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Synthesis and Structural Properties of Lanthanium Doped Tin Dioxide Nanocrystalline

L.P.Chikhale*[1], F.I.Shaikh[2], A.V.Rajgure[3], S.S.Suryavanshi[4]

*[1]Department of Physics, Venkatesh Mahajan Senior College, Osmanabad, Maharashtra, India

[2]Department of Physics, Government Institute of Forensic Science, Aurangabad, Maharashtra, India

[3]G.S.Tompe Arts,Commerce & Science College, Chadur Bazar, Dist-Amravati 444704, Maharashtra, India

[4]Department of Physics, Solapur University, Solapur, Maharashtra, India

ABSTRACT

The present work to study effect of pure and La doped tin dioxide thick films on their optical properties and the oxide powders were prepared by the chemical co-precipitation method. The thick films of La doped SnO_2 were prepared by the screen-printing technique. Thick films of functional material were prepared by using a screen printing technique and then sintered at 750 °C for 2 h. The samples were characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDAX) techniques. XRD studies reveal formation of fine nanocrystalline material. The particle size of sintered powder of pure and La (2 and 4 mol %) doped SnO_2 powders were 15, 10 and 9 nm respectively.

Keywords: TEM; SEM; Screen printing;Thick Film

I. INTRODUCTION

Nanostructured semiconductor oxides are attracting a greatdeal of attention due to their exclusive properties and novel applications. In the recent years, atmospheric pollution has become a major global concern. Gases released from automobile exhausts and industries are severely polluting the environment. Thus, it has not only imperative to develop sensors for the detection but also the quantitative measurement of the toxic and inflammable gases to determine the exact concentration which could be ascertained before some natural disaster may occur. The gas sensor would also facilitate on line monitoring of the hazardous gases and providing safety of working environment [1]. In this paper we are reporting the effect of La doping on the structural and optical of SnO_2 thick films prepared by chemical co-precipitation technique. We have chosen this technique because of the easy of selectivity of resistivity and transmittance by controlling preparative parameters[2-4]

II. EXPERIMENTAL PROCEDURE

2.1 Preparation of undoped and doped SnO_2 powder:

We have opted the simple and economical co-precipitation method for synthesis of undoped and La doped SnO_2.The analytical grade stannic chloride ($SnCl_4.5H_2O$) and ammonia ($NH_3.H_2O$) was used as raw materials. The stannic chloride was dissolved in distilled water to form a transparent solution. Ammonia was added drop wise into the solution under continuous stirring until pH attains around 8 where the precipitation was occurred. The precipitate was washed with de-ionized water for several times to remove chlorine ions [5]. The precipitate was dried under IR lamp for about 2 h and then calcined at 600°C for 2 h in air to obtain fine nanocrystalline powder. The sample was labeled as S1.The as synthesized powder was subjected to TGA to know the phase formation temperature and to decide calcination or sintering temperature. The 98 mol% stannic chloride and 2 mol% lanthanum nitrate were added together in distilled water to form precursor solution. Dilute ammonia was added to the solution to precipitate the constituents together. The precipitates were filtered and washed using de-ionized water and further procedure was same as adopted in case of L1. In a similar way 2 mole and % 4 mol% La doped SnO_2 was prepared and the sample was labeled as L2 and L3 respectively.

2.2. Preparation of thick films:

The thixotropic paste was formulated by mixing the fine powder of SnO_2 with a mixture of organic solvents (a temporary binder) namely ethyl cellulose, butyl carbitol acetate and terpineol. In formulating the thixotropic paste, the ratio of the inorganic to organic part was kept at 75:25 [6]. This thixotropic paste was further used to deposit thick films on ultrasonically cleaned alumina substrate (10mm X 20mm) by using screen printing technique with the nylon cloth of 140 mesh counts. The details of the technique are described elsewhere [7]. The thick films were sintered at 750°C for 2 h in air.

III. RESULTS AND DISCUSSION

3.1. XRD analysis

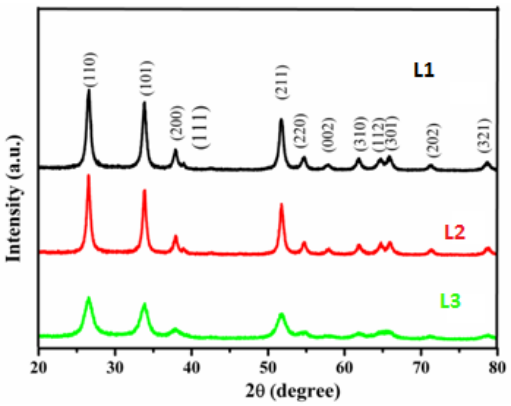

Fig. 1 XRD patterns of undoped (L1) , 2 mol % La–doped (L2) , and 4 mol % La–doped SnO_2 (L3).

Fig.1 show the XRD patterns of SnO_2 thick films with different La concentrations. All patterns exhibit peaks corresponding to the rutile structure of polycrystalline SnO_2 with the maximum intensity peak corresponding to (110) planes and are indexed on the basis of joint committee on powder diffraction standards (JCPDS) data [8]. However, the relative intensity of the peaks decreases with an increase in the La content. It is also observed that the full width at half maxima (FWHM) of the diffraction peaks increases with increasing La content which is in good agreement with the earlier studies [9].The increase in FWHM along with a decrease in peak intensity suggests that La incorporation into the SnO_2 lattice results in a decrease in crystallite size of the films. The decrease in the crystallite size is further supported by the decrease in the sharpness of the peaks with the incorporation of La. Pure SnO_2 has an average crystallite size of 15nm, while 2 mol % La doped SnO_2 shows reduced crystallite size of 10 nm and 8 nm in case of 4mol % La-doped SnO_2. A small crystallite size provides a larger surface area for adsorption of oxygen species and exposure to the test gas, which increases the probability of gas solid interaction, thereby increasing the response. The presence of La in the crystallographic structure increases the formation of oxygen vacancies as required by the charge balance. This effect is in conjunction with the smaller ionic radius of La^{3+} (250 pm) as compared to Sn^{4+} (71 pm) [10].These end result suggest that, the increment of the doping concentration leads to decrease of the lattice parameter and crystalline size, and prevent the growth of SnO_2 Crystallites. The role of La as grain growth inhibitor is also supported by earlier studies [11].

3.2. SEM and EDAX analysis:

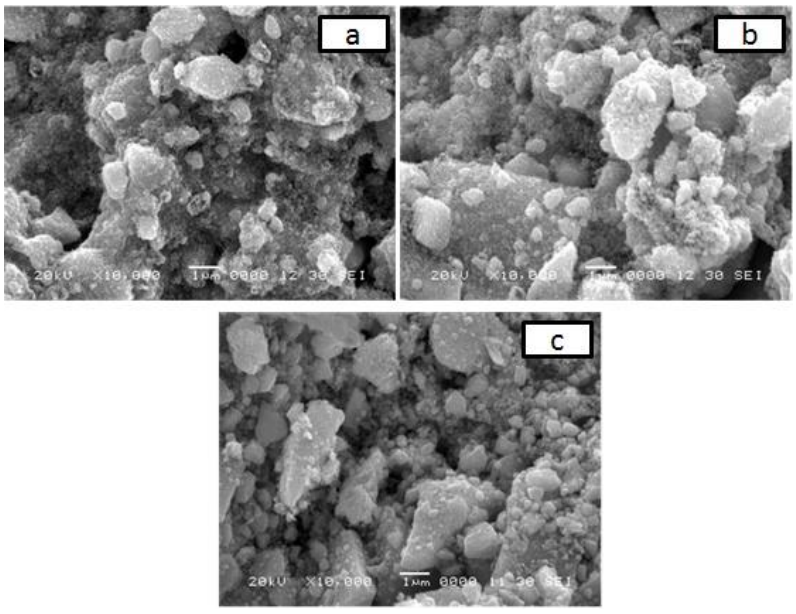

Fig. 2 SEM images of the surface of SnO_2 thick films: (a) undoped SnO_2 (b) 2mol % La doped SnO_2 (c) 4 mol % La–doped SnO_2.

Fig.2 illustrates SEM images of sintered thick film samples. It shows evidence of splintered surface morphology, covering the micro pores on the surface. Pure SnO_2 shows large pore with non-uniform grains (Fig. 2(a)), however; agglomerates can be seen for L2 sample (Fig. 2(b)). In case of 4 mol % doped SnO_2, dispersed nanoparticles with significant porosity were observed. (Fig.2(c)). It is clearly seen that with

incorporation of La the particle size of SnO_2 decreases changes distinctly suggesting La playing imperative role in the growth embarrassment for the SnO_2 material. The decrease of crystallite size caused by doping of La results in high surface energy, thereby agglomerates are favored to form. A chemical composition of thick film was investigated by energy dispersive X-ray analysis (EDAX) and is shown in Fig.3.

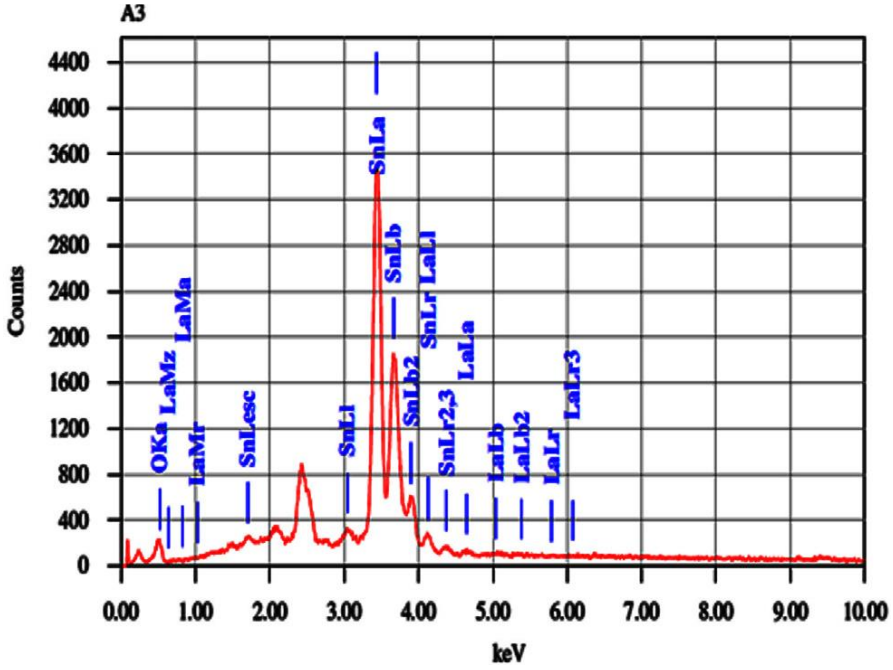

Fig. 3 EDAX spectrum of 2 mol % La–doped SnO_2 thick film.

The EDAX data is in good agreement with the initial precursor concentration. It was found that the La doped SnO_2 thick film was tin deficient and oxygen rich, indicating that the La doped SnO_2 thick film was non-stoichiometric.

3.3. TEM analysis:

The morphology of powders was examined with TEM and the images of pure, 2 mol% and 4 mol% La doped SnO_2 sintered powders are shown in Fig 4(a-c) respectively. There is a noticeable reduction in average particle size of SnO_2 when La is doped into the SnO_2. For pure SnO_2, the size of the crystallites was about 15 nm while the crystallite size decreases to 10 and 8 nm for SnO_2 doped with 2mol% La and 4mol% La respectively. The average crystallite size estimated from XRD is comparable with the size observed from TEM images. In addition, a significant amount of agglomeration was observed in the micrographs especially for L2 and L3 sample.

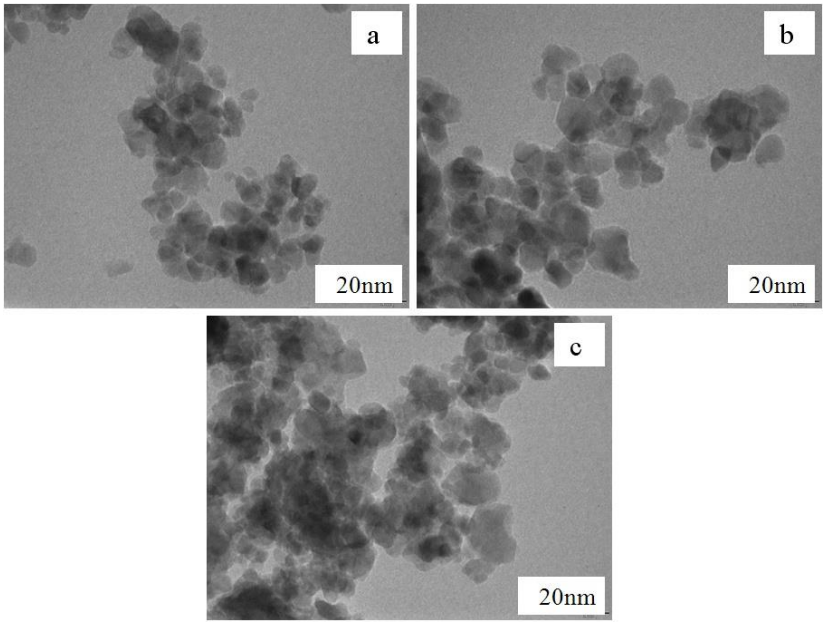

Fig. 4 TEM images of SnO2 thick films: (a) undoped SnO2 (b) 2mol % La –doped SnO2 (c) 4 mol% La – doped SnO2.

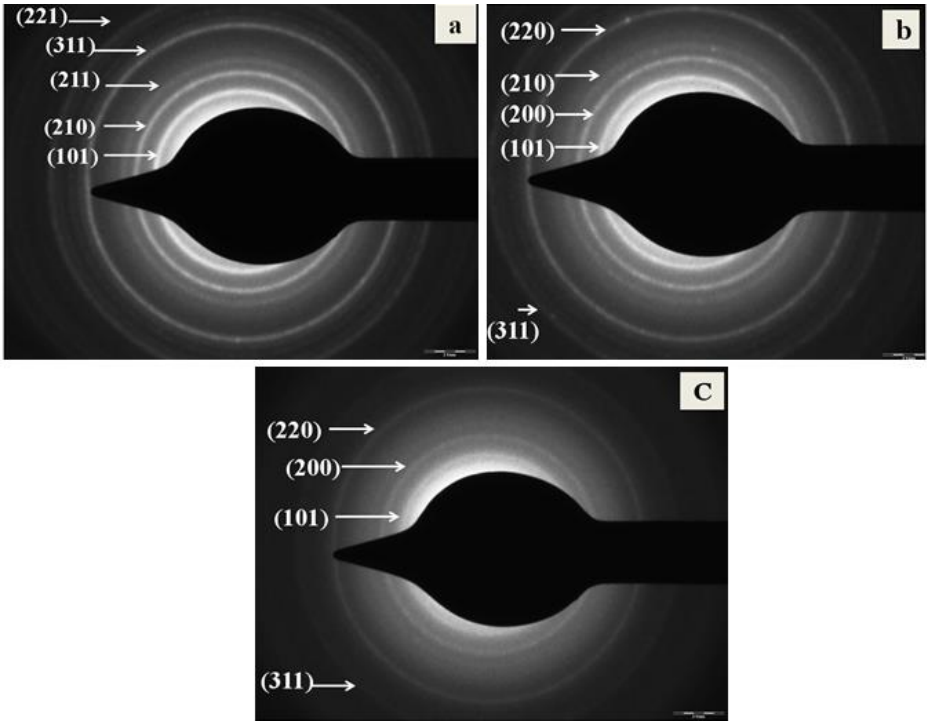

Fig. 5 SAED pattern of SnO_2 thick films: (a) undoped SnO_2 (b) 2mol % La –doped SnO_2 (c) 4 mol% La – doped SnO_2.

The selected area electron diffraction (SAED) pattern of an isolated particle for various SnO_2 thick film samples is shown in Fig.5.The SAED shows bright rings corresponding to the (101), (210), (211), (311), (221) lattice planes of SnO_2 which are in good agreement with the X-ray diffraction pattern.

IV. CONCLUSIONS

The films of co precipitation synthesized undoped and La doped SnO_2 were successfully deposited using screen printing technique. XRD and TEM studies confirmed the nanocrystallinity of undoped and La doped SnO2 material. The structural and morphological studies were proved the role of La as grain growth inhibitor. The XRD, SEM-EDAX, TEM, SAED analysis confirmed that the La doping inSnO_2 can significantly affects morphology and particle size of La doped SnO_2.

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