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Properties of Stacked Cobalt: Ruthenium Oxide Thin Film

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Abstract: In the present work stacked Cobalt: Ruthenium oxide thin films have been prepared by sol-gel spin coat deposition technique. The suitable annealing temperature was 900^{0}C. Various thin film properties were studied by using XRD, SEM, FTIR and EDAX analysis.
The XRD showed dominant peaks of cobalt oxide and ruthenium oxide indicating crystalline nature. The SEM revealed the porous and mud-cracked morphologies with random and rough surface.
FTIR depicts strong absorption band at 876.70cm^{-1} indicating the stretching mode of Ru-O and O-Ru-O. EDAX showed presence of ruthenium, cobalt and oxygen in the sample.
Keywords: Cobalt: Ruthenium oxide, XRD, SEM, FTIR, EDAX

I. INTRODUCTION

Transition metal oxides have attracted a huge amount of attention [1]. Transition metal oxides show a broad structural variety due to their ability to form phases of varying metal to oxygen ratios reflecting multiple stable oxidation states of the metal ions [2].

Transition metal oxides are used for many electrochemical applications, such as gas sensors and chlorine or oxygen evolving electrodes [1,2].

One of the most commonly used transition metal oxide in catalytic electrode materials is ruthenium dioxide (RuO_2). Ruthenium is a relatively expensive metal and is therefore often used with a variety of other metal oxides for example Ce [3], Sn [4], Co [5-9] or Ni [10] to improve the catalytic activity, enhance the selectivity and increase the stability of the coating.

The interest for using Co_3O_4 as an electrode coating component started in the late 1970s. Agapova and Kokhanov [11] studied the preparation and activity of Co_3O_4 and found a high selectivity for chlorine evolution.

Boggio et al. [12] investigated the electrochemical surface properties of Co_3O_4 coated electrodes. Further studies were performed in the 1990s, looking at mixed oxides of Co_3O_4 and RuO_2 coated on Ni electrodes for hydrogen [13-14] and chlorine evolution [15].

B. Ch. Kim et.al have reported the role of Co in providing enhanced electronic conduction in Ru–Co mixed oxides deposited by electrochemical deposition method.

They also reported that mixed metal oxide electrode showed superior performance when compared to that of the pure RuO_2 electrode [7].

At the beginning of the 2000s more studies appeared on surface characteristics, stability and activities for oxygen and chlorine evolution of mixed coatings of RuO_2 and Co_3O_4 [6].

More recently, nanocrystalline coatings of RuO_2 and Co_3O_4 were investigated using a variety of surface characterization techniques and differential electrochemical mass spectrometry (DEMS) experiments [9,16]. Thus in the present work various properties of composite ruthenium oxide and cobalt oxide thin films have been studied.

II. EXPERIMENTAL

A. Synthesis Process of Ruthenium -Cobalt oxide Thin Film

The schematic presentation of the synthesis process and various deposition parameters of stacked Cobalt:Ruthenium oxide thin film is as shown in figure.1 and table. 1 respectively.

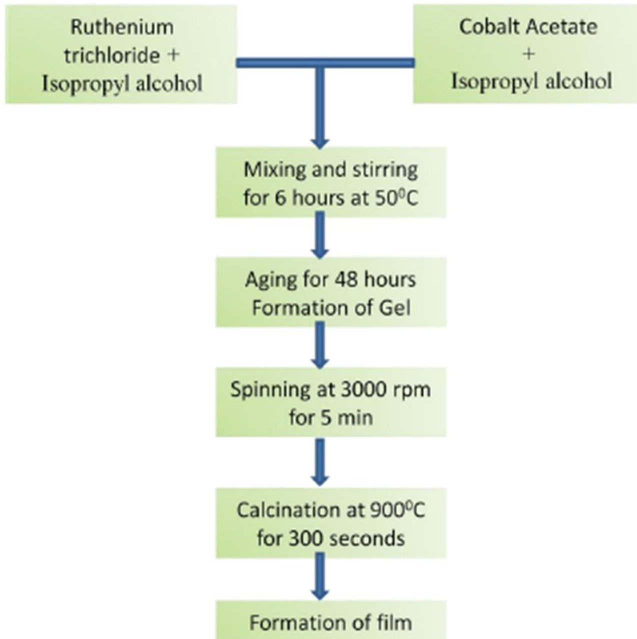

Fig. 1 Flow diagram of synthesis process

Sr. No.	Parameters	. Conditions
1	Solution	Ruthenium + Cobalt Gel
2	Substrates	Glass and stainless steel
3	Spin time	180 sec.
4	Spin speed	3000 rpm
5	Annealing temperature	900^0 C
6	Annealing time	300 sec
7	Number of coating	5

Table.1 various deposition parameters

III. RESULT AND DISCUSSION

A. Physical Studies

The as deposited films were uniform, greyish black in colour and well adherent to the substrate. The thin film electrodes having high surface area will result in low interfacial resistance, high power density and long life cycle.

B. X-Ray Diffraction Studies

The structural analysis of as deposited film was carried out using X-ray diffractometer varying diffraction angle 2θ from 10° to 80°. Figure. 2 shows the X-ray diffraction pattern (XRD) of as deposited (Co:Ru) oxide thin films. XRD pattern showed the dominating peaks at (101), (211) of ruthenium oxide. Crystalline nature and tetragonal structure of ruthenium oxide was confirmed by sharp intense peaks. Similar results were observed by W.C. Fang et.al [17] and Park et.al [18] by electrodeposition. The calculated values of the lattice parameters for tetragonal structure are ($a=b\neq c$) a =4.514 Å, b = 4.683 Å, c = 3.068 Å which are in good agreement with the JCPDS data (88-0322). In XRD pattern the peaks corresponding to cubic phase of cobalt oxide are also observed for the planes (400), (440), (444) and (620) the lattice parameter a= 8.118 Å is in good agreement with standard value compared with JCPDS 78-1969 of cobalt oxide. Table. 2 gives the details of calculated and standard "d" values and planes of RuO_2, Co_3O_4 deposited thin film.

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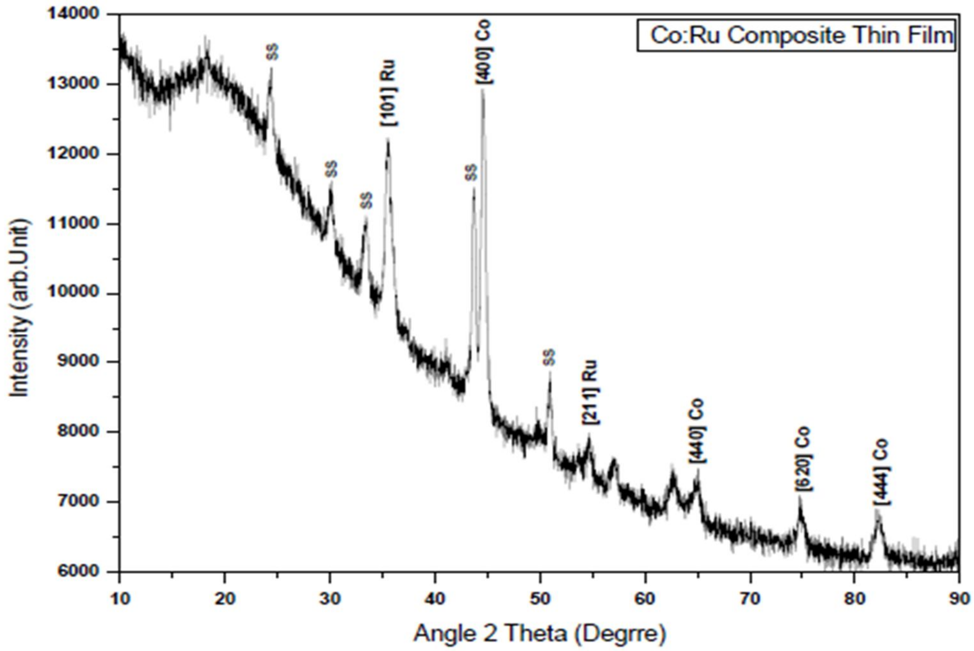

Fig. 2 XRD Pattern of stacked cobalt oxide and ruthenium oxidethin film

Peaks	Standard 'd'	Observed 'd'	Plane	Intensity
Cobalt	2.033	2.021	400	12935
JCPDS 78-1969	1.435	1.429	440	7380
(Cubic)	1.269	1.278	620	7105
	1.170	1.167	444	6833
Ruthenium	2.527	2.555	101	12233
JCPDS 88-0322	1.676	1.687	211	7993
Tetragonal				

Table. 2 Standard and observed‘d’ values of stacked cobalt oxide and ruthenium oxide thin film

C. Surface Morphological Studies

The surface morphological study of the stacked Co_3O_4:RuO_2 thin film has been carried out from SEM image. Figure. 3 shows scanning electron microscopic (SEM) photographs of stacked oxide thin films at different magnifications. It showed that the substrate is well covered with Co_3O_4:RuO_2 material.

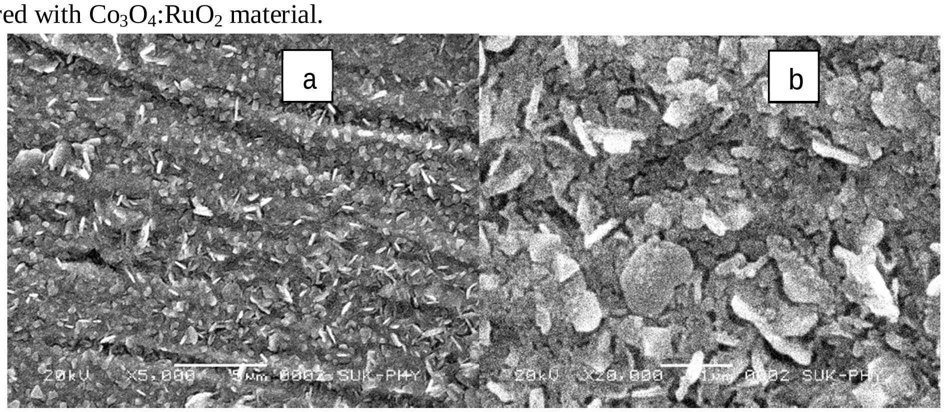

Fig. 3 SEM images of stacked Cobalt:Ruthenium oxide thin film at different magnifications

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The SEM images shows non-uniformly distributed aggregates giving rise to a high surface roughness. The porous and "mud-cracked" morphologies (fig. 3. a) clearly found on these films which is favourable for penetration of electrolyte. The large cracks are attributed to the presence of inner stress in forming a crystalline RuO_2 film during the high temperature preparation step. Similarly the cubical grains corresponding to cobalt oxides (fig. 3. b) are also observed, we 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 pores lay in the range 40-50 nm.

D. Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy (FT-IR) gives the information of the functional groups of compounds. Generally, metal oxides give absorption bands below 1000 cm^{-1}, due to interatomic vibrations. Cobalt: Ruthenium oxide thin films were studied under IR spectroscopy. For the examination of absorbed molecules on a solid surface, FTIR is a well-known technique. IR spectroscopy was used to obtain additional information on the phases as well as structure transformations of RuO_2 phases. Figure.4 describes the dependence of optical spectra in the range 500 to 4000 cm^{-1} for Cobalt: Ruthenium oxide stacked thin film. The infrared spectrum depicts strong absorption band at 876.70cm^{-1} indicating the stretching mode of Ru-O and O-Ru-O.

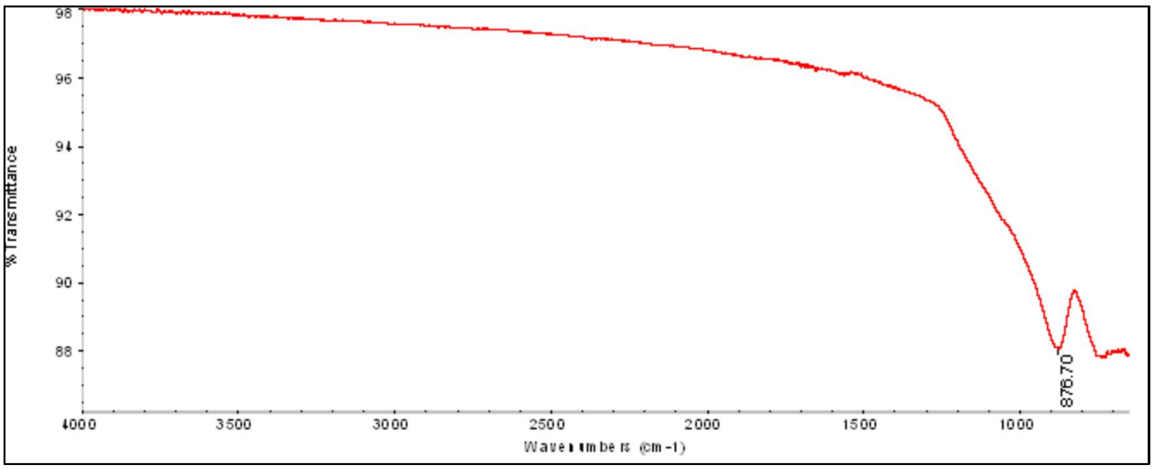

Fig. 4 FTIR Spectrum of stacked Cobalt:Ruthenium Oxide thin film

E. EDAX Analysis

The EDAX spectroscopy was used to know the percentage of the element present in the sample. EDAX analysis is carried out using Quanta 200 ESEM instrument. The EDAX spectrum of stacked Co:Ru binary thin film is shown in figure. 5. As shown in figure, ruthenium cobalt and oxygen elements existed in the sample

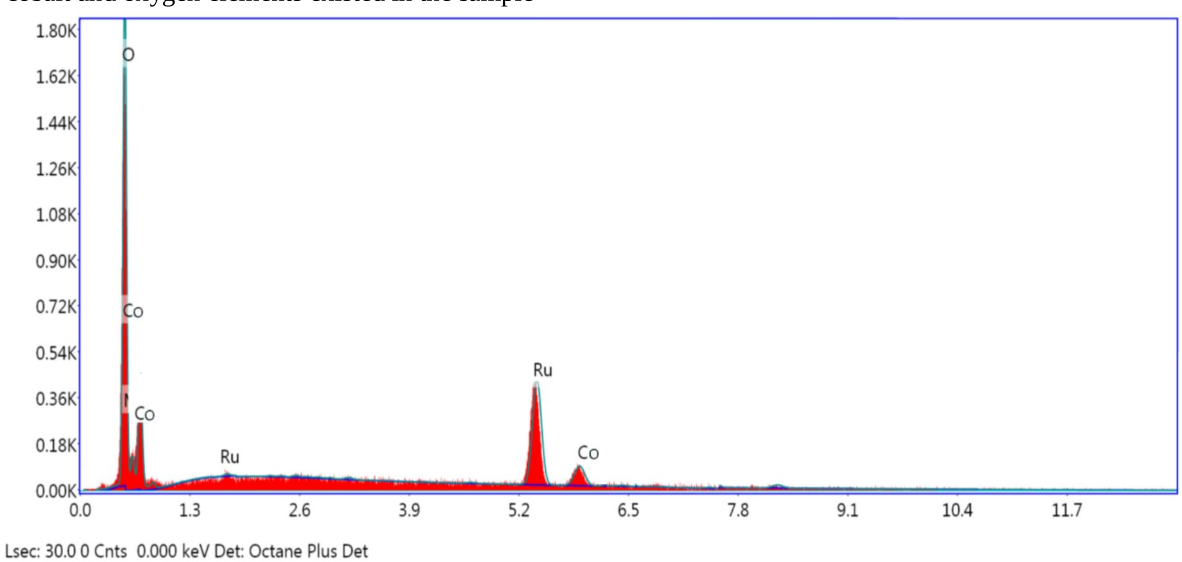

Fig. 5 EDAX Spectrum of stacked Cobalt:Ruthenium Oxide thin film

IV. CONCLUSION

The thin film of stacked Cobalt oxide: Ruthenium oxide deposited on steel substrate by sol-gel spin coating technique at annealing temperature 900^0 C. In conclusion, the as deposited films were uniform and well adherent to the substrate. The XRD showed dominant peaks of cobalt oxide cubic structure and ruthenium oxide with tetragonal structure. The SEM images showed the porous and mud- cracked morphologies with random and rough surfaces with cubical grains. EDAX showed presence of ruthenium, cobalt and oxygen in the sample.

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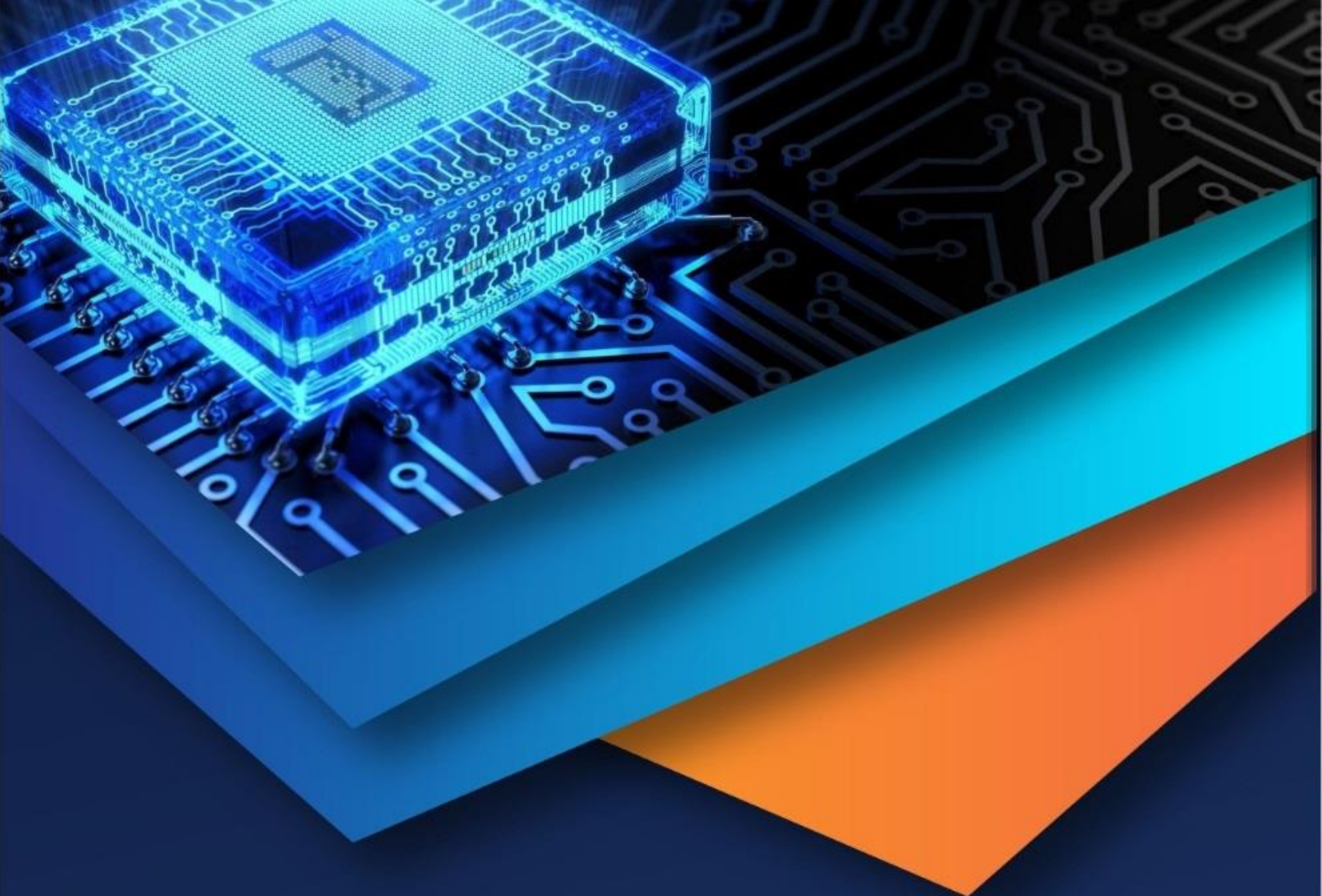

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