PRINT ISSN : 2395-6011
ONLINE ISSN : 2395-602X

INTERNATIONAL JOURNAL OF SCIENTIFIC RESEARCH IN SCIENCE & TECHNOLOGY

VOLUME 8, ISSUE 1, JANUARY-FEBRUARY-2021

Web Site : www.ijsrst.com

Email : editor@ijsrst.com

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International Journal of Scientific Research in Science and Technology
Print ISSN: 2395-6011 | Online ISSN: 2395-602X (www.ijsrst.com)
© 2021 | IJSRST | Volume 8 - Issue 1

Synthesis and Characterization of Pure and Ce Modified $SmFeO_3$ Thick Films

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ABSTRACT

In present work, $SmFeO_3$ perovskite oxide powder was synthesized by sol gel method. The crystalline structure and surface morphology were analyzed by X-ray diffraction and Scanning Electron Microscopy techniques. Pure $SmFeO_3$ thick films were then prepared onto a glass substrate in desired pattern by screen printing technique followed by firing at 500 ^{0}C for 30 min. As-prepared pure $SmFeO_3$ thick films were dipped into 0.1 M aqueous solution of Cerium Chloride for 1 min and fired at 550 ^{0}C for 30 min to obtained 'Ce modified $SmFeO_3$ thick films'. Both pure and surface modified $SmFeO_3$ thick films were characterized by Energy Dispersive X-Ray Analysis (EDAX) and Field Effect Scanning Electron Microscopy (FE-SEM) techniques. The effect of cerium doping on microstructure and surface morphology of pure $SmFeO_3$ thick film was discussed.

Keywords : $SmFeO_3$, Surface modification, Perovskite, Gas sensor.

I. INTRODUCTION

Detection and monitoring of hazardous gases emitting from auto and industrial exhaust is strongly required all over the world in order to provide better air quality for the survival of human being. Different techniques have been accepted for environmental monitoring. Among them solid state metal oxide gas sensor has been proved to be one of the most promising devices for the detection of variety of pollutants. After the first semiconductor metal oxide gas sensor reported by Seiyama et al in 1962, different semiconducting gas sensing materials along with complex materials have been studied for ethanol, benzene, NO_2 and VOCs[1-3]. But, their gas sensing characteristics such as sensitivity, selectivity, stability and operating temperature are still not meeting the ever increasing requirements of sensor in complicated systems.

Now a day, perovskites of type ABO_3 (A: rare earth, B: transition metal) have created a great deal of interests for chemical sensor because of their interesting novel and improved physical and chemical properties. The perovskite structure can accommodate most of the elements of periodic table in order to produce a series of potential compounds while retaining the perovskite structure. Doping of perovskite oxides with dopants of different oxidation states result in the creation of oxygen vacancies. Partial substitutions at A-site, B-site and/or both A-site and B-site can easily tailor the material properties for specific applications. Therefore

perovskites have wide range of applications including solid oxide fuel cell [4], catalysis [5] and gas sensors [6-8]. Among various perovskite oxides, $SmFeO_3$ has outstanding merit of higher sensitivity and stability in thermal and chemical atmosphere. $SmFeO_3$ is reported to be p-type semiconducting material with orthoferrite phase [9]. Its resistance decreases in presence of oxidizing gases like O_3 ,NO_2 ,ethanol and increases with the adsorption of reducing gases like CO and H_2 [10-12]. For the synthesis of $SmFeO_3$ perovskite different methods like sol-gel method [13] and hydrothermal method [14] have been adopted. Sol-gel method in citric system has advantage of providing $SmFeO_3$ perovskite powder with high sensitivity and selectivity [15].

ABO_3 type perovskite structure of $SmFeO_3$ permits the modification in microstructure and thereby sensitivity and selectivity by partial substitution at A-site and/or the B-site. Electrical conductivity of doped perovskite is affected by different factors like nature of dopants, amount of dopants and the surrounding environment. With the doping of bigger A-site cation, greater reduction stability is possible whereas doping at B-site affects electrical conductivity as well as thermal stability. Researchers have reported the advantages of introducing Ce at A-site and Co, Ni and Mg at B-site [16-19]. S.M. Bukhari et al have reported that the partial substitution of Sm by Ce within the solubility limit improves the electrical conductivity of perovskite as well as prevents it from decomposing under reducing conditions [19]. This creates the possibility of using Ce doped $SmFeO_3$ as a gas sensor for reducing gases.

Generally, doping and dipping techniques are employed for incorporating additives in pure material. Doping method was frequently reported for surface modification in $SmFeO_3$. In the present work, surface modification in $SmFeO_3$ thick films prepared by screen printing method was achieved by dipping technique.

II. METHODS AND MATERIAL

2.1 Preparation of $SmFeO_3$ powder

Sol-gel method was employed for the preparation of fine powder of $SmFeO_3$ perovskite oxide from samarium nitrate $Sm(NO_3)_3.6H_2O$, iron nitrate $Fe(NO_3)_3.9H_2O$ and citric acid monohydrate. Stoichiometric amounts of samarium nitrate, iron nitrate and citric acid monohydrate were mixed in the ratio 1:1:1.provided with grounding in Agate mortar for 30 minutes. To this mixture, ethylene glycol was added under constant stirring at 75°C for 2 hours to yield a sole which was then dried into a gel. The gel was dried in oven at 110°C for 12 hours and allowed to cool naturally. Finally, sample was calcined at 800°C for 4 hours.

2.2 Preparation of SmFeO3 thick films

Pure powder was added to the solution of ethyl cellulose and the mixture of organic solvent in ratio 75:25 to prepare the paste. This paste was then screen-printed on glass substrate in desired pattern.

2.3 Preparation of Ce surface modified thick films

In order to obtained surface modified thick films, as-prepared pure thick films were dipped into 0.1 M aqueous solution of cerium chloride for 1 min, 3min and 5 min. After drying, these films were fired at 550 °C for 30 min and then used for further characterization.

III. RESULTS AND DISCUSSION

3.1 X-ray diffraction and surface morphology analysis of pure $SmFeO_3$ powder

X-ray diffraction pattern of synthesized pure $SmFeO_3$ powder is shown in Fig. 1. This X-ray diffraction pattern matches well with the standard JCPDS card number 39-1490 conforming that the prepared powder has perovskite phase and orthorhombic symmetry. Importantly single phase perovskite structure was observed without presence of secondary

phases. The lattice parameters of the sample were calculated from XRD pattern according to the formula $d= (h^2/a^2+k^2/b^2+l^2/c^2)^{-1/2}$, where (h, k, l) are indices of crystallographic planes, d is the interplanar distance and (a, b, c) are lattice parameters. The values of lattice constants a, b and c are 5.604 A^0, 7.704 A^0 and 5.397 A^0 respectively for $SmFeO_3$ powder prepared by sol-gel method. By means of Scherer's formula, $D= 0.89\lambda/\beta\cos\theta$ where λ is wavelength of X-ray, θ is diffraction angle and β is true half-peak width, the crystalline particle size was estimated and is found to be 50.08 nm. The volume of unit cell of prepared sample is 233.05 A^{03}

Fig 2 represents the SEM images of the sample to study its surface morphology. The micrograph indicates that the morphology of the particle is irregular because sintered material being crushed until powder form is obtained. The average size of particle is 100 nm.

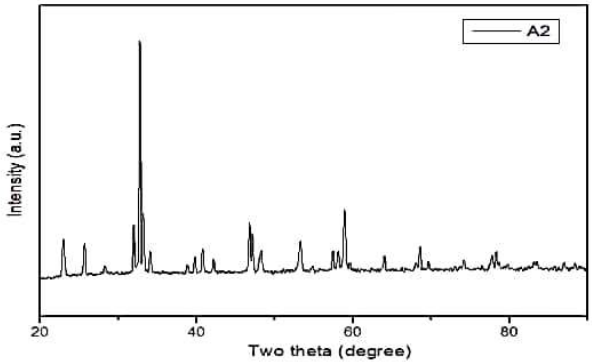

FIG. 1: XRD PATTERN OF $SmFeO_3$ POWDER

3.2 Surface morphology of pure and modified SmFeO3 thick films by FESEM analysis

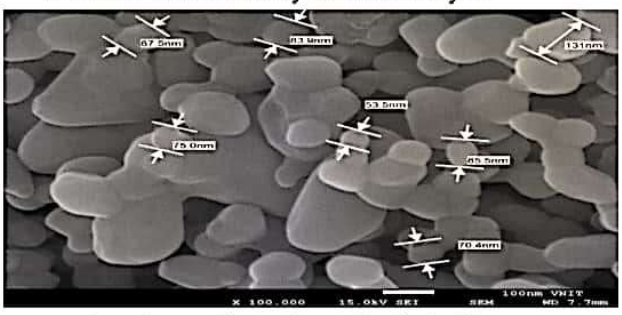

Fig. 2: FESEM of pure $SmFeO_3$ thick film

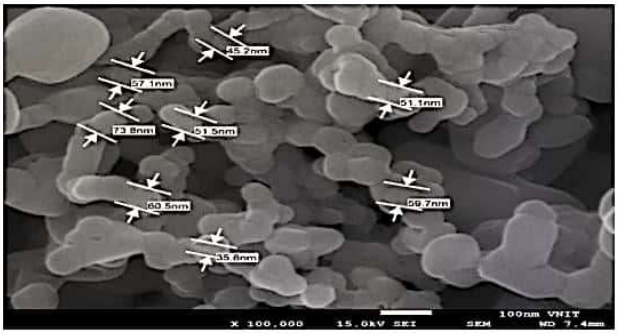

Fig. 3: FESEM of Ce doped $SmFeO_3$ thick film.

Fig. 2 represents the FESEM images of pure $SmFeO_3$ thick film fired at 500 °C for 30 min. The micrograph shows the presence of large number of grains with grain size ranging from 53 nm to 131 nm on the film. The films were highly porous with inner layer of perovskite type oxide adhere to the substrate. Due to the firing temperature, sintering proceeded and growth of the grains was observed. The composition of organic vehicle also influences the morphology of film. The presence of α-terpineol favored the sintering of grains. Fig. 3 depicts FESEM image of Ce doped $SmFeO_3$ thick film for dipping time interval 1 min and fired at 550 °C for 30 min. The micrograph shows the distribution of smaller particles around the larger grains. The smaller particles may be attributed as Ce species. The modified thick film appears to have comparatively high porosity and large surface area for oxygen adsorption.

3.3 Elemental composition of pure and modified SmFeO3 thick films by EDX analysis

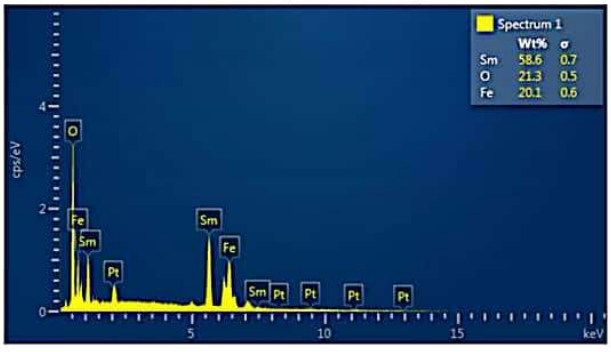

Fig 4: EDX of pure SmFeO3 thick film.

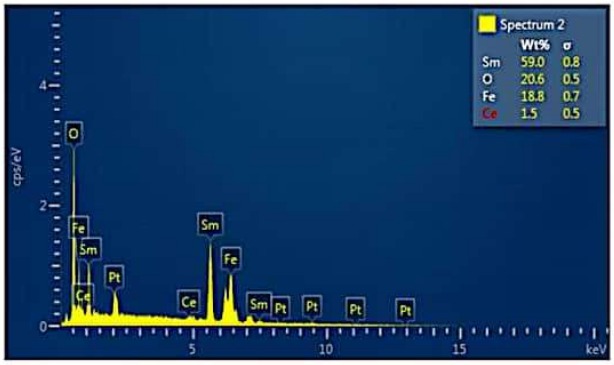

Fig 5: EDX of Ce doped SmFeO3 thick film.

Elemental analysis of pure and Ce doped thick films was carried out by EDX technique. Table 1 represents the wt % of constituent elements of both pure and modified films.

Table 1: Quantitative elemental analysis.

	Pure $SmFeO_3$ thick film	Ce modified $SmFeO_3$ thick film
Sm (wt.%)	58.6	59
O (wt.%)	21.3	20.6
Fe (wt.%)	20.1	18.8
Ce (wt.%)	---	1.5
Total	100	100

It is observed from table 1 that weight percentage of oxygen decreases due to Ce doping. Further it was observed that both the samples are oxygen deficient but oxygen deficiency is more in Ce doped thick film than pure $SmFeO_3$ thick film. Therefore, Ce doped $SmFeO_3$ thick film may facilitate increased oxygen adsorption.

IV. CONCLUSION

The results demonstrated here depict the possibility of synthesis of fine powder of $SmFeO_3$ perovskite oxide by sol-gel method in citrate system. XRD pattern confirms the presence of single phase orthorhombic perovskite structure. Surface modification of $SmFeO_3$ thick films can be achieved by dipping technique. Moreover Surface modification promotes increased oxygen adsorption. FESEM analysis and EDX analysis respectively confirm the structural morphology and the elemental composition of both pure and modified thick films.

V. ACKNOWLEDGEMENT

Authors would like to acknowledge VNIT, Nagpur for providing characterization facilities.

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