SnO_2 Thin Film: XRD, SEM and CV Analysis

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Abstract
Tin oxide (SnO_2) thin films were grown on steel substrate via sol-gel spin coating deposition method at temperature 650^0C. The structural and morphological properties of the films were studied by X-ray diffractometer (XRD) and Scanning Electron Microscopy (SEM) respectively. The XRD revealedmicro crystalline nature of tin oxide with orthorhombic structure and SEM images showed very small circular grains distributed throughout the film surface.Specific capacitance of the film was calculated using Cyclic Voltammetry (CV) technique. From CV analysis, maximum specific capacitance was observedforminimum scan rate. The specific capacitance of $138Fg^{-1}$ at scan rate of $100mVs^{-1}$ whereas $334Fg^{-1}$ was obtained at scan rate of $10mVs^{-1}$.
Key words: SnO_2, Spin Coating, XRD, SEM, CV

I. INTRODUCTION

Among various metal oxides, transition metal oxides attracting much attention due to their wide range of applications. SnO_2 in thin film form has engrossed considerable interest. Tin oxide (SnO_2) has promising technological applications in various fields such as catalysis [1], gas sensors [2], solar cells [3], Li-ion battery [4] and supercapacitors [5]. It is an important n-type semiconductor with large band gap and high sensitivity to various toxic or flammable gases [6, 7]. SnO_2 exhibits a number of interesting functional properties such as optical transparency in the visible spectrum [8], chemical stability at high temperatures [9], good surface adsorption properties of oxygen and availability of numerous oxygen species and active acid sites on its surface [10], high specific theoretical capacity [11], and excellent electrical characteristics [9, 12]

Many methods have been developed to synthesize SnO_2 nanoparticles[13] including evaporation of SnO_2 or SnO powders at elevated temperatures [14], hydrothermal routes [15], homogeneous precipitation [16], microwave-assisted aqueous solution using a kitchen oven [17], sol–gel route [18] and sol-gel method [19, 20].

II. EXPERIMENTAL

2.1 Preparation of Gel

The samples of SnO_2were prepared from 0.05M solution. Precursor used was Stannous Chloride[$SnCl_2.2H_2O$]. To prepare 0.05M solution, 0.564gm of $SnCl_2$ in appropriate proportions were dissolved in double distilled water and isopropyl alcohol and stirred at 50ºC on magnetic stirrer for four hours and then aged for two days. Then the sol is spin coated on cleaned steel substrate.

2.2 **Deposition by spin coat method**

In this method, excess amount of the solvent is placed on the substrate, which is then rotated at high speed in order to spread the fluid by centrifugal force. The spin coating process can be broken down into the four stages they are deposition, rotating, annealing and repeating the same process for multilayers. For annealing, films were placed into furnace. Before deposition, the steel substrates were polished with zero grade polish paper and washed with double distilled water in an ultrasonic bath for about 20 min. All deposition conditions and parameters are as shown in table-1.

Sr. No	Parameters	Conditions
1	Precursor	Stannous Chloride
2	Substrate	Steel
3	Spin Time	120sec
4	Spin Speed	3,000 RPM
5	Furnace Temperature	650^0c
6	Annealing Time	60sec

Table1. Deposition parameters and conditions

III. Result and Discussion

3.1 Structural Analysis by XRD

Phase identification was carried out by XRD. A D2 PHASER diffractometer with source CuKα1 with λ =1.54184, the 2θ angle is varied from 10^0 to 80^0. Fig-1 shows the X-ray diffraction (XRD) patterns of SnO_2thin film. The XRD pattern imply that as-deposited film is SnO_2[JCPDS card number 78- 1063] with orthorhombic crystal structure. It is clear from the XRD pattern that the diffraction peaks at angle (2θ) of 33.883, 35.31, 43.485and 50.361 are assigned to (006), (023), (124) and (221) planes of the SnO_2crystal lattice respectively.The values of lattice parameters a =4.677 Å, b = 5.821 Åand c = 15.005 Å were calculated by theXRD data, which are in good agreement with thestandard diffraction pattern [JCPDS card number 78- 1063].

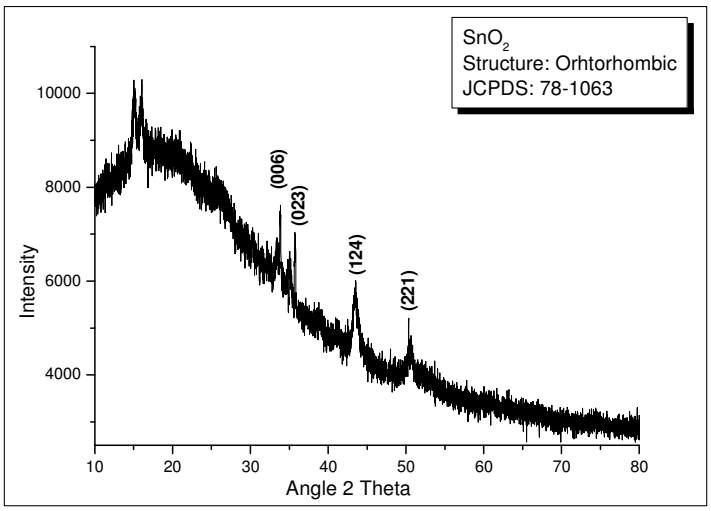

Fig. 1 XRD of SnO2 thin film

3.2 Morphological Analysis by SEM

Surface morphological studies have been carried out by Scanning Electron Microscopy (SEM) using a JEOL JSM-6360 instrument.

The SEM images revealed the formation of thin film well adherent to the substrate and porous structure of the film. Such type of morphology provides greater surface area, which is the prime requirement in supercapacitor [21]. Fig-2 shows SEM micrographs of SnO_2 at two different magnifications (x3,000 and x10,000). At x3,000magnification we can observe very small grains distributed throughout the film surface. The inner image shows the magnified SEM morphology where circular shape of grains can be seen.

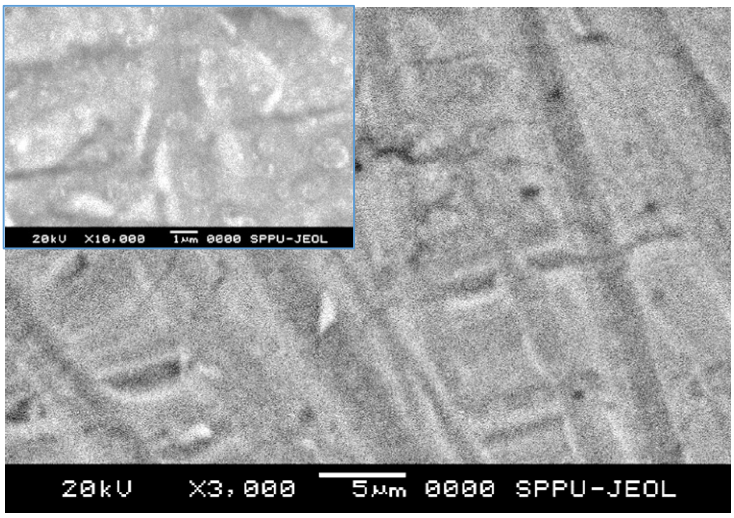

Fig. 2 SEM morphology of SnO2 thin film

3.3 Electrochemical Properties

Cyclic voltammetry is considered to be an ideal tool for indicating the capacitive behaviour of any material. The cyclic voltammetry study is carried out with SnO_2 thin film as a working electrode and platinum wire as counter electrode and SCE as a reference electrode in 0.1M KOH electrolyte. Fig – 3 shows the cyclic voltammograms for SnO_2 thin film electrode with potential window of 1V to -1V/SCE at various scan rates 10, 20, 50, 80 and 100mV/sec.

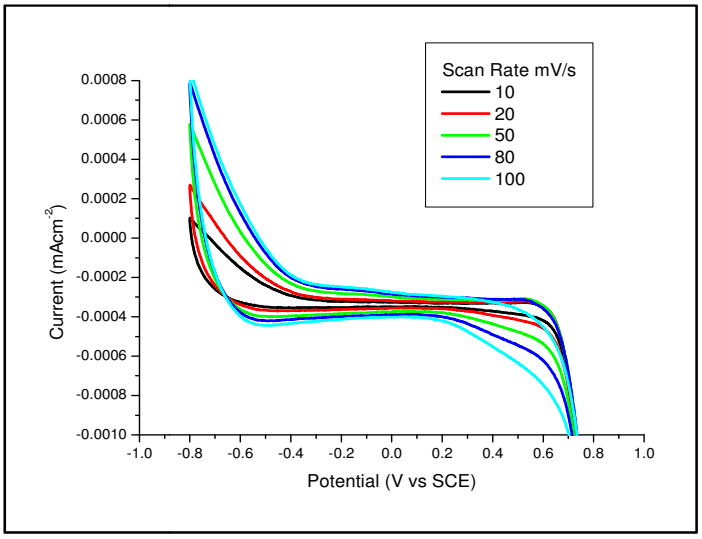

Fig. 3 Graph of Cyclic voltammetry of SnO2 thin film

From the CV curves, it is observed that the reduction and oxidation peaks are visible. This indicates that the electrochemical capacitance of the SnO_2 thin film electrode mainly results from pseudocapacitance. It is observed from the figure, the measured current densities increased and oxidation and reduction peaks are shifted in opposite direction with increase in scan rate. As current under curve slowly increased with scan rate, we conclude that the voltammetric current is directly proportional to scan rate and this is good indication of supercapacitive behaviour [22].

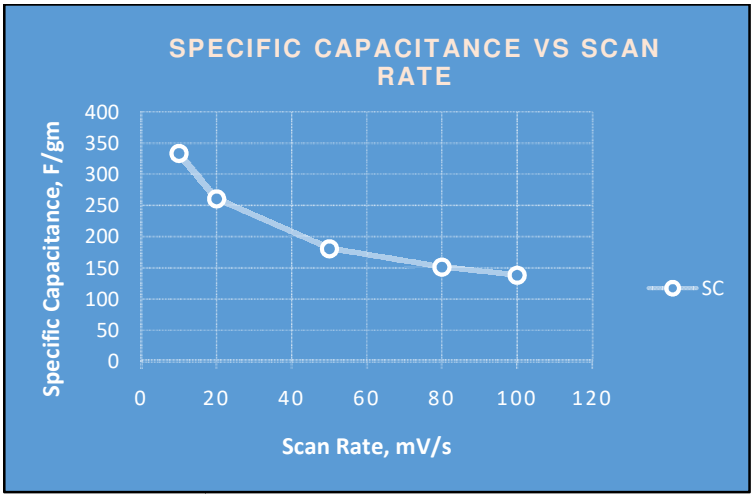

Fig. 4 Specific capacitance vs scan rate

Fig -4. shows the graph of specific capacitance versus scan rate.SnO_2 thin film electrode exhibited a common trend of decreasing specific capacitance values against an increasing scan rate. It is well known that for very low scan rates, the specific capacitance values are higher because the ions have a much longer time to penetrate and reside in the electrode pores and form electric double layers, which are needed to generate higher capacitance[23]. The specific capacitance of $334Fg^{-1}$ obtained at minimum scan rate of $10mVs^{-1}$.

IV. Conclusion

In the present work,SnO_2 thin films weresuccessfully synthesizedby sol gel spin coating deposition technique. XRD revealed crystalline SnO_2 phase with orthorhombic structure. SEM images exhibited the formation of well adherent and small circular grains distributed throughout the film surface. SEM morphology also revealed the porous nature of the film, which is needed for the supercapacitor application. The SnO_2thin film electrode showed good supercapacitive behaviour with specific capacitance of $334Fg^{-1}$ at minimum scan rate of $10mVs^{-1}$.

V. References

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