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Annealing induced enhancement in characteristics of screen-printed cobalt oxide (Co_3O_4) thick films

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Abstract
Co_3O_4 thick films are deposited on glass substrate by screen printing technique. All characterization was carried out for unannealed, annealed at 250℃-400℃ . The XRD analysis indicates prepared films are polycrystalline nature with cubic structure having preferential orientation through (311) plane. Crystallite size is found to be 18.516nm. The lattice parameter found to be 8.036-8.138 A^0. approaches to standard value. SEM analysis films show agglomeration of nanoparticles, occurrence of spherical-shaped grain aggregations. Spherical grain size increases 47.66 to 77.33nm with annealing temperature. The link between structural and morphological properties are noted. The EDAX analyses indicates that all compositions have desired stoichiometric ratios. Besides electrical measurements, film D.C. resistance, resistivity was measured that assured material has semiconducting nature. Calculated Specific surface area, TCR, activation energy decreases with increases in annealing temperature. It was shown that structural, morphological, and electrical properties of Co_3O_4 films were improved by increasing annealing temperature.

Keywords: *Co_3O_4 Thick Films, XRD, SEM-EDAX, Resistivity, TCR, Activation energy.*

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I. Introduction

Recently material science has become more promising field in the catalysis and sensors technology and having diverse applications in different areas such as smart windows, negative electrodes in Li- ion batteries and mirrors with variable reflectance. As properties (such as structural, magnetic, optical, catalytic, and electronic) of nanomaterials depend strongly on their size, structure, and shape so research is presently being carried to enhance these properties and to add to novel functionalities to the metal oxides.

Mentioned material (Co_3O_4) is an electrochromic material and would be classified into two categories: cathodic (i.e., coloured in the reduced state) like molybdenum oxide, tungsten oxide etc, and anodic coloured materials (i.e., coloured in the oxidized state) like, ruthenium oxide, cobalt oxide, nickel oxide, etc. [1] However, Cobalt oxide films is a promising anodically colouring electrochromic material. After oxidation it becomes grey in colour, which is opposite to the reduced tungsten oxide.

Among transition metal oxides, spinel type tricobalt tetra oxide is versatile metal oxides because of its two characteristics namely variable valance state and existence of oxygen vacancy defects. It exists in different forms such as CoO_2, Co_2O_3, CoO (OH), Co_3O_4 and CoO. Of these, Co_3O_4 acquires the normal spinel structure magnetic Co^{2+} ($3d^7$) cations are placed in tetrahedral sites and nonmagnetic Co^{3+} ($3d^6$) cations have occupied octahedral ones. In bulk crystal structure, Co_3O_4 exhibits antiferromagnetic property while in nanostructured it shows weak ferromagnetism with an energy bandgap of 1.4 – 1.8 eV [2].

Owing to their enhanced and exclusive properties such as increased surface to volume ratio, good chemical stability and high specific surface area, semiconductor nanoparticles are used in field of gas sensors for detection purpose[3].

In most of the research work the Co_3O_4 has been selected for investigation, because of its chemical stability, desired electrochemical property, and high annealing temperature. Among different techniques, screen printing technique has many advantages such as low cost, easy to handle, convenient for large area deposition, uniform film deposition and less deposition time. This technique has been employed by many researchers to fabricate the films of tin oxide, cerium oxide, zinc oxide and indium oxide. [4-8]. After preparation, films are ready for characterization by X-ray diffraction, Scanning electron microscope, energy dispersive x-ray analysis (EDAX), electrical conductivity measurement. Efforts have been taken by researchers during synthesis of Co_3O_4 nanostructures to prepare different morphological structures like hollow spheres, nano porous, nanospheres, nanotubes cubic single crystals, nano particles, nano rods, nano plates, nano wires, and nano cubes structures [9] To improve electrical conductivity, transmission stability and practical feasibility different processes like doping, annealing, and aging are employed [10].

Screen-printing technology allows to control the dimensional factor, the thickness of the film elements and so it is used for mass production of thermal modules [11]. For structural characterization, XRD technique, Surface morphology scanning electron microscopy (SEM) instrument while the stoichiometric characteristic was proved from energy dispersive X-ray (EDAX) analysis [12].

In the report preparation of screen printed Co_3O_4 thick film is described and effect of annealing temperature on structural, morphological, and electrical characterization has been investigated and elaborated by a simplified screen-printing technique.

II. Materials method and measurements

The commercially available AR grade with 99.99% purity cobalt oxide nano powder was used for preparation of thick films and acetone, ethyl cellulose, B.C.A., etc are required. To verify structural properties and purity of samples, the X-ray diffraction (XRD) technique is used. The X-ray diffraction patterns of all the prepared samples are recorded for analysis purpose. They were plotted using Bruker D8 advance diffractometer, Germany with CuKα (λ=1.5404 A.U.) radiations operated at 40 KV and 40 mA in the scanning range of (2θ) between 20° and 80°. To observe chemical compositions and surface morphology, scanning electron microscope, SEM-JEOL JSM-6360A with OXFORD EDAX attachment is used. For electrical characterization simple half bridge method was used.

2.1. Preparation of thick films of cobalt oxide nanoparticles

The cobalt oxide based thick film sensor was constructed by standard screen-printing technique. The powder nanoparticle of Co_3O_4 converted into paste form was used to prepare thick films by maintaining the inorganic to organic binder ratio at 70:30. The inorganic part consists of AR grade nanoparticles of Co_3O_4**.** The organic binder contains of 8% ethyl cellulose (EC)which is in a powder form and 92% butyl carbitol acetate (BCA) present in a liquid form. All these stoichiometric amounts of Co_3O_4 and binders then mixed into mortar and pestle and crushed continuously for nearly 40 minutes. BCA was added to crushed powder of cobalt oxide (functional material) and binders drop by drop to get correct viscosity for screen printing. Prepared gel like paste is coated over glass substrate of dimensions 1.5 X 2 cm then films were air dried for 30 minutes followed by IR drying for 30 minutes. Finally, the prepared binary oxide Co_3O_4 film sensor was kept in muffle furnace for calcinations process at various temperatures 250 ^{0}C, 300 ^{0}C 350 ^{0}C ,400 ^{0}C nearly 2 hours. On the next day the thick films of used oxide were removed from muffle furnace and utilize for further study. Annealed films were black in colour and found to be uniform, pinholes free and strongly adherent to the glass substrates. Such a annealed films are now ready for characterization.

2.2. Measurement of the film thickness: The thickness of the prepared films calculated by the using gravimetric (weight-difference) method assuming the samples were approximately uniform as compared to bulk form of cobalt oxide. Thickness measurement equation is given by $t = \Delta w / A * \rho$ (1) where, Δw = actual weight of the prepared film, ρ = density of Co_3O_4 = 6.11 gm/cm^3
A= area of the film (length*breadth)
After calculations thickness observed in terms of μm. Here it varies from 33 to 38 μm.

2.3. Characterisation of screen printed Co_3O_4 thick film sensor

The physical, chemical properties and performances of cobalt oxide NPs intensively depends on size, shape geometry, morphology etc**.** To characterise the prepared samples mainly X-ray diffraction technique (structural analysis), scanning electron microscopy with EDAX (morphological and elemental analysis) and half bridge method (electrical analysis) were used. Using characterisation techniques, the success of the synthesised nanoparticles was confirmed.

III. Results and discussion

3.1. Structural characterisation: XRD analysis: To determine the quality of crystal structure and analyse phases of transition metal oxides nanoparticles and composite nanoparticles XRD technique is used[13]. For recognition of the crystalline phase, JCPDS (Joint Committee on Powder Diffraction Standards) data and using Debye-Scherrer equation the crystallite size was calculated.

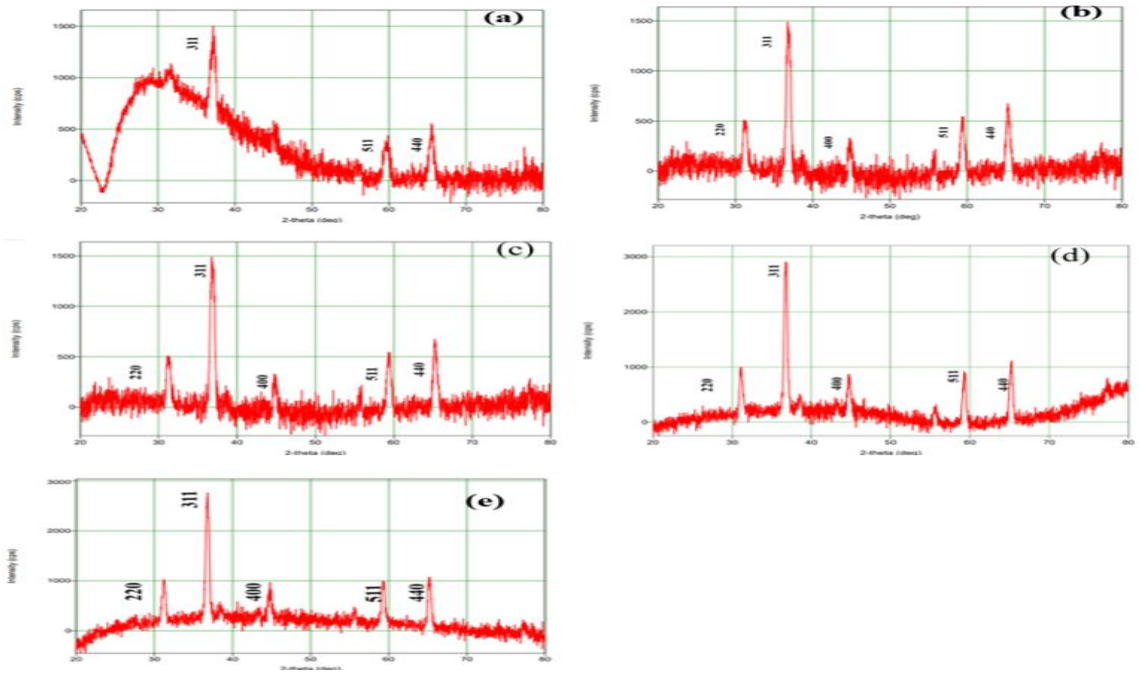

FIG. 1. XRD patterns of Co_3O_4 thick films (a) as deposited (unannealed), annealed at (b) 250°C, (c) 300°C, (d)350°C, (e)400°C respectively.

FIG.1(a-e) shows the XRD patterns of Co_3O_4 thick films as deposited at room temperature (unannealed) and annealed at different temperatures namely 250 ,300, 350 400℃. It indicates the purity of the product .From XRD study, pattern was exploited in the 2θ range of 20– 80°and revealed the formation of Co_3O_4 with cubic crystal structure .All main diffraction peaks are located at 2θ of 31.073^0 ,36.749^0 , 38.47^0 , 43.13^0 ,44.71^0 , 55.52^0 ,59.25^0 , 65.16^0 and 77.94^0 indicating their polycrystalline nature corresponding to the (220), (311), (222), (400), (422), (511),(440) and (622)crystal planes, respectively and demonstrated the formation of Co_3O_4 with cubic crystal structure [JCPDS card no. 42- 1467]. The peak intensity is strong, indicating high crystalline structure of the products [14] The average crystallite size of the Co_3O_4 thick films is estimated from the X-ray diffraction patterns using the Debye Scherrer formula,

$$D = \frac{0.9\lambda}{\beta cos\theta} \qquad (2)$$

where 'D' is crystal size, 'β' is Full Width at Half Maximum of XRD peak
'λ' is wavelength =1.54 A.U. of X-ray used and 'θ' is Bragg's diffraction angle.
The crystallite size of Co_3O_4 calculated from the strongest peak, locating at (311) plane at $2\theta \approx 37°$, are estimated 18.516 nm. The average crystallite size for pure cobalt oxide was found to be 18.2033nm [15,16] and its distribution range is 18.52 to 36.38nm depending on annealing temperature.
The obtained value of d_{hkl} (interplanar distances) values and the standard values confirms that the deposited and annealed films are nano crystallized in the spinel type cubic structure of Co_3O_4. The lattice constant 'a' of the unit cell is determined from the peak positions using the formula of cubic system. This value is found to be in the range 8.036-8.138 Å, which is close to standard value given by JCPDS 42-1467 (a = 8.084 Å). When the annealing temperature increases, the intensity of the peak corresponding to the (311) plane is found to decrease due to the thermal strain. Sharp diffraction peaks are observed at 350 ◦C annealed temperature [17]**.**
From XRD data, the crystalline parameters like lattice constant (a), crystallite size (D), dislocation density (δ), and micro strain (ε) were determined. The dislocation density is the length of dislocation lines per unit volume of the crystal. Greater D and smaller δ values mean better crystallization of the films. The dislocation density and lattice strain were calculated from the diffraction pattern to obtain more information about structural properties. The observed changes of strain are due to point defects and crystallite size. The lattice strain is calculated using Stokes-Wilson equation and to know more on the number of defects in the film, the dislocation density, δ is calculated by using relation mentioned in TABLE 1. The induced strain causes reduction in crystallite size and increase in peak broadening. The average micro strain screen printed Co_3O_4 thin film sensor is 0.27014 %. Micro strain decreases from 0.00611 to 0.0009 due to good crystallinity.

TABLE 1: Shows Dislocation density and Micro Strain for different annealing temperatures.

Annealing temperature (°C)	Dislocation density $\delta = \frac{1}{D^2}$ (lines/m^2) 10^{15}	Micro Strain $\varepsilon = \frac{\beta cos\theta}{4}$, 10^{-3}
Unannealed	13.18	6.11
250	0.75	0.952
300	5.10	2.475
350	2.92	1.870
400	3.695	2.1

The XRD spectrum is in full agreement with the standard spectrum of cubic crystalline Co_3O_4 (JCPDS card no: 42–1467). The diffraction peaks revealed to the cubic nature of nanocrystalline Co_3O_4.

3.2. Morphological characterization

SEM with EDS analysis (surface morphology)

The Scanning electron microscopy (SEM-JEOL JSM 6360 Model) images of prepared nanomaterial Co_3O_4 thick film are displayed in FIG.2. These 2D images with high magnification show surface texture, color and porosity Co_3O_4 nanoparticles and indicate the formation of particles with different shapes and sizes. This porosity may be due to the evaporation of organic binder during the growing process and the annealing treatment [17]. The instrumental parameters, accelerating voltage, spot size, and magnification and working distances are shown on SEM image. The surface characteristics of the prepared films investigated by scanning electron microscopy shows heterogeneous, porous nanoparticles with varying dimensions. Some particles are appeared in spherical shape. The observed particles are highly agglomerated, and they are essentially cluster of nanoparticles. The surface morphology of screen printed Co_3O_4 thick films shows small sphere-shaped particles are distributed some of them having heterogeneous surface, microspores and mesopores as seen from its surface micrographs.[10] It's greyish black in color, various sized nanoparticles imaged can be seen from SEM images as shown.

Thick films deposited of Co_3O_4 on glass substrate annealed at unannealed, 250 ^{0}C, 300 ^{0}C, 350 ^{0}C, 400 ^{0}C, surface morphology was determined with high resolution. It showed polycrystalline nature with number of voids (pores)distributed on the surface of film. The particle size was obtained as the length of the segment joining the two ends of the particle and passing through its mass centre and the average particle size is increases from 73 nm to 145nm with increase in annealing temperatures. It is maximum when film is unannealed. The average particle size is 116.702 nm. Spherical grain size increases with respect to annealing temperature and its change from 47.66 to 77.33nm and its maximum when thick film is unannealed. Surface area decreases from 20.60 to 12.698 m^2/gm with increase in annealing temperature and is a minimum for unannealed film as shown in table 2. Surfaces of Co_3O_4 thick films are cubical, spherical grains, so such a film can adsorb more atmospheric oxygen due to more exposed surface area of the film. Highly porous film shows high sensitivity.

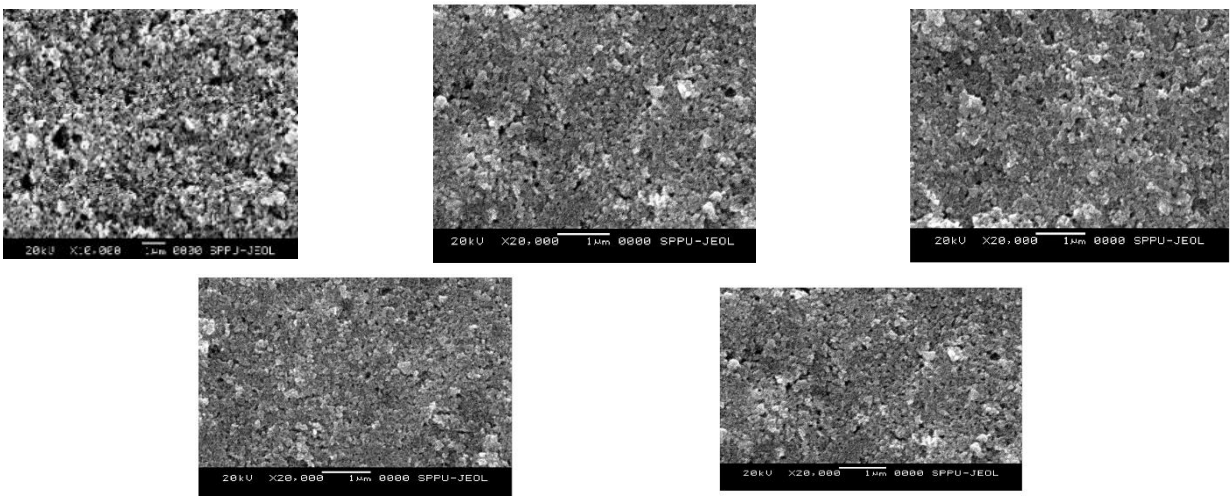

FIG. 2. SEM micrographs as deposited (unannealed), annealed at 250 ^{0}C, 300 ^{0}C, 350 ^{0}C, 400 ^{0}C Co_3O_4 thick films respectively.

TABLE 2: Mean particle size, grain diameter and Surface area at various annealing temperatures.

Annealing temperature (° C)	Mean Particle size (nm)	Spherical grain diameter 'd ' (nm)	Specific surface area $S_w = \frac{6}{\rho d}$ (m^2/gm)
Unannealed	127.2	83.5	11.76
250	73.11	47.66	20.60
300	108.2	66.66	14.73
350	130	73.66	13.331
400	145	77.33	12.698

EDS Analysis: The elemental quantitative composition of grown Co_3O_4 nanoparticles was determined using the spectra obtained by energy dispersive analysis of X-rays (EDAX). The weight percentage and atomic percentage of cobalt oxide nanoparticles are shown in TABLE 3. This tells proof that that synthesized nanoparticles are non-stoichiometric. In Co_3O_4 this nonstoichiometric is supplemented by change in colour with the stoichiometrically correct cobalt oxide being grey and non-stoichiometric being black.

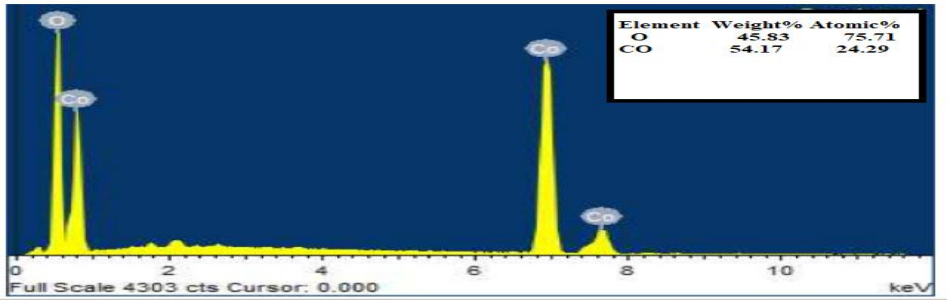

FIG. 3: EDAX results of Co_3O_4 nanoparticle

TABLE 3: Atomic % elemental composition of Pure Co_3O_4 thick films as measured by EDS

Sample Annealed Temperature	Elements				Co/O Ratio
	Co		O		
	At. wt.%	*At.Mass %*	*At.wt.%*	*At.Mass %*	
Unannealed	53.90	24.09	46.10	75.91	1.17
250^0 C	52.21	22.88	47.79	77.12	1.09
300^0 C	53.76	23.99	46.24	76.01	1.16
350^0 C	54.43	24.24	45.57	75.71	1.19
400^0 C	54.17	24.29	45.83	75.51	1.18

EDAX results of Co_3O_4 nanoparticles As annealing temperature increases weight percentage of cobalt is increases but mass percentage of oxygen decreases due to release of excess oxygen. decreases and it also indicates the oxygen adequate nature of the films. [18].

TABLE 3 shows quantitative elemental analysis of Cobalt oxide thick films. In figure, the major peaks of Co and O elements are observed. The observed compositional data from the EDX analysis responds positively with theoretically calculated values, representing a good elemental compositional homogeneity across the cobalt oxide nanoparticles. The EDX spectrum shows sharp peaks between 6.6 and 7.8 KeV for Co and between 0.4 and 0.6 KeV for O corresponding to crystalline Co_3O_4 NPs. The EDAX results obtained for spinel type cobalt oxide thick are in good agreement with reported output. Due to high electron affinity of surface molecules, oxygen element gets attached to the molecules on the surface of the prepared thick films.

Crystallinity: It describes degree of structural order in a solid. Crystallinity index is denoted by I_{cry} can be calculated through comparison of crystallite size obtained from XRD and that obtained by SEM.

$$I_{cry.} = \frac{D_p}{D} \tag{3}$$

Where, D_p =116.702 nm $D = 18.2033$ nm particle size from SEM and XRD respectively.

When I_{cry} is close to 1, it is assumed that the crystallites represent monocrystalline units while larger value of I_{cry} *means* the particles are of polycrystalline type. From XRD and SEM, $I_{cry} = 6.4110$ for cobalt oxide thick film, it's much greater than 1 implies that the prepared thick films show polycrystalline type. Result shows particle has polycrystalline nature and good crystallinity.

3.3. Electrical characterization The cobalt oxide film sample D.C. resistance was measured by using half bridge method as a function of temperature in home built static gas measurement system. This system consists of glass chamber and heater of nichrome wire having Resistance-120ohm at room temperature. The heater was used to

change the film sample temperature from room temperature 30^{0}C to 350^{0}C. In half bridge method the value of R_{ref} =10 MΩ and 30 V_{DC} power supply were used.

3.3.1. Sample resistance The resistance of the thick film samples was determined using following equation

$$R_{sample} = R_{ref}\{\frac{V_{in}}{V_{out}} - 1\} \quad (4)$$

Readings of temperature verses output voltage were taken by changing temperature 10° C. The D.C. resistance of the films was measured by half bridge method in air atmosphere at 30℃-350 ℃ .The resistance of cobalt oxide thick films found to be decreases with increase in temperature. This gives the confirmation of semiconducting behaviour of Co_3O_4 material by obeying $R = R_0 e^{-\Delta E/kT}$ in the 30° C- 350° C temperature range. FIG.4 shows the change in resistance of pure Co_3O_4 thick films with respect to change in temperature(k).

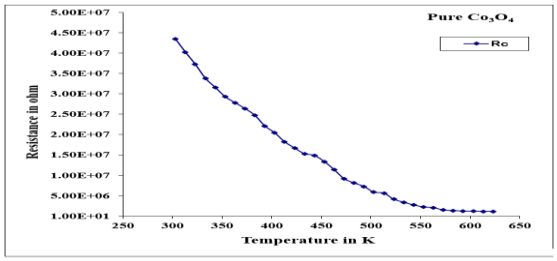

FIG .4a. Plot of resistance against temperature for Co_3O_4 thick films

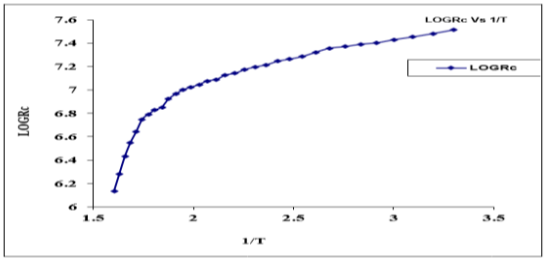

FIG .4b. Plot of logR against inverse temperature for Co_3O_4 thick films

3.3.2. Resistivity of Co_3O_4 thick films: The resistivity of prepared thick films was calculated as function of temperature within temperature range. From nature of graph it is observed the resistance decreases steadily to lowest saturation level with respect to increase in temperature. The resistivity of thick films at constant temperature is calculated using equation

$$\rho = \frac{Rbt}{l} \quad (5)$$

Where, ρ = Resistivity of the film, R = resistance at room temperature,
b = breadth of film=1.25cm, t = thickness of the film, l = length of the film=2.5cm.
To calculate resistivity of unannealed thick film, R = 4.791x10^7Ω ,t= 38μm were taken. Resistivity is 910.38 Ωm and is maximum and corresponding conductivity 1.098x10^{-3} $\Omega^{-1}m^{-1}$ and is a minimum. The resistivity of pure Co_3O_4 thick films decreases with increase in annealing temperature. It represents the development of a semiconducting nature in the film. The conductivity is inverse of resistivity and is computed from the relation σ = 1/ρ, where σ is the conductivity. The electrical resistivity and conductivity values are found out at various annealing temperatures and are mentioned in table. From electrical properties we observed the electrical conductivity of pure Co_3O_4 thick films increases with increase in annealing temperature, films deposited onto nonconductive substrate (250-400°C) as mentioned in TABLE 4. Actually, annealing treatment provides sufficient thermal energy to crystal which facilitates the electron delocalization through the removal of a large number of defects. [17].

3.3. 3. Temperature Coefficient of Resistance (TCR) of Co_3O_4 thick film

The temperature coefficient of resistance (TCR) of pure Co_3O_4thick films is calculated by using equation (6). Temperature coefficient of resistance is found negative to pure Co_3O_4 thick film samples. The negative sign indicates the semiconductor behaviour of the prepared pure Co_3O_4 thick films. Low resistivity of cobalt oxide thick film samples corresponds to a high TCR value or vice versa. TCR of pure Co_3O_4 unannealed thick film is found -0.011523/k. The observed value of TCR decreases as annealing temperature increased.To obtain the value of T.C.R. using graph following equation is used

$$\text{TCR } (\alpha) = \frac{1}{R_0}\frac{\Delta R}{\Delta T} = \frac{\text{Slope}}{R_0} \text{ ,per degree kelvin.} \quad (6)$$

3.3.4. Activation energy of Co_3O_4 thick films The activation energies at the low temperatures and high temperatures of prepared spinel type Co_3O_4 thick films film were calculated using Arrhenius plot. FIG 4b. shows graph of log R in ohm versus reciprocal of temperature (1/T) in Kelvin for Co_3O_4 thick films. The Arrhenius plot has two distinct regions of temperature namely low and high temperature region. The annealing temperature of films is an important quantity to decide the transition temperature from low region to high region. The observed values of activation energy for Co_3O_4 unannealed thick film found1.88735 x10^{-4} eV and 5.6158 x10^{-4} eV at low and at high temperature respectively. The energy of activation within the low temperature region is low as

compared to energy within the high temperature region because material passes from one conduction mechanism to a different. This region are regions of temperature conduction, during this region energy of activation decreases because a tiny low thermal energy quite sufficient for the activation of charge carriers to require part within the conduction process. Hence increase in conductivity within the lower temperature region may be attributed to the increase of charge mobility. It shows more thermal durability with increase in annealing temperature.

TABLE 4: Electrical parameters of Co_3O_4 thick film sensor annealed at different temperatures.

Annealing temperature °C	Resistivity Ωm	Electrical Conductivity$10^{-3}\Omega^{-1}m^{-1}$	T.C.R. per kelvin	Activation energy x10^{-4} eV	
				At low temp.	At high temp.
250	782.57	1.2778	-0.003575	0.777366	2.7044
300	634.42	1.5763	-0.004409	0.3546	6.4341
350	570.74	1.7521	-0.00476	0.31887	9.5916
400	365.55	2.7356	-0.005936	0.29339	12.440

Using the known Arrhenius relation, the activation energies are calculated as

$$R = R_0 e^{-\Delta E/k_B T}, \quad (7)$$

where, R_0 = pre-exponential factor, ΔE = activation energy,

k_B = Boltzmann constant and T = absolute temperature.

The activation energy ΔE for Co_3O_4 films are calculated from the slope of the plot the Log R vs (1/T) and is given by $\Delta E = 2.303x$ K_B x slope of the graph. At low and high temperature its values in electron volt are mentioned in table. [18].

It shows that the electrical behaviour of the NPs was studied by measuring the dc conductivity as a function of temperature T ,this results reveals that the materials are characterized by semiconductor behaviour i.e. by increasing the temperature, more and more charge carriers can overcome the energy barrier and participate in the electrical conduction is assigned to elevated temperatures provide enough energy for charge carriers to hop among sites or even from defects involving singly charged oxygen ions[19]. These oxygen defects would add additional charge carriers to increase conductivity.

IV. Conclusion

Properties of Cubic structured Co_3O_4 thick films of different thickness deposited on glass substrate by screen-printing technique was investigated for different annealing temperature. The films deposited at room temperature (unannealed) and unannealed in the temperature range 250^0C to 400^0C. Polycrystalline nature of crystal oriented along the (311) plane was confirmed using XRD analysis and an increase in crystallinity as well as morphological properties was observed with increase in thickness of Co_3O_4 films. Also, an increase in grain size and a simultaneous decrease in surface area of grain was observed as annealing temperature increases from 250^0C to 400^0C. Co_3O_4 thick films are highly porous and sensitive in nature. EDAX pattern reveals their purity and spinel type cobalt oxide structure and several other structural properties namely the dislocation density, lattice strain etc. But the presence of point defects and change in crystal size causes change in properties. The higher peak intensities are due to better crystallinity. From electrical characterization, resistivity decreases from 910.576 Ωm to 365.5466 Ωm with increase in annealing temperature it means cobalt oxide thick films shows semiconductor behavior. The value of electrical conductivity and activation energy at high temperature increases $1.098x10^{-3}\Omega^{-1}m^{-1} to\ 2.7356x10^{-3}\Omega^{-1}m^{-1}$ and $5.6158\ x10^{-4}$ eV to $12.44x10^{-4}$ eV as annealing temperature increased respectively. The electrical characterisation confirms semiconductive nature of crystal and shows that conductivity and activation energy increase with increase in annealing temperature. From these results, we conclude that the annealing temperature strongly affects the structural, morphological, and electrical properties of cobalt oxide thick films. However further investigation and optimization still need to be done for doped cobalt oxide thick films.

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