



Research Paper

Cadmium Sulphide (CdS) Quantum Particles: Synthesis, Characterization and Properties

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Abstract-Yellow colored CdS quantum particles were prepared by template synthesis method. The particles are passivated by thiophenol as template was confirmed by FTIR technique. HRTEM image showed that the wurtzite polycrystalline nanoparticles have size of the order of 15-50 nm. The compound nano-semiconducting sample was subjected to VSM and Impedance measurements to reveal the magnetic and electrical nature of the sample respectively. The properties varied significantly from that of bulk grain sized sample.

Keywords: CdS quantum particles, template, VSM etc.

Introduction

In this materials age nano-semiconductors play a vital role because of their relevance to the development of miniature technology. Their specific behavior to external constraints, like magnetic field and electric field finds applications in many devices ^[1]. Since the discovery of germanium transistor ^[2], research on germanium and silicon has revolutionized the electronic industry. But the demand for new materials with flexible properties for higher efficiencies, compactness and low cost was realized recently ^[3]. Many important properties of this sample have not been studied so far systematically, especially electrical conductivity and the scattering mechanisms in this sample are not yet established. In this present investigation, CdS nanoparticles capped with thiophenol have been synthesized and the structural, magnetic and electrical properties of this sample were studied.

Material and Methods

1g of respective precursors were suspended in 100 ml of 0.1M aqueous solution of cadmium acetate, thiophenol and sodium sulphide, were prepared and equal volume of each solution was used for the reaction. First, the cadmium acetate solution was taken in a reaction vessel and thiophenol was added to it drop wise while stirring it continuously with magnetic stirrer, then sodium sulphide solution was added to same reaction vessel. Solutions were mixed at the rate of 1ml per minute. The end product was washed thoroughly in double distilled water to remove the by-product, sodium acetate salt. Finally the solution was centrifuged and the precipitate obtained was dried at room

temperature. By properly adjusting the concentration of the starting reagents, free-standing powder of CdS quantum dots can be synthesized. The as-prepared yellow color sample was calcined at 300^oC and subjected to different characterization techniques.

The compositions of the sample was estimated using Energy Dispersive Analysis of X-rays (EDAX) attached to Scanning Electron Microscope (SEM, Model: JSM 840A). The role of capping agent was confirmed by IR spectroscopy (Model: MIR 8000TM). Particle size and structure were obtained from High Resolution Transmission electron microscopy (HRTEM, Model: JEOL JEM 3010). Magnetic study was performed on Vibrating Sample Magnetometer (VSM, Model: PAR 4500) and impedance plot was recorded on an Impedance Analyzer (Model: Solatron SI 1260).

Results and Discussion

Elemental analyses and HRTEM

The atomic percentage of the chemical species found in the sample was given in Table 1. The concentrations in the sample agreed well with nominal concentrations. SEM image of CdS is shown in Figure 1. The rough and spongy surface morphology has been clearly seen for the sample, which make it difficult to estimate the crystallite size. In general the powders were isolated as agglomerates of primary crystallites with dimensions of approximately a few micrometers. These agglomerates in turn, are composed of the primary 15-50 nm sized crystallites as evident from HRTEM. Fine particles, particularly nanoscale particles, since they have large surface areas, often agglomerate to

form either lumps or secondary particles to minimize the total surface or interfacial energy of the system^[4]. Figure 2 shows HRTEM micrograph of the CdS sample where (a), (b) and (c) show selected-area electron diffraction (SAED) pattern, lattice fringes in CdS and bright-field image of CdS respectively. The size distribution in the bright-field image was measured by manual counting from a population of about 50 particles. The average particle size measured is close to 50 nm. Besides, coarse particles of a few hundred nm in diameter were also occasionally observed. Further, the quasi-spherical shape is evident. Also, the presence of a 2 to 5 nm thick layer on surface of the particles has been observed. This belongs to the thiophenol group as is evident from FTIR studies. The rings in SAED pattern confirms all of the 'd' spacings of CdS. The three brighter dot-rings indicated by arrows correspond to the (101), (110) and (103) diffractions of wurtzite structure of CdS. Further, the SAED pattern shows that diffraction fringes are diffraction spots and not diffraction rings for the nanoparticles. This confirms the good crystallinity of the present CdS nanoparticles. The lattice fringes of CdS reveal that the CdS nanoparticle cores are polycrystalline in nature. The observed lattice fringe spacing of 0.375 nm corresponds to the 'd' value of (100) plane of CdS^[5].

Table 1: Elemental analysis for CdS nanoparticles

Starting elements in at. %		Elements from EDAX analysis in at. %	
Cd	S	Cd	S
50	50	47.23	52.77

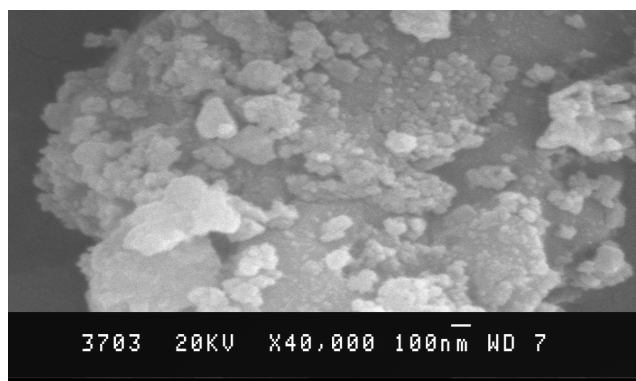


Figure 1: SEM image of CdS Nanoparticles

Infrared spectroscopy

FTIR spectrum of CdS is shown in Figure 3. In the higher energy region the peak at 3429 cm⁻¹ is assigned to O-H stretching of absorbed water on the surface of CdS. Again, presence of water is confirmed by its bending vibration at 1627 cm⁻¹. The peak at 1440 cm⁻¹ is assigned to bending vibration of methanol used in the process. It is also verified by its CH₃-stretching vibrations occurring as very weak peaks just below 3000 cm⁻¹. The C-O stretching vibration of absorbed methanol gives its intense peak at 1117 cm⁻¹. In addition to surface coverage of CdS by methanol, presence of trace amounts of template ligand namely thiophenol is also partly evident from this spectrum.

Its ring C-H vibration occurs at about 3000 cm⁻¹, it is a very weak peak. Similar such weak peaks due to C-H bending vibrations are observed at about 617 cm⁻¹. Hence, in addition to absorbed methanol on the surface of CdS, presence of thiophenol in its dissociation form is also evident. This observation therefore suggests that control of size of CdS is dominated by both methanol and thiophenol, hence even after sintering presence of such compounds is clearly evident. So these observations convincingly support template role of thiophenol in the control of the size of CdS particles.

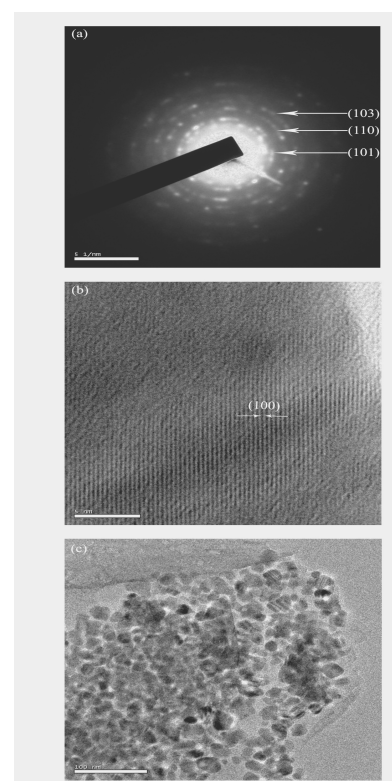


Figure 2: HRTEM micrograph of the CdS sample where (a) selected-area electron diffraction (SAED) pattern (b) lattice fringes in CdS (c) bright-field image of CdS

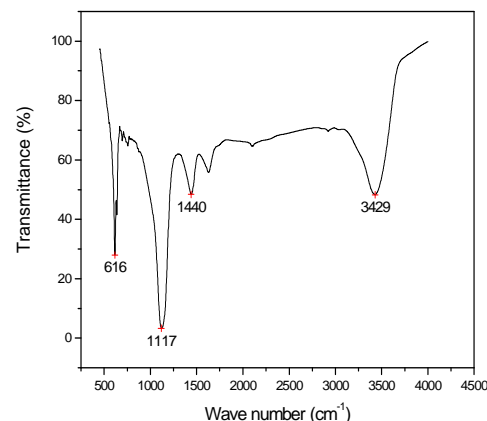


Figure 3: FTIR spectrum of CdS nanoparticles

Magnetization study

The magnetization curve for CdS sample as shown in Figure 4 is about a straight line passing through the origin. No change in magnetization was observed when the

field was reversed i.e. no hysteresis was noticed. This shows that the sample is in paramagnetic state. The nature of Paramagnetism in CdS might be attributed to super paramagnetic nature of the small grains present in the sample and also to spin canting i.e. surface pinning effect. When the size of the particles is reduced below the single domain limit, they exhibit super Para magnetism at room temperature followed by a spin-glass like transition at low temperature [6]. Super Para magnetism is essentially a result of non-interacting, thermally fluctuating nanoparticle moments where the volume dependent effective anisotropy energy of each particle is easily overcome by the thermal energy ($K_B T$). The effect of this is that the nanoparticles have a large moment with high saturation magnetization (as expected in Ferro magnets) but a non hysteretic M-H curve with zero remanance and coercivity.

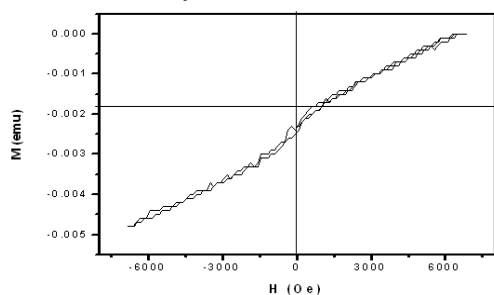


Figure 4: Magnetization versus Field (M vs. H) hysteresis loop

a.c Impedance study

The complex-plane impedance spectrum of CdS sample is shown in Figure 5. It can be seen that the spectrum consists of two overlapping semicircular arcs with the center depressed below the Z' axis. The intra-grain and the inter-grain polarization effects are observed via the semicircular arc in the complex impedance plane. The overall impedance behavior appears to be mainly the outcome of the contribution made by the grain boundaries. The impedance behavior is explained with the equivalent circuit shown in Fig. 6. The parameters R_g , C_g and $(\omega_p)_g$ denote the resistance, capacitance and the relaxation frequency ($1/\tau$) of the grain and R_{gb} , C_{gb} and $(\omega_p)_{gb}$ are the corresponding quantities for the grain boundaries. These parameters are obtained for room temperature measurement by analyzing the data using the non-linear least-squares (NLLS) fitting routine. The depression observed in the semicircle indicates the non-Debye nature of the material, since a depressed semicircle with centre below the real axis is related to the distribution of relaxation time among more than one relaxation mechanism. The calculated resistivity of this

sample is $5.064 \text{ M } \Omega\text{-cm}$, which is very much higher than the bulk or coarse grained CdS ($0.12 \text{ } \Omega\text{-cm}$ at room temperature). The electrical resistivity of the nanocrystalline materials is expected to be higher than that of coarse grained polycrystalline materials, because of the increased volume fraction of atoms lying in the grain boundaries. Besides, if the crystallite size is smaller than the electron mean free path, the grain boundary scattering dominates and thus its electrical resistance is expected to increase.

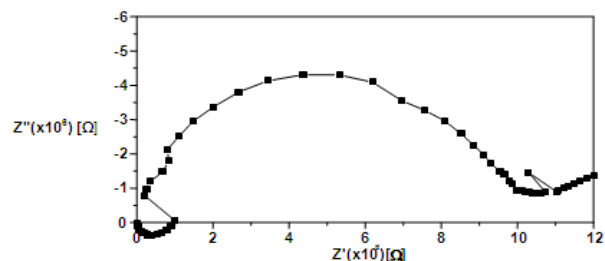


Figure 5: The complex impedance spectrum of CdS

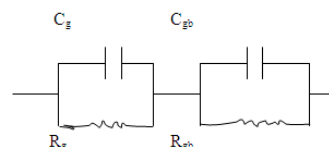


Figure 6: The equivalent circuit of impedance spectrum

Conclusion

CdS nanocrystalline sample was prepared by chemical route using thiophenol as template. The Presence of template on CdS nanoparticle was confirmed by FTIR technique. HRTEM study revealed the wurtzite structure of the sample and the particle size of 15-50 nm. VSM measurements depicted the super Para magnetic state of the sample. Finally a.c impedance study disclosed the insulating nature of the sample.

References

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