Synthesis of Lead Sulfide Nanoparticles by Chemical Precipitation Method

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Abstract. Lead sulfide (PbS) nanoparticles were prepared by chemical precipitation method (CPM) with the assistance of H_2S gas. The microstructure and morphology of the synthesized nanoparticles have been investigated using X-ray diffraction (XRD) and transmission electron microscopy (TEM). The XRD patterns of the PbS nanoparticles reveal formation of cubic phase. To investigate the quality of prepared nanoparticles, the particles size, lattice constant, strain, dislocation density etc. have been determined using XRD. TEM images reveal formation of cubic nanoparticles and the particle size determined from TEM images agree well with those from XRD.

1. Introducation

Lead sulphide(PbS) is an important IV-VI group semiconductor and bulk lead sulfide has cubic crystal structure and narrow direct band gap 0.41 eV at room temperature (300k) has a large excitonic Bohr radius about 18nm [1-2]. It has high dielectric constant and very high carrier mobility [3]. Hence PbS nanoparticles (NPs) show strong quantum size effects for relatively large size. Also, their absorption edge can be tuned to anywhere between red to violet covering the entire visible spectrum. PbS nanoparticles have been prepared by various techniques but they require sophisticated instruments. Keeping in mind the importance of PbS nanoparticles development of a simple and economic method of preparation is highly desirable. In the present work we have prepared PbS nanoparticles by a simple, time and cost effective method. To the best of knowledge of authors this method has not been reported earlier. The prepared nanoparticles have been characterized using x-ray diffraction (XRD), transmission electron microscopy (TEM) and selected area electron diffraction (SAED).

2. Experimental details

2.1 Synthesis

Lead nitrate $Pb(NO)_3.5H_2O$ procured from Merck India was used for synthesis of PbS nanoparticles. Lead nitrate was dissolved in double distilled water to make N/20 aqueous solution. NH_4OH solution was slowly added with constant stirring to obtain required pH value. Then H_2S gas was bubbled through the solution for a few seconds. The resulting black precipitate was collected and washed with distilled water many times and then centrifuged for fifteen minutes. The nanoparticles were dried by heating for half an hour at 373 K and then allowed to cool at room temperature. Finally we get black powder which is PbS. The PbS nanoparticles were obtained with pH value = 8. The samples were prepared at room temperature (308K). The as-prepared samples without any further processing were used for all the experiments.

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2.2. Characterization

XRD patterns were recorded using Burker D8 Advance X-ray diffractometer with Cu Kα radiation (λ =1.54 Å) operated at 40 kV, 100 mA with a step size of 0.1792°. TEM images were recorded with Philips CM 200 microscope operated at 200 kV.

3. Results and discussion

XRD patterns of PbS nanoparticles are presented in Figure 1. All the patterns show well defined but broad diffraction peaks indicating the formation of nanocrystalline materials. It can be seen that in case of PbS (Figure1) all the major crystalline planes of PbS are present. The interplanar distance d were determined by Bragg's law [4] given below

$$n\lambda = 2d \sin\theta$$

where n is the order of diffraction, λ is the wavelength of the incident x-ray, d is the interplanar distance, θ is the Bragg angle The peaks corresponding to (111), (200), (220), (311) and (222) planes of PbS with cubic phase are present. Further, no peaks other than those corresponding to PbS are observed, which indicates the high purity of synthesized nanoparticles. The lattice constants were estimated using the formula [4]

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2 +}}$$

Where a = lattice constant; h, k, l = miller indices

The interplanar distances as well as the lattice constants match well with the values reported in JCPDS Card No.65-0132 and can be attributed to cubic phase and confirm the formation of PbS nanoparticles.

The average crystallite size D was calculated by Scherrer formula [5] given below.

$$D = 0.9\lambda / B \cos\theta$$

where λ is the wavelength of x-ray used, B is the full width at half maximum (FWHM), θ is Bragg angle. The average crystalline size of PbS nanoparticle is estimated were found to be 25.6 nm.

The origin of strain is related to lattice "misfit" which depends upon the growing condition of nanoparticles [6]. The strain **ε** value have been calculated the following formula

$$\varepsilon = \beta \cot\theta / 4$$

Where θ = Bragg angle, β = Full width of half maxima

The calculated value of strain has been shown in Table 1. From this table the strain data indicate the strain values increases corresponding to pH value such type of change in the strain may be due to the predominant recrystallization process in nanoparticles.

The dislocation density is known as the length of dislocation lines per unit volume of the crystal. A dislocation is a crystallographic defect or irregularity in the crystal structure [7]. The presence of dislocation strongly influences many of the properties of materials.

Table 1. Particle size and lattice constant, dislocation density and strain of PbS nanoparticles prepared by chemical precipitation method with assistance of H_2S gas.

Sample name	2θ (degree)	d-Spacing (Å)		Lattice constant **a** (Å)		hkl	Dislocation density Lines/m^2	Strain
		Present study	Reported*	Present study	Reported*			
PbS	26.05	3.41	3.41	0.5921	0.59	111	0.0012	0.0039
	30.16	2.96	2.95	0.5924	0.59	200	0.0008	
	43.12	2.09	2.09	0.5931	0.59	220	0.0012	
	51.06	1.78	1.78	0.5930	0.59	311	0.0016	
	53.50	1.71	1.70	0.5930	0.59	222	0.0020	

* JCPDS Card No.65-0132

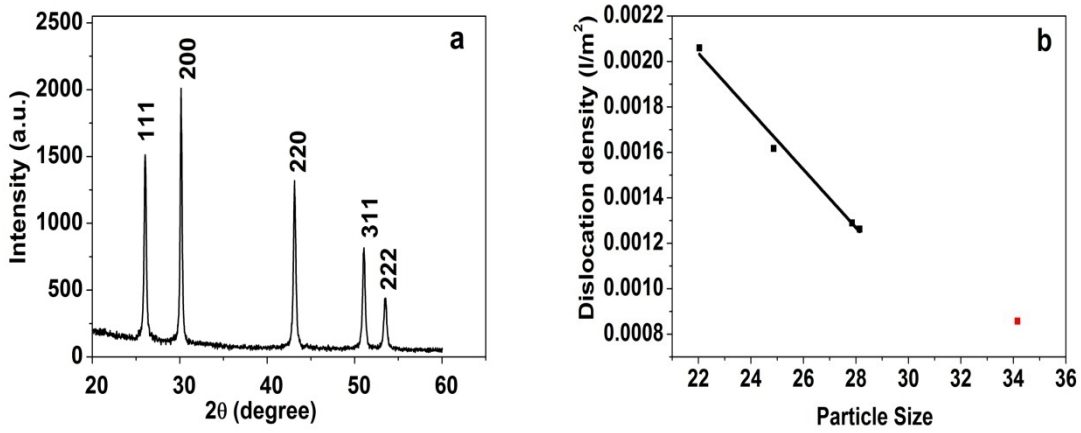


Figure 1.(a) XRD pattern and (b) Particles size Vs dislocation density of Lead sulfide nanoparticles prepared by chemical precipitation method

The X-ray line profile analysis has been used to determine the intrinsic stress and dislocation density [8]. The dislocation density (δ) in the sample has been determined using following expressions

$$\delta = 1/ D^{2}$$

where D is the size of the nanoparticle. Figure 1b shows the relation between particle size as obtained from each XRD peak and its corresponding dislocation density.

TEM image of PbS nanoparticles is presented in Figure 3. The images of PbS nanoparticles reveal formation of cubic structure. The size of PbS nanoparticles is found to between 29-49 nm. Furthermore, the selected area electron diffraction (SAED) patterns are also included in Figure 3. These SAED patterns index well to the cubic rock salt PbS crystal structure and diffraction rings match well with corresponding XRD patterns of the PbS nanoparticles. The SAED pattern shows concentric rings with bright spots, which indicates the nanocrystalline nature and good crystallinity of PbS NPs.

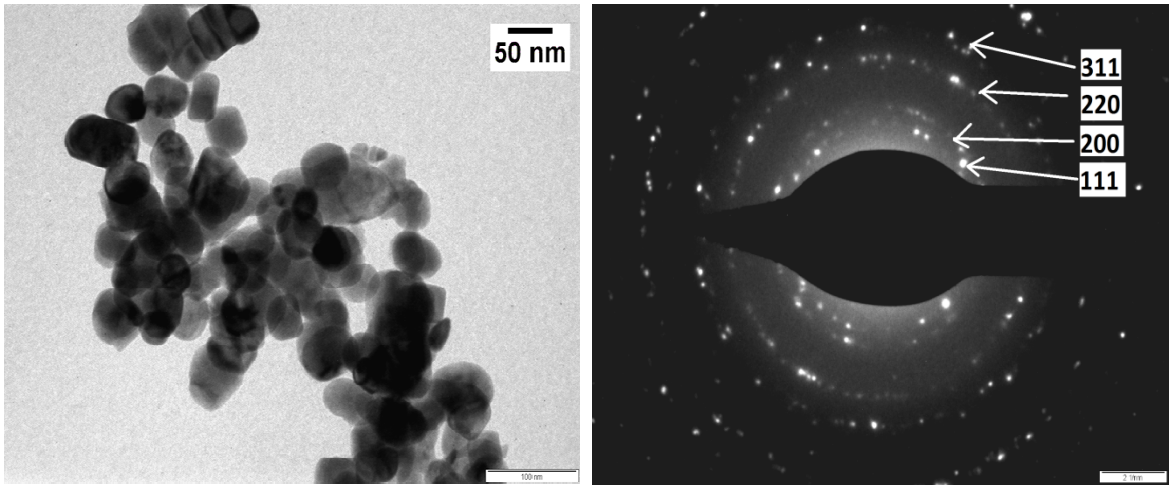


Figure 2. TEM image and SAED pattern of Lead sulfide nanoparticles

4. Conclusion

The present technique is a simple, cost effective as well as time effective method for producing good quality lead sulfide nanoparticles which is confirmed by XRD, TEM and SAED study.

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