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Growth and surface morphological studies of highly pure Lead Selenide nanocrystalline structures

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***Abstract*—Lead Selenide has shown potential application in the field of energy. In order to harvest energy from Lead Selenide the studies of its various nanostructures is very crucial. In this research Lead Selenide in the form of highly pure pallet is developed at suitable temperature and pressure. The another Lead Selenide nanostructure is Lead Selenide thin film which is developed by very efficient and low cost Chemical Bath Deposition (CBD) technique. The thin film is grown in the self-developed instrument for CBD technique. The purity of both the developed nanostructures have been checked by the widely used Energy Dispersive X ray Spectroscopy (EDAX). The EDAX analysis shows that both the developed nanostructures are highly pure having no foreign elements present in them.**

Keywords— PbSe, Pallet, Thin film, Chemical Bath Deposition method, EDAX.

I. Introduction

21st century has made the human kind to think about other available energy sources rather than about to end fossil fuels. The Transition metal chalcogenes are very important materials that can be researched to resolve the recent energy crisis. Lead Selenide is a one of a kind transition metal chalcogene (TMCs) that have shown their potential applications in solar thermal and other areas deals with energy.[1,2,3] The nanocrystalline structures have very unique electrical, optical, chemical and also the mechanical properties and hence they must be researched widely.[4,5] There are variety of Lead Selenide nanostructure a researcher can work on such as nanocrystalline pallet, thin film, quantum wells, quantum wires, quantum dots, etc.[6,7] because of they shown promising applications in the field of solar thermal, energy, IR sensors and biomedicines. [8,9] The past research of Lead Selenide nanostructure includes very few work on pallet form. Here in this research author has discussed some important properties of pallet form of PbSe and compared it with thin film of Lead Selenide grown by Chemical Bath Deposition (CBD) Method.

Here the Lead Selenide pallet is grown by using highly pure powders of Lead and Selenium and pressure controlled highly accurate pallet making machine. Now the nanocrystalline Lead Selenide thin film is developed by most suitable and low cost Chemical Bath Deposition (CBD) Method. For studying the energy applications of any material the purity of that material will play very crucial role, so here the purity of both the developed nanostructures have been studied by the widely used and most trusted Energy Dispersive X ray Spectroscopy (EDAX).

II. Growth of Lead Selenide Nanostructures

Any researcher can work on the growth and characterization any of the available nanostructures of Lead Selenide . In this research the author has carried out the research on two of the below given Lead Selenide nanostructures.

A. Highly pure pallet of Lead Selenide.

B. Lead Selenide thin film grown by Chemical Bath Deposition (CBD) method.

A. Highly Pure Pallet of Lead Selenide (PbSe)

Previously resarchers have done very few works on Lead Selenide (PbSe) pallet form so there is a wide scope of opportunity to work in this area. Hence here in this research the author has included vital work on Lead Selenide pallet form.

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A.1 Pallet Preparation for Experimentation

Lead and Selenium powders having purity 99.99 % are used for the development of the pallet. In order to prepare the pallet of Lead Selenide pallet following procedure has to be done precociously.

A.1.1 Material weight calculation

A.1.2 Pallet weight calculation.

A.1.1 Material weight calculation

(1) Atomic mass of Lead (Pb): 207.2 gm/mol, Atomic mass of Selenium (Se): 78.96 gm/mol
(2) Atomic mass of Lead Selenide (PbSe): 207.2+78.96 = 286.16 gm/mol

A.1.2 Pallet weight calculation

(1) Weight of Pb (Lead) in 10 gm powder = $\frac{\text{Atomic mass of Lead} \times \text{Sample weight}}{\text{Atomic mass of Lead Selenide}}$

$= \frac{207.2 \times 10}{286.16}$ = 7.2404 gm.

(2) Weight of Se (Selenium) in 10gm powder = $\frac{\text{Atomic mass of Selenium} \times \text{Sample weight}}{\text{Atomic mass of Lead Selenide}} = \frac{78.96 \times 10}{286.16}$ = 2.7592 gm.

(3) Total weight of prepared sample of PbSe = Weight of Pb+ Weight of Se= 7.2408+2.7592 = 10.0000 gm

As calculate above PbSe sample having weight 10 gm is taken in the pallet making machine as shown in Fig-1. Now 5 ton pressure is applied for 2 minutes and the grown pallet is collected in sample collector for further experimentation. To make the Lead Selenide pallet stoichiometrically perfect it is annealed in the instrument as shown in Fig-2. Lead Selenide pallet is annealed at 200 oC for 1 Hour in the Hot air oven. After cooling of the pallet it is collected in sample holder and ready for further characterization.

Fig-1 Instrument for the development of pallet

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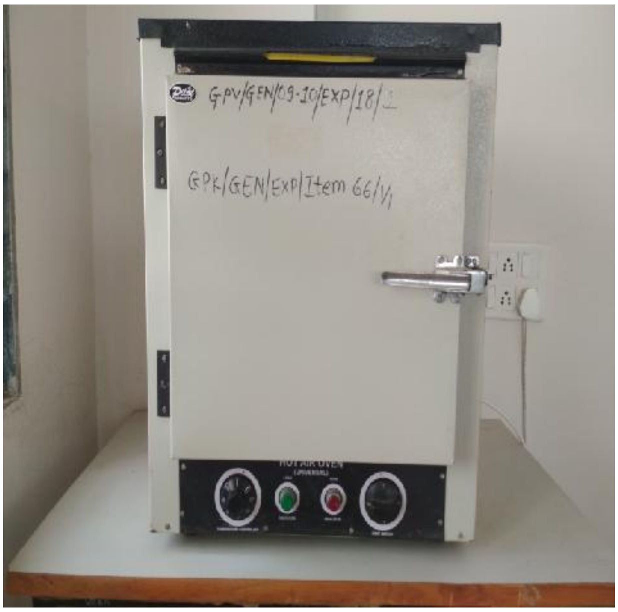

Fig-2 Instrument for the annealing of Pallet

B. Lead Selenide thin film grown by Chemical Bath Deposition (CBD) method.

Among the available variety of methods for the development of thin film of Lead Selenide Chemical Bath Deposition (CBD) is the most suitable method. Now the Chemical Bath Deposition method does not need complicated instruments such as high pressure or vaccum or very high or very low temperatures so it is easy to carry out at room temperature and in a single room facility. This technique can be easily done with the simple instruments like hot plate, magnetic temperature gauge (Thermometers) and Glassware's. The chemicals compositions which are to be used here are commonly available and cheap. Using this method in a single run large number of substrates can be coated. There is no requirement of electrical conductivity of the substrate for deposition. Depositions of thin films at low temperature gives the researcher to expel corrosion and oxidation of metallic substrates from the developed thin films.[10]

B.1 Self-Developed Instrument for CBD Method:-

Chemical Bath Deposition (CBD) method is very efficient and low cost but some special setup is required to develop thin film by it. Generally researchers use experimental setup developed by well-known firms for the growth of thin film but here the author has developed instrument for the growth of thin film by CBD method using simple equipment's available in most physics laboratories. Following are the important equipment's used by the author for the development of instrument for CBD method.

1. Hot plate.
2. Acrylic sheet with suitable made holes in it.
3. Burette Stand.
4. Mercury Thermometer.
5. Steel Pan.
6. Normal tap water.

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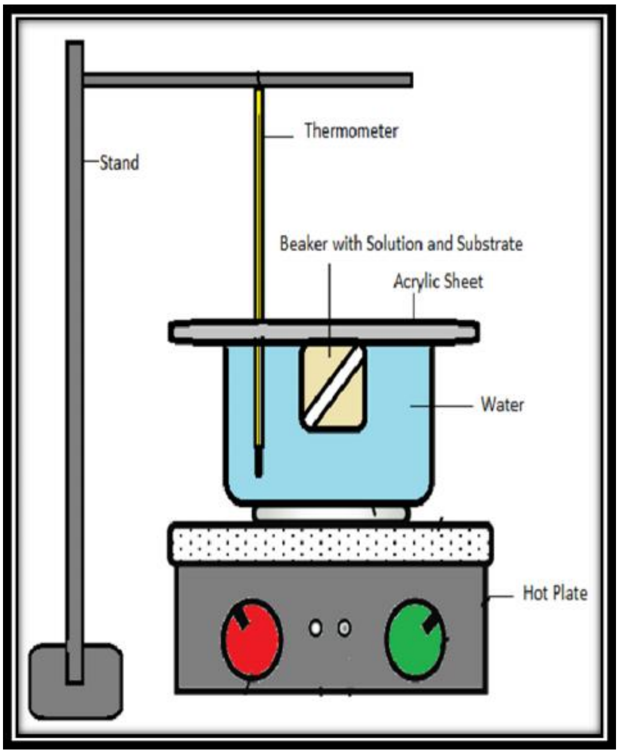

Fig- 3 Schematic diagram Self Developed Instrument for CBD method

Fig- 4 Self Developed Instrument for CBD method

Above Fig-3 & Fig-4 shows the schematic diagram and actual image of the self-developed instrument for the growth of thin film by Chemical Bath Deposition (CBD) method. The developed instrument has worked efficiently for the growth of thin film as very nice temperature control has been achieved by it.

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B.2 CBD Method Experimantation :-

The author has carried out several unsuccessful attempts to grow thin film of Lead Selenide by Chemical Bath Deposition method and finally succeeded in 3rd attempt of the experimentation. Following is the step by step procedure for the growth of Lead Selenide thin film by CBD method.

- 0.5 M 50 ml Lead Nitrate- Pb(NO3)2 solution is prepared as shown below.
  50 ml double distilled water + 8.30 gm Pb(NO3)2 = 0.5 M 50 ml Lead Nitrate
- The prepared composition of Lead Nitrate is taken in 250 ml borosilicate glass beaker.
- 0.5 M 25 ml Tartaric Acid (C4H4O6) solution is prepared as shown below
  25 ml double distilled water + 1.8761 gm C4H4O6 = 25 ml 0.5 M Tartaric Acid
- This Complexing agent is mixed with Lead Nitrate in 250 ml glass beaker by stirring constantly with glass rod.
- 0.5 M 50 ml Sodium Selenate (Na2SeO4) solution is prepared as below
  50 ml double distilled water + 4.7235 gm Na2SeO4 = 0.5 M 50 ml Sodium Selenate
- This Selenium source in mixed with Lead Nitrate solution by stirring constantly with glass rod.
- The pH of the solution is adjusted by adding 15 ml 30% Ammonia (NH4OH) with constant stirring the solution.
- Now a cleaned glass substrate is vertically immersed into the solution and the Glass beaker is closed with watch glass.
- The prepared solution beaker with substrate is introduced into the indigenously developed hot water bath at temperature 90 0C.
- The temperature is kept 90 0C constant for 120 minutes.
- After 120 minutes the glass beaker with substrate is taken out of the hot water bath substrate is removed from the solution.
- The substrate is washed with double distilled water and dried in the air for 30 minutes.
  Following all the steps shown above precociously a thin film as required by the need of characterization has been developed. Below Fig-4 shows the uniform and adherent PbSe thin film grown by the experimentation.

Fig-5 Developed PbSe thin film by CBD method.

The developed thin film is then collected in suitable sample holder for further characterization.

III: PURITY VERIFICATION OF DEVELOPED NANOCRYSATLLINE STRUCTURES:-

The title of the paper get the idea that here the research includes work on highly pure Lead Selenide (PbSe) nanocrystalline structures. So there is no doubt that only highly pure nanocrystalline structures are to be considered for the characterization. Now the purity of the nanocrystalline structures have been verified by the most trusted and widely used Energy Dispersive X ray Spectroscopy (EDAX).

A: ENERGY DISPERSIVE X RAY SPECTROSCOPY (EDAX):-

The Energy Dispersive X ray Analysis (EDAX) is the technique that reveals the stoichiometry of constituent elemnets in any material by the configuration of X rays and it is the fundamental rule for the EDAX analysis.[11] So basically EDAX analysis is a stoichiometrical analysis that gives the constituent elements present in the sample. Hence EDAX will reveal that weather the sample is pure or it have impurities present in it. Here the EDAX analysis is done by the instrument Field Emission Gun Scanning Electron Microscopy (FEG-SEM) Make: FEI Ltd Model: Nova Nano SEM 450 for the experimentation. Following Fig-6 shows the instrument of EDAX.

Fig-6 Instrument for the EDAX analysis

B: Results obtained by EDAX Analysis:-

The EDAX analysis of the naocrstalline structures have given us three important parameters to analyze for the result.
(B.1) Area-1 Image
(B.2) Full Area-1 Graph
(B.3) eZAF Smart Quant Results

B.1:Area-1 Image:-

The following Fig-7 shows the area-1 image of the Lead Selenide nanocrystalline pallet form at 5 micrometer resolution. Fig-8 shows the area-1 image of Lead Selenide thin film at 20 micrometer resolution. As we can see in both the images that spherical shaped structures are present in both the images at the resolution of micrometer which clearly indicates that both the nanocrystalline Lead Selenide structures consist of spherical shaped atomic structures.

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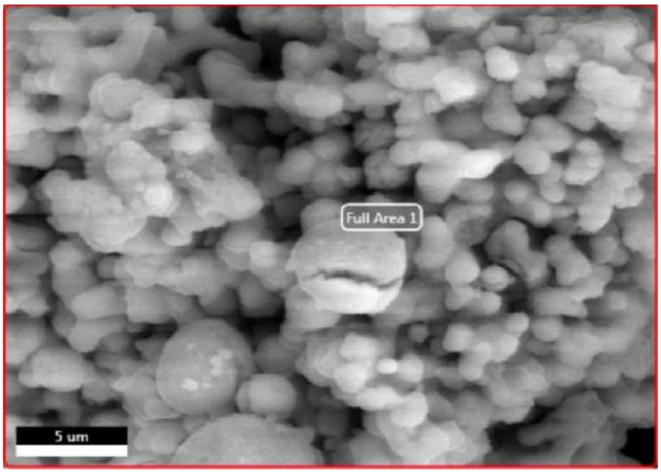

Fig-7 Area-1 Image of PbSe Pallet

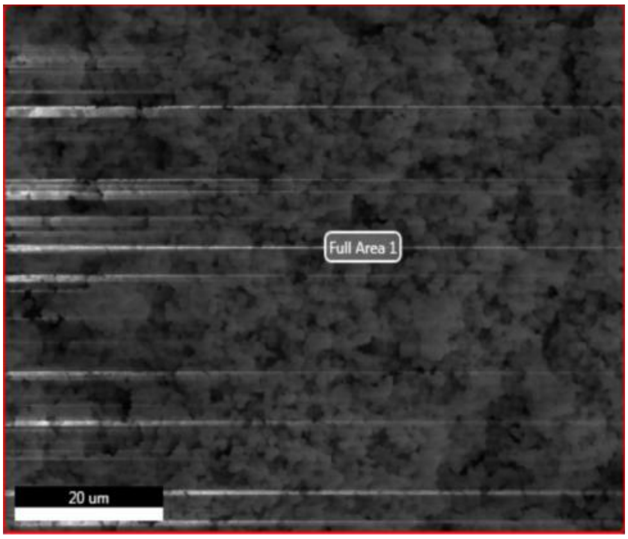

Fig-8 Area-1 Image of PbSe Thin film

B.2: Full Area-1 Graph:-

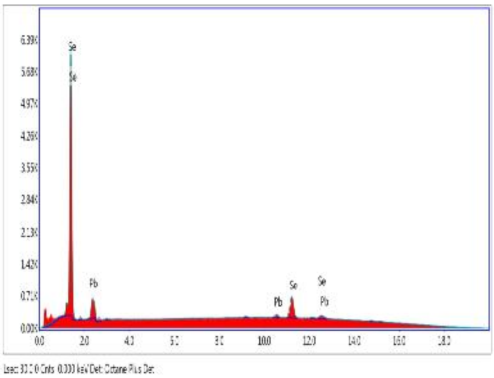

Fig-9 Full Area-1 Graph of PbSe Pallet

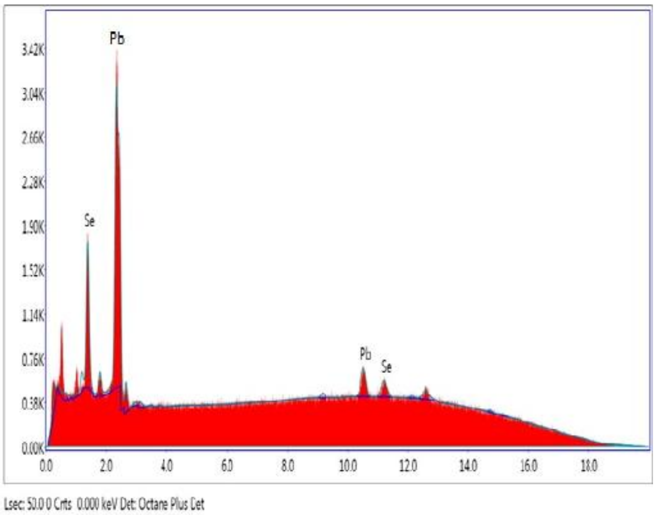

Fig-10 Full Area-1 Graph of PbSe Thin film

Above Fig-9 indicates the Full Area-1 Graph of Lead Selenide nanocrystalline pallet form and Fig-10 indicates the Full Area-1 Graph for the Lead Selenide thin film. As analyzing both the graphs the important facts can be achieved for the constituent elements present in the material sample of PbSe. The peaks seen in both the graphs are the peaks for the elements present in the sample and as it is seen that in both the graphs the peaks seen are only indicating presence of two elements one is Lead (Pb) and other is Selenium (Se). So there are no foreign elements present in both the nanocrystalline structures, Hence it is proved that developed Lead Selenide nanocrystalline pallet and the thin film by CBD method are highly pure.

B.3: eZAF Smart Quant Results:-

The eZAF Smart Quant Results obtained by the EDAX analysis gives the weight percentage, atomic percentage, various correction parameters (Such as Z-atomic number correction, A- absorption correction, F-fluorescence correction, etc).These results are very important as they indicates the weight percentage of the elements present in the sample and it can be compared with the previously taken amount of the elements for experimentation to verify the purity of developed nanocrystalline structures.

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	R	A	F
PbM	64.55	40.96	1,046.8	4.19	0.53	0.94	1.09	0.87	1
SeK	35.45	59.04	110.62	17.24	0.41	1.18	0.95	0.98	1.01

Table-1 eZAF Smart Quant results of PbSe Pallet

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	R	A	F
SeK	10.21	4.16	312.78	8.95	0.06	0.86	1.21	0.74	1
PbM	89.79	95.84	600.77	8.63	0.92	1.03	0.99	1	1

Table-2 eZAF Smart Quant results of PbSe Thin Film

Above Table-1 shows the eZAF smart quant results obtained by the EDAX analysis of Lead Selenide pallet. Here as we compare the constituents of elements with the previously taken amount it is matching accurately which suggest the high purity of developed pallet of Lead Selenide. Table-2 shows the eZAF smart quant results for Lead Selenide thin film developed by Chemical Bath Deposition (CBD) method. The weight percentage and atomic percentage of only two constituents Lead (Pb) and Selenium (Se) is seen which shows that the developed thin film by CBD method is highly pure.

IV-CONCLUSION:-

Recently the energy crisis have made the researchers to thought about other important materials for harvesting energy from solar thermal. Currently available solar cell efficiency is low and it is very important to increase the efficiency of solar cells so that maximum solar thermal energy can be converted into electrical energy. Lead Selenide have shown its ability to be used in the solar cells. So it is very important to carry out research on PbSe nanocrystalline structures to study their efficiency in the field of energy. Here Lead Selenide nanostructure in the form of pallet and thin film are studied. Highly pure Lead Selenide pallet is successfully developed at suitable temperature and pressure. The stoichiometry of the developed pallet is perfected by annealing. It is observed that the Chemical Bath Deposition(CBD) is the most suitable method for the growth of Lead Selenide thin film. Here the instrument for the growth of Lead Selenide thin film is self-developed and succefully tested for the growth of the film. Energy Dispersive X ray Analysis has shown that the developed nanocrystalline structures are highly pure and only having two constituents Lead (Pb) and Selenium (Se) without any impurities in it.

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