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Synthesis and Characterization of LSM Thin Films as Cathode for SOFC

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ABSTRACT

Strontium-doped lanthanum manganite (La0.9Sr0.1MnO3-δ-LSM) have synthesized by using spray pyrolysis techniques. The deposition of thin films via spray pyrolysis involves spraying metallic salt solution on a heated substrate. The solution droplets reach the substrate surface, where solvent evaporation and the decomposition of the salt occurs, forming a film. The film morphology and thickness depends on the volume of solution sprayed and substrate temperature. Strontium-doped lanthanum manganite (La0.9Sr0.1MnO3-δ-LSM) were synthesized by spray pyrolytic technique in thin film form on glass (quartz) substrate optimizing the spray parameters to obtain pin-hole free, crack free and porous thin film sintered at 650oC and thin films were characterized by thermo gravimetric analysis, X-ray diffractometry (XRD), Scanning electron microscopy. Film thickness measurement was carried out by using surface profilometer.

Keywords: Spray Pyrolysis, Cathode, LSM, SOFC.

I. INTRODUCTION

It has important to the preparation of thin film size in nanometre because of reduced operating temperature of fuel cell. The use of LSM cathode in bulk form is not applicable for a low-temperature solid oxide fuel cell (SOFC) due to its low oxygen ion conductivity and high activation energy. Hence the attempts are made that to form LSM thin film at the minimum operating temperature. The LSM thin films which are used as cathode for SOFC are synthesized by chemical spray pyrolysis. The Spray pyrolysis is the cheap and effective Technique. This method is convenient for preparing pinhole free, homogenous and porous thin film with required thickness. Numbers of researchers have employed the spray pyrolysis technique for preparation of nanoparticles. In the spray pyrolysis technique various parameters such as spray rate, substrate to nozzle distance, air pressure and deposition rate and substrate temperature also affect structural and optical properties of the thin film.

In the present work we have synthesized LSM thin films by using spray pyro lytic technique. The final material of LSM film was characterized by Bruckner X-Ray Powder diffract meter, the Morphological imaging had also done by Jeol Scanning Electron Microscope.

II. METHODS AND MATERIAL

Experimental:

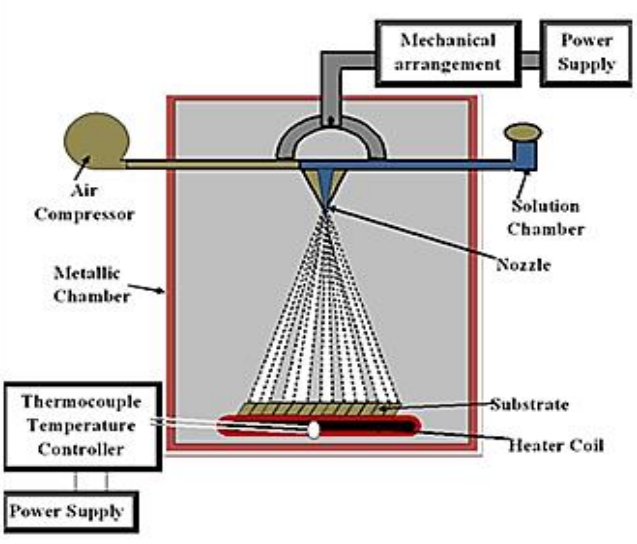

Figure 1. Systematic Diagram of Spray pyrolysis system

Systematic diagram and experimental set up of spray pyrolysis system (Fig.1 and 2) contains a spray nozzle, hot plate for heating substrate and mechanical system for rotor, thermocouple included temperature controller and air compressor. The spraying system and hot plate are kept inside the air tight box is fitted with an exhaust fan to remove the toxic gases produced during the decomposition of the spray solution.

The Lanthanum Strontium Manganite ($La_{1-x}Sr_xMnO_{3-\delta}$) thin films are deposited on glass substrate by using Chemical Spray Pyrolysis technique. The basic principle used in chemical spray pyrolysis technique is that, spraying a metallic salt solution on a heated substrate. The solution droplets reach the substrate surface, where solvent evaporation and decomposition of the metal salt occurs, forming a film. The film morphology and thickness depend on the volume the solution sprayed and substrate temperature.

Figure 2. Photograph of Spray pyrolysis system.

The stoichiometric amounts of precursor (Loba Chmie and Alfa Aesar grade) Lanthanum Nitrate Hexa hydrate La $(N_3O_9).6H_2O$, Strontium Nitrate Sr $(NO_3)_2$, Manganese Nitrate tetra hydrate $MN(NO_3)_2.4H_2O$ were dissolved in double distilled water and stirred with magnetic stirrer for 15 minutes. This precursor solution is used to deposit LSM thin films on glass substrate at deposition temperature of 350^{o}C, this further leads to pyro lytic decomposition of these metallic salts and formation of Lanthanum Strontium Manganite thin film. The precursor flow rate is maintained as 3 ml/min, the nozzle to substrate distance is kept 20 cm and adherent thin films were obtained. The post deposition annealing conditions were chosen to be 2 hours at 650^{o}C in air. The deposited $La_{0.9}Sr_{0.1}MnO_{3-\delta}$ (LSM) thin film was used for structural and morphological characterization. Morphology of the composite was measured by scanning electron microscope (Jeol Scanning Electron Microscope) on glass substrate. The phase identification of the thin film was performed using X-ray diffractometry with cu k_α radiation (λ = 1.5414A^o) with 2θ ranging from 20^o to 55^o. TGA/DSC was carried outby SDT Q600 V20.9 Build 20 instrument in air at 10^{o}C/min.

Table 1. Deposition parameters of LSM thin films.

Spray parameter	Quantity	Unit
Spray solution volume	40	ml
Carrier-Air pressure	10	LPM
Nozzle to substrate distance	20	cm
Spray nozzle diameter	0.1	mm
Substrate temperature	350	°C
Spray rate	3	*ml*/min

III. RESULTS AND DISCUSSION

3.1. Thermo Gravimetric Analysis:

In order to predetermine the phase formation temperature of LSM, the TG-DTA has been carried out in the temperature range from room temperature to1200^{o}C. For the LSM thin film the thermal decomposition process occurs in the four steps between 30^{o}C to 1000^{o}C. The first step of thermal decomposition between 30^{o}C to 100^{o}C is related to the mass loss of water. Gas absorbed on the surface of substrate and polymer degradation occurs in the second step up to 330^{o}C. In the third step between (330^{o}C-650^{o}C) the mass loss is attributed to the dust particles. Further weight loss from 650^{o}C to 700^{o}C shows the formation of carbon us phase. Finally the material begins to stabilize at 700^{o}C (fourth step) when perovskite phase formation has been confirmed from the XRD study.

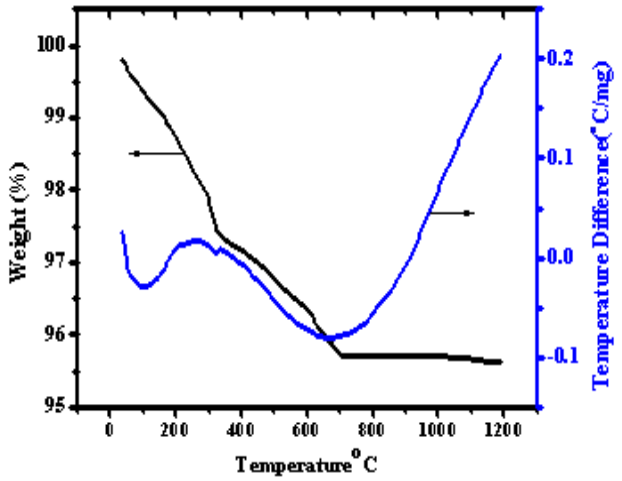

Figure 3. TG/DTA curve of LSM

3.2 XRD Studies

The structural analysis of LSM was performed using an X-ray diffractometry with 2θ ranging from 20° to 55°. The X-ray diffraction (XRD) pattern of LSM sample (fig.5) shows that after deposition the film is amorphous. However, after the heat treatment at 650°C for 2 hours, the crystallization of LSM was observed (fig.6). All diffraction patterns of La0.9Sr0.1MnO3-d perovskite nanostructures show characteristic peak of the perovskite phase. The XRD patterns are good agreement with standard data for hexagonal symmetry with a = 5.53A°, b = 5.53A° and c = 13.35A°. It is observed that material is phase pure without any impurity. The unit cell is rhombohedra with hexagonal lattice parameter. Similar results are reported by R. Chiba et al. The Scherer's relation is used to calculate the crystalline size,

$$D = \frac{K\lambda}{\beta \cos\theta}$$

Where D is crystallite size, λ the wavelengths of X-ray, β is the full width at half maximum of (110) plane. The observed crystallite size is in the range of200-400nm.

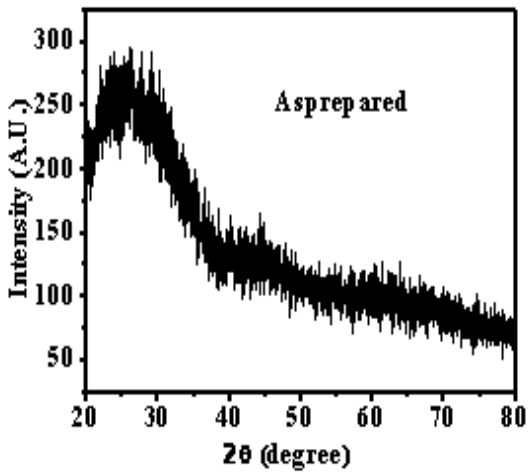

Figure 4. The XRD of LSM thin films asprepared

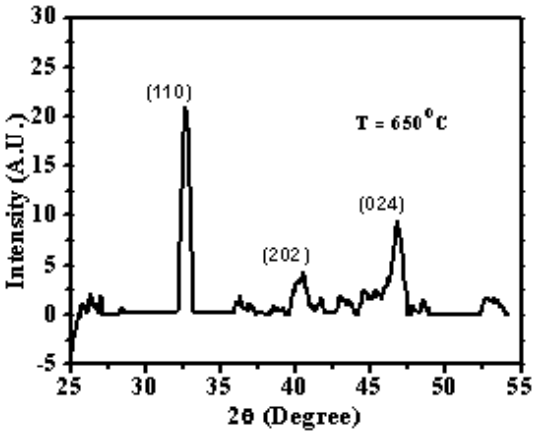

Figure 5. The XRD of LSM thin films **sintered at 650°C.**

3.3 Scanning electron micrograph:

The scanning Electron Microscope images of LSM thin film from fig.6 and fig.7 showing porous morphology composed by the many crystallites of Lanthanum Strontium Manganite. The adherent thin film of LSM observed after sintering at temperature 650°C for 2 hours. A SEM photograph (figure 8) of the film surface shows the required porous structure which is applicable as cathode for solid oxide fuel cell as it is necessary in order to pass oxygen ions at cathode-electrolyte interface. The phase formation takes place after annealing at 650°C for 2 hours in air as compared to before sintering the LSM thin film. The crystallite size from sintered sample shown in fig.8 is approximately 200 nm. The film thickness is about 200 nm to 400 nm and roughness of the sample is found to be 4000A° to 5000A°.

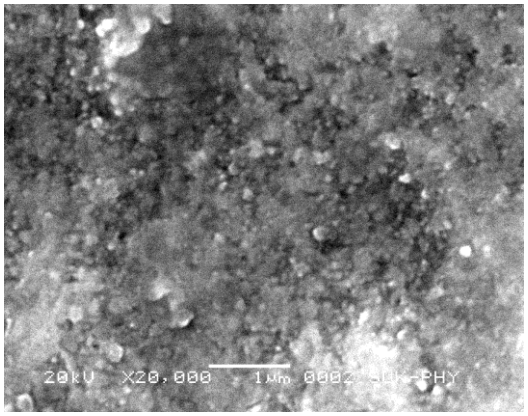

Figure 6. The SEM images of asprepared LSM film

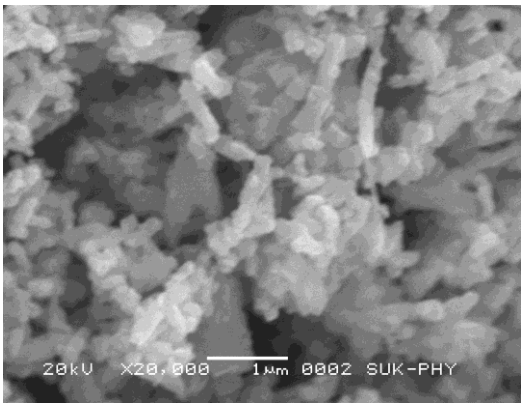

Figure 7. The SEM images of LSM thin films sintered at 650^0C.

IV. CONCLUSION

The spray pyrolysis system has been used for preparing reasonably good quality of LSM thin films. The films thus prepared have high homogeneity. XRD reveals the confirmation of LSM phase formation of a main phase with identification of hexagonal crystalline structure. The microstructure analysis carried out by electron microscopy showed that obtained material has homogenous structure with well formed crystals and also porous.

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