

Structural and Optical Properties of NiS Nanoparticles Synthesized by Chemical Precipitation Method

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ABSTRACT: In the present work, orthorhombic NiS nanoparticles were obtained by chemical precipitation method using tri ethanol ammine (TEA) as a capping agent. The nickel sulfide nanoparticles were characterized by X-ray diffraction (XRD), UV-visible spectrophotometry (UV-vis), photoluminescence (PL) and micro Raman techniques. The X-ray diffraction pattern of the sample indicated the formation of orthorhombic structure of NiS. Absorption due to band edge and defect levels was observed in the absorption spectrum of nickel sulphide nanoparticles. Emission peaks due to defect levels was observed in the photoluminescence spectrum.

KEYWORDS: Nickel Sulfide nanoparticle, micro Raman, UV Visible absorption, Photoluminescence.

I. INTRODUCTION

In the recent years, transition metal sulfide nanoparticles are being studied extensively owing to their unique electrical, optical and catalytic properties. These materials find applications in fields of research interest such as fluorescence devices, solar energy conversion devices, electroluminescence devices, super conductors, flat panel displays, semiconductor and optical devices, etc. [1-6]. Nickel sulfide proves to be a promising material for application as cathode material for the rechargeable lithium battery and catalyst in the degradation of organic dyes and different magnetic devices and certain non-linear optical devices. [7-12]. NiS films have been used as catalyst and coatings in photovoltaic cells [13&14]. Catalysts have applications in the oil industry for the separation of elements with hydrocompounds from insulators [15]. Due to the various applications, different synthesis methods were employed for the preparations of the nanoparticles such as sol-gel method, laser ablation, solvothermal process, UV irradiation, colloidal microemulsion method [16&17]. Nickel sulfide exhibits complicated compositional, structural, optical, electrical and magnetic phase behaviour. Depending on the preparation method, a number of binary nickel sulphides such as Ni₃S₂, Ni_{3+x}S₂, Ni₄S_{3+x}, Ni₆S₅, Ni₇S₆, Ni₃S₄ and NiS have been reported [18-22]. To the knowledge of present authors, only few reports are available on the synthesis, photoluminescence and micro Raman study of nickel sulphide nanoparticles

II. EXPERIMENTAL DETAILS

All the chemicals reagents used in our experiments were of analytical grade, commercially purchased from Merck used as received without further purification. 10 ml of 1M nickel acetate and 2 ml of 0.1 M tri ethanol ammine (TEA) is taken in a beaker. To this solution, 10 ml of 1M Na₂S is added dropwise while the reactants are continuously stirred. The precipitate formed is centrifuged, washed several times with water and finally with acetone, and then air dried to obtain nano powders of nickel sulfide. The oxidation mechanism of nickel sulfide from ambient up to 1000 °C was investigated by thermogravimetric (TG) and differential thermal analysis (DTA) using Perkin Elmer, Diamond TG/DTA set up. X-ray Diffraction patterns of the nickel sulfide samples were recorded using a Philips XpertproDiffractometer and using CuK α radiation over the diffraction angles (2θ) from 10 to 90°. The micro Raman spectra of nickel sulfide nanoparticles were recorded using Horiba Labram-HR with an excitation at 514.5 nm. UV-vis spectrophotometry and photoluminescence spectra of nanoparticles of NiS were recorded using Varian, Cary 5000 spectrophotometer and JASCO FP 8200 Spectrofluorometer, respectively.

III.RESULT AND DISCUSSION

A.X-ray Diffraction Studies

Fig. 3.1 shows the of XRD pattern of nickel sulfide nanoparticles of present study. The diffraction pattern reveals that the nickel sulfide nanoparticles prepared is polycrystalline in nature .The diffraction peaks appear at 2θ values of approximately 19.6, 20.7, 26.1, 30.4, 33.1, 35.1, 38.9, 42.1 and 52.3°corresponding to (200), (201), (221), (113), (041), (312), (241), (242), (205) planes, which well matched with orthorhombic phase of Ni₉S₈ (JCPDS card No. 22-1193). The interplanar distance ‘d’ were calculated using Bragg’s law $n\lambda=2d\sin\theta$ where n is the order of diffraction, λ is the wavelength of the incident X-ray, d is the interplanar distance, θ is the Bragg’s angle, The observed 2θ values, interplanar distance and corresponding planes are tabulated in table1.

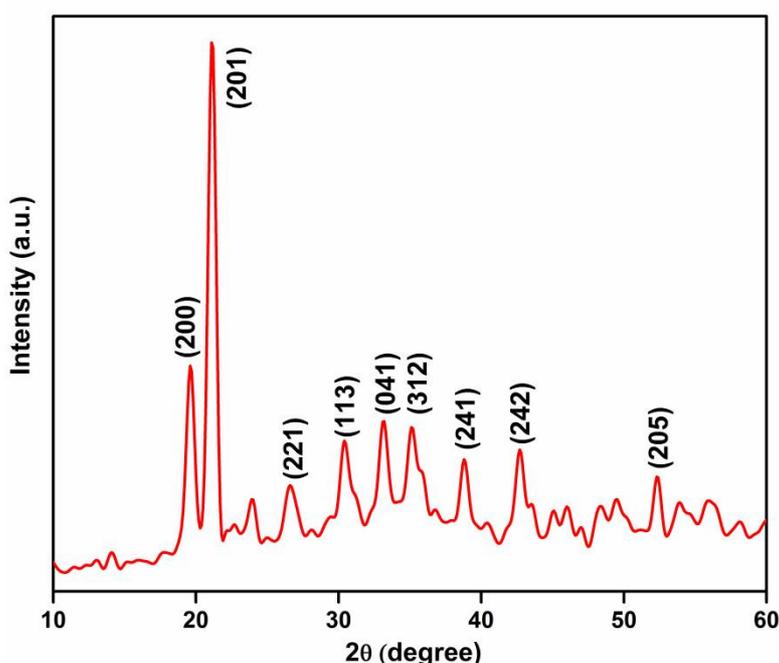


Fig. 1 XRD pattern of Ni₉S₈nanoparticles

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

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The average crystallite size is calculated from the full width at half maximum (FWHM) of the diffraction peaks using the Debye-Scherrer formula [23] $D = k\lambda/\beta\cos\theta$, where D is the mean grain size, k is a geometric factor (equal to 0.89), λ is the X ray wavelength, β is the FWHM of diffraction peak and θ is the diffraction angle. The grain size of the NiS particles calculated from the most intense peak is approximately 20 nm.

Table 1

Observed 2θ values, interplanar distance and corresponding planes

S.NO	2θ (degree)	d (nm)	Plane
1	19.723	0.4495	200
2	21.235	0.4180	201
3	26.699	0.3335	221
4	30.431	0.2934	113
5	33.407	0.2679	041
6	35.384	0.2533	312
7	38.872	0.2314	241
8	42.848	0.2108	242
9	52.288	0.1748	205

B. Micro Raman studies

The micro Raman spectrum of nanoparticles of nickel sulfide of present study is shown in the figure 2. The spectrum shows peaks around 205, 465, 608, 984 and 1087 cm^{-1} . These peaks positions are observed to be red shifted indicating phonon confinement effect.

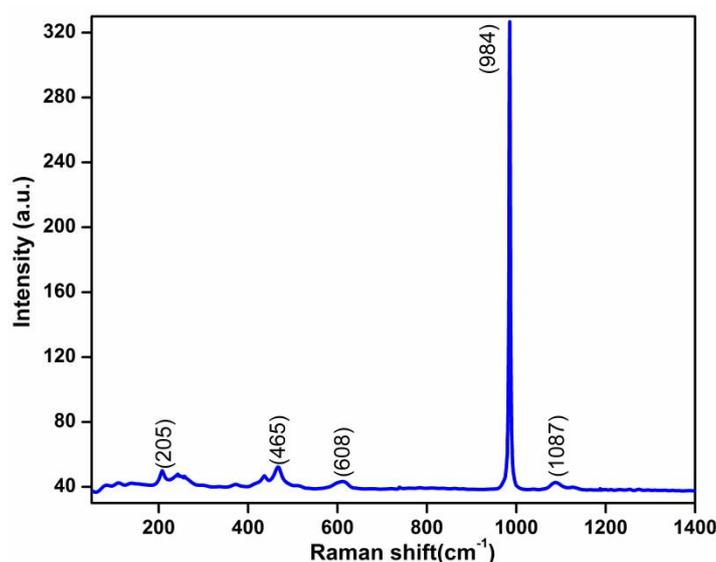


Fig. 2 Raman spectrum of nickel sulfide nanoparticles

C. UV-Visible absorption studies

Figure 3 shows the UV-Visible absorption spectrum of NiS nanoparticles. The spectrum shows absorption

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 5, Issue 8, August 2016

peaks around 225 nm, 269 nm, 355 nm and 435 nm. The peak at 225 nm can be ascribed to band edge absorption and that at 269, 355 and 435 nm to absorption due to defect levels.

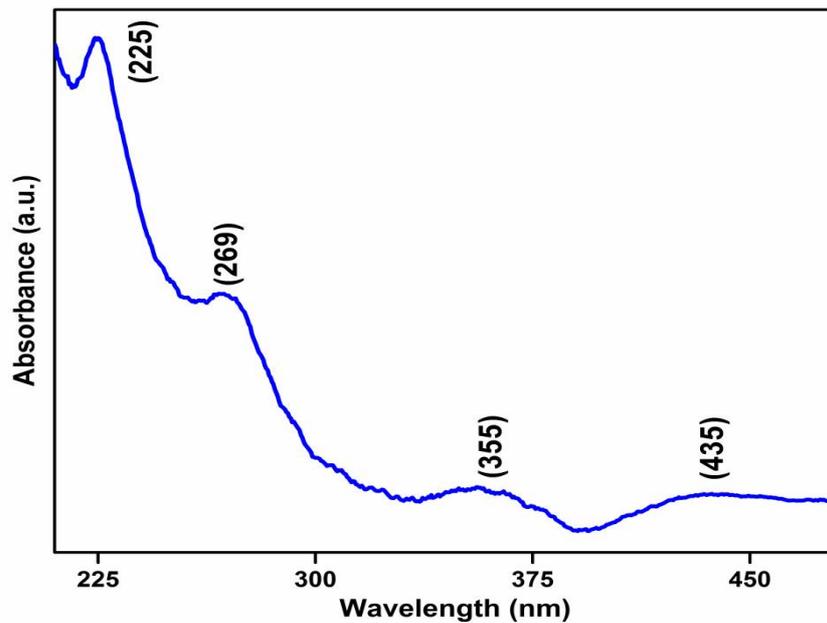


Fig. 3 UV-Visible absorption spectrum of nanoparticles of nickel sulfide

D. Photoluminescence studies

The photoluminescence spectrum of nickel sulfide nanoparticles is shown in the figure 4. The excitation wavelength is 350 nm. The emission spectrum shows two broad peaks around 418 and 437 nm. The peaks were attributed to emissions from defect levels.

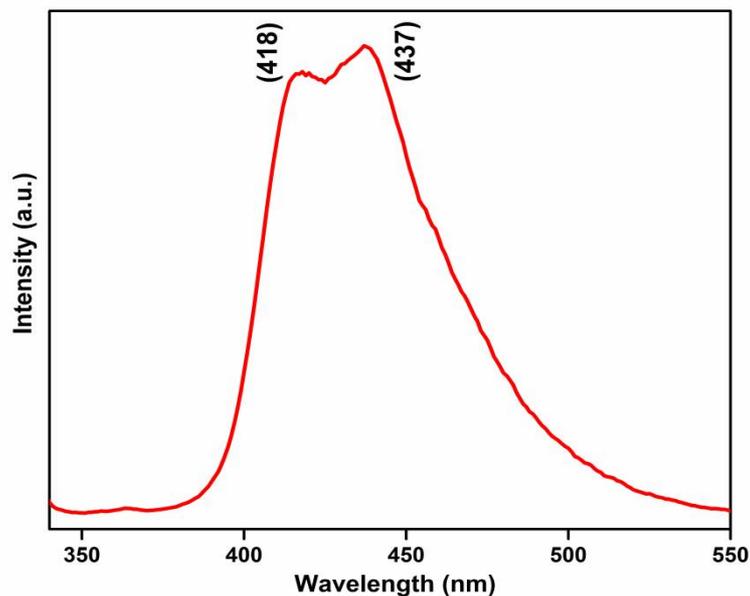


Fig. 4 Photoluminescence spectrum of NiSnanoparticles

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 5, Issue 8, August 2016

IV.CONCLUSION

In conclusion, nickel sulfide nanoparticles (Ni_9S_8) have been prepared by chemical precipitation method and were annealed at 700 °C. The XRD pattern show that the nanoparticles of NiS are in orthorhombic phase and the grain size estimated using Debye Scherrer equation was approximately at 20 nm. The UV-Visible absorption spectrum showed four absorption peaks. The band edge absorption peak shows a blue shift compared to that of bulk NiS indicating quantum size effect. The PL spectrum show peaks around 418 nm and 437 nm, which were attributed to transitions due to defect energy levels.

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