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SYNTHESIS AND CHARACTERIZATION OF NANOPOROUS NIO NANOFLAKES SYNTHESIZED USING CHEMICAL BATH DEPOSITION

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ABSTRACT:

In the present work, NiO nanoflakes are developed using the simple chemical bath deposition (CBD) method. The structural, morphological, and electrochemical properties are investigated for deposited NiO nanoflakes. The NiO nanoflakes observed a cubic structure, and Ni-O bonds are confirmed the formation of NiO using CBD. The FESEM results confirm the formation of nanoflakes. The nanoflakes deposited at 120 min shows better electrochemical properties compared to other deposited NiO thin films. In the present study, variation in deposition time has been studied.

Keywords: - Nanoflakes, Nickel oxide, chemical bath deposition

INTRODUCTION:

Environmental pollution and energy consumption have become more and more severe with industrial development. Therefore, dealing with industrial pollution and finding a substitute for traditional energy has become a significant challenge [1-2]. The super capacitors are considered as one of the most promising energy storage devices due to their excellent properties such as fast charge-discharge rate, high power density, low cost, and long cycle life. According to an energy storage mechanism, supercapacitors (SCs) can be divided into electrical double-layer capacitors (EDLCs) and pseudocapacitors [3-4]. Pseudocapacitors materials mainly contain transition metal oxides, hydroxides, sulfides. Among transition metal oxides, NiO has attracted wide attention due to its high theoretical capacity, wide operating potential window, natural abundance, and environmental compatibility [5-6]. The NiO is also able to form different nanostructures by changing any chemical as well as a physical parameter. By considering the advantages of NiO in energy storage devices as well as the need for energy storage devices, many research groups published literature in recent years.

The morphological investigation for improving the supercapacitor is an important task, and a different research group develops recently different nanostructures. Zhang et al. [7] developed NiO based hierarchical hollow nanofibers for improving the specific capacitance. Also, observed specific capacitance 700 f/g with 96% efficiency. Yang [8] et al. synthesized NiO based flake using chemical bath deposition and studied electrochemical properties in 1 M KOH and used for an electrochromic application. Das et al. [9] synthesized NiO nanostructures using the chemical bath deposition and studied structural, optical, and electrical properties. Also, it investigated the effect of thickness on its consequence properties, the three-dimensional nonporous NiOthin films deposited by Liang et al. [10]. For electrochemical supercapacitor and observed high specific capacitance 1776 F g^{-1} in KOH.

By observing literature, it is necessary to develop NiO based nanostructures and study the electrochemical properties.

In the present work, NiO nanoflakes are developed using the simple chemical bath deposition (CBD) method. The structural, morphological, and electrochemical properties are investigated for deposited NiO nanoflakes.

The NiO nanoflakes observed a cubic structure, and Ni-O bonds are confirmed the formation of NiO using CBD. The FESEM results confirm the formation of nanoflakes. The nanoflakes deposited at 120 min shows better electrochemical properties compared to other deposited thin films. In the present study, variation in deposition time has studied.

EXPERIMENTAL:

Analytical grade chemicals usedfor the experiment. For the synthesis of NiO thin films, 0.3M nickel nitrate was used as a precursor.The pH of the nickel solution has maintained at 12 using the ammonia solution. The temperature was maintained at 90°C. The thin films were deposited with three different times, such as 30, 60 min, and 90 min. The as-deposited thin films were annealed at 450°C for 4 hours. For the deposition of NiO, steel substrates were used. The FTIR study has been made using the FTIR from Lamda instruments PVT ltd. The field emission scanning electron microscopic study has been made using the FESEM from Tescan, Pvt. Ltd. The electrochemical study has been made using the biologic Instruments Pvt Ltd.

RESULT AND DISCUSSION:

3.1 X-ray diffraction and Fourier transform infrared spectroscopic study -

Fig. 1 shows the structural investigation of nickel oxide using the XRD and FTIR. Fig. 1 (a) shows the XRD pattern of NiO thin film, The observed d values matched with stander d values mentioned in JCPDS card 00-047-1049. The Nio thin films show the cubic structure with space group Fm-3m. The values of a=b=c=4.177 and α=β=γ=90°. The peak at 37.07, 43.12, 62.80, 75.36 and 79.40 represents orientation along (111), (200), (220), (311) and (222), respectively. The FTIR spectra is an important characterization tool for finding atomic and molecular boding in nanomaterials. Fig. 1 shows the FTIR spectra of NiO nanoflowers. The FTIR spectra are recorded for samples A, B, and C. The peak at 557, 627, 1102 cm^{-1}have belonged to characteristic vibration of Ni-O. The peak near about 3500 cm^{-1}has belonged to the O-H group. As a deposition time increases for NiO, change has been observed in the intensity of NiO peaks [11-12].

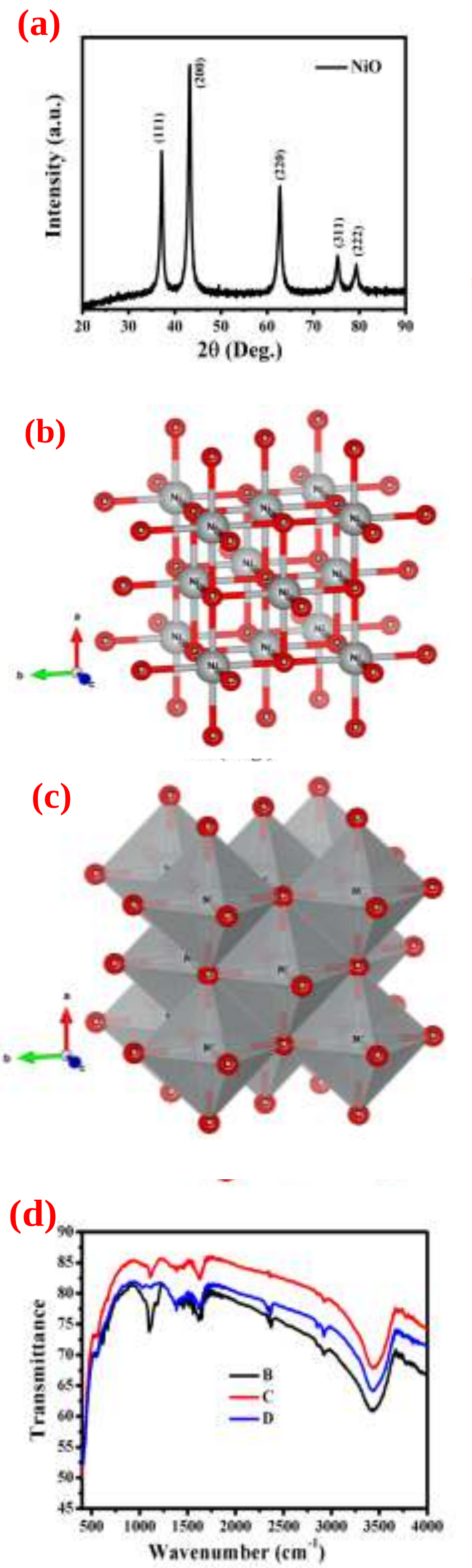

Fig. 1 - FTIR spectra of NiO thin films, (a) XRD pattern of NiO thin film, (b) crystal structure of NiO thin film, (c) Polyhedron of NiO thin film, (d) FTIR of NiO thin film.

3.2. Field Emission Scanning Electron Microscopy -

Fig. 2 shows the FESEM image of a NiO thin film. The NiO thin film shows nanoflakes like nature. The FESEM images recorded for a different time, and as time increases, the size of flex was observed to decrease [13]. The nice nanoflakes are observed in Fig. 2. For 60 min (Fig.2 (a)), the size of flakes is observed to more; also, the distance between two flakes is more as compared to other samples. But as time increases,the distance between two or more flakes observed to be decreased (Fig. 2 (b)). For the 120 min, the size of the flake is very less because ofthe growth of NiO in free space. The bunch of nanoflakes observed like a flower and observed in Fig. 2 (c).

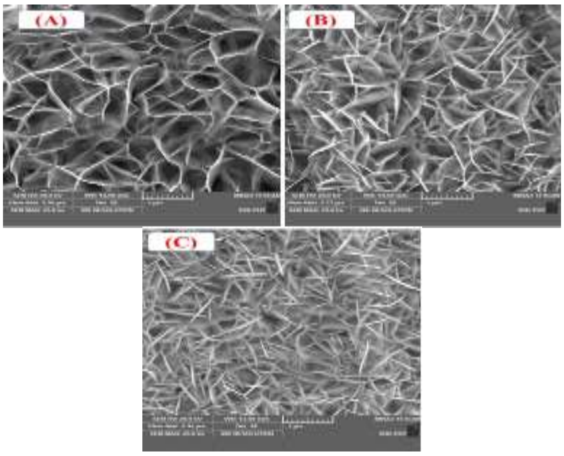

Fig. 2 FESEM images of NiO thin films

3.3 Electrochemical Impedance spectroscopic study –

The electrochemical impedance spectroscopy is a tool used to investigate the charge transfer process at the solid-liquid interface [14-15]. Fig. 3 shows the Nyquist plot (Fig.3 (a)) and equivalent circuit (Fig.3(b)) diagram for NiO thin film. Table. 1 shows the observed parameters from the equivalent circuit diagram. The observed solution resistances are 7.52, 5.60, and 4.68 Ω for 60, 90, and 120 min, respectively. The solution resistance is near about the same for all samples. The charge transfer resistances are 49.05, 85, and 36.8 Ω for 60, 90, and 120 min, respectively. The NiO thin films deposited at 120 min shows the relatively better charge transfer resistance as compared to other deposited samples. Also, these results are consistent with FESEM results. The thin film deposited at 120 min can be used as efficient supercapacitor material.

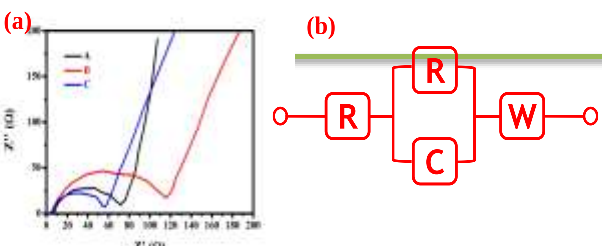

Fig. 3 - Electrochemical impedance study of NiO thin film, (a) Nyquist plot NiO Nanoflex, (b) equivalent circuit diagram

Table. 1 Observed parameter from EIS

Samples/ parameters	R1 (Ω)	C (μF)	R2 (Ω)	W ($\Omega.s^{1/2}$)
30 min	7.52	17.3	49.05	97.7
60 min	5.60	13.9	85.0	130.2
90 min	4.68	13.8	36.8	112.0

3.4 Cyclic voltammetry study –

The cyclic voltammetry (CV) is a powerful and popular electrochemical technique commonly employed to investigate the reduction and oxidation processes of molecular species [16-17]. The cyclic voltammetry study is shown in Fig. 4 (a, b, c) for NiO thin films for 60 min, 90 min, and 120 min, respectively. From Fig.4,it is observed that the maximum current and area under the curve is for 120 min. The NiO sample deposited at 120 min shows the more area under the curve than other deposited samples. It indicates that nanoflakes at 120 min having more specific capacitance than other deposits.

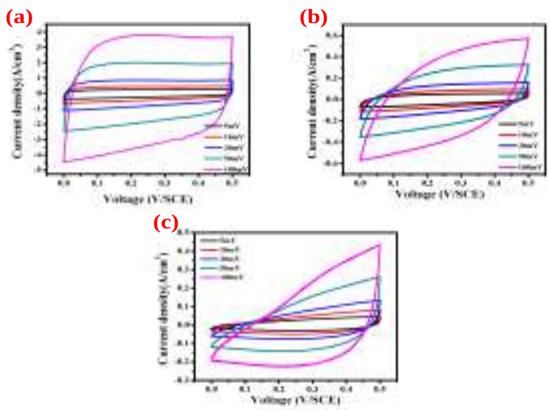

Fig. 4 Cyclic voltammogram for 60, 90, and 120 min.

CONCLUSION: -

In conclusion, nickel oxide thin films have deposited using the chemical bath deposition method. The NiO nanoflakeshave successfully synthesized. The XRD study confirms the formation of NiO thin film. The peaks at 557, 627, 1102 cm^{-1}have belonged to the characteristic vibration of Ni-O. The FESEM study shows the thin NiO having nanoflakes like nature. The electrochemical impedance spectroscopic study exposes the less charge transfer resistance for the thin film deposited at 120 min. The cyclic voltammetry study indicates the more area under the curve for sample deposited at 120 min. In conclusion, NiO thin films deposited at 120 min are more useful for supercapacitor applications.

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