Solution Route Synthesis of Strontium Ruthenate ($SrRuO_3$) with Cetyl Trimethyl Ammonium Bromide (CTAB) as a Surfactant and Study its Characterization through TGA/DTA, XRD and SEM-EDXA

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ABSTRACT: We have under taken the synthesis and characterization of a novel nanomaterial Strontium Ruthenate and explored the possibility of its role in design of a supercapacitor. Strontium Ruthenate is synthesized by solution route method and it is characterized by TGA, XRD and SEM-EDXA.
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Introduction

Strontium Ruthenate ($SrRuO_3$) is considered as perspective oxide electrodes, e.g. for ferroelectric and high-k materials. The close chemical and structural similarity between electrode and functional material in this case minimizes interface electrochemical reactions, charge injection in oxide and other detrimental processes, thus improving retention performance, fatigue resistance, number of operational cycles, etc. However, device integration utilizing SRO electrodes necessitates high surface stability of the material. Indeed, the interface properties in the SRO based heterostructure will be strongly dependent on the interface properties and atomically smooth interface without interface states [1, 2], etc. Another reason for our interest to this material is that it is one of the end members for the familily of layered strontium ruthenates $Sr_nRu_{n-1}O_{3n-2}$, which exibit a range of interesting bulk electronic properties, ranging from *p*-wave superconductivity in Sr 214 to metamagnetic behavior in Sr 327 and ferromagnetism in 113.

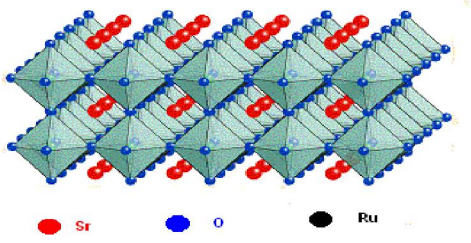

Fig 1: Structure of $SrRuO_3$

These materials exhibit extremely interesting surface behavior, including surface specific Mott transition, etc. However, while the surfaces of layered members of strontium ruthenate family (n = 2,3,4) can be prepared in-situ by cleaving, perovskite 113 is not amenable to this procedure and thus atomically clean surface must be prepared using alternative ways [3,4]. The structure of $SrRuO_3$ as shown in the figure 1.

Pervoskite Structure:

The perovskite structure is adopted by many oxides that have the chemical formula ABO_3. The structure is very versatile having many useful technological applications such as ferroelectrics catalysts, sensors, thermopower and superconductors. The general crystal structure is a primitive cube, with the B cation in the middle of the cube, the A cation in the corner and the anion, commonly oxygen, in the centre of the face edges. The structure is stabilized by the 6-fold coordination of the B cation (octahedron) and 12-fold coordination of the A cation. The packing of the ions can be thought of as the A and O ions together forming a cubic close-packed array, where the B ions occupy a quarter of the octahedral spaces [5, 6].

Although the primitive cube is the idealized structure, differences in radius between the A and B cations can alter the structure to a number of different so-called distortions, of which tilting is the most common one. With perovskite tilt the BO_6 octahedron twists along one or more axes to accommodate the difference as shown in the figure 2. Complex perovskite structures contain two different B-site cations. This results in ordered and disordered variants.

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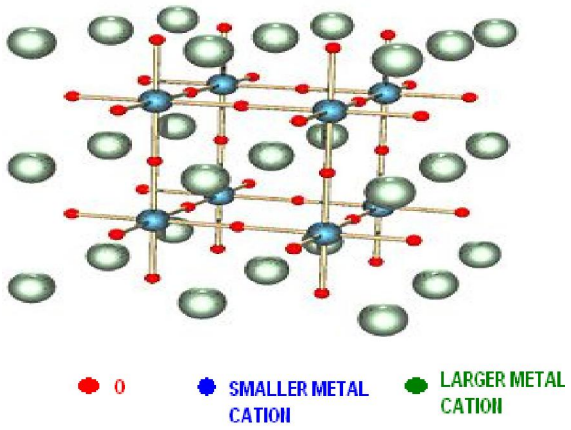
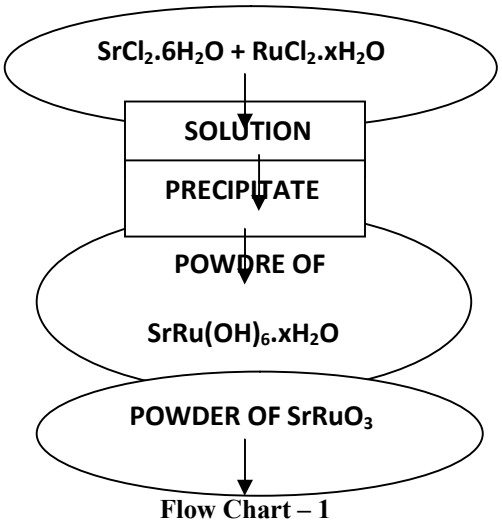


Fig 2 : Pervoskite Structure

Synthesis of Strontium Ruthenate ($SrRuO_3$):

The solution synthesized nano-powders of strontium ruthenate were characterized by the following instruments. Thermal stability of ruthenate powder was investigated by using thermal analyzer (Mettler, Toledo, Model 851e) in dynamic nitrogen atmosphere in the temperature range 25 ^oC to 800 ^oC. Surface morphology, particle size and elemental composition were determined by using Scanning Electron Microscope-Energy Dispersive X-ray Analyser SEM-EDX, Model XL 30, Philips, Holland). X-ray diffractograms (XRD) were recorded on X-ray diffractometer (X-ray Generator, Model PW 1729, Philips, Holland) by using CuKa (k =1.5405 A°) and Ni as the filter. Stoichiometric amount of $SrCl_2.6H_2O$ and $RuCl_3.xH_2O$ are dissolved separately in 0.04% (w/w; 1.0 mM) Cetyl Trimethyl ammonium bromide (CTAB) solution as surfactant. The two solutions were mixed, stirred and the pH of the mixture was slowly increased up to 9.41 by addition of dilute NaOH.



Flow Chart – 1

The reaction mixture was maintained at pH between 9.41 by using dilute HCl and NaOH solutions for about 1 h by constant stirring. The reaction mixture was allowed to settle for an hour, filtered and washed with distilled water and the powder was dried at 100^{o}C. The dried sample was ground to fine powder. This powder was further dried in vacuum oven at 250 ^{o}C for 4 h. The synthesis is shown in the flow chart flow chart-1.

Result and Discussion:

Thermal stability i.e. weight loss/original with different temperatures of the prepared samples were investigated by using Thermal Analyzer (Mettler Toledo 851e) in dynamic nitrogen atmosphere in the temperature range 30°C to 900°C at 10°C/min. The surface morphology, particle size and elemental composition were determined by using Scanning Electron Microscope with Energy Dispersive X-Ray Analysis (SEM-EDXA, Model XL 30, Philips, Holland Phase identification was carried out by X-ray Diffractometer (X-Ray generator, Regaku Miniflex, Japan) employing a scanning rate of 2°/minute in diffraction angle 2θ range from 10° to 80° and CuKα radiation (λ=1.5405 A°).

Thermal Analysis (TGA/DTA):

Thermal analysis of strontium ruthenate synthesized by solution route method as shown in the figure 3. The first weight loss is due to water elimination and the second weight loss is due to the formation of Sr and Ru complex hydroxides. The third weight loss is due the formation of $SrRuO_3$.

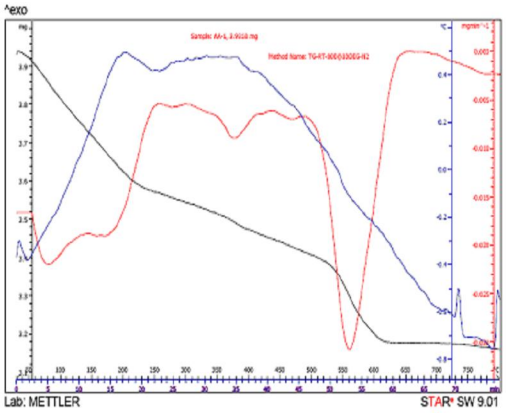


Fig.3: TGA/DTA result of nanomaterial $SrRuO_3$

X-Ray Diffraction Studies:

Figure 4 is the XRD of strontium ruthenate (SRO) prepared at pH 9.41 by solution route and dried at 250 ^{o}C in air. According to the theoretical and experimental values of $SrRuO_3$ the majority of the peaks matches with the reported strontium ruthenate. The three XRD major peaks of $SrRuO_3$ which are matching with the RuO_2 peaks and the above XRD pattern matched

with JCPDS data (JCPDS No. 43-1027)

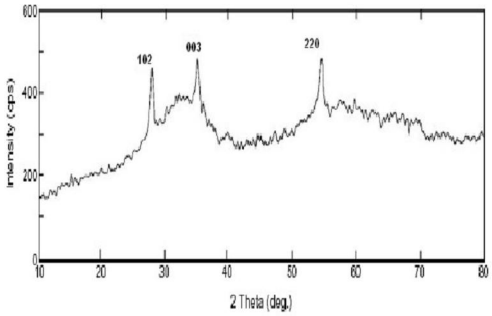


Fig 4:XRD result of nanomaterial $SrRuO_3$

Scanning Electron Microscopy with Energy Dispersive X-Ray Analysis(SEM-EDXA):

The scanning electron microscopy with energy dispersive X-ray analysis (SEM-EDXA) for prepared materials are explained here. Figure 5 shows the SEM image of nanocmaterial $SrRuO_3$ synthesized by solution route method. This SEM image clearly shows the morphology of $SrRuO_3$ nanoparticles with an average particle size of 50-100 nm. Figure 6 is the EDX spectrum of nanomaterial $SrRuO_3$. Table 1 gives the element composition of $SrRuO_3$ which matched with standard reported value.

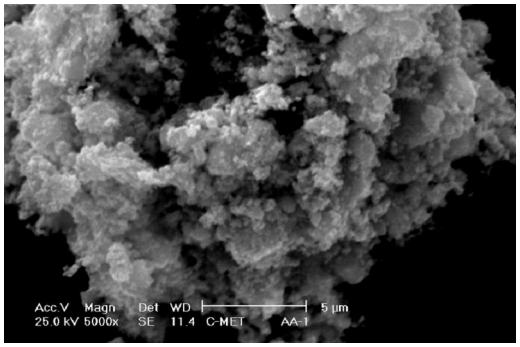


Fig 5 (a)

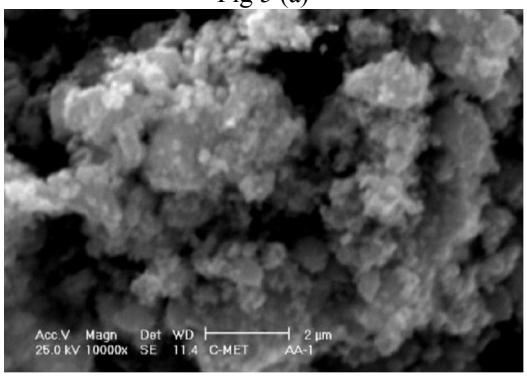


Fig 5 (b)

Fig. 5. SEM micrographs of a) nanosized $SrRuO_3$ (× 4000), b) nanosized $SrRuO_3$ (×8000).

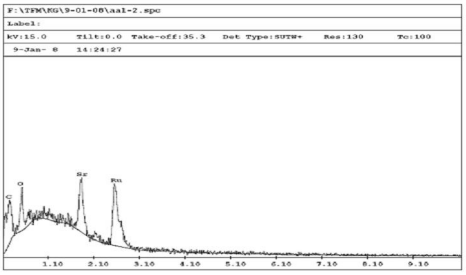


Fig 6: EDX spectra of $SrRuO_3$

Table1

EDAX ZAF Quantification (Standardless)
Element Normalized

Element	Wt %	At %	K-Ratio	Z	A	F
C K	29.92	59.09	0.1055	1.1338	0.3110	1.0001
O K	19.09	28.31	0.0383	1.1118	0.1805	1.0001
SrL	17.68	4.79	0.1464	0.8345	0.9850	1.0070
RuL	33.31	7.82	0.2701	0.8393	0.9663	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
C K	26.59	9.35	3.80	2.84
O K	21.66	17.09	4.84	1.27
SrL	43.74	25.26	3.20	1.73
RuL	56.41	12.41	2.48	4.54

4. Conclusion

We successfully synthesized Strontium Ruthenate nanomaterial at room temperature and pressure. The formation of pure phase of $SrRuO_3$ was confirmed by XRD. TGA/DTA is used to analyse the material and stepwise weight loss was correlated with formation of various intermediates. SEM images clearly showed the morphology of $SrRuO_3$ nanoparticles with an average particle size of 50-100 nm.

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