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Nanocrystalline and monophasic thin films of metal chalcogenide (FeS, ZnS) and oxide (ZnO) by chemical bath deposition (CBD)

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Abstract

FeS, ZnS and ZnO nanostructured thin films have been deposited by Chemical Bath Deposition (CBD), a simple and cost effective technique. These materials with different morphologies such as nano-flakes, nanoparticles and nanorods for FeS, ZnS and ZnO, respectively were deposited onto the glass substrates. The crystallite sizes have been estimated from Scherrer's equation and were found as 14 nm for nano-flakes of FeS, 9.09 nm for nanoparticles of ZnS and 49 nm for nanorods of ZnO. The average values of the band gaps were found to be 1.97, 3.43 and 3.74 eV for FeS, ZnO and ZnS thin films, respectively. These nanostructured semiconducting materials have wide applications in advanced optoelectronic devices.

Keywords: CBD; FeS; ZnS; ZnO; nanostructures; morphology; thin films

1. Introduction

Metal chalcogenides (metal sulfides and oxides) when grown as thin films on various substrate exhibit novel properties which make them viable for potential applications in solar technology, optoelectronic devices and sensors ¹⁻¹¹. Growth and deposition of cost effective and non-toxic materials by any of the simple chemical technique have gained an enormous research interest owing to their applications in advanced photovoltaic devices ^{12, 13}. Nanostructures of metal chalcogenides especially, FeS, ZnS and ZnO are most important among all sulfides and oxides ^{2, 14, 15}. There are several physical and chemical techniques reported to date for the deposition of nanostructured thin films on variety of substrates including thermal evaporation ¹⁶, electro-deposition ¹⁷, hydrothermal method ^{18, 19}, sputtering ²⁰, chemical spray-pyrolysis ^{21, 22}, chemical bath deposition (CBD) ^{2, 23-26} and aerosol assisted chemical vapor deposition (AACVD) ^{27, 28}. Simple and cost effective nature of the CBD makes it the most attractive method of deposition among the techniques mentioned above. This technique can produce high quality nanostructured thin films and has the benefit of several features such as straightforward processing, large scale deposition and low temperature synthesis in contrast to the difficulties associated with conventional higher temperature and physical methods ²⁹. Nanostructured thin films of ZnS and ZnO have been widely studied but FeS is the least studied phase of iron sulfide. The reports published on CBD of FeS thin films mostly showed amorphous or multi-phases growth of iron sulfide. Kassim *et al.* in 2010 reported the deposition of iron sulfide thin films by CBD on ITO substrates and observed variation in structural properties as a function of bath temperature ²³. Most recently, authors have published the pioneer work on FeS by CBD and studied the structural and optical properties of thin films. Energy dispersive X-ray spectroscopy revealed the deposition FeS with iron to sulfur ratio close to 1:1 ²⁴.

ZnO is a material studied most via CBD regarding deposition of thin films and nanostructures. There are several studies presenting the effects of pertinent factors for achieving the favorable conditions of the synthesis process to achieve uniform growth of ZnO nanostructures especially, nanorods. The major factors studied to date are; substrates, seed layer thickness, temperature, concentration of precursors and most importantly the role of stabilizing agent ³⁰⁻³⁹. Shi and Walker reported recently that stirring produces smoother but less reproducible ZnO films than sonication-assisted CBD ⁴⁰. Rathore *et al.* presented a growth mechanism of polarity controlled ZnO nanorods array on a glass/FTO substrate by chemical bath deposition and found that anions of the Zn-salt affect the preferred orientation of growth ⁴¹. Lee *et al.* reported a novel approach for the synthesis of ZnO nanotube arrays (ring-like structures) using the polymer-based seeding solution through the Marangoni mechanism, guiding the growth of ZnO nanotubes with aqueous chemical bath deposition ⁴².

Deposition of uniform ZnS thin films by CBD is bit tricky compared to the deposition of CdS and ZnO by the same technique owing to the chemistry of reactions taking place to produce ZnS thin films by CBD. The role of complexing agent along with pH of the solution has crucial importance in deposition of oxygen free ZnS thin films by CBD ⁴³⁻⁴⁶. Optimized conditions for the growth of nanocrystalline ZnS thin films by CBD have been reported recently by the authors ⁴⁷. Herein, we report a simple, cost effective and reproducible method to deposit monophasic and nanocrystalline thin films of FeS, ZnS and ZnO on glass substrates without seed layers. It is believed that, without seed layer, the growth of films or nano-structures may be slower or miss-oriented due to lattice-mismatch, adhesion, stress etc. ^{48, 49}. Large lattice mismatch between glass substrate and deposited nanostructures obstruct the oriented growth of nanostructures. However,

in the current research work focus on the optimization of synthesis parameters instead of using seed layers grow nanostructures especially vertically aligned ZnO nanorods.

2. Experimental

2.1 Chemicals

All reagents, iron chloride tetra-hydrate, zinc chloride, thioacetamide, hexamethylenetetramine and urea were purchased from Sigma-Aldrich and used without further purification. De-ionized water was used as solvent in all experiments.

2.2 Instruments

Thermostat bath connected with jacketed beaker was used for deposition of thin films on glass substrates. Bruker D8 advance Diffractometer with Cu-K α radiation was used for X-ray powder diffraction measurements. SEM analysis was carried out using Hitachi S3400N microscope. TEM and SAED images were collected from a Tecnai 20 F30 transmission electron microscope with accelerating voltage of 200 kV. The calculations of band gaps were performed after acquiring data from an Agilent HP 8453 UV-Vis spectrophotometer. Photoluminescence data were obtained on a Fluorolog 22, HORIBA PL using excitation wavelength of 340 nm.

2.3 Substrate preparation

Diamond scribe was used to cut glass slides into substrates with dimensions 1 x 3 cm². Glass substrates were degreased with ethanol and then cleaned ultrasonically by acetone, 2-propanol and de-ionized water for ten minutes each, respectively. Two glass substrates were joined together and hanged vertically into the solution bath with help of Teflon substrate holder in order to avoid deposition of thin films on both sides of the substrate.

2.4 Synthesis of FeS thin films

The FeS thin films were deposited from chemical bath having solutions of thioacetamide, urea and iron trichloride as reported earlier²⁴. The solutions of iron trichloride (0.15M), urea (1M)

and thioacetamide with molar concentrations of 0.15, 1 and 2 M were prepared separately in de-ionized water under stirring for half an hour to get 30 mL for each precursor solution. Urea solution was added dropwise to the iron trichloride solution followed by the addition of thioacetamide solution under stirring. After two hours of stirring, a transparent solution was achieved. To adjust the pH of solution, 0.5M hydrochloric acid was prepared and added dropwise to the solution till the pH reached to 2.5. The reaction mixture was kept at 85°C in thermostat bath. Color of the reaction mixture starts changing from transparent to blackish and after 30 minutes of reaction turns dark black (Figure 1). At this stage, glass substrates were immersed vertically into the bath and removed after 3 hours. Deposited thin films were washed with de-ionized water immediately after removing from the bath to remove slackly adherent deposits and then dried at room temperature. No post deposition heat treatment was carried out before characterizations.

Figure 1: Transformation of the color of reaction mixture from (a) transparent to (c) dark black and (d) as-deposited thin film of FeS on glass substrate

2.5 Synthesis of ZnS thin films

For growth of ZnS thin films, solutions (30 mL each) of urea, thioacetamide and zinc chloride were prepared in de-ionized water with concentrations of 0.15, 1 and 5M, respectively ². Solution of urea was added dropwise to zinc chloride solution followed by addition of thioacetamide solution under continuous stirring at room temperature. The pH of reaction mixture was fixed to 4.0 by adding 0.5M HCl drop wise. The reaction mixture was then transferred to the chemical bath and maintained at 80°C. Pre-cleaned glass substrates were held vertical by substrate holder into the reaction beaker just after five minutes of turbidity and removed after three hours (Figure 2). As-deposited thin films were washed with de-ionized water and dried at ambient atmosphere before characterization.

Figure 2: (a) Reaction mixture for deposition of ZnS (b) as-deposited thin film of ZnS on glass substrate

2.6 Synthesis of ZnO thin films

The ZnO thin films were grown on the glass substrates from chemical bath having equimolar solutions of zinc chloride and hexamethylenetetramine. The solutions of zinc chloride and hexamethylenetetramine, 50 mL each prepared separately in de-ionized water. These solutions were then mixed gradually to have the bath solution of 100 mL. The solution was maintained at a

temperature of 95°C in thermostat bath (Figure 3). Ultrasonically cleaned glass substrates were held vertically into the bath and withdrawn after 3 hours followed by washing with de-ionized water. The deposited thin films after removal from the bath appeared white in color which were then dried at room temperature before further characterization.

Figure 3: (a) Reaction mixture for deposition of ZnO (b) as-deposited thin film of ZnO on glass substrate

3. Results and Discussion

3.1 Structural studies

Figure 4 shows the XRD pattern of as-deposited FeS thin films is in good agreement with that of tetragonal phase of FeS (ICDD pattern # 01-086-0389). Black vertical lines in Figure 4 represent the standard pattern of FeS. The existence of an intense and distinct diffraction peak divulges the well crystallized and monophasic growth of FeS preferentially grown along (001) plane ²⁴. Additional peaks are not observed indicating that no impurity phases are formed during the synthesis of FeS thin films. Scherrer's equation was used to estimate the crystallite size and found to be 14 nm.

Figure 4: XRD pattern of FeS thin film.

Figure 5 reveals the XRD pattern of ZnS thin films. The broad diffraction peaks correspond to the lattice planes of cubic phase *i.e.* (111), (220) and (311) well accorded to the standard data (ICSD # 01-080-0020). No diffraction peaks corresponding to hexagonal phase of ZnS were detected. The observed XRD pattern for ZnS is in close agreement with the observations reported earlier^{50, 51}.

Figure 5: XRD pattern of ZnS thin film.

Broad diffractions peaks represent the nano-crystallinity of the films ⁴⁷ rather than deposition of amorphous material as reported previously by several authors ⁵²⁻⁵⁴. The average crystallite size in case of ZnS deposition was 9.09 nm and is close to that observed by Man and Lee ⁵⁰.

XRD pattern of ZnO thin films reveal the deposition of polycrystalline material exhibiting hexagonal wurtzite structure. The obtained diffraction peaks (Figure 6) correspond to (002) and (004) lattice planes which are well matched with reported data (JCPDS card # 00-001-1136). Intensity of (002) peak indicates the growth of ZnO along preferred orientation of c-axis ³⁰. It is important to mention here that ZnO thin films are grown onto the bare glass substrates without any seed layer of ZnO nanoparticles. The average crystallite size for ZnO nanorods estimated through Scherrer relation is 49 nm.

Figure 6: XRD pattern of ZnO thin film.

The preferred orientation of ZnO thin films was confirmed by Harris analysis using the formula

of texture coefficient⁵⁵; $P(hkl) = \frac{I(hkl)}{I_o(hkl)} \left[\frac{1}{n} \sum_{i=1}^n \frac{I(hkl)}{I_o(hkl)} \right]^{-1}$, where I is the observed

relative intensity with respect to the most intense diffraction peak, I_o is the standard relative intensity for (hkl) plane obtained from JCPDS card # 00-001-1136 and n is the number of peaks in the diffraction pattern. For preferentially oriented sample the P (hkl) value should be greater than 1 and in the case of ZnO thin films, P(002) is greater than '1' showing the preferred orientation of deposited thin films.

The dislocation density (δ), defined as the length of dislocation lines per unit volume was

estimated using the equation⁵⁶; $\delta = \frac{1}{D^2}$ and obtained as 0.0051, 0.0625 and 0.0028 nm⁻² for

FeS, ZnS and ZnO thin films, respectively. Where 'D' is crystallite size calculated from Scherrer's equation. The low values of dislocation line density indicating progressive annihilation of linear defects that would confirm the compatibility of thin films with substrates

and ultimately resulted in enhanced optical and mechanical properties of deposited thin films. Thin films with minimum dislocation densities or virtually dislocation-free can have applications in devices.

3.2 Morphology of thin films

The SEM images of FeS, ZnS and ZnO thin films at same magnification are shown in Figure 7 and reveal growth of nanostructures with different morphologies. In case of FeS, anisotropic growth of nano-flakes occurred with size ≤ 200 nm resulted from nucleation of crystallites on the surface of substrate. The SEM micrograph for ZnS thin film shown in Figure 7 indicates the growth of nanoparticles which are distributed uniformly onto the substrate surface. These nanoparticles are in the form of spherical agglomerates⁵⁷ with an average size of 100 nm. These agglomerated grains are formed by combination of very small crystallites as estimated by XRD analysis^{58, 59}.

The nanorods of ZnO (Figure 7) were grown on glass substrates with a hexagonal prism shape facets⁶⁰. Note that the nanorods are dense enough even without use of any seed layer and grown along the c-axis (002) orientation. A well aligned vertical growth of these nanorods can be possible by using seed layer of ZnO nanoparticles before chemical bath deposition as reported in literature^{30, 61, 62}. However, the observed morphology of ZnO nanorods grown on glass substrates without seed layer is in close agreement with that observed earlier with seed layer⁶⁰.

Figure 7: SEM micrographs of FeS, ZnS and ZnO thin films

Further microstructural study was carried out by typical TEM and HR-TEM. Thin films were scratched from the glass substrate and were dispersed in ethanol followed by casting and drying on copper grids. Figure 8 illustrate that FeS nanocrystals are anisotropic while ZnS nanocrystals are in the form of spherical clusters made up of small nanoparticles. Inset image of selected area electron diffraction pattern for ZnS depict the appearance of diffraction rings. The observed diffraction rings correspond to electron diffraction from three planes as observed in XRD of ZnS thin films. In case of ZnO, TEM reveals the growth of nanorods with average length and diameter as 350 and 100 nm, respectively. Inset image (HRTEM) shows the d-spacing of 0.26

nm correspond to the most intense XRD peak *i.e.* (002) as observed in XRD analysis of ZnO thin films confirming the growth along c-axis.

Figure 8: TEM images of FeS, ZnS and ZnO

3.3 Optical studies

To evaluate the optical properties of deposited thin films, optical band gaps were determined. The band gap calculation was performed simply by inferring the linear part of the $(\alpha h\nu)^2$ Vs $h\nu$ curves to the energy axis, when $(\alpha h\nu)^2 = 0$. The band gaps were found to be 1.97, 3.43 and 3.74 eV for FeS⁶³, ZnO⁶⁴ and ZnS⁵⁸ thin films, respectively (Figure 9). The observed values of band gap are found to be slightly higher than those reported earlier for these materials^{24, 59} which might be attributed more to the increment in surface area of deposited nanocrystals depending on their geometrical shape and less to the quantum confinement phenomenon. The variation of $(\alpha h\nu)^2$ with photon energy $h\nu$ (Figure 9) depicts a direct type of transition in all materials.

Photoluminescence of deposited metal chalcogenide thin films under excitation wavelength of 340 nm was observed at room temperature. Emission spectra are shown in Figure 10. The broad emission peak observed at 641 nm for FeS can be assigned as its characteristic peak confirming the monophasic nature of deposited material.

Figure 9: Bandgaps of FeS, ZnS and ZnO thin films deposited on glass substrate by CBD

The photoluminescence spectra observed at room temperature for ZnS and ZnO thin films show two emission bands in each case. For ZnS thin films, emissions bands at 397 and 498 nm were observed and for ZnO thin films, emissions bands at 389 and 504 nm were observed. The emissions at 397 and 389 nm are attributed to the well-known near band edge (NBE) emission of the wide band gap of semiconductors and mainly came from the exciton recombination of the photo-generated holes in the valence band and the electrons in the conduction band through an exciton–exciton collision process when materials are chemically synthesized at nano scale ⁶⁵⁻⁶⁸. The emissions at 498 and 504 nm are attributed to the zinc interstitial and oxygen vacancy related defect levels which act as luminescence centers for deposited thin films ^{69, 70}. These luminescence centers enhance the radiative recombination processes and substantially increase PL intensity of green emission. The observed optical properties revealed that the deposited nanocrystalline, monophasic and pure thin films can have applications in advanced optoelectronic devices. Both the surface recombination and structural parameters have great

impact on designing of thin film solar cells in order to obtain the maximum conversion efficiency

71.

Figure 10: PL spectra of FeS, ZnS and ZnO thin films deposited on glass substrate by CBD.

4. Conclusions

In summary, growth of monophasic and nanocrystalline thin films FeS, ZnS and ZnO has been accomplished by CBD on glass substrates. The XRD revealed that the deposited thin films exhibit tetragonal (mackinawite) phase for FeS, cubic (zinc blende) phase for ZnS and hexagonal (wurtzite) phase for ZnO, without evidence of other phases of these materials. The SEM images show the growth of nano-flakes of FeS, nanoparticles of ZnS and nanorods of ZnO. The deposited films show high crystallinity and good adhesion. Optical studies revealed that nanostructured thin films exhibit direct band transitions with enhanced photoluminescence and have potential applications in advanced optoelectronic devices.

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Conflicts of Interest

The authors would like to declare no conflict of interest.

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