

Electrical Measurements on Cadmium Selenite Nano Composites

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ABSTRACT

By simple microwave irradiated solvothermal process using microwave oven Cadmium Selenite (CdSeO₃) nanoparticles were prepared. Cadmium acetate and Sodium selenite were used as precursors in the molar ratio 1:3. The powder XRD result shows that particles are purely crystallized in hexagonal phase with the broadening of diffraction peaks attributed to nanoscale size of the particles. The dielectric measurements were carried in two frequencies for temperatures ranging from 40 to 150oC. The present study indicates that the polarization mechanism in the nano particles considered is mainly contributed by space charge polarization. It can be understood that the space charge contribution plays an important role in the charge transport and polarizability in all the systems considered in the present study. The dielectric constant, dielectric loss, AC conductivity and DC conductivity increases with increase in temperature.

Key Words: Nanoparticles, Microwave, AC conductivity, DC conductivity, Dielectric constant, Dielectric loss

INTRODUCTION

Semiconductor nano crystals have attracted impressive attention, because of their novel optical and electronic properties [1]. Varying the size of the particle and changes in the degree of confinement of the electron and affects the electronic structure of the solid in particular band edges, which are tunable with particle size. One of the most important II - VI group semiconductors and nano crystalline Wurtzite structured Cadmium Selenide has attracted great interest in their various promising optoelectronic applications owing to its excellent optical conductivity such as photoelectron chemicals, photo-conductors, thin film transistor [2-4]. Blue shift in the band gap of this material, with decreasing grain size has led to many applied investigations. Many strategies have been utilized to prepare 1-D nano structural materials, where wet chemical method is considered as a practical and effective method for the synthesis of 1-D nano materials because it is more convenient and facile to be compared with most physical methods and need little expensive equipment by which CdSe nanorods, nanowires and nano tubes have been prepared successfully.

CdSe has been considered in many applications such as optoelectronic devices [5], light sensors [6], biological labels [7], chemical libraries [8], etc. The nanopowder of CdSe provides excellent and unique properties which depend upon the shape and size of the nanostructures [8-12]. Various methods such as hydrothermal, sol–gel approach, surfactant-assisted approach, etc. had been utilized for the synthesis of nanoparticles [13,14]. Synthesis, structural, and optical properties of CdSe nanoparticles have been reported [15]. In the present study, the main focus is on the electrical properties of pellets of nanoparticles of CdSeO₃ at different temperatures. The temperature dependence of dielectric constant, dielectric loss ac conductivity and dc conductivity was also investigated.

Experimental

The chemicals such as cadmium acetate dehydrate and sodium selinite were used as precursor materials to prepare CdSeO₃ nano particles. Cadmium acetate dihydrate and sodium selinite in 1:3 ratio were dissolved in Ethylene glycol solvent and water and stirred well using a magnetic stirrer and this solution mixture is kept in a microwave oven till the solvent evaporates completely, then the colloidal precipitate

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is filtered and washed by water and acetone several times. The precipitate thus formed is collected and dried.

AC conductivity measurements

The prepared cadmium selenite samples were palletized using a hydraulic press (with a pressure of about 5 tons) and used for the AC electrical measurements. The flat surfaces of the cylindrical pellets were coated with good quality graphite to obtain a good conductive surface layer. Using a traveling microscope the dimensions of the pellets were measured. The capacitance (Cc) and the dielectric loss factor (tan δ) were measured using the conventional parallel plate capacitor method [16-19] using an LCR meter (APLAB MODEL 4912) for all the samples with two frequencies 100 Hz and 1 kHz at various temperatures in the range $30 - 150^{\circ}$ C. The observations were made while cooling the sample. The temperature was controlled to an accuracy of $\pm 1^{\circ}$ C. Air capacitance (Ca) was also measured for the thickness equal to that of the pellet. The area of the pellet in contact with the electrode was same as that of the electrode. The air capacitance was measured only at room temperature because the variation of air capacitance with temperature was found to be negligible. The dielectric constant of the pellet sample was calculated using the relation,

$$\varepsilon_r = Cc / Ca.$$

where C_e is the capacitance of the crystal and C_a is the capacitance of the air medium of the same dimension as the crystal. As the crystal area was smaller than the plate area of the cell, parallel capacitance of the portion of the cell not filled with the crystal was taken into account and, consequently, the above equation becomes

$$\varepsilon_{\rm r} = \left\{ \frac{C_{\rm crys} - C_{\rm air} \left(1 - A_{\rm crys} / A_{\rm air} \right)}{C_{\rm air}} \right\} \frac{(A_{\rm air})}{(A_{\rm crys})}$$

where C_{crys} is the capacitance with crystal (including air), C_{air} is the capacitance of air, A_{crys} is the area of the crystal touching the electrode and A_{air} is the area of the electrode. From these measurements, ε_r and tan δ (dielectric loss factor) are available for the evaluation of AC conductivity.

The AC electrical conductivity (σ_{ac}) was calculated using the relation,

$$\sigma_{ac} = \varepsilon_0 \varepsilon_r \omega \tan \delta.$$

Here, ε_0 is the permittivity of free space (8.85 x 10⁻¹² C² N⁻¹ m⁻²) and ω is the angular frequency ($\omega = 2\pi f$, where f is the frequency).

DC Conductivity Measurements

The DC electrical conductivity measurements were carried out to an accuracy of $\pm 3\%$ using the conventional two-probe (parallel plate capacitor) technique at various temperatures in the range 40–150 °C [20-22]. The sample was prepared as done for the AC conductivity measurement. The resistance of the sample was measured using a million megohumeter. The observations were made while cooling the sample. Temperature was controlled to an accuracy of ± 1 °C. The dimensions of the sample were measured using a traveling microscope (least count = 0.001 cm). The DC conductivity, σ_{dc} , of the crystal was calculated using the relation

$$\sigma_{dc} = d/(RA)$$

where R is the measured resistance, d is the thickness of the sample, and A is the area of the face in contact with the electrode.

RESULTS AND DISCUSSION

Powder XRD analysis

The powder XRD pattern for the as prepared sample was done using Bruker AXS D8 Advance Diffractometer with monochromatic Cu (λ =1.5406 A⁰) radiation. The XRD pattern of as prepared CdSe nanoparticles are shown in Fig.1. The diffraction peaks indicates the nanocrystalline nature. The intensity of the peaks shows that the CdSe nanoparticles are highly crystalline. The diffracted peaks (100),(002), (101),(102),(110), (103), (112),(004), (202), (210), (211), (105) and (300)are corresponding to Hexagonal phase with Wurtzite structure which are in very good agreement with hexagonal (P63mc) structure in (Join Committee on Powder Diffraction Standards) JCPDS CAS No. 08-0459. The corresponding lattice constants are a = 4.299 Å and c =7.010 Å. The size of the nanocrystallites was estimated using the Debye-Scherrer formula (Sagirani et al. 2015)

$$2d = \frac{0.9\lambda}{\beta \cos\theta}$$

where, A is coherence length, β is the full-widths-at-half maximum (FWHM) of the diffraction peak, λ (1.5418 Å) is the wavelength of X-ray radiation, and θ is the angle of diffraction. From different θ values, the calculated average particle size is about 28 nm.



Figure 1: Powder XRD patterns of cadmium selenite nanoparticle.

The dielectric parameters viz. ϵ_r , tan δ and σ_{ac} observed are shown in Figures 2-4. All the parameters increase with increase in temperature. The dielectric constant is attributed to four types of polarization which are space charge, dipolar, ionic and electronic [23]. At lower frequencies at which all four types of polarizations contribute, the rapid increase in dielectric constant is mainly due to space charge and dielectric polarizations, which are strongly temperature dependent [24]. In the case of space charge polarization which is due to the accumulation of charges at the grain boundary, an increase in polarization results as more and more charges accumulate at the grain boundary with the increase in temperature. Beyond a certain temperature, the charges acquire adequate thermal energy to overcome the resistive barrier at the grain boundary and conduction takes place resulting in decreasing of polarization. This interfacial polarization occurs up to frequency of 1 kHz with possibly some contribution from the dipolar polarization also as the temperature increases. The grain size observed for the system considered in the present study is significantly small (not more than 26 nm). So, it can be understood that the polarization mechanism is mainly contributed by the space charge polarization[25-29].



Figure 2: Variation of dielectric constant with temperature.

The observed DC electrical conductivities of cadmium nanocomposite are shown in Fig 5. Nanoparticles lie between the infinite solid state and molecule. The electrical resistivity of nanocrystalline material is higher than that of both conventional coarse grained polycrystalline material alloys. The magnitude of electrical resistivity and hence the conductivity in composites can be changed by altering the size of the electrically conducting component. The σ_{ac} values observed in the present study are very small. When the grain size is smaller than the electron mean free path, grain boundary scattering dominates and hence electrical resistivity is increased [30]. Thus the space charge contribution plays an important role in the charge transport process and polarizability for the system considered in the present study.



Figure 3: Variation of dielectric loss with temperature.



Figure 4: Variation of AC conductivity with temperature.



Figure 5: Variation of DC conductivity with temperature.

CONCLUSION

Semiconductor nanoparticles of cadmium selenite were successfully prepared by simple microwave assisted solvothermal process. The powder XRD result shows that particles are purely crystallized in hexagonal phase with the broadening of diffraction peaks attributed to nanoscale size of the particles. The dielectric parameters, viz. dielectric constant (ε_r), dielectric loss (tan δ) and AC electrical conductivity (σ_{ac}) and DC conductivity (σ_{dc}) measurements were also carried out at various temperatures in the range 40-150°C and these electrical parameters are found to increase with the increase in temperature.

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