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A facile microwave synthesis of PbS:Sr nanoparticles and their key structural, morphological, optical, photoluminescence, dielectric and electrical studies for optoelectronics

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Abstract

Herein, various Strontium (Sr) concentrations doped Lead Sulfide (PbS) (PbS:Sr) nanoparticles (NPs) were synthesized and subjected to key parameters analysis. EDX/SEM mapping analysis confirms the presence of Sr and its harmonized allocation in the final product. Spherical NPs morphology was approved by field emission scanning electron microscopy (FE-SEM) analysis with tiny dimension grains formation. The grain size was observed to be approximately 55 nm; crystallite sizes calculated was found to be in the range of 18–21 nm. Slight shift in vibrational modes was noticed owing to low dimension NPs and approved a single phase. Diffused reflectance spectra were recorded, and direct energy gap was found in the range of 0.70–0.78 eV, which was much larger compared to the bulk (0.41 eV) one. PL emission spectra contained three emission peaks at 519 nm (broad) when excited at 450 nm. Also, the quenching in PL intensity was observed with Sr doping. The value of dielectric constant was noticed in the range of 25 to 57. The enhancement in total electrical conductivity was observed from $-11.67$ to $-7.83 \text{ S cm}^{-1}$ at 10 kHz; and from $-4.30$ to $3.42 \text{ S cm}^{-1}$ at 10 MHz. The enrichment of physical properties of Sr doped PbS signify its suitability in optoelectronics.

1. Introduction

In recent past, amongst the IV-VI group of binary chalcogenides, lead sulfide (PbS) is in lime light for research and development owing to its tremendous wide range applications in the field of solar cell, fiber optics, lasers, IR detectors, biological, as photocatalyst, antifungal etc PbS possess tiny energy gap i.e. 0.41 eV at 300 K and it can be varied up to 5 eV through particle size variation [1–10]. It also has good electrical and optical characteristics [11–13]. Owing to have high absorbing capacity of light from visible to IR region, quantum efficiency, large excitonic life time in PbS, makes it a model contender in solar device production. Also, it is utilisable in several needful devices like: visible light photo detector, photo-diodes, photo-transistors, sensor for humidity and temperature, optical switching etc [10, 14, 15]. The synthesis of PbS have attained through several techniques in different forms like: nanoparticles [16–18], nanosheets [19–21], nanorods [22], nanoribbons [23], nanocrystals [24, 25], quantum dots [26–28] etc. After going through the documented literature, author found that there is number anionic/cationic dopants has been tried with PbS and its key physical characteristics were significantly improved/modified [29–34].

In the past, Strontium (Sr) have been used as a doping agent to improve the photocatalytic, electrical, magnetic characteristics of ZnO, BiFeO3, CdS [35–38] and decrease resistivity of Cu2O [39]. Recently, synthesis
and fabrication of Sr doped PbS nanoparticles (NPs), nanopowders and films have documented with enhanced antifungal, photocatalysis, optical transmission, energy gap etc. by Suganya et al. [10] and Yucel et al. [40], correspondingly. Suganya et al synthesized the 0, 5, 10 and 15% Sr doped PbS NPs through chemical sol-gel route using Pb(NO$_3$)$_2$, SC(NH$_2$)$_2$ and SrCl$_2$.6H$_2$O precursors. As per documented literature to the best of our knowledge, there is no report on the microwave synthesis process using 0, 0.5, 1, 2.5 and 5 wt% Sr doped PbS NPs. Microwave synthesis has its own merits compare to the conventional one which can prepare the nanoscale materials within a short period of time [41–47]. Hence, herein we report on the synthesis of homogeneous NPs of PbS:Sr (Sr = 0, 0.5, 1, 2.5 and 5 wt%) using a microwave route; characterized them using numerous instrumentation techniques like FE-SEM, EDX/SEM mapping, XRD, FT-Raman, DRS, and impedance analyzer and discussed hereafter.

2. Experimental procedure

2.1. Materials and synthesis

PbS: Sr synthesis has been carried out using lead nitrate [Pb(NO$_3$)$_2$], sodium sulfide (Na$_2$S), CTAB [C$_{16}$H$_{33}$BrN] and strontium nitrate [Sr(NO$_3$)$_2$]; procured from Alfa Aesar, Sigma Aldrich and Fisher Scientific Pvt. Ltd. Five well-cleaned beakers were taken, in each beakers 0.5 M Pb(NO$_3$)$_2$ (16.5605 g) was dissolved in 50 ml distilled water (DW) and referred as A1, A2, A3, A4, A5. In another five beakers 0.5 M Na$_2$S (3.902 g) was dissolved in 50 ml DW and referred as B1, B2, B3, B4 and B5. A constant amount of CTAB i.e. 10 g/100 ml was prepared and 100 ml from it was added to A1, A2, A3, A4, A5 solutions and stirred well for 15 min and then 0.0, 0.5, 1.0, 2.5, 5.0 wt% Sr(NO$_3$)$_2$ was added to it as Sr source under regular stirring. At the end, the prepared B1, B2, B3, B4 and B5 solutions were added slowly into their respective A1, A2, A3, A4, A5 solutions then a black precipitate was formed which indicates the formation of PbS. The whole process was carried out at constant temperature i.e. 60 °C and stirring continued at 400 rpm for another 1 h. At the end, all the solutions were transferred to 1L capacity cylindrical flasks and introduced to an indigenously tailored domestic microwave [48] for 10 min, irradiated at 700 W, finally cooled to ambient temperature. All the black precipitates were carefully washed, with DW and the powder samples were collected and dried in an oven at 90 °C for 24 h.

2.2. Characterization details

A JEOL JSM 7600 F/6360 LA, Japan, FESEM/EDX system was used to study the elemental confirmation, mapping and morphology of the final products. Shimadzu X-600 Japan, XRD and DXR THERMO SCIENTIFIC FT-RAMAN systems were employed to investigate structural and vibrational parameters. A UV-3600 Shimadzu UV–Vis–NIR, DRS was employed to estimate the energy gap. For dielectric and electrical analyses, a KEITHLEY 4200–SCS, Keithley a Tektronix company, USA was used on primed 0.2 mm thick circular pellets of all the synthesized materials.

3. Results and discussion

3.1. EDX/SEM mapping and morphological studies

Figures 1(a1), (b1) and (a2), (b2) represents the respective SEM mapping images and EDX spectra for pure and 5.0 wt% PbS: Sr, correspondingly. SEM mapping images recorded in 5 min of duration confirm that Pb (red) and S (green) is homogeneously distributed in pure PbS (figure a1), however in doped PbS samples Sr is homogeneously distributed throughout the sample and Pb, S and Sr are mapped with red, blue and green colors, respectively. The combinations of all the three colors can be seen in inset figure (figure b2). Therefore, it is confirmed a homogeneous doping of Sr in the sample. Furthermore, both pure and doped products contained Pb, S and Sr, correspondingly is shown in figures (a2), (b2). The concentration of Sr in 5.0 wt% PbS: Sr was found to be 2.72 (mass%) from the EDX analysis.

Figures 2(a)–(e) represents the surface morphology of the final products i.e. 0.0, 0.5, 1.0, 2.5, 5.0 wt% PbS: Sr captured by FE-SEM at two different resolutions. It is clearly visible from the following figures that all the prepared products are at nanometer ranges. The morphology looks like nanoparticles (NPs) of small dimensions in the form of clusters/agglomerated are seen. All the samples exhibited nanoparticles of similar morphology with low dimensions and the size of the nanoparticles are in range of 40 to 55 nm forming clusters or agglomerated structures. A slight increase in particle size can be observed in the FE-SEM images are due to the Sr; as Sr promoting the growth of NPs during the formation of PbS. The clusters contain a number of small dimension grain of sizes less than 20 nm however, a minute variation can also be seen in the higher magnification view (80,000 X) that is displayed adjacent to each images. The grain size of pure PbS (see figure 2(a), (a’)) is smaller as compared to the Sr doped samples. The grain size is in accordance to the crystallite
size estimated by XRD data. As a conclusion, based on the high-resolution FE-SEM images, the homogeneous PbS:Sr NPs synthesis can be achieved facilely, which will have applications in optoelectronic devices.

3.2. Structural analysis

XRD patterns for all the synthesized PbS:Sr NPs are exposed in figure 3(a) along with standard XRD from CIF #9013403. These patterns reveals that all the prepared PbS:Sr nanostructures are polycrystalline in nature and grown along its well-known planes which obey FCC cubic system as reported in the standard card no., JCPDS #03-065-0692 and 05-0592 [20, 49]. Additionally, the absence of any extra peaks owing to free of metallic Sr or its complexes approved the purity of the synthesized products. From the XRD patterns, it can be noticed that the PbS:Sr NPs are preferentially grown along (200) planes. Moreover, from the XRD plots it can be seen that the crystalline quality is not much affected by Sr doping in PbS. However, in the previous reports the decreased in crystalline quality/size was observed for PbS:Sr nanopowders and thin films [10, 40]. In their reports, they used different precursors and doping sources than the current one. Moreover, the lattice constant (a = b = c) and cell volume (V) were estimated form two prominent planes noticed in all products which are (111) and (200) using the relation:

\[
\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} \quad \text{and} \quad V = a^3
\]

here, d stand interplanar spacing and all estimated values are presented in Table 1; these values are in well agreement to the standard reports JCPDS #5-0592 [20] and 65-0692 [49]. The listed values showed that the lattice constant is increased with increasing Sr concentrations in PbS hence, cell volume (see figure 3(b)). The increased values of PbS:Sr was also reported earlier [10]. The expansion of these parameters gives a clear cut idea about the incorporation of Sr in PbS lattice or owing to the occurrence of intrinsic type of defects and stoichiometry loss, that may be discussed with the help of Vegard’s law [50, 51]. As per the above law, if the doping is taken place interstitially the cell parameters expands, and this is the case in the current work. Additionally, we can explain with the help of ionic radii, as the ionic radii for Sr$^{2+}$ (118 pm) is lower compare to the Pb$^{2+}$ (119 pm), both the interstitial and substitutional incorporation of Sr in PbS are possible [10, 40, 52]. All the diffraction peaks in XRD patterns were used to estimate the average values of crystallite size (L_{ave}), density of dislocations (\(\delta\)) and strain (\(\varepsilon\)) through well-known relations [46, 53]:

\[
L_{\text{ave}} = \frac{0.9\lambda}{\beta \cos \theta}, \quad \delta = \frac{1}{L_{\text{ave}}} \quad \text{and} \quad \varepsilon = \frac{\beta \cot \theta}{4},
\]

in respective way (here \(\lambda\) stand for x-ray wavelength, \(\beta\) for FWHM and \(\theta\) is angle), and listed in table 2. The listed
values revealed that the $L_{\text{ave}}$ value is increased from 18 to 22 when Sr doping increased to 5.0 wt%. These values are in correlation with grain size in FE-SEM. As $L_{\text{ave}}$ is increasing the value of $\delta$ and $\varepsilon$ are decreasing. The reduction in $\delta$ and $\varepsilon$ indicates low defect in PbS:Sr NPs.

**Figure 2.** FE-SEM images of pure PbS (a-a'), 0.5 (b-b'), 1.0 (c-c'), 2.5(d-d'), and 5.0 wt% (e-e') PbS:Sr NPs.
3.3. Vibrational spectroscopy

Presentation of measured Raman spectra for pure and Sr doped PbS NPs are shown in Figure 4. It is evident from the figure that the Raman intensity is constantly increased with increasing Sr doping. This enhancement might be owing to increase in crystallinity and size of the NPs as discussed in XRD and FE-SEM sections. There are about 5 Raman peaks for all the samples measured. These peaks are positioned at 59 ± 1, 69 ± 1, 129 ± 3, 273 ± 2, 429 ± 2 and 600 ± 3 cm⁻¹ which are assigned to phonon and combinatorial modes as TO, TO + LO, LO, 2LO, 3LO [20, 54–58]. Here, primary and secondary modes present in the synthesized PbS:Sr NPs are due to LO phonon modes at Γ Brillouin zone point. Raman modes situated at 429 ± 2 cm⁻¹ is related to LO phonon overtone at L Brillouin zone point in all NPs. All observed Raman modes in PbS:Sr samples are found to be red shifted when compared to the bulk PbS [20, 54–58]. The possible reasons behind the red shifts are small size used wavelength for Raman measurement and direction of NPs growth [48, 57, 58].

3.4. Optical analysis

For energy gap estimation the diffused reflectance spectra were recorded from UV to NIR region as depicted in figure 5(a). It is visible from the figure that the absorption edge wavelength is varying with varying the Sr doping in PbS and possess blue shift. The blue shift signifies the enhancement of energy gap. Also, there is a clear variation in the % of reflectance owing to Sr doping in PbS. Hence, the Kubelka-Munk theory is employed to estimate the energy gap (Eg) for all PbS:Sr NPs samples. The modified Tauc’s relation by Kubelka-Munk is
expressed as \([59, 60]\): 
\[
\left( \frac{Fh\nu}{t} \right)^{1/m} = B(h\nu - E_g)
\]
here all symbols are we recognized. As PbS is well known direct band gap material, so \(m = 1/2\) has been take in this case. The plot between \(\left( \frac{Fh\nu}{t} \right)^{1/2}\) and \(E\) (eV) has been drawn and exposed in figure 5(b). For determining the \(E_g\) values for all PbS:Sr samples, a simple straight line has been sketched towards energy axis, where \(\left( \frac{Fh\nu}{t} \right)^{1/2} = 0\). The observed \(E_g\) values are on range of 0.70 to 0.78 eV. Highest energy gap value was observed for 1.0 wt% Sr:PbS sample. There is a clear enhancement in \(E_g\) value with Sr doping in PbS, however all values including for pure and also \(\sim 2\) times larger compare to bulk value of PbS viz. 0.41 eV \([20, 22, 34]\). Such enhance provides a clear-cut idea about the presence of quantum confinement effect in grown NPs.

3.5. Photoluminescence analysis

PL excitation and emission -spectra were recorded for all the PbS:Sr samples by preparing their colloidal solutions in DMF. The measured PL excitation and emission spectra are displayed in figures 6(a) and (b) correspondingly. For recording the excitation spectrum, the NPs were emitted at 519 nm and for emission spectra the NPs were excite at 450 nm. It can be clearly seen from the following figures that the PL intensity for both excitation as well as emission are quenched by Sr doping in PbS. The reduction in PL intensity may be due to the fact that Sr ions are acting as electron sink which result in inhibition of electron-hole recombination, so very few/less electrons will be back to valence band and emit lower energy in form of light intensity. Figure 6(b) represents the PL emission spectra which contain three emission peaks at 519 nm (Major) and 556 nm and 702 nm (broad) \([61, 62]\). The PL emission intensity is systematically reduced with Sr doping concentration of all
bands, this indicates that the charge carriers are enhanced in PbS by Sr doping and photocatalytic performance may also enhance. Similar type of observations are also documented previously in I:Fe2O3 [63].

3.6. Dielectric permittivity and ac electrical conductivity studies
To investigate the effect of Sr doping concentration on dielectric and electrical activities of PbS, the capacitance (C), impedance (Z) and loss tangent (tanδ) values were recorded over a high frequency region. Using these parameters we have estimated the part of dielectric permittivity in real (ε′) and imaginary (ε′′) modes using the well-known equations: ε′ = C × 1/ε0 × A and ε′′ = tan δ × ε′, here, all the symbols like: t and A, ε0 are related to the thickness and area of the pellet and space permittivity, correspondingly. The calculated values of ε′ and ε′′, are plotted as a function of frequency in figures 7(a) and (b), correspondingly. The values of ε′ shows the enhancement with Sr doping and the highest value was noticed for 1.0 wt% PbS: Sr NPs, the value was noted in the range of 25 to 57. These values are much higher to the reported values for PbS nano and crystal [20, 64–66]. With 0.5 wt%, 2.5 wt% and 5.0 wt% PbS: Sr its value was reduced but still higher than that of the pure PbS NPs. Similar trend has been followed by ε′′ (figure 7(b)) with frequency as of ε′. At lower region of frequency, the value of ε′′ is higher; however, at higher range it is very low. This shows that the synthesized NPs of PbS may be applicable over higher frequency regions as they will possess fewer defects. The reason behind such changes that high/low values at high and low frequency regions could be understood from the previous reports [60, 67, 68]. It may also be mentioned here that the values of ε′′, are lower compared to the ε′′, similar type of behavior for ε′ and ε′′ values have reported earlier [69]. The ac electrical conductivity in total (σac,total) was studied using the relations: σac(total) = 1/2A and σac(total) = σdc(ω) + σac(ω → 0) [70, 71] and displayed in figure 7(c). It was found that the σac,total(ω) value is increased with increasing the applied frequency and also enhanced with Sr doping concentration in PbS. The largest value was noticed for 1.0 wt% PbS: Sr NPs; however, observed the similar trend as of ε′′. To recognize the conduction mechanism in the synthesized NPs of PbS: Sr and the effect of Sr, the Jonscher’s rule was employed [71]: σac = σdc + Bωs, here B, ω and s are known as constant, angular frequency and frequency exponent, respectively. The value of s was computed from the slope of ln σac,total versus ln ω in linear part of the curve. The noted values are plotted in figure 7(d) with standard error and revealed that the value of s < 1, which indicates the transport mechanism of charge is covered under correlated barrier hopping (CBH) [72, 73]. Moreover, to know and understand the exact nature of charge transport these studies must be carried out under varying temperatures by obtaining the values of s [73]. The variation in these values signifies the effect of Sr doping in PbS as well as orientation of grown nanoparticles.

4. Conclusion
Microwave synthesis process was employed successfully to achieve homogeneous shape and narrow size dimension NPs of pure and Sr doped PbS. The presence of Sr and its homogeneous distribution in the final product was confirmed by EDX/SEM mapping analysis. FE-SEM approved the spherical shape NPs formation in all the final products and their sizes are in the range of less than 55 nm. Sr doping effect on PbS was clearly observed in structural and vibrational properties. The Scherrer’s rule employed to estimate size of crystallites and noticed in the range of 20–35 nm. Vibrational modes are noticed to be shifted due to low dimension NPs and high intense Raman peak was observed in the case of 0.5 wt% PbS: Sr sample. The value of energy gap

Figure 6. PL spectra (a) excitation and (b) emission for the synthesized PbS: Sr NPs samples.
evaluated by Kubelka-Munk method was found to be in the range of 0.70 – 0.78 eV. The energy gap for the synthesized product is almost 2 times higher than the bulk PbS (i.e. 0.41 eV). Three emission peaks at 519 nm (major), 556 nm and 702 nm (broad) were observed and quenching in PL intensity occurred was observed in Sr doped samples. PL quenching might be owing to the reduction of defects/color centers. The dielectric constant loss and total electrical conductivity was studied and found to be enriched with Sr doping. The value of dielectric constant was found to be in the range of 25 to 57. The total electrical conductivity was enhanced from $-11.67$ to $-7.83$ S cm$^{-1}$ at 10 kHz and from $-4.30$ to $3.42$ S cm$^{-1}$ at 10 MHz. Results suggest that the physical properties of PbS:Sr are enriched compare to the pure PbS, hence may be a good candidate for optoelectronic applications.

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Compliance with ethical standards

Conflict of interest

None.

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