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SYNTHESIS AND STUDIES OF VARIATION OF STRUCTURAL AND OPTICAL PROPERTIES OF NICKEL OXIDE NANOPARTICLES PREPARED BY CHEMICAL PRECIPITATION METHOD

Science

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ABSTRACT

NiO nanoparticles have been successfully prepared by chemical precipitation method. The crystalline nature of the nanoparticles are confirmed by X-ray diffraction (XRD) analysis and High Resolution Transmission electron Emission (HRTEM).The particle size increases from 22 nm to 47 nm respectively with increase in calcination temperatures of NiO nanoparticles from 673K to 873K. Strain is found to decrease with increase in calcination temperature. The optical band gaps are found to be of the order of 5.43 eV, 5.36 eV and 5.26 eV corresponding to calcination temperatures of NiO at 473K, 673K and 873K respectively.

KEYWORDS:

Nanoparticles, XRD and UV-visible

Introduction:

In the past few decades, the nanostructure materials have shown increasing research interest due to their unique physical and chemical properties. These properties are greatly affected by the morphology, size, shape and crystallography of nanomaterials [1-3]. One of the most commonly used metal oxides for wide range of applications is NiO. Because of the novel physical and chemical properties of NiO [4–7], various nanostructures such as nanoparticles [8], nanosheets [9], nanorods [10], nanowires [11], hollow spheres [12] and porous solids [13] of nickel oxide are synthesized. Recently, three-dimensional nanostructures are also reported [12-18].

Nanostructure NiO particle shows stable wide bandgap in the range from 3.6 eV to 4.0 eV [11-20]. When particle size decreases, nanoparticles show blue shift but in some reported work the values of optical band gap of nanostructure are found to be less than the optical band gap of bulk NiO nanoparticles [21-24]

Preparing of NiO nanoparticles

0.02M, 100ml alcoholic solution of $Ni(NO_3)_2.6H_20$ in ethanol is prepared. The solution is stirred constantly at 60°C until it is dissolved completely to form a transparent green solution. A 0.25M solution of NaOH is also prepared in 80ml ethanol. These two solutions are sonicated for 30 minutes to remove dissolved oxygen. NaOH solution is added drop by drop to the alcoholic solution of $Ni(NO_3)_2.6H_20$ under constant stirring at room temperature for 2 h. A black precipitate is obtained and the precipitation is centrifuged at 14000 rpm at an ambient temperature for 15 min to clear the supernatant of the reaction. Then the precipitate is washed with distilled water followed by ethanol and is further sonicated for 15 min. The successive sonication and centrifugation process are repeated up to four times. Finally, the black precipitate is separated from supernatant liquid and washed with ethanol and distilled water.

Characterisation

X-ray diffraction analysis

The XRD spectra shows the formation of NiOH nanoparticles at room temperature but as we increase the calcination temperature to 673K, the NiO phase is achieved which is confirmed by XRD study and the prepared nanoparticles matched with the standard data of JCPDS No. 01-1239. The sample shows polycrystalline nature and the peaks are found which are indexed as (111), (200), (220), (311) and (222).The broadening of peaks is observed which is mainly due to the size-shape of the nanoparticles and presence of strain. The particle size is calculated using Scherer's formula,

$$D = K\lambda / \beta\cos\theta \quad \text{.....(1)}$$

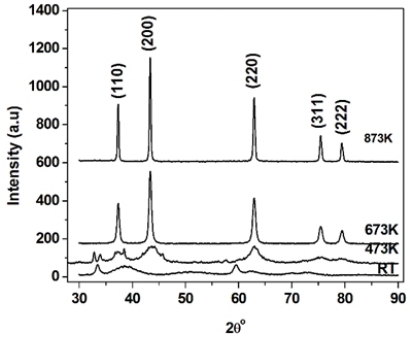

Fig.1 XRD spectra of NiO nanoparticles prepared at 0.02M and at calcination temperature (A) RT (b) 473K (c) 673K and (D)873K

Where λ is the wave length of the radiation used, 2θ is the Bragg angle and K is a constant whose value is 0.9and β is the FWHM in radians.

The patterns are characterized by classical W-H plot to calculate the strain present in the sample using the following relation,

$$\beta\cos\theta = K\lambda/D + 4\varepsilon\sin\theta \quad \text{.....(2)}$$

The slope of the curve βcosθ vs sinθ(Fig.2) gives the values of strain present in the sample. The strains calculated from W-H plots are shown in Table.1.

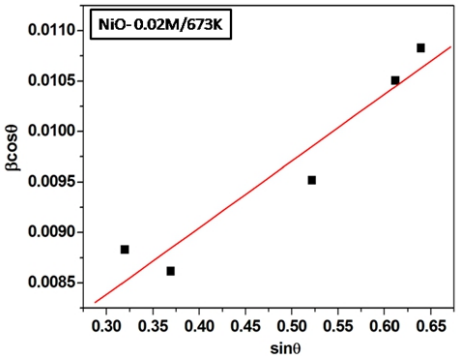

Fig.2 W-H plot of NiO nanoparticles prepared at 673K and at 0.02M

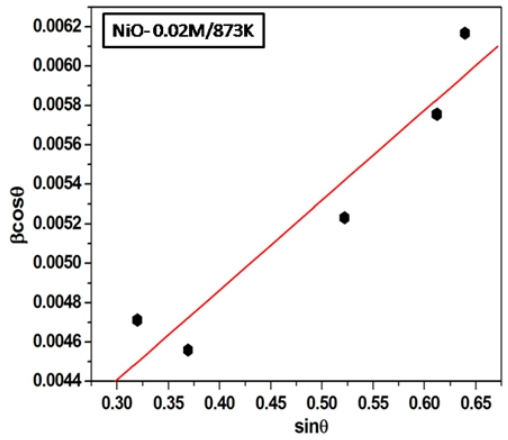

Fig. 3 WH plot of NiO nanoparticles prepared at 873K

Table .1 particle size and lattice strain variation

Sample code	2 θ values	(hkl)	Size obtained by scherer formul (nm)	Size obtained by W-H plot(nm)	Lattice Strain By W-H plot
NiO-673K 0.2M	37.318	(100)	16	22	0.001653
	43.356	(200)	17		
	62.959	(220)	15		
	75.5	(311)	14		
	79.495	(222)	14		
NiO-873K 0.2M	37.318	(100)	31	47	0.001140
	43.356	(200)	32		
	62.959	(220)	27		
	75.5	(311)	25		
	79.495	(222)	23		

TEM analysis:

The HRTEM image of the synthesized nanoparticles recorded in a JEOL JEM 2100 (200 kV) shows that the particles size distribution is non-uniform. The average crystallite sizes of the NiO nanoparticles obtained by TEM analysis are compared with those obtained from XRD analysis. The inter planar spacing (d-value) are also obtained from TEM analysis and are matched with that obtained from the XRD spectra for each SEAD images of the nanoparticles. XRD is widely used to determine the particle size of nanoparticles but TEM is the best way for the measurement of nanoparticle size. The Scherrer's method for calculating particle size gives an average value of the entire particle responsible for diffraction perpendicular to a particle set of plane. However, by TEM, besides directly measuring particle size, the morphology of the particles can also be observed. Fig. 4a shows the uniform particles of the NiO nanoparticles and average particle size from TEM analysis are found to be 26 nm. Fig.4b shows the Lattice plane (002) and Fig. 4c shows the selected area diffraction pattern which shows the crystalline nature the nanoparticles.

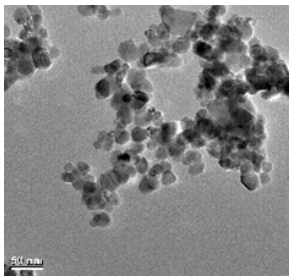

Fig.4a HRTEM image of NiO nanoparticles prepared at 0.02M and 673K

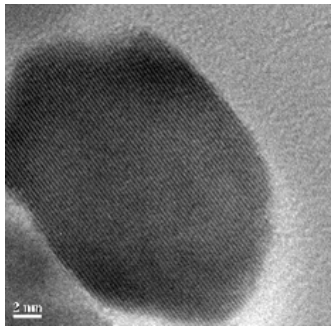

Fig.4b HRTEM image of lattice plane (200) of NiO nanoparticles prepared at 0.02M and 673K

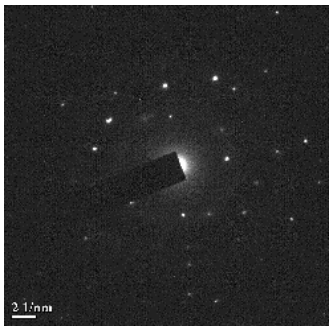

Fig.4c SEAD Image of NiO nanoparticles prepared at 0.02M and 673K

UV analysis:

UV-visible absorption spectroscopy is one of the most important techniques to investigate the energy structures and optical properties of nanocrystalline semiconductors and has widely been studied [25]. The strong absorption in the UV region can be assigned to the band gap absorption of nanostructure NiO [26]. Fig.5 shows the plots of $(\alpha h\nu)^2$ vs hν for NiO nanoparticles corresponding to different annealing temperatures calculated from absorption data. The linear nature of the plots indicate the involvement of direct transition and thus the prepared NiO nanoparticles fall on the group of direct band gap Metal Oxide [27]. The calculated value of optical band gap of 0.02M NiO nanoparticles prepared at calcination temperatures 673 K, and 873 K found to be 5.43eV, 5.36eV and 5.26eV corresponding to calcination temperatures of NiO at 473K, 673K and 873K respectively.

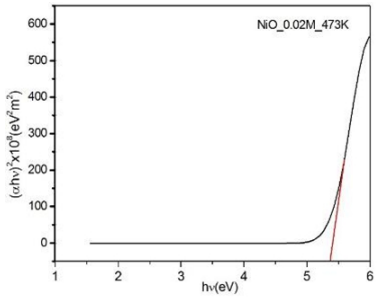

Fig.5a shows the plots of $(\alpha h\nu)2$ vs $h\nu$ for NiO nanoparticles prepared at 0.02M and 473K.

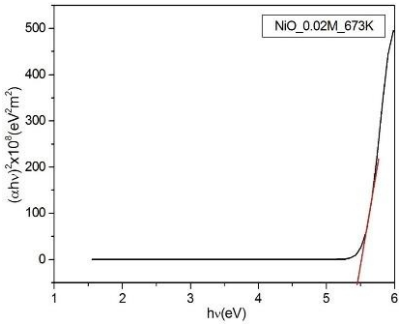

Fig.5b shows the plots of $(\alpha h\nu)^2$ vs $h\nu$ for NiO nanoparticles prepared at 0.02M and 673K.

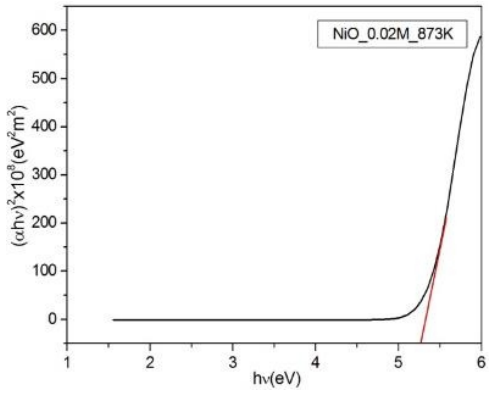

Fig.5c shows the plots of $(\alpha h\nu)2$ vs $h\nu$ for NiO nanoparticles prepared at 0.02M and 873K.

Conclusions

It is found that with increase in calcination temperature from 673K to 873K, the particle size increases from 22 nm to 47 nm respectively. As the particle size increases from 22nm to 47nm, the values of lattice strain calculated by W-H plot decreases from 0.001653 to 0.001140 respectively. The optical band gap of NiO nanopartilce prepared at 0.02M are found to be 5.43eV, 5.36eV and 5.26eV corresponding to calcination temperatures of NiO at 473K, 673K and 873K respectively. Particle size obtained from TEM analysis are found to be 26nm at calcination temperature 673K of NiO nanoparticles prepared at 0.02M.

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