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Chemical Synthesis of Cobalt Oxide and Study of Structural Properties

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Abstract: ***The Cobalt Oxide (Co_3O_4) thin film was deposited directly on a stainless-steel substrate by simple hydrothermal method. Optimized thin films were further characterized for Phase, morphologies, elemental analysis, quantitative compositional information and functional groups were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), The prepared Cobalt Oxide samples were studied for super capacitor application by using cyclic voltametric technique.***

Keywords: thin film, Supercapacitor

1.Introduction

Supercapacitors are new emerging in the field of energy storage devices. It bridges the gap between battery and conventional capacitor[1]. Supercapacitors have higher energy density than capacitors and have higher power density than batteries. Transition metal oxide have been most attracted research area. Now a days researcher is fabricating the electrode with composite material[2]

Supercapacitors with carbon material show excellent cyclic stability and have high specific capacitance. Metal oxide suffers from low value of specific energy density and limited cell voltage. These problems are overcome by making a composite working electrode[3]. Various transition metal oxide such as MnO_2[4], NiO_2[5], Co_3O_4[6], RuO_2[7], Fe_3O_4[8], Bi_2O_3[9], $NiFe_2O_4$[10] have been studied and reported as electrode material in supercapacitor applications. The cobalt hydroxide-based materials are widely used as a precursor for the synthesis of various mixed oxide catalysts and absorbents, activity of which is highly depend on structure, morphology, composition and treatment method of the hydroxide precursor[11]. The metal oxide $Co_3O_4$are most prominent electrode materials for supercapacitor owing to their attractive properties like high conductivity and good cyclic stability. Generally, literature study revealed that the cobalt oxide thin films have been deposited using chemical bath deposition[12], successive ionic layer adsorption and reaction (SILAR) [13], spray pyrolysis[14], chemical vapour deposition[15], Spin coating[16], and pulsed laser deposition methods[17]. Among these synthetic methods the electrodeposition technique has one advantage over the others; it is easy, economical, low temperature process, and the growth rate of the film can be controlled by depositing film in potentiostat, galvanostatic, modes. On the other hand, electrode composed of nano particles are more difficult to fabricate by traditional slurry coating methods, because nanoparticles have poor dispersibility in slurry. Thus, it is more advantageous to have an electrode of nano sized cobalt oxide fabricated by electrodeposition directly on to the copper substrate at room temperature without any template or catalyst.

In this work, Cobalt oxide thin films with nanostructured morphology were synthesized by simple hydrothermal method. The Structural and Morphological characteristics of these films were examined.

2.Experimental Section

2.1 Materials preparation

The cobalt oxide (Co_3O_4) films were prepared on a copper substrate through electrodeposition in an electrolyte containing 0.1M cobalt chloride (CoCl2) and it was complexed with 30% aqueous ammonia by adjusting the pH around 12. The resultant solution was then stirred for another 2 h to get complete homogeneity and transferred to 100 mL of Teflon lined stainless steel autoclave, which was kept in an oven at 70^oC for 18 hours. The well-polished and cleaned stainless steel substrate was placed in the autoclave bottle. After the reaction time the autoclave was then allowed to cool to room temperature and the thin film was formed on the substrate and annealed at 400^oC. The prepared thin film was further characterized by various characterising techniques.

3.Material characterizations

3.1 X-ray diffraction

The structural properties of the films were carried out using X-ray diffraction analysis comprising copper (Cu Ka target wavelength = 1.54060 Å) target at a diffraction angle 2θ from 20º to 80º. The surface morphology of the film was studied by scanning electron microscopy (SEM).

Figure 1 shows the XRD spectrum of the electrodeposited cobalt oxide thin film on copper substrate. The high intensity peaks at 2θ=44.14°, 51.30° and 72.91° were due to the (400), (331) and (620) planes of Co_3O_4 respectively (JCPDS card number 80- 1545). The peaks are very sharp which indicate good crystallinity of material. The crystallite size is obtained as follows,

$$\beta_T = \frac{k\lambda}{D\cos\theta} + 4\epsilon\tan\theta$$

where, λ is wavelength of Cu ($k_\alpha = 0.154060\, nm$) radiation, ϵ is strain, shape factor $k = 0.9$ and θ is angle of diffraction. Crystallite size (D). From the XRD data of Co_3O_4 the average crystallite size is calculate as $\mathbf{51.56\, nm}$, the strain $\boldsymbol{\epsilon = 0.00224}$ and dislocation density $\boldsymbol{\delta = 1.976869 \times 10^{-3} (nm)^{-2}}$.

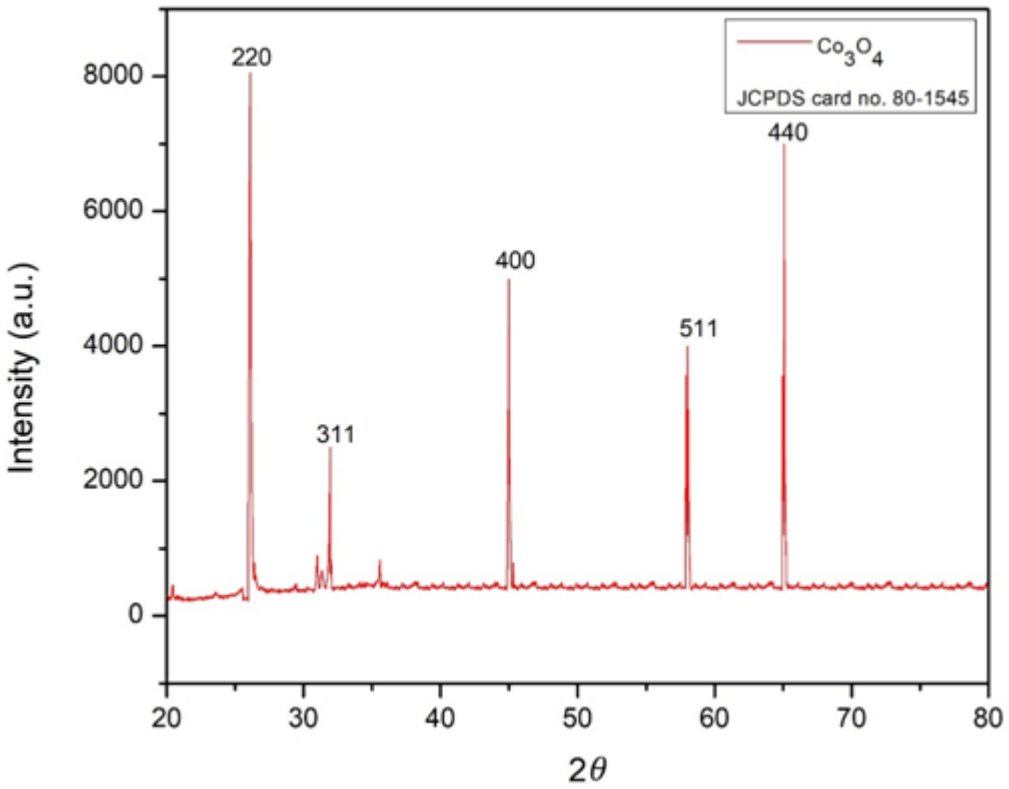

Figure 1: XRD phot of Cobalt Oxide

4.Surface Morphology Study

Scanning Electron Microscopy (SEM) was used for the morphology study of nanoparticles of Co_3O_4 samples. Figure 2 shows the SEM image with formation of clusters. These analyses show the nanoparticles are appeared in the samples by increasing annealing temperature. The SEM images clearly show the formation of large-scale, dense, uniform and well-ordered nanochains of Co_3O_4, which are interconnected to each other.

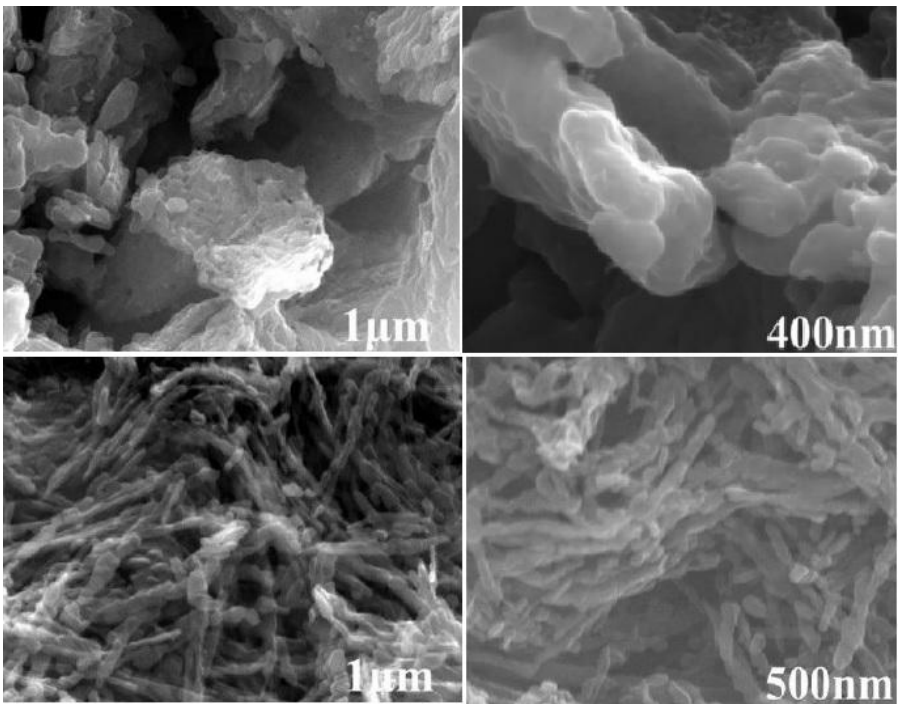

Figure 2: SEM of Co_3O_4

5.Result and conclusion

The Co_3O_4 thin films were Electrodeposited onto the copper substrates. The XRD measurements revealed that the electrodeposited Co_3O_4 thin film was nanocrystalline Co_3O_4 on highly textured Copper substrate. The FESEM micrographs confirm the surface morphology of the prepared films and it is uniformly deposited all over the substrate. It showed that the cobalt oxide thin film was covered with cubical and hexagonal shaped particle. Micro porous spaces between the particles were also observed.

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