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Effect of matrices on size and morphology of HgS nanoparticles

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ABSTRACT

Mercury sulfide (HgS) nanoparticles embedded in different matrices were synthesized. In the reaction, Mercury nitrate was used as Mercury source; thioacetamide was employed as chalcogenide source. This method has been used to achieve good control over composition and morphology of the products.and the influence of matrices on the dispersion of HgS products was briefly discussed. X-ray powder diffraction (XRD), Transmission electron microscopy (TEM) and scanning electron microscope (SEM) were used to characterize the size and morphology of obtained HgS nanoparticles. The results revealed uniform crystallite aggregates for HgS nanoparticles, with a narrow size distribution.

Key words: Transmission electron microscopy, nanoparticles, thioacetamide, mercury sulfides, semiconductor materials, sol–gel.

INTRODUCTION

Semiconductor nanostructures are promising candidates for the photonic and electronic applications [1,2]. Nanostructures overwhelm films and even bulk crystals in terms of luminescence efficiency. Also due to its large surface-to volume ratio (S/V ratio), surface-oriented luminescence phenomena characterize it from bulk crystals [3].

Optical properties of composite thin films consisting of nanometer-sized metal or semiconductor particles dispersed in solid dielectric materials such as polymers or glass have been of increasing interest for both fundamental and practical reasons, largely because of their novel applications as photonics and electronics devices based on quantum size effects. It is well known that the properties of such composite films are dependent on their microstructure, e.g. size, shape, composition, and also spatial distribution of the particles in the film [4-8]. Therefore, in order to describe the relationship between optical properties and microstructure of the composite thin films, it is necessary to obtain the composite films with well-defined microstructure. Recently, various techniques to fabricate such composite thin films, especially polymeric thin films containing nanoparticles, have been proposed and their optical and electrical properties have been studied by many researchers [9-14].

As one of the most important chalcogenides, HgS is a semiconductor that has a variety of potential applications, such as photoconductors, photovoltaic cells, near-IR photon detectors, and solar-selective coating [15]. Great efforts have been devoted to the synthesis of HgS nanoparticles to enhance its performance in currently existing applications in photography and luminescent devices [16].

In this paper, we report the preparation of HgS nanoparticles in different substrate. For the characterization of the obtained composite films, transmission electron microscopy (TEM) and X-ray powder diffraction (XRD) were used.It was shown that different substrate play essential role on the morphology and mean size of HgS nanoparticles.

MATERIALS AND METHODS

The chemical materials all were of analytical grade reagents: Mercury nitrate (Aldrich, 99%), Tetraethoxysilan (TEOS) (Fluka, 98%), mercaptopropyl trimethoxysilane (MPTMS) (Fluka, 97%), poly vinyl alcohol(PVA) (Fluka, 98%), poly vinyl pyrrolidine(PVP) (Fluka, 98%) thioacetamide (TA) (BDH, 98%), and methanol (BDH, 99%) were used as the starting ingredients.

Synthesis of HgS -SiO_2/ nanocomposite was carried out by sol-gel technique through the following steps:

A mixture of 9.6 mMol of Tetraethoxysilane (TEOS), 2ml of methanol, 26.4mmol of water and 3 drops HCl (0.1M) was prepared. After stirring the solution for 30 min at 30˚C, a mixture of 19.2 mMol of MPTMS with 13.9 mmol ethanol, 28.8 mmol water and 1.5 mmol of $Hg(NO_3)_2$ were added and stirred for 10 min .

In order to obtain HgS /SiO2 nanocomposite, 1.5 mmol of thioacetamide was added dropwise to the above cited solution and the mixture was vigorously stirred for 30 min at 60˚C. The filtrate was dried and heated at 80^{o}C for 24 h and calcined at a temperature of 550^{o}C for 6 h.

Synthesis HgS /PVA nanocomposite:In a typical preparation process, an appropriate amount of $Hg(NO_3)_2$ (ca.10% based on PVA weight) was dissolved into a 10% (by weight) homogeneous PVA distilled aqueous solution. This solution and Na_2S (20% excessive based on $Hg(NO_3)_2$ was put into a Teflon-liner autoclave of 80 ml capacity.

The autoclave was maintained at 100°C for 6 h, and then cooled to room temperature. The resulting viscous solution was cast on the glass plates and dried at room temperature for 12 h, and then dried at 80°C for 12 h for the complete removal of water. The films with thickness about 30 mm were peeled off from the plates.

Synthesis HgS /PVP nanocomposite**:**In a typical process, 0.05 g PVP was dissolved into 10 ml water, then 5 ml of 0.001 M aqueous $Hg(NO_3)_2$ solution was added to the solution under magnetic stirring. After 3 h, 5 ml 0.002 M aqueous thioacetamide was added to the solution and the mixture was stirred in dark for sulfuration reaction for 3 h. This reaction mixture was then constantly heated at 80 C for 8 h for the aging of HgS particles and then cooled to room temperature naturally.

The final products were obtained after being centrifuged and washed with water several times. Finally, they were dried less than 60 °C for 24 h for characterization later.
The morphology and structure of the HgS nano-sized powders were further investigated by transmission electron microscope (TEM), TEM (Philips EM208) were operated at 100 kV .SEM photographs for the nano-sized HgS particles were recorded (LEO 435) at 30 kV from samples covered with a gold thin film. To determine crystalline phases of the nanocomposite The Powder X-ray diffraction (XRD) pattern was recorded on a Seisert Argon 3003 PTC using nickel-filtered XD-3a $CuK\alpha$ radiations (λ=0.1542 nm).

RESULTS AND DISCUSSION

The X-ray phase analysis of the samples prepared is given in Fig(1) . The crystallite size is calculated from Scherrer equation i.e. $d = \lambda/\beta \cos(\Theta)$ Where b is the integral breadth of the diffraction peak, l is the wavelength of the incident X-ray and d is the volume weighted average crystallite size.

The interplannar spacing d obtained from the XRD peak are in good agreement corresponding to planes of the standard literature data of a-HgS . In pattern (a) PVA is well known as a crystalline polymer, and the diffraction peaks at 2Θ= from 11° till 40° correspond to the PVA crystalline phase. The crystalline nature of PVA results from the strong intermolecular interaction between PVA chains through the intermolecular hydrogen bonding.

It is noted that the relative intensities of the peaks differ from the standard pattern of the bulk material, which should be caused by preferred orientation and distribution of the HgS crystals on the substrate surface and the diverse morphologies of samples. From the patterns of sample (a&c), no peak of PVP and SiO2 was detected, suggesting that SiO2 was amorphous. The average crystallite size calculated using above formula is in PVA matrice 32nm , in PVP matrice 31 nm and in SiO_2 matrice is 14 nm.

The obtained product was further characterized by transmission electron microscopy (TEM). The TEM images of the as-prepared samples is shown in Fig(2). The size of the nanocrystals generated appears to be dependent to the matrices that have been used.

It was found that in silica matrice ,the distribuition and size of the HgS nanoparticles (Figure-3c) are more homogenous than the other matrices. While the morphology of HgS /PVP is cubic and HgS / PVA is hexagonal nanoparticles , HgS /SiO2 nanoparticles have mixtures of Nanospheres and hexagonal shapes.

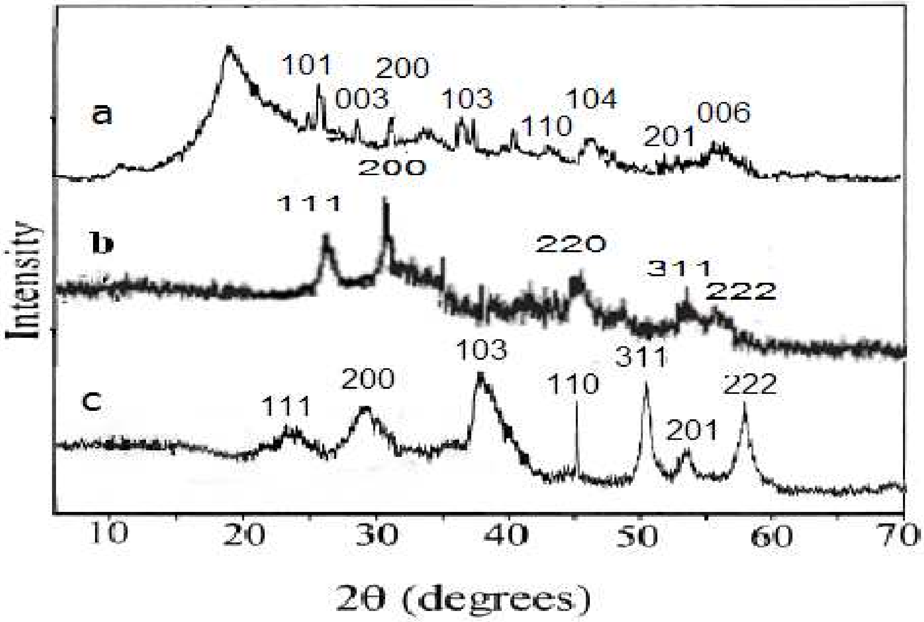

Fig. 1. XRD patterns of (a) HgS / PVA; (b) HgS PVP and (c) HgS /SiO2 nanocomposites

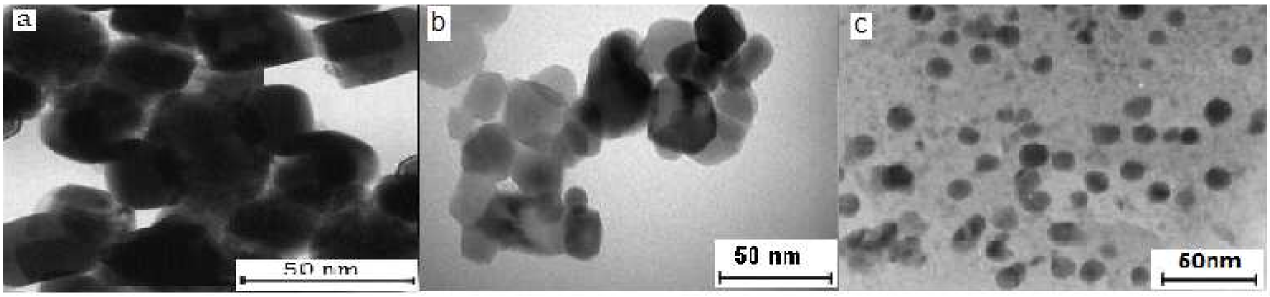

Fig.2 TEM images of (a) HgS / PVA; (b) HgS/ PVP and (c) HgS /SiO2 nanocomposites

CONCLUSION

HgS nanoparticles have been synthesized in PVP,PVA and SiO_2 matrices under mild conditions. Three different synthesize methods were used but in all the methods mercaptopropyl trimethoxy silane was used as capping agent. In PVA matrice HgS nanoparticles with smaller size and homogeneity were obtained, so It was mentioned thar silica matrice is more proper than PVP and PVA. Our preliminary investigations show that this general route can also be applied to the preparation of other chalcogenide nanocrystals.

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