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A simple hydrothermal route for synthesizing copper Selenide Nano-Flakes

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ABSTRACT

Cu_2Se particles have been synthesized by the hydrothermal method at a temperature of 200° C. The synthesized nanoparticles were characterized by a variety of techniques like XRD, UV, FTIR, SEM and EDAX. Experimental data reveals that the final product almost consists of hexagonal flakes in the [111] plane. The estimated optical band gap for the particles was found to be within the reported range.

Introduction

Recently, nano structured and micro structured materials have attracted great attention in the research field because of their inbuilt chemical and physical properties and due to its numerous applications [1-6]. Cu_2Se compounds which is a well unknown p- type semiconductor has a wide range of applications in various areas of research such ad optical filters, super ionic conductors , window materials , thermal electric conductors and photo electric devices [7,8]. The band gap of $Cu_{2-x}Se$ nanostructure varies between 1.0 to1.4ev. Materials which are in this band range are well suited for solar energy conversion [7].

Several synthesis, techniques have been reported for copper selenide nanostructures. Some of the methods being sol-gel technique [9] chemical vapour deposition [10], electrochemical deposition method [11] and hydrothermal synthesis.

The hydrothermal technique is one of the most effective methods, in terms that it is simple and cost effective. By using this method the morphology and size of the nano particles are well controlled and they also produce highly crystalline and pure products.

In this paper, Cu_2Se nanoparticles were synthesized by hydrothermal method and their characteristic properties were analyzed and discussed.

Experimental

The precursors taken were copper sulphate pentahydrate ($CuSO_4.5H_2O$) and selenium dioxide (SiO_2). 7.5 gm (0.2 moles) of $CuSO_4.5H_2O$ has taken and stirred for 15 minutes to get homogeneous solution. 1.6 gm (0.1 moles) of Selenium dioxide was added into it, followed by 30 minutes stirring. Hydrazine hydrate was used as the reducing agent and 2 ml of N_2H_4 was added to the above solution. The solution was subjected to continuous stirring for 15 minutes.

The pH value of the solution was 3. The prepared solution was transferred into a stainless steel autoclave with a Teflon liner. The autoclave was filled with deionized water up to 80% of the total volume. The autoclave was sealed and heated at temperatures of 200° C for 24 h in an electric furnace. After heating, it was cooled down to room temperature naturally. The black product was collected by filtration, washed with deionized water and absolute ethanol, and then dried at 60°C.

Results and discussion

The powder x-ray diffraction pattern of the synthesized sample was recorded using powder x-ray diffraction system. X-ray diffraction was recorded for the sample by means of a slowly moving radiation detector in the range of 10° -70° where monochromatic wavelength of 1.5405Å (Cu) was used. The figure 1 shows the x-ray diffraction pattern of copper selenium nanocrystals.

The peaks of x-ray diffraction can be compared with the standard available data for the confirmation of the structure, with the use of JCPDS (Joint Committee on Powder Diffraction Standards) card no.88-2043. Thus in the observed pattern peak positions exhibit cubic structure of Cu_2Se with lattice constants of $a = b = c = 5.736$Å and $\alpha = \beta = \gamma = 90°$.

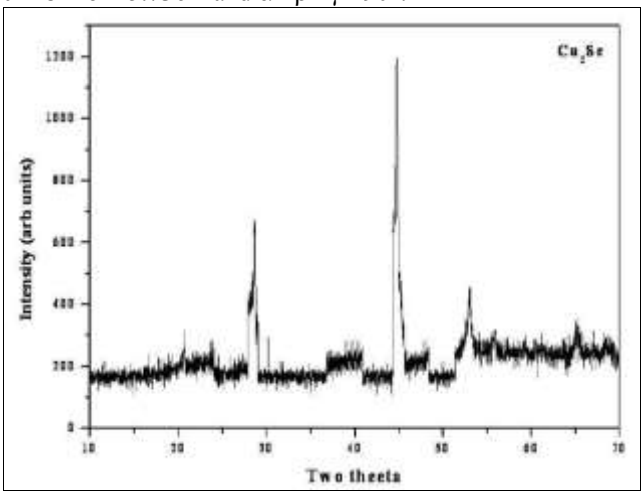

Figure 1: Typical X-Ray diffraction pattern of Cu_2Se powder.

The optical absorption of Cu_2Se nanoparticles are shown in Fig 2 .The absorption peak was found to be at 382.94 nm. The band gap energy thereby calculated was found to be 1.2eV.

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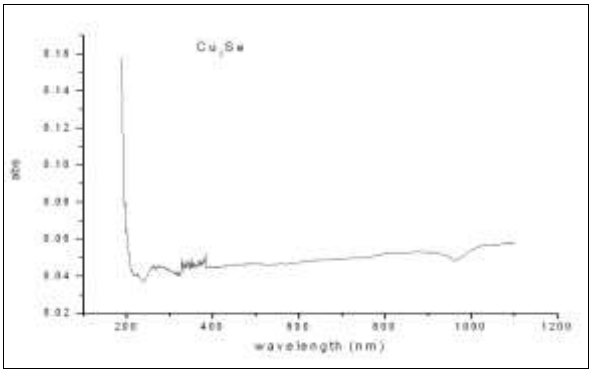


Figure 2: UV-Vis Spectrum of the nanoparticles

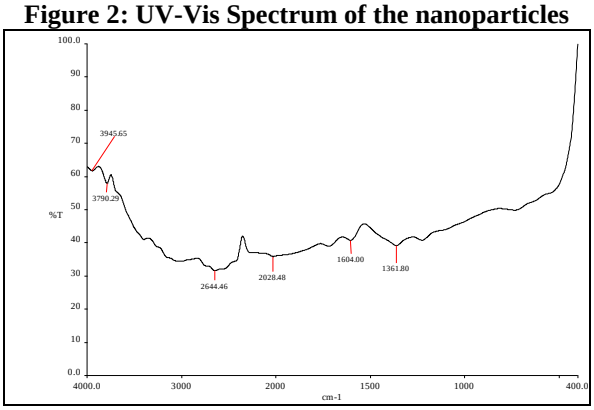
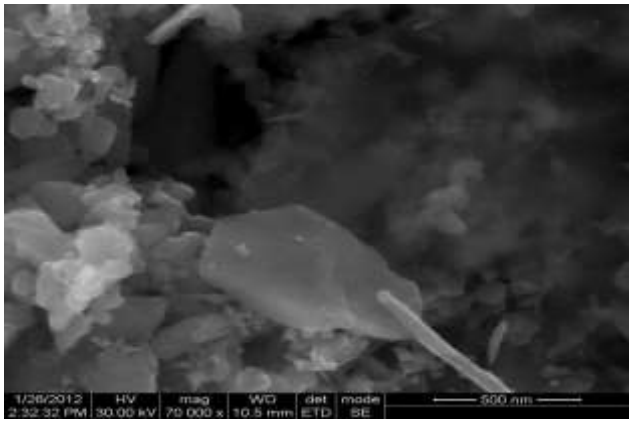


Figure 3 : FTIR spectrum of Cu_2Se sample prepared by hydrothermal method

Fourier transform infrared (FTIR) spectroscopy was employed to further characterize the as-synthesized Cu_2Se nanoparticles. The peak at 1604.00 cm^{-1} may be attributed to the presence of Cu^{2+} ion in the present system. The peaks at 1361.80 cm^{-1} are assigned to the O-H characteristic vibrations resulting from small quantity of H_2O in the sample. The peak at around 3100 cm^{-1} may corresponds to N-H stretching vibration band which can be attributed to the interaction of N_2H_4 with copper ions.

The structural morphology analysis was carried out by SEM. The figure 4 shows hexagonal flake –like morphology. On a higher resolution is seen the formation of many small particles and flakes which are possibly formed by the interface nucleation at the low crystal nuclear growth rate. Energy dispersive analysis of X-rays (EDAX) confirmed the presence of Copper (45.07%) and Selenium (32.82%) in the sample. The morphology can be further controlled by adjusting the reaction temperature or by the use of surfactants.



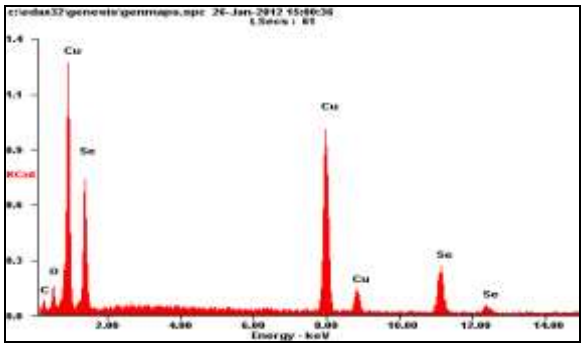


Figure 4 : SEM and EDAX images of the copper selenide powder obtained.

Conclusion

Copper selenide nanopowders have been synthesized by low cost and effective hydrothermal method. XRD and SEM analysis confirms that the synthesized particles to be of the cubic structure exhibited a hexagonal flake –like morphology. A UV and FTIR study informs us about the optical properties of Cu_2Se nanoparticles. EDAX reveals the composition of the synthesized material.

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