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RESEARCH ARTICLE

STUDIES ON $CdSeO_3$ NANOPARTICLES SYNTHESIZED BY MICROWAVE ASSISTED SOLVOTHERMAL PROCESS

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ABSTRACT

Cadmium Selenite ($CdSeO_3$) nanoparticles were prepared by simple microwave irradiated solvothermal process using a domestic microwave oven with frequency 2.45 GHz. Cadmium acetate and Sodium selenite were used as precursors in the molar ratio 1:2. 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 150°C. 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.

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INTRODUCTION

The semiconductor nanoparticles belong to the state of matter in transition between molecules and bulk solids in which the relevant physical dimensions changes on the length of a few to a few hundred nanometers. Both equilibrium and dynamic properties of nanomaterials can be very different from those of their corresponding bulk materials or isolated atoms and molecules. The dielectric constant of a semiconductor is one among its most important properties. Its magnitude and temperature dependence are significant in both fundamental and technological considerations. Recently, many extensive studies are going on in the semiconductor nanocrystals because they exhibit strong size dependent optical properties. These will be the key structural parameters in the fabrication of novel electronic nanodevices and nanocircuits. Semiconductor particles exhibit size dependent properties such as the scaling of the energy gap and corresponding change in the optical properties. CdSe is one of such materials, shows strong fluorescence which can be tuned according to the particle size.
CdSe has been considered in many applications such as optoelectronic devices (Nazzal *et al.,* 2003), light sensors (Bruchez *et al.,*1998), biological labels (Colvin *et al.,*1994), chemical libraries (Gaponik *et al.,*2002), etc.

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The nanopowder of CdSe provides excellent and unique properties which depend upon the shape and size of the nanostructures (Haram *et al.,* 2001; Wang *et al.,* 2004; Datta and Das 1990; Peng *et al.,* 2000). Various methods such as hydrothermal, sol–gel approach, surfactant-assisted approach, etc. had been utilized for the synthesis of nanoparticles (Tang *et al.,* 2003; Busbee *et al.,* 2003). Synthesis, structural, and optical properties of CdSe nanoparticles have been reported (Dwivedi *et al.,* 2011). In the present study, the main focus is on the electrical properties of pellets of nanoparticles of $CdSeO_3$ at different temperatures. The temperature dependence of dielectric constant, dielectric loss ac conductivity and dc conductivity was also investigated.

Experimental

Cadmium Selenite nanoparticles were synthesized by microwave assisted solvothermal method. The chemicals such as cadmium acetate dihydrate and sodium selinite were used as precursor materials to prepare $CdSeO_3$ nano particles. Cadmium acetate dihydrate and sodium selinite in 1:2 ratio were dissolved in Ethylene glycol solvent and stirred well using a magnetic stirrer for 30 minutes and this solution mixture is kept in a microwave oven for 45 minutes then the solution 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 (Joseph John *et al.,* 2007, 2008, 2011, 2016) 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°C. The observations were made while cooling the sample. The temperature was controlled to an accuracy of ±1°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,

$\varepsilon_r = Cc / Ca$.

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 (8.85×10^{-12} C^2 N^{-1} m^{-2}) and ω is the angular frequency (ω= 2πf, where f is the frequency).

DC Conductivity Measurements

The DC electrical conductivity measurements were carried out to an accuracy of ±3% using the conventional two-probe (parallel plate capacitor) technique at various temperatures in the range 30–150 ºC in a way similar to that followed by Mahadevan and his co-workers (Jayaprakash Manoharan *et al.,* 2012, 2012a, Sivakala *et al.,* 2015, 2016). The sample was prepared as done for the AC conductivity measurement. The resistance of the sample was measured using a million megohmmeter. The observations were made while cooling the sample. Temperature was controlled to an accuracy of ±1 ºC. 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 A^0) 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 (JoinCommittee on Powder Diffraction Standards) JCPDS CAS No. 08-0459. The corresponding lattice constants are

$\varepsilon_r = Cc / Ca$.

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).

where, λ 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 26 nm.

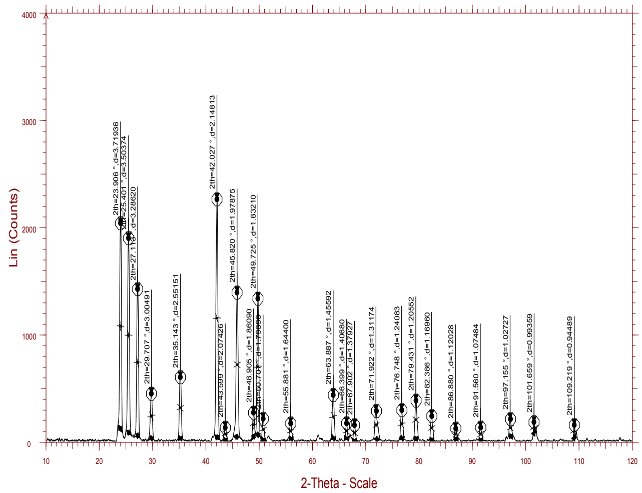

Fig. 1. Powder XRD patterns of $CdSeO_3$ nanoparticle

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 (Joseph John *et al.,* 2007). 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 (Joseph John *et al.,* 2008, Jayaprakash Manoharan *et al.,* 2011, 2011a). 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 (Sivakala P et al 2014, 2016). 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 (Ajin Sundar 2016c, 2016d, 2016e).

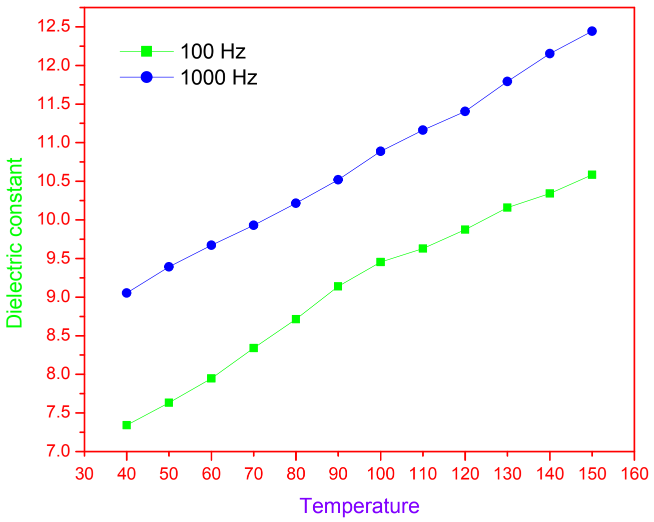

Fig. 2. Variation of dielectric constant with temperature

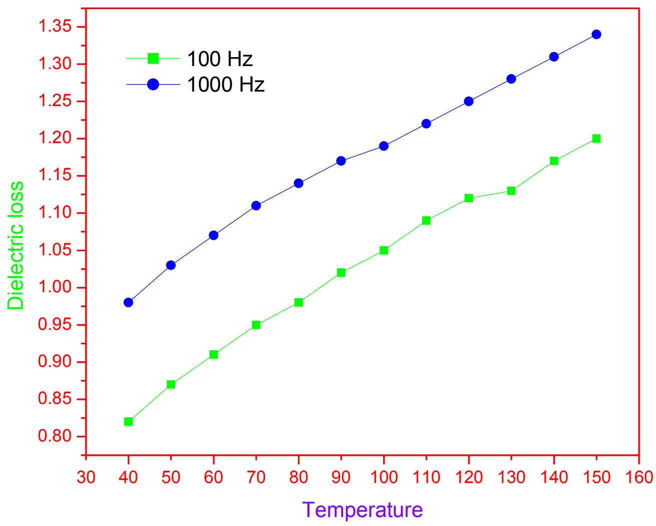

Fig. 3. Variation of dielectric loss with temperature

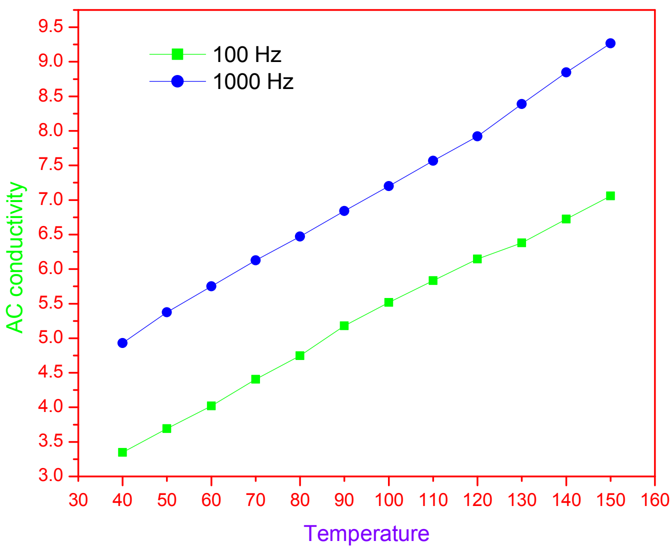

Fig. 4. Variation of AC conductivity with temperature

The observed DC electrical conductivities of cadmium nanocomposite are shown in Figure 9. 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 (Ajin Sundar *et al.,* 2016, 2016a, 2016b). Thus the space charge contribution plays an important role in the charge transport process and polarizability for the system considered in the present study.

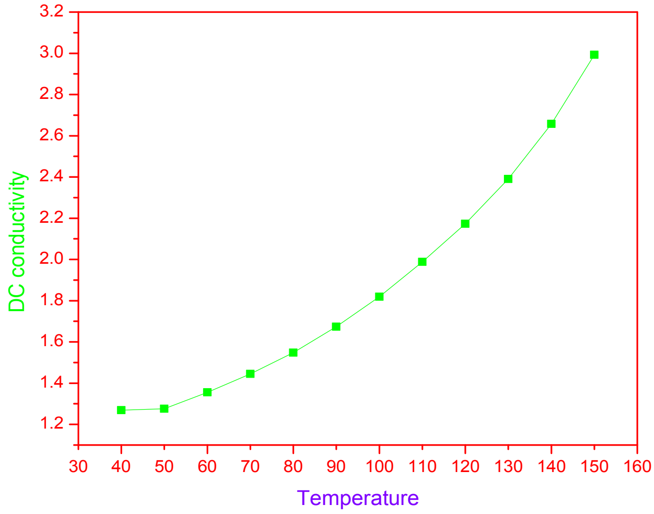

Fig. 5. Variation of DC conductivity with temperature

Conclusion

Semiconductor nanoparticles of $CdSeO_3$ are successfully prepared bysimple 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 40-150ºC and these electrical parameters are found to increase with the increase in temperature. Much work remains to be done, however, in developing reliable characterization methods that will aid in unambiguous structural identification as well as synthetic procedures that lead to relatively uniform products.

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