



Synthesis and characterization of strontium titanate using chemical solution deposition method

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ABSTRACT

Strontium titanate has been synthesized successfully by using solution growth and Chemical solution deposition method in powder and thin film form. The chemical composition of the resulted samples were analyzed by EDX and the morphology was observed by Scanning electron microscope. The structure and phase composition were characterized by X-ray diffraction.

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Introduction

Strontium titanate (SrTiO_3) is one of the most fascinating perovskite systems having a high dielectric constant of quantum paraelectric nature that has been giving surprises in materials research for several decades. In MOS devices, the dielectric layer used between the gate metal and silicon substrate is SiO_2 . High capacitance is required to produce high transistor current. This is very difficult with silicon dioxide as it has low dielectric constant. For future high speed applications of the MOS devices, strontium titanate may replace silicon dioxide, if the material properties are slightly modified to get higher band gap and lower leakage currents.

In the electronic ceramic industry Strontium Titanate is used as semiconductor material at high temperatures or as dielectric material for internal boundary layer capacitors (IBLC), due to its high dielectric constant and excellent stability with temperature and applied voltage [1-3].

Thin films of strontium titanate are used as tunable high temperature superconducting microwave filters. They are used as substrates and buffer layer for coating of superconducting substances. Strontium titanate has also been used widely for special optical windows due to its high refractive index (2.41) and as high quality sputtering target. STV (Super strontium Titanate Varistors) are symmetrical voltage-dependent non-linear resistors that are made primarily of strontium titanate and sintered at high temperatures.

They have excellent non-linear coefficient characteristics of voltage and high capacitance for reducing transient voltages. These varistor rings are especially suitable for absorbing micromotors spark noise and protecting the micromotors contact point.

SrTiO_3 also has superconductivity when small amount of electron carriers are added by oxygen vacancies.

Among various synthesizing techniques of SrTiO_3 , Verneuil method is the oldest and most common method conventionally used for synthesis of bulk crystalline materials of strontium titanate. The other common method of preparation is the solid

state reaction method to get bulk polycrystalline samples. STO thin films are usually prepared by CVD, PVD, hydrothermal synthesis method, sputtering and MOCVD or by sol gel methods.

In this paper, we report a simple process to prepare Strontium Titanate powder and thin films by the solution growth and Chemical bath deposition method which will be the first of its kind and is never reported in literature.

Experimental procedures

An aqueous solution of a metal fluoro complex which is slowly hydrolyzed by adding water, boric acid (H_3BO_3) or Al (III) nitrate or Fe (III) chloride.

The addition of water forces precipitation of the oxide, and boric acid (BA) or Al(III) nitrate or Fe(III) chloride acts as a fluorine scavenger destabilizing the fluoro complex and promoting precipitation of the oxide.

Solution Growth method

The starting materials employed were ammonium hexafluorotitanate $[(\text{NH}_4)_2\text{TiF}_6]$ (AHFT), BA and strontium nitrate (SN). They were separately dissolved in deionized water, and then mixed to form a homogenous solution containing different molar ratios of AHFT: SN:BA. The typical molar ratios are 1:1:3, 1:1:4, 1:1:5, 1.3:1:3, 1:1.3:3, 1.5:1:3, 1:1.5:3.

Chemical solution deposition

The same precursors like solution growth was used and the deposition of a solid phase was conducted by floating the substrate on the surface of the solution with the ground side upside down at 50°C and 60°C for 3-/4.5 h. The substrate was floated to prevent particles formed in the solution from accumulating on the substrate surface. After deposition, the substrate was allowed for air-drying.

The chemical composition of the resulted samples were analyzed by EDX and the morphology was observed by Scanning electron microscope. The structure and phase composition were characterized by X-ray diffraction.

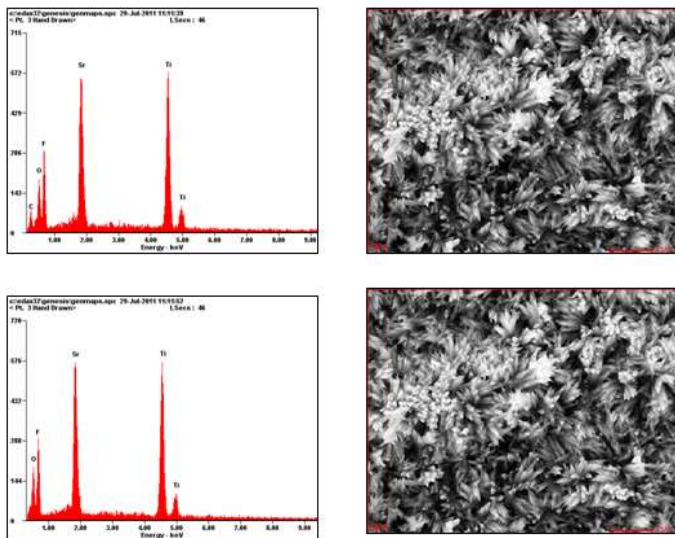


Fig.1 EDX and SEM pictures of as-grown samples grown in Solution Growth method

Results and Discussion

Solution Growth method

The molar ratio of AHFT:SN:BA are taken in 1:1:3, 1:1:4, 1:1:5, 1.3:1:3, 1:1.3:3, 1.5:1:3 and 1:1.5:3. In this obvious precipitation occurred after 30 min soaking but it was left over about 3-4.5 hours for the reaction. Only 1:1:3, 1.3:1:3 and 1:1.3:3 shows remarkable results. EDX and SEM (Fig.1) shows the presence of excess fluorine in the samples before annealing which could be removed when annealed to the temperature above 500°C. Boric acid, the fluorine scavenger shows no change when its ratio was increased.

Chemical bath deposition

Deposition on Glass Substrate

The glass substrate was floated upside down above the liquid and the remaining procedures were followed as before. The rate of deposition slightly increases as the exposure time increases. But the most exposed samples had more impurities like ammonia, fluorine, etc. During annealing the deposited films gets departed from the glass substrate due to its low wetting factor.

Deposition on ITO coated glass substrates

When the glass substrates are replaced by the ITO coated glass substrates the wetting factor was slightly increased. Indium Tin Oxide is basically a conducting material with low resistivity, transparency in the visible and high infrared reflectivity, raises the hope of developing a device for FET applications.

The SEM pictures shows when the annealing temperature increases from 500°C to 600°C the crystal shows a flower like structure from the needle like structure. The sample taken in the ratio of 1.3:1:3 shows low wetting factor (fig.2(e)) in the ITO coated glass substrates.

When the proportion of Sr(NO₃)₂ increases instead of (NH₄)₂TiF₆, the crystal shows remarkable growth. More addition of fluorine scavenger shows no change in the above samples. /

Fig.3 shows the XRD peak results of the different samples which greatly coincides with the JCPDS data.

Table.1 for the identification of the samples in XRD profile

Sample id	IA1	IB1	IA2	IB2	IA3	IB3
Ratio	1:1:3	1:1:3	1.3:1:3	1.3:1:3	1:1.3:3	1:1.3:3
Annealed temp°C	50	60	50	60	50	60

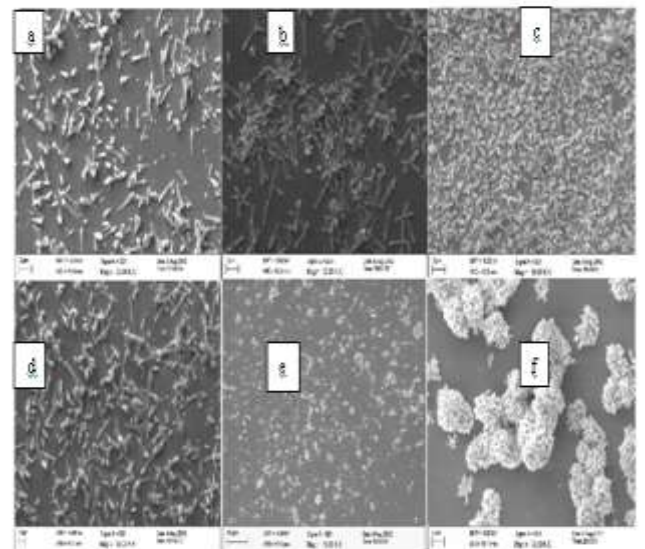


Fig.2 SEM photographs of (a) sample in the ratio 1:1:3 annealed @500°C (b) sample in the ratio 1.3:1:3 annealed @500°C (c) sample in the ratio 1:1.3:3 annealed @500°C (d) sample in the ratio 1:1:3 annealed @600°C (e) sample in the ratio 1.3:1:3 annealed @600°C (f) sample in the ratio 1:1:3 annealed @600°C.

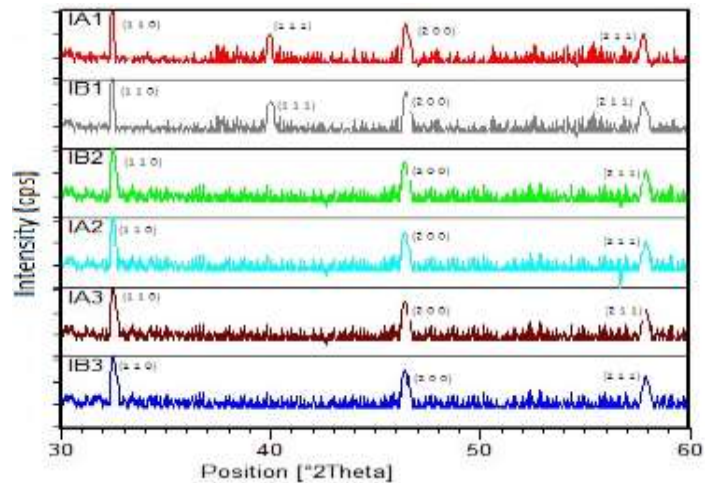


Fig.3 XRD profiles of the samples grown in different proportion and annealed temperatures

Conclusions

Strontium Titanate was successfully synthesized by using solution growth and chemical solution deposition method in the low temperature of about 50 to 60 degrees. The ITO coated glass substrate shows greater wetting factor than in ordinary glass substrate. Increase in annealing time shows change in crystal structure.

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