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STUDIES ON SYNTHESIS, STRUCTURE AND OPTICAL PROPERTIES OF NICKEL NANOPARTICLES

M. HEMALATHA[a*], N.SURYANARAYANAN[b].
[a]*Department of Physics, Vivekananda College of Engineering for Women, Elayampallayam, Tiruchengode*
[b]*Department of Physics, Government College of Technology, Coimbatore-641659, India.*

The formation of nanocrystalline nickel particles has been investigated via a chemical reduction method. The powder XRD study confirms that the average size of the particle is 12nm.The quantum size confinement of the crystallites is evident from the blue-shift of the absorption edge in the UV-visible absorption spectrum. Result of the thermo gravimetric analysis(TGA) and differential thermal analysis (DTA) indicate that the thermal stability of nickel nanoparticles.

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Keywords: Synthesis; Nanoparticles; Nickel; Chemical reduction method

1. Introduction

In the past two decades, considerable attention has been devoted to the synthesis of metal nanoparticles because of their unusual properties and potential applications in optical, electronic, catalytic and magnetic materials [1-3]. Until now, only a few works on the preparation of Nickel nanoparticles have been reported and they usually were performed in Organic media to avoid the formation of nickel oxide or hydroxide [4]. Szu-Han Wu et al [5] and Yang long Hou et al [6] prepared Nickel nanoparticles by chemical reduction method and characterized Nickel to be pure crystalline with a FCC structure. Xiao-Min Ni et al [7] had prepared the Nickel nanorods in Water-in-oil micro emulsion technique with a FCC structure.

Our objective in this paper is to study the chemical reduction method to synthesize nanosized nickel nanoparticles using nickel chloride and hydrazine in an aqueous solution.

2. Experimental details

10ml of Ethylene glycol was chosen as a solvent and 2.5mM of $NiCl_2$ was dissolved in it. Then, an appropriate amount of hydrazine (0.05-0.9M) was added. No particles were formed in ethylene glycol without adding sufficient amount of hydrazine. This reveals that Nickel ions were reduced by hydrazine instead of ethylene glycol. NaOH, which acts as a catalyst of 10-72 μ/ml, were added in sequence and stirred well.

The resultant product was taken and kept in the room temperature. There was no change in that product even after two days. Then, the resultant product was kept in the water bath above the room temperature and heated. No significant change takes place. When the temperature raised to above 90^o C, there was a change in the resultant product. That is, the bluish green colour precipitate slowly changed in to black colour, indicating the formation of Nickel nanoparticles.

* Corresponding author: mhlhemu@gmail.com

Then after half-an hour the precipitate is separately obtained at the bottom of the solution. By washing the precipitate with ethanol and dried in the room temperature. Then precipitate is taken in the silica crucible and heating the precipitate in the water bath at the temperature of ~96^{o} C to get the dried black Ni nanopowders..

The structural aspects of the Nickel powder were analyzed using X-ray diffractometer with filtered CuKα radiation (λ=1.5418Å). The optical absorption measurements were recorded in the wavelength range of 200-800nm using JASCO V-570 Spectrophotometer.

3. Results and discussion

3.1Structure studies

Fig.1shows a typical XRD pattern of the Nickel sample. From the Fig three characteristic peaks for Nickel [2θ = 44.6˚, 51.9˚, 76.4˚] corresponding to Miller indices (111), (200), (220) were observed. This revealed that the resultant particles were pure face-centered cubic (FCC) Nickel.

No obvious peaks of Nickel oxides and hydroxides were detected, possibly attributed to the observed phenomenon that Nitrogen gas was produced and bubbled up continuously during the reaction. It could be suggested that the above said gas produced might auto create an inert atmosphere; hence the input of extra Nitrogen gas was not necessary for the synthesis of Nickel nanoparticles [4,5].

In Fig 1, the intensity ratio of the peaks [111]: [200]: [220] were calculated to be [100, 26, 16] which exactly coinciding with the JCPDS [no: 040850] values. This confirms the presence of Nickel particles in the sample and it is in good agreement with the earlier reports [7, 10-12].

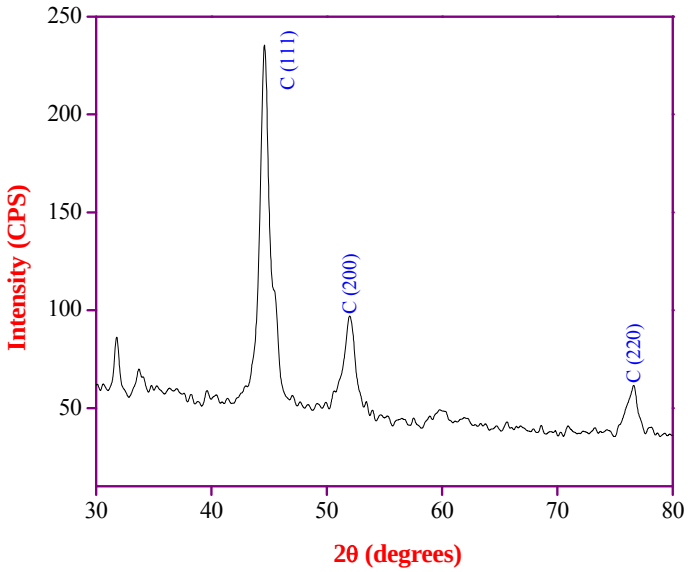

Fig. 1. XRD Spectra of Nickel Nanoparticles

3.2. TG/DTA Analysis

The DTA and TGA curve of the prepared powder is given in Fig.2. The DTA curve has two exothermic peaks at 277^{o}C and 435^{o}C and one endothermic curve at 342^{o}C as evidenced by the earlier work [13].

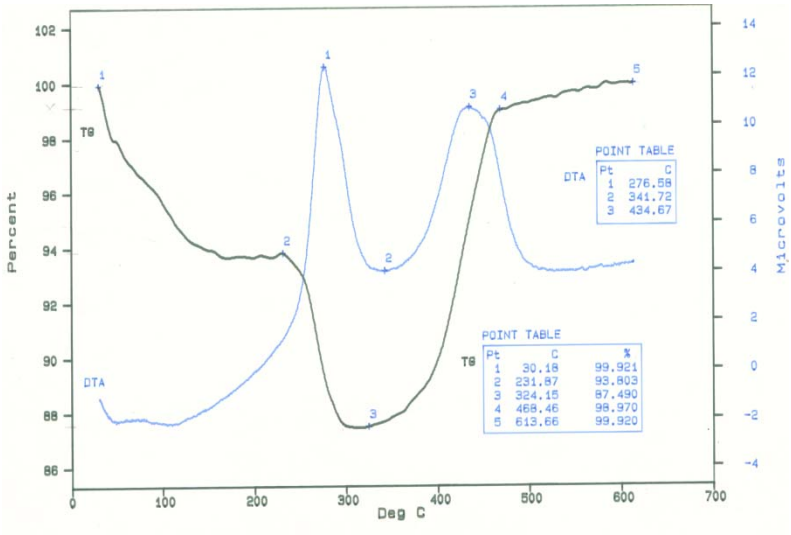

Fig.2.DTA and TGA curves of Ni nanoparticles.

In TG curve, the colloidal nanoparticles undergoing weight loss and weight gain is indicated in comparison with DTA curve. At 232^{o}C , the sample has lost 6% weight of the initial amount. This corresponds to the sharp exothermic peak at 277^{o}C[14]. This exothermic peak suggests the combustion of organic residue and at the same time the nucleation and growth of the crystallites of NiO.

On further heating, the powder decomposes endothermic ally at 342^{o}C, with weight loss corresponding to 12.5% of the original weight [8]. This may be due to decomposition of organic component and the reduction of Ni^{2+} to $Ni^{(0)}$. Here, the nickel is present in the cubic crystalline form, which is in agreement with XRD pattern shown in Fig.1.

On continuation of heating again, the resultant Nickel powder undergoes weight gain to the extend of 11.5% at 468^{o}C. This corresponds to exothermic change at 435^{o}C. At 600^{o}C, this is fully oxidized to NiO. The final weight gain was 12.5%. So this indicates that the particle consists of pure nickel, with crystallite size of 12 nm. As the size of the particles is smaller it increases the oxidation rate of the particle Nickel [15].

3.3. Optical studies

From the Fig. 3. the absorption band is observed at 220nm for the particle size of 12 nm. Galo Cardenas et al. have reported that Nickel particles of size 4.46 nm in Ni-2-propanol system present absorption band at 212 nm [16]. Curtis et al. [9] found that Nickel bulk absorption presents a band centered near 230 nm. The difference in position of the absorption band in the present investigation and that observed in the earlier reports may probably due to the effect of quantum size confinement [17]. It conforms the presence of Nickel nanoparticles.

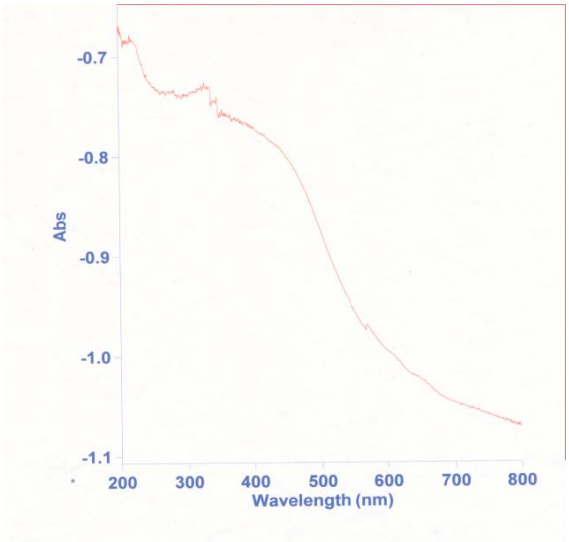

Fig.3. Optical absorption spectrum of Nickel nanoparticles

3.4. PL Studies

Fig.4.represents the photoemission characteristics of Nickel nanoparticles emission at 383 nm. Two characteristic emission bands were observed at 214 nm and 240 nm with intensities 47and 117 arbitrary units respectively. The emission peaks found in the PL spectra may be the characteristic features of Nickel nanoparticles occurring due to the effect of quantum size. Further investigations are going on.

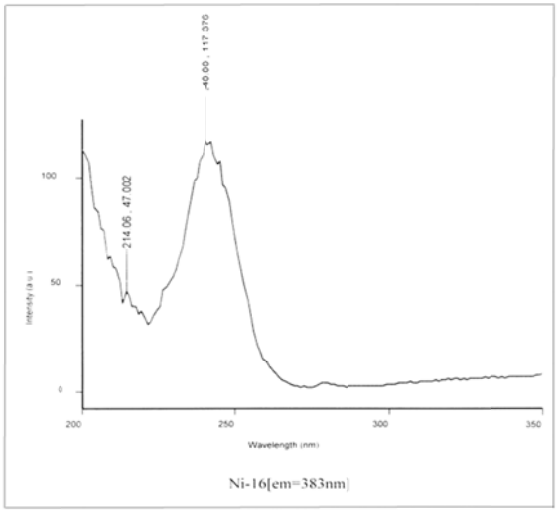

Fig.4. Photoemission spectrum of Nickel nanoparticles

4. Conclusions

The Nickel nanoparticles have been synthesized by chemical reduction method. As per the XRD analysis the structure of the Nickel nanoparticles is found to be FCC crystalline in nature, with the average particle size calculated as 12 nm. Thermal analysis confirms the presence of Nickel. With the exhibition of two exothermic and one endothermic curves and is in conformity with the previous investigation. The optical absorption spectra and photoluminescence characteristics strongly reveal the presence of quantum confinement effect in Nickel nanoparticles.

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