International Conference on Systems, Science, Control, Communication, Engineering and
Technology 2016 [ICSSCCET 2016]

ISBN	978-81-929866-6-1	**VOL**	02
Website	icssccet.org	**eMail**	icssccet@asdf.res.in
Received	25 – February – 2016	**Accepted**	10 - March – 2016
Article ID	ICSSCCET168	**eAID**	ICSSCCET.2016.168

Structural Analysis of Nickel Oxide Nanoparticles

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Abstract: *Nickel oxide (NiO) nanoparticles were produced via a solution-phase precursor route method. Guanidine metal citrate precursor was obtained by the addition of solid guanidium carbonate to the citric acid and nickel nitrate hexahydrate solution at the temperature of 95 °C in water bath. By calcinating the precursor at different temperature and different time period, the porous nickel oxide nanopowders were produced. X-ray diffraction patterns revealed that the product had a well-crystallized/high-purity nanostructure with grain size of ~35 nm and it was confirmed by Scherrer's formula and William and Hall plot methods.*

Keywords: *NiO nanoparticles, XRD, Scherrer's formula and William and Hall plot*

1. INTRODUCTION

Nanoparticles of transition metal oxides have attracted the attention of many materials researchers due to their exceptional properties stimulating advanced applications [1-2].Nickel oxide is a p-type semiconductor with a wide band gap (3.6–4.0 eV) and large exciton binding energy. [3,4] Among the various preparation techniques, the solution-phase precursor route has many advantages and much promising. The advantages of this solution based technique are that it is simple, cost effective, appreciable reduction in the processing temperature and the resulting material being of nanostructure. Also, it provides excellent stoichiometry, low impurity content compared to the other methods.

In this work, Guanidine metal citrate precursor was first produced by solution-phase precursor route method. Then it was post heated to form nickel oxide nanopowders. The stoichiometry, purity and grain size were confirmed by XRD.

2. Experimental Procedure

Sample Preparation

In this method the synthesis of NiOnanopowder was composed of two stages: (a) the formation of guanidine nickel citrate precursor and (b) subsequent heat treatment of precursor to transform into NiO. To synthesis the precursor, solid Guanidinium carbonate was added to an aqueous solution of Citric acid with continuous stirring. To this acid-base solution an aqueous solution of Nickel nitrate hexahydrate was added. Here the metal, acid and base ratio was maintained at 1:1:4.

There is a precipitate formation taking place while adding the metal to acid base solution. This precipitated solution became clear with the prolonged stirring. The resulting clear solution was evaporated in the water bath to reduce half of its volume. The concentrated solution was left aside over night at room temperature for re-crystallization. Finally a highly crystalline substance was separated from the solution. The separated complexes were washed with distilled water and dried in air. By heating this citrate prcursor at various

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temperature from 600°C to 1000°C and different time duration one hour and two hours, nickel oxide nanoparticles have been obtained.

Results and Discussions

As shown in Fig (i), the TG/DTA analysis confirmed that the transformation of NiO from its metal complexes taking place after 500°C. It showed that dehydration takes place at 170 and removal of base followed by citrate takes place around 320-460°C.

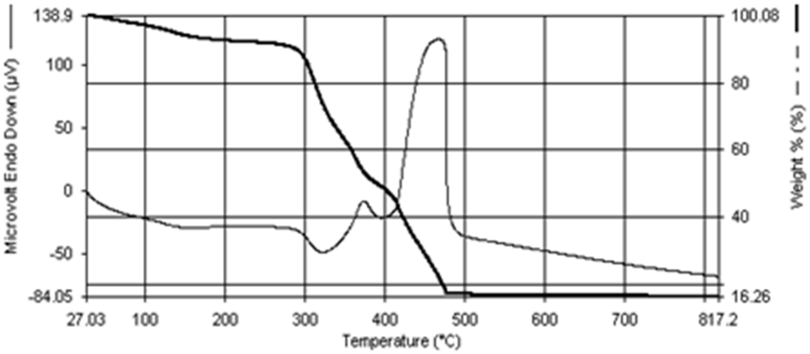

Fig (i) TG/DTA curves for the metal complex

Then the formation of NiO by thermal decomposition of the metal complex has been confirmed by the IR spectrum of the residues. The IR spectrum for NiO is shown in Fig (ii). The strong absorption band at 419 cm^{-1} is corresponding to the stretching vibration of NiO. The band at 3419 and 1383 cm^{-1} are due to the calcinated powder tends to physically absorb water and carbonated ions [5,6].

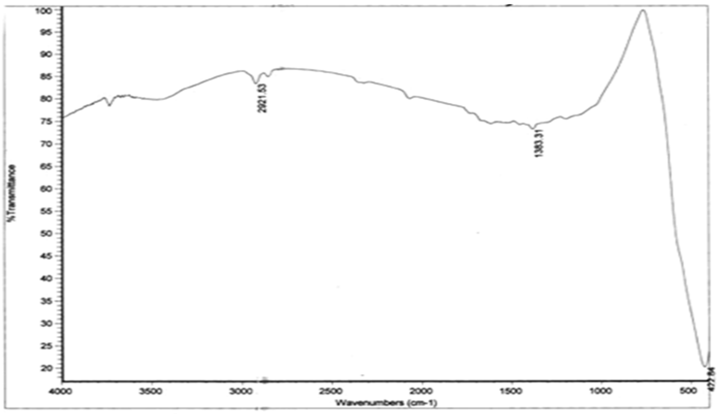

Fig (ii). IR spectrum of NiO

The phase purity and crystallinity of the prepared samples were studied through the powder X-ray diffraction method. The powder XRD pattern of NiO nanoparticles prepared from the complex precursor method is shown in Fig (iii) and (iv).Fig (iii) shows that NiO nanoparticles obtained by thermal decomposition of the complex precursor with different calcinations temperatures, varied from 600 to 1000 °C at zero hour.The XRD pattern for the 600 to 800 °C calcinated sample shows, few hydroxide peak in the product sample. This confirms the sample in the form of nickel hydroxide upto 800°C calcinations. While increasing the temperature to 900 to 1000 °C the hydroxyl group disappears from the complex precursor and it form a single phase *face centered cubic* (*fcc*) structure of NiO. The prominent and sharp peaks of the diffractogram indicate the formation and high crystallinity of the prepared NiO [7-9]. These peaks are very well match with the JCPDS # 78-0423.

Fig (iv) shows the XRD pattern for the NiO obtained by calcinating the complex precursor with the temperatures from 600 to 1000 °C at two hours. Here also, the 600 and 700 °C calcinated samples shows the peaks corresponding to hydroxide. The pure NiO was obtained after 800 °C annealing for 2 hours. At higher calcinations temperatures, the samples shows pure NiO phase with high crystalline nature. Again, the crystallinity is further improved compared to the sample annealed at 1000 °C for zero hour.

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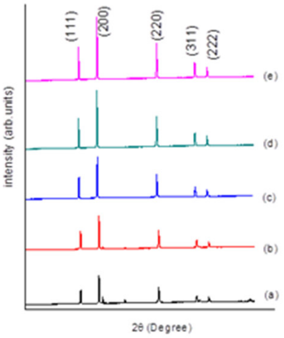

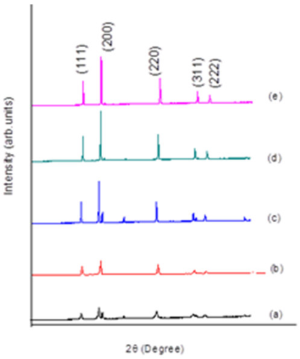

The average crystallite sizes of the samples were calculated by two different methods extracted from the XRD pattern. The (111), (200), (220), (311), and (222) planes are used to calculate average crystallite size. The average crystallite size of the NiO prepared by thermal decomposition of complex precursors are calculated by using Scherer's formula and compared with the William-Hall plot method.

Scherrer's formula is given by, $$D = \frac{K\lambda}{\beta\cos\theta} \qquad (1)$$

Where, **λ**is the wavelength of the X-rays used,**θ** is a Bragg angle,**K** is the geometrical shape factor and **β** be the full width half maximum of the sample in radian.

For the entire experiment, the plot is drawn only for the preferred orientation of the peaks. From the linear fit to the data, the crystallite size D was extracted from the y-intercept and the strain ε from the slope of the fit. W-H plot for the samples are shown in fig.(v) and (vi).

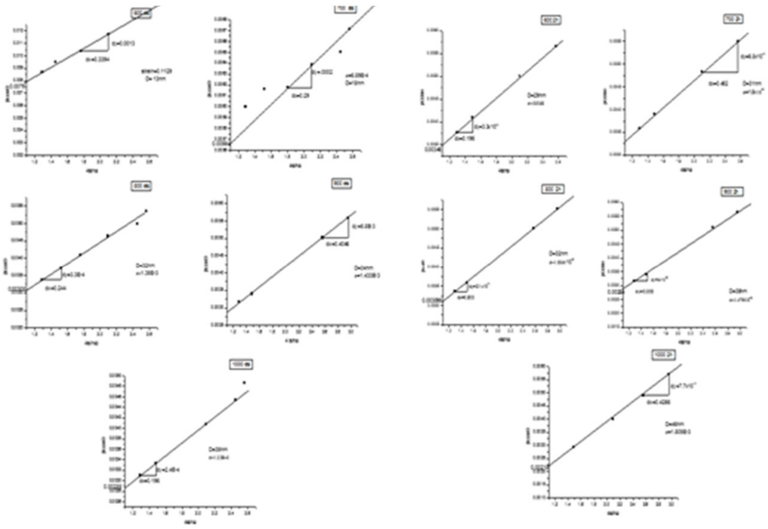

Fig (v) William and Hall plot for the samples prepared at various temperature with zero hour calcination time. Fig (vi) William and Hall plot for the samples prepared at various temperature with two hour calcination time.

The calculated average crystallite sizes were tabulated.

The parameters extracted from the X-Ray diffraction pattern

Calcination time - zero hour				
Samples calcination temperatures (□C)	Avg.Grain size calculated from (nm)		Strain lin^{-2}m^{-4}	Dislocation Density lines/m^{2}
	Scherrer formula	W-H plot method		
600	20	13	0.1129	2.50E-03

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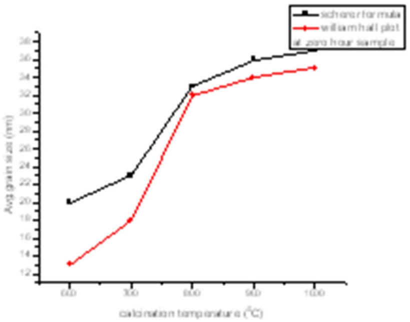
700	23	18	6.89E-04	1.89E-03
800	33	32	1.35E-03	9.20E-04
900	36	34	1.43E-03	7.70E-04
1000	37	35	1.22E-03	7.30E-04
Calcination time - two hours				
600	33	29	0.0168	9.00E-05
700	34	31	1.50E-03	8.70E-04
800	35	32	1.03E-03	8.20E-04
900	38	35	1.48E-03	6.90E-04
1000	39	46	1.81E-03	6.60E-04



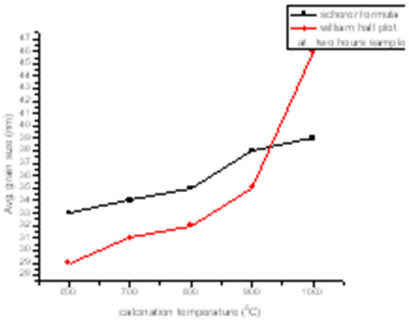


Fig (vii) Calcination temperature vs average grain size for zero hour calcinated samples Fig (viii) Calcination temperature vs average grain size for two hour calcinated samples

By W-H models the strain due to the dislocation received at the time of synthesis were calculated and tabulated. In both cases the increase in the calcination time and temperature leads to the increase in the average grain size and it is shown in fig. (vii) and (viii). The increase in the calcination temperature and time leads to the reduction in the lattice strain and which is reported in Table.

The lattice parameters are calculated by using the following formula,

$$d_{hkl} = \frac{a}{(h^2 + k^2 + l^2)^{1/2}}$$

The *d* spacing values corresponding to the planes such as (111), (200), (220), (311), and (222) are 2.41, 2.09, 1.48, 1.26 and 1.20 at lattice constant *a* = *b* = *c* = 4.178 Å which is good agreement with the reported value. The cell volume of the sample was also calculated.

Conclusion

Highly-crystallized pure nickel-oxide nanoparticles with average crystallite size of ~35nm were synthesized by a solution-phase precursor route method. Morphology of the resulted powder showed NaCl-like cubic structure. The average crystallite size increased with increasing the calcination temperatures and the strain was decreased. The range of crystallite size varied from 30 to 40 nm. The structural properties were analyzed by both Scherrer formula and the William and Hall methods.

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