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SPECTRO-STRUCTURAL-MORPHOLOGICAL STUDY OF RUTHENIUM OXIDE THIN FILM

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ABSTRACT

There is a growing necessity of transition metal oxide thin film for many important technological applications such as smart windows gas sensors, solar cells, super capacitors etc. Among the other transition metal oxides, ruthenium oxide is a potential material as it exhibits interesting structural, optical, chemical, electrical properties. In this investigation ruthenium oxide thin films have been synthesized using spin coating technique. Here ruthenium oxide thin films have been deposited on stainless steel substrate by sol-gel spin coating method. Thin film properties of deposited samples were studied by XRD, SEM, FTIR, and EDAX.AFM. The XRD pattern showed sharp intense peaks conform crystalline structure and indicating tetragonal structure of ruthenium oxide. The SEM images of ruthenium oxide thin films showed total coverage of thin films. The thin film had a dense layer covered by agglomeration of particles forming a porous structure. At higher magnification (X 40,000) a porous structure of ruthenium oxide

KEYWORDS: Supercapacitor, Molybdenum oxide, Sol-gel Spin-coating, CV, Galvanostatic charge-Discharge.

INTRODUCTION

Ruthenium oxide is superior due to the unique combination of characteristics, such as metallic conductivity, high chemical and thermal stability, catalytic activities, electrochemical redox properties, highly reactive with reducing agents due to its oxidizing properties and field emitting behaviour etc. [1]. It has been widely used in supercapacitor because of its good catalytic properties [2]. Ruthenium oxide thin films have been prepared using various techniques, including organometallic chemical vapour deposition [3], sol gel [4], and electro deposition [5]. Here, attempts are made to deposit RuO_2 thin films using sol-gel spin coating deposition technique. The structural, morphological, vibrational properties were presented for as deposited films. The cyclic voltammetry study and chronopotentiometry was carried out with 0.1M KOH electrolyte to study the supercapacitor properties. EDAX pattern of the RuO_2 shows the RuO_2 composite film is in non-stoichiometeric form. AFM gives the roughness of the sample.

Experimental Deposition of RuO2 Thin Films:

RuO2 thin films had been synthesized by a sol-gel spin coating technique using ruthenium trichloride as a source of Ruthenium oxide. In a typical experiment, 0.01 M solution of ruthenium trichloride was prepared. To obtain homogeneous solution a magnetic stirrer was used. After aging for 24 hours a gel was formed and then deposited on steel substrate by Spin coating unit. The sample was then rotated about 3000 rpm and films were annealed at a temperature of 900°C for 3 minutes. The deposition was repeated for number of time to increase the thickness of the film.

$$RuCl_3.nH_2O + (CH_3)_2CHOH \xrightarrow[900\,^{\circ}C]{\Delta} RuO_2 + CH_3CH_2OH + HCL$$

RESULTS AND DISCUSSIONS

Structural Characterization:

The as deposited films were uniform, well adherent to the substrate and black in colour. Film crystallinity was analyzed using X-ray diffraction. The XRD patterns of RuO2 films on to the stainless steel substrate are shown in figure 1. The sharp intense peaks confirm the crystalline nature and tetragonal structure of the ruthenium oxide (JCPDS Card Number 65-2824) .These results are consistent with the results obtained by M. Khorasani-Motlagh, M. Noroozifar, M. Yousefi et.al.[6] The peaks having star mark corresponds to stainless steel. Table 1. gives the details of calculated and standard 'd' values and planes of RuO2 deposited thin films. The obtained values for the lattice parameters are a=b= 4.5200 A° and c = 3.1272A° which are in good agreement with the JCPDS data (65-2824).

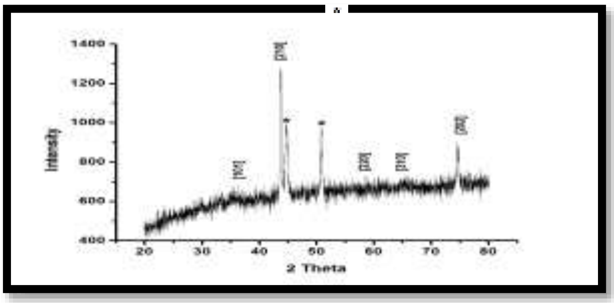

Figure 1. X-ray Diffractogram for RuO_2 thin films on stainless steel substrate

Table 1: Comparison of observed 'd' values, obtained from XRD data with the standard'd' values, from JCPDS card No-65-2824

RuO_2 (Films for this work)				RuO_2 (Card No -65-2824)		
Peak No	2Θ	d	Intensity	d	Intensity	Plane
1	36.0323	2.5035	603	2.5654	715	[101]
2	44.8134	2.02084	985	2.0214	16	[210]
3	58.2545	1.5825	731	1.5980	11796	[220]
4	65.2598	1.4285	739	1.4293	96	[310]
5	73.5845	1.2861	727	1.2827	50	[202]

Surface Morphological Analysis:

The surface morphological study of the RuO_2 thin film has been carried out from SEM image. Figure 2. shows scanning electron microscopic (SEM) photographs of ruthenium oxide thin films at different magnifications. It showed that the substrate is well covered with RuO_2 material. The SEM image shows non-uniformly distributed aggregates giving rise to a high surface roughness. The porous morphologies clearly found on these annealed RuO_2 films which is favourable for penetration of electrolyte. In the inset, one can see the particles are well connected yet provide porous structure, which is much required for supercapacitors. The rough texture represents the grain boundary surfaces. The size of grains laid in the range 170 – 182.2 nm. In electrochemical supercapacitors, an increased amount of charge can be stored on the highly extended surface area created by large number of pores within a high surface area electrode material. Nano crystalline and porous materials as electrode material exhibit good electrochemical performance because these materials possess both a high surface area and pores which are adapted to the size of ions.

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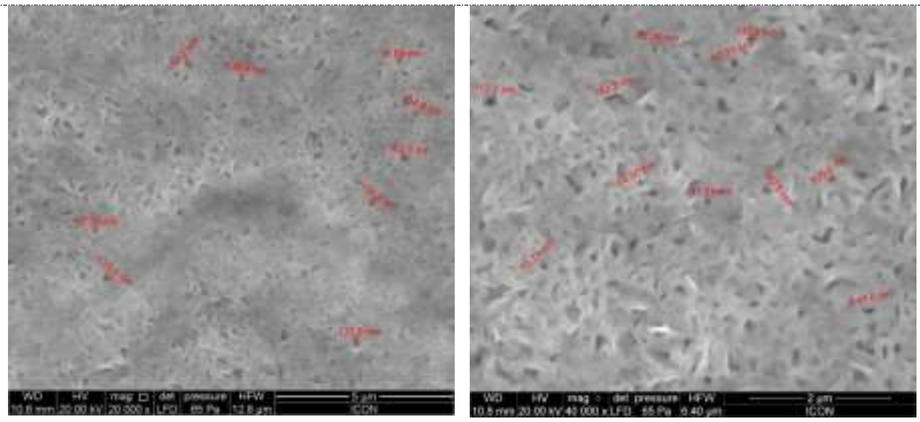

Fig.2 SEM micrographs of RuO_2 thin films at (a) (X20,000) and (b) (X40,000) magnifications

2.3 EDAX analysis of RuO_2 film:

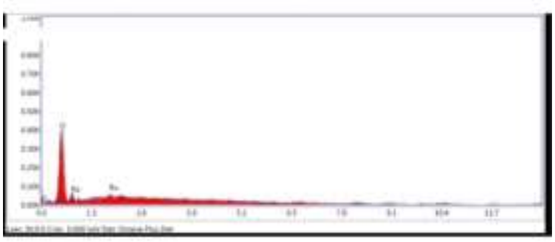

Fig. 3 The energy dispersive X-ray (EDAX) analysis of RuO_2 film

Fig. 3 shows typical EDAX pattern of the RuO_2 film The peaks for Ru and O were present in the spectrum confirming the formation of RuO_2.The observed atomic percentages for Ru and O are shown in Table 4.1, which showed the RuO_2 composite film is in non-stoichiometeric form.

Element	Atomic %
O K	51.91
Ru	0.23

Table 2: Elemental composition analyses of RuO_2 film

FTIR Spectroscopy:

The FT-IR absorption spectrum of as-deposited RuO_2 thin films in the range 4000–400 cm^{-1} is shown in fig.4. From these spectra, it can be observed apparently that strong band Ru and O around at 759.70 cm^{-1}, 896.75 cm^{-1} are associated with the characteristic vibrational mode of rutile RuO_2. This result indicated that, as deposited film contained hydroxide and other bonds, which indicates that formation of hydrous ruthenium oxide that may play important role in capacitive behavior

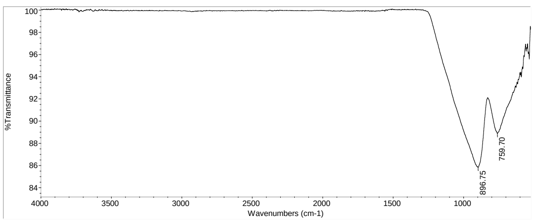

Fig.4 FT-IR spectrum of RuO_2 deposited by spin coating method

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The infrared spectrum of as deposited RuO_2 thin film depicts strong absorption bands at 759.70 cm^{-1}, 896.75 cm^{-1} indicating the stretching mode of Ru=O. The dominant band at 896.75$cm^{-1}$is associated with the vibration of Ru=O stretching [7-8] and band at 764 cm^{-1} indicates the weak O-Ru-O stretching. Bouzidi et al observed the similar results.[9]

AFM Study:

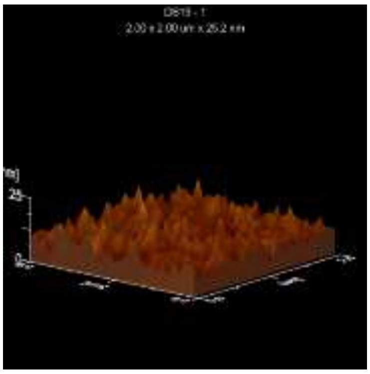

Fig.5: AFM image of sample Ru

AFM scanning of the sample pure Ru was made to study the change in the surface morphology of the films (Fig.5). For measuring the surface roughness of the films, 2×2 μm^2 surface area was utilized. The roughness of the sample is observed to be 25.2 nm.

CONCLUSIONS

In conclusion, the amorphous hydrous ruthenium oxide (RuO_2) thin films have been successfully synthesized at low temperature on stainless steel substrates using sol-gel method. Porous and spherical grained morphology was observed for sol gel deposited RuO_2 thin films.

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