth of YSZ thin films using RF magnetron sputtering technique. Sputtering is a versatile technique for obtaining good quality epitaxial thin films as well as large area depositions with good uniformity. However, very few research reports are available on STO buffer layers [6]. For better understanding on the growth conditions of YSZ and STO buffer layers a detailed study about the preparatory conditions for reproducible results is needed .

## EXPERIMENTAL

A diode RF magnetron sputtering system (NORDICO 2000) was used to grow buffer layers on Si $\langle 100 \rangle$  sapphire and  $\text{LaAlO}_3$  $\langle 100 \rangle$  substrates. The YSZ (10% Yittria) and STO sputtering targets were of 99.9% pure (10cm dia obtained from Cerac.Inc., USA). Si wafers were degreased followed by etching with 6:1 mixture of high purity HF and deionised water for removing the native oxide layer

on Si. Other substrates were cleaned by usual methods. Using CT-8 Cryotorr cryopump(CTI) the chamber was pumped upto a base pressure of  $1 \times 10^{-7}$  Torr in all our experiments. A halogen lamp was used to heat the substrates upto  $800^{\circ}\text{C}$ . Thickness of the films was measured after deposition using surface profilometer (stylus).

Films were deposited at room temperature(RT),  $600^{\circ}\text{C}$ ,  $700^{\circ}\text{C}$  and  $800^{\circ}\text{C}$ . Studies also were made on buffer layers in different sputtering gas environments (pure Ar, Ar +  $\text{O}_2$  mix gas and pure oxygen). STO layers were deposited at  $700^{\circ}\text{C}$  and  $750^{\circ}\text{C}$ . Films were characterized by XRD and SEM. A set of films were post annealed at  $800^{\circ}\text{C}$  -  $1000^{\circ}\text{C}$ . YBCO thin films were deposited on these buffer layers and growth conditions were optimized by using pulsed laser deposition (PLD) technique [7]. Microstructural studies were done on the fractured Si<100> substrates.

## RESULTS AND DISCUSSION

### 1. Optimum deposition conditions for YSZ buffer layers :

For obtaining reproducible results and high quality oriented films, we have observed that irrespective of the nature of substrate, 24 - 30 mTorr Ar +  $\text{O}_2$  gas mixture (in the ratio 9:1) and  $800^{\circ}\text{C}$  substrate temperature are the best conditions. We have realized that it is difficult to produce <hoo> oriented thin films of YSZ on Si substrates as reoxidization of Si surface is unavoidable in sputtering technique. However, the films deposited on Sapphire,  $\text{LaAlO}_3$  substrates yielded <hoo>oriented films. [Fig. 1,2, 3.]. A set of YSZ films deposited on various substrates are post annealed and YBCO thin films have been deposited on these substrates with optimized conditions. Post annealed films resulted with good crystalline quality with single phase. YBCO thin film deposited on polycrystalline buffer layer (deposited at  $600^{\circ}\text{C}$  and post annealed at  $850^{\circ}\text{C}$ ) has yielded 80 -84 K and on <hoo> oriented YSZ film has yielded 86 -88K reproducibly on Sapphire substrate.

## 2. SrTiO<sub>3</sub> buffer layers :

The optimum deposition conditions for STO buffer layers on Si and Sapphire are 700° C and 15 -20 mTorr sputtering gas pressure ( Ar+ O<sub>2</sub> in 9:1 ratio ). However, we couldn't achieve highly <hoo>oriented STO films on Sapphire or Si due to lattice mismatch between STO and the substrates. Since, LaAlO<sub>3</sub> has good lattice match with STO, we have deposited STO on LAO <100> substrate and have obtained highly <hoo> oriented films using the above sputtering conditions. This clearly indicates the importance of lattice match for depositing <hoo> oriented films. However, we could achieve oriented STO films on YSZ buffered sapphire substrate which is due to the lattice match between YSZ and SrTiO<sub>3</sub>. YBCO thin films are deposited on these buffer layers which have yielded reproducible T<sub>c</sub> values in the range 85K-88K.

## 3. Microstructure of Buffer layers.

It has been pointed out by R.Pinto [8] et al that in the case of MgO films that columnar growth is a common phenomenon above 15 mTorr sputtering gas pressure. Similar columnar growth has been realized in the case of YSZ and STO buffer layers above 15m Torr sputtering gas pressure. Our scanning electron micrographs (fig.4) have confirmed this observation on fractured Si surfaces.

## CONCLUSIONS

We have realized the effect of growth conditions on the microstructural properties of YSZ and STO thin films on Si, Sapphire and LaAlO<sub>3</sub> substrates. We have optimized the growth conditions of buffer layers for reproducible results. The microstructural studies of YSZ and STO films on Si<100> substrate have revealed a tendency for columnar growth above 15 mTorr. It is noticed that even with polycrystalline YSZ buffer layers it is possible to get highly c-axis oriented YBCO superconducting thin

*films.*

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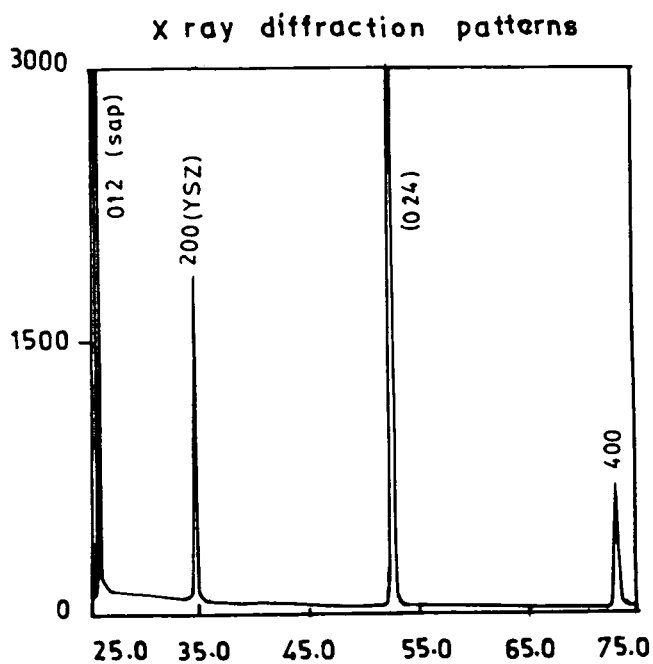
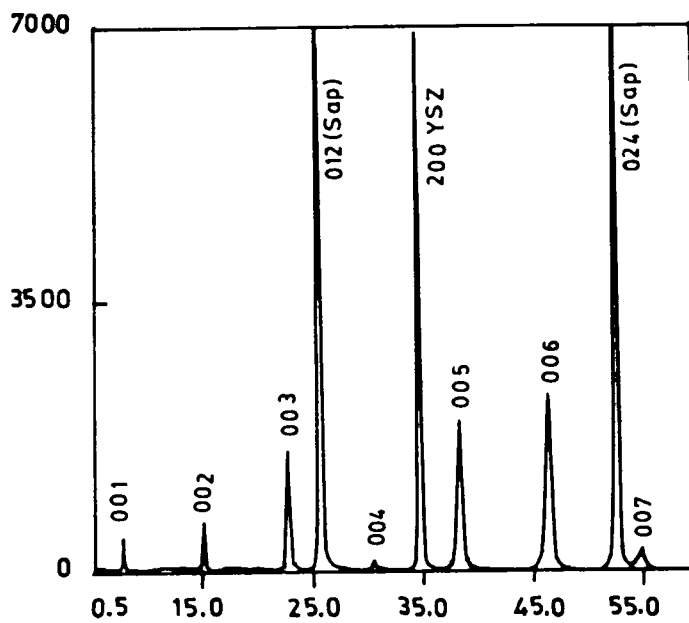


Figure 1.a)- XRD pattern of YSZ buffer layer on Saphire substrate deposited 800° C and 25 mTorr sputtering gas pressure.



b)- XRD pattern of YBCO on YSZ buffered on SI <100>

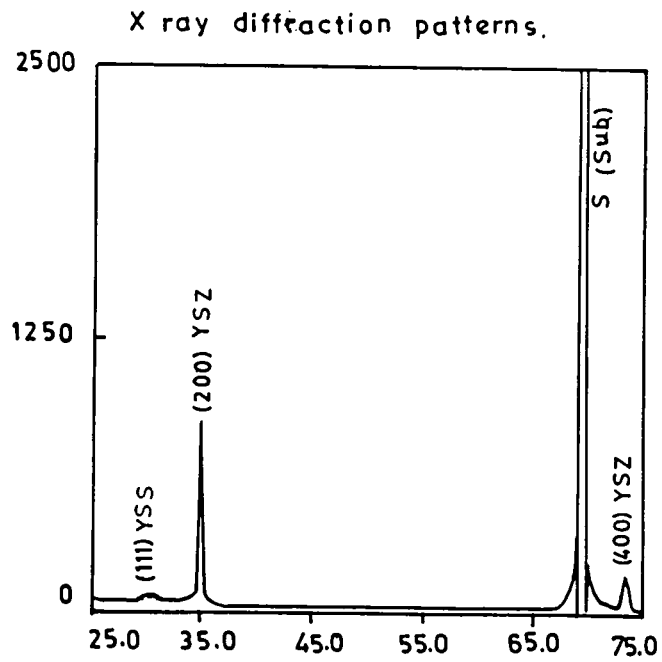
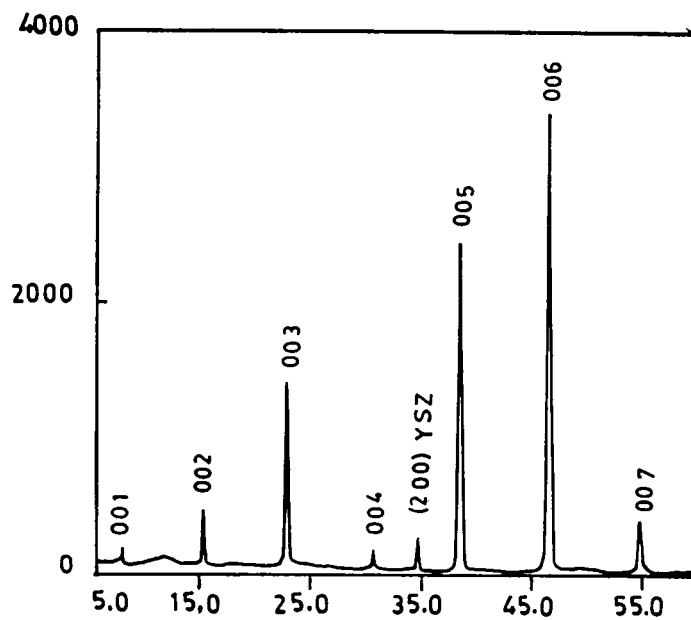


Figure 2.a)- XRD pattern of YSZ buffer on SI <100> deposited at 800° C and 30 mTorr sputtering gas pressure.



b)- XRD patter of YBCO on YSZ buffered si <100>.

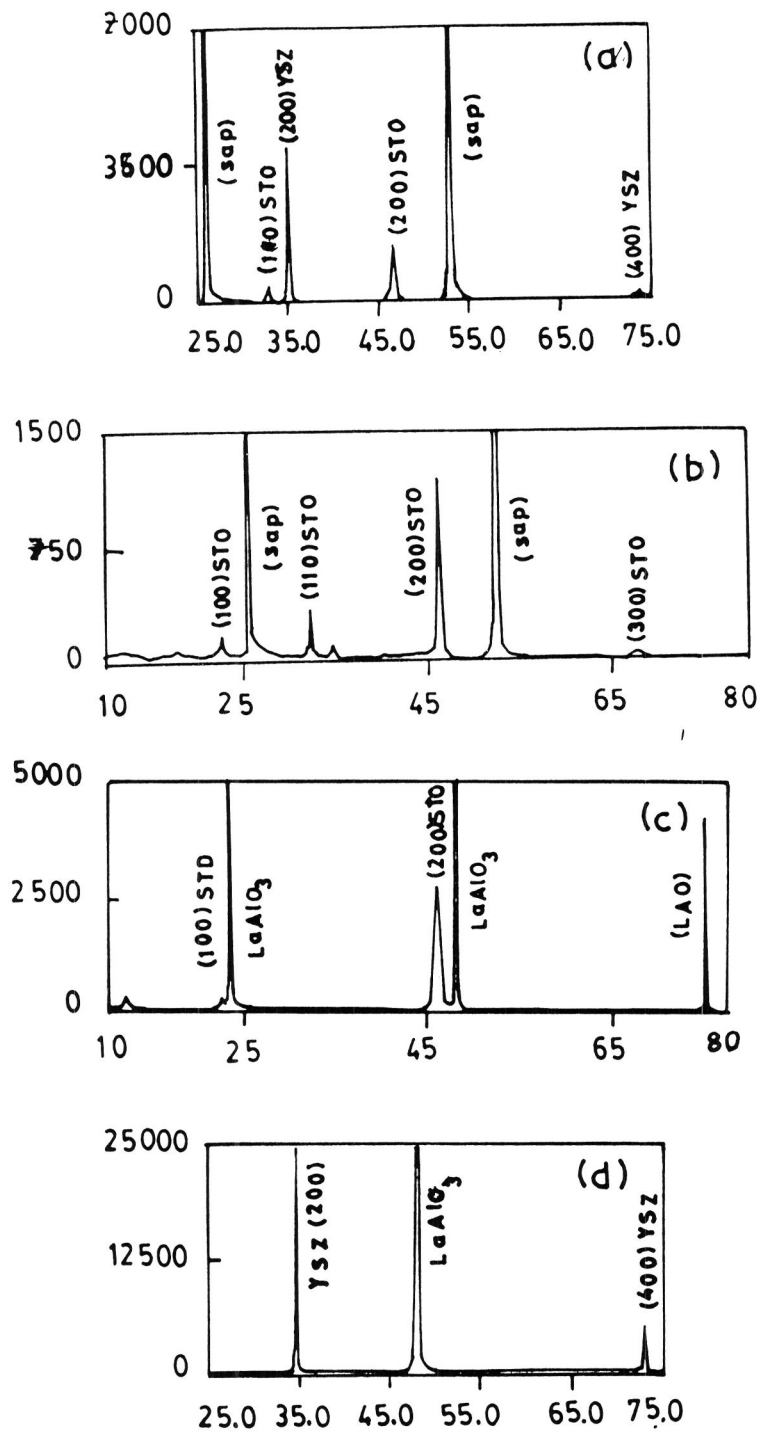


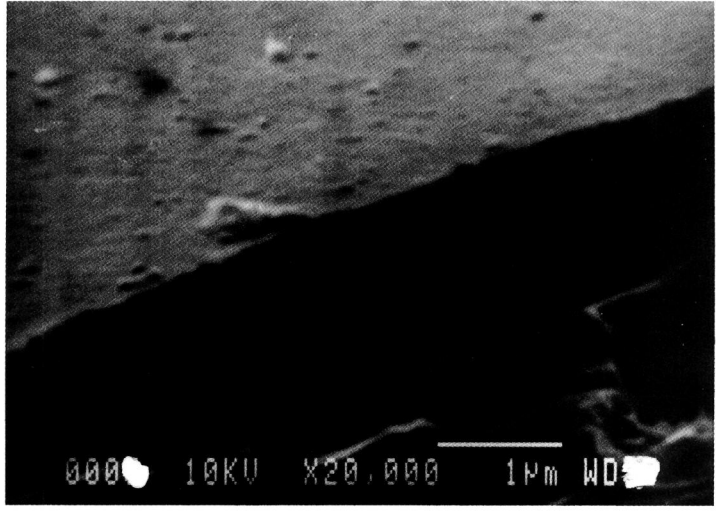
Figure 3.a)- XRD pattern of STO film on YSZ buffered Sapphire substrate deposited at 700° C and 20 mTorr sputtering gas pressure.

b)- XRD pattern of STO film on Sapphire substrate deposited at 700° C mTorr sputtering gas pressure.

c)- XRD pattern of STO buffer layer on  $\text{LaAlO}_3$  substrate deposited at 700° C and 20 mTorr sputtering gas pressure.

d)- XRD pattern of YSZ thin film on  $\text{LaAlO}_3$  substrate deposited at 700° C and 20 mTorr sputtering gas pressure.





(a.)



(b.)

Figure 4.- Scanning electron micrographs which shows the columnar growth for  
a) YSZ film on Si substrate deposited at 25 mTorr.  
b) STO layer on SI substrate deposited at 20 mTorr.