Structural and Surface morphology of Lead Selenide (PbSe) thin films

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Abstract. Lead Selenide (PbSe) thin films are deposited by chemical bath deposition for 2 hours in different temperature. The precursors used are 0.2M of Lead (II) nitrate, 0.5M of Sodium Selenosulfate, 2.0M of Sodium hydroxide and 4ml of Triethanolamine. The film deposition is carried out at 50˚C, 60˚C, 70˚C, and 80˚C for 2 hours. The thickness of the film varied in the range from 1400 to 5200Å. The structural characterization of these films is carried out by X-ray diffractometer (JEOL-Japan, JDX 8030 model). The XRD pattern of PbSe films deposited at different temperature exhibit the polycrystalline structure. In the present study, Scanning Electron Microscope (JOEL 840 SEM/EDAX) is employed to analyze the surface morphology of the films. In addition, the compositions of the films are estimated from EDAX Spectrum. Therefore, it is observed that the films deposited in this work, possess strong peaks for Pb and Se and no other impurities are detected through the EDAX Spectrum, confirming high purity of the PbSe thin film.

1. Introduction
The thin film systems have gained considerable interest over the years, due to their special properties. The properties of any solid-state substance and bulk or thin-film rely on the compositions of the atoms. In addition, the existence of the chemicals connections and simple excitations exist inside the structure. On three fundamentals, thin film production is focused viz., fabrication, usability and applications. Substantially, the dependent on a large number of repositioning parameters may also depend on thickness [1, 2], the structural, chemical, methodological and physical properties of such material. There are other benefits of chemical bath deposition, such as easy operation which does not need any specialized tools or heat facilities, zero waste content, cost-effective methods for any wide-range deposition. For this current element, the substrate is submerged directly in the reaction bath for a specific temperature deposition at 2 hours, whether metallic or nonmetallic [3,4]. In this research study, for the deposition of PbSe films, the chemical depositing technique is used. Moreover, this examination presents the structural and surface morphology of these films.

2. Experimental methods
In 100 ml glass beaker strength, 10 mL of Pb (NO₃)₂ solutions is taken, gradually 4 ml of TEA is added with continuous stirring. In the beginning, the solution becomes milky turbid. Further, 7 ml NaOH is used to transform lead nitrate into lead hydroxide, it should be dissolved in sodium hydroxide before a stable solution is achieved. 10ml of freshly formulated solution Na₂SeSO₃ is applied gradually with a constant stirring to sustain an alkaline pH media of approximately 10. For a few minutes, using a magnetic stirrer the final solution is separated from the substrates are then
washed, tilt vertically at 20° to the beaker surface. The bath water is left open and the deposition is allowed for 2 hours without stirring at various deposition temperatures. For about (50°C, 60°C, 70°C and 80°C) the different deposition is carried out at 2 hours in session.

3. Results and discussion

The research examination employs an accurate electronic balance for weight measurements to decide film thickness, the SHIMADZU-ADY220 digital electronic balance. The mass difference between the slides before and after the film is the m' mass. The deposition area is determined in the knowledge of the length (l) and width (b) of the deposited film. When μ is the density of the film content, then the film’s thickness (t) is as shown by Table 1.

\[
t = \frac{\text{mass of the deposited film}}{\text{area of the film}} \times \text{density of the film}
\]

\[
t = \frac{m}{\text{Density of PbSe}} \quad \text{[the density of PbSe is 8.10 g/cm}^3]\]

| Different deposition Temperature(˚C) at 2hours | Thickness (Å) |
|-----------------------------------------------|---------------|
| 50                                           | 1400          |
| 60                                           | 2600          |
| 70                                           | 4100          |
| 80                                           | 5200          |

A ray diffractometer (JEOLJapan JDX 8030) conducted the structural characterization of these films. Figure1.1 illustrates the XRD pattern in chemically deposited films PbSe at various deposition temperatures (50°C, 60°C, 70°C and 80°C) for 2 hours. The thin films of Lead selenide are observed to be polycrystalline in nature, with a preferred direction around (200) the point. The amplitude of the peak (200) rises considerably and more rapidly than the other peaks (111), (220), (311), (222), (400), (420) and (422). Likewise, CuInSe2 and CdS thin films are previously recorded [5-8].

A procedure is employed to determine the magnitude of the preferred orientation factor 'f' in relation to certain planes (peaks) of material [7]. The preferred orientation factor f(200) of (200) in PbSe thin films is measured using this tool by measuring a fraction of (200) the level amplitude over the cumulative intensities of all peaks inside that specific plane. Similarly, all other peaks for the four films are assessed. In PbSe films are prepared at 50°C (Figure 1.1.a) f (111) =0.3042, f (200) =0.7869, f (220) =0.1748, f (311) =0.0649, f (222) =0.0769, f (400) =0.0474. In PbSe films prepared at 80°C (Figure 1.1.d) f (111) = 0.3190, f (200) = 0.8305, f (220)=0.1573, f (311)=0.0331, f (222)=0.0675, f (400)=0.0266, f (420)=0.0283,f(422)=0.0201. As f (200) is higher than other orientations in all four films, PbSe thin films have the preferred (200) orientation. Increased crystallinity and the bath deposition temperature are exceptional for the change of (200) the design and switch on, independent of chemical environment. The predicted peaks of the thin PbSe films are recorded by many researchers [5]. In the Figure 1.1(a) broad peaks at 2θ ≈ 20° and 34° is given as the quantity of unreacted precursor oil plants [9]. PbSe thin films are identified as peaks of the character of the PbSe thin films by many researchers [5]. Table 2. displays measured and normal values 'a' and 'd' for dominant peak diffraction angles and the values are seen to be in accordance with PbSe normal values. Table 3 measured the
volume of grain, strain and dislocation rate. As a result of the greater mobility of atoms at high temperatures, it is found that the rising grain size depends on the deposition temperature. Therefore, the abundance of nuclear centers is reduced, and a smaller number of centers continue to rise under the circumstances [10].

![Figure 1 (a)](image1)

![Figure 1 (b)](image2)

![Figure 1 (c)](image3)

![Figure 1 (d)](image4)

**Figure 1.** XRD pattern of PbSe thin film at Different deposition temperature (50°C, 60°C, 70°C, and 80°C) for 2 hours time period.

The declining diffusion intensity suggests the creation of high-quality films at a temperature 70 degrees Celsius [11], subsequently the substrate is kept at a temperature around 70 degrees Celsius. It is mainly because of the dislocalisation, which absorbs more thermal energy and has better mobility. The additional increase in temperature by 80 °C is also observed as a consequence of an increased stress and dislocation density due to the re-evaporation of certain Pb atoms [6].
Table 2. Comparison of calculated and standard ‘d’ and ‘2θ’ values for PbSe thin films for different deposition temperature at 2 hours.

| Deposition Temperature (°C) | hkl planes | 2θ values (degree) | d- spacing values (Å) | Lattice constant (Å) | FWHM (β) |
|-----------------------------|------------|-------------------|---------------------|---------------------|---------|
|                             |            | JCPDS | Expt | JCPDS | Expt | JCPDS | Expt | JCPDS | Expt | JCPDS | Expt | JCPDS | Expt | JCPDS | Expt | JCPDS | Expt |
| 50                          | 111        | 25.125 | 25.114 | 3.5414 | 3.5430 | 6.1366 | 0.2481 |
|                             | 200        | 29.092 | 29.076 | 3.0670 | 3.0685 | 6.1370 | 0.1856 |
|                             | 220        | 41.610 | 41.604 | 2.1687 | 2.1689 | 6.1345 | 0.3137 |
|                             | 311        | 49.227 | 49.237 | 1.8494 | 1.8491 | 6.1327 | 0.5987 |
|                             | 222        | 51.573 | 51.628 | 1.7707 | 1.7689 | 6.1276 | 1.9507 |
|                             | 400        | 60.307 | 60.468 | 1.5335 | 1.5297 | 6.1188 | 1.1535 |
| 60                          | 111        | 25.125 | 25.129 | 3.5414 | 3.5409 | 6.1330 | 0.1774 |
|                             | 200        | 29.092 | 29.103 | 3.0670 | 3.0658 | 6.1316 | 0.1164 |
|                             | 220        | 41.610 | 41.642 | 2.1687 | 2.1671 | 6.1294 | 0.2268 |
|                             | 311        | 49.227 | 49.270 | 1.8494 | 1.8479 | 6.1287 | 0.2282 |
|                             | 222        | 51.573 | 51.644 | 1.7707 | 1.7684 | 6.1259 | 0.3871 |
|                             | 420        | 68.334 | 68.398 | 1.3716 | 1.3704 | 6.1286 | 1.9900 |
| 70                          | 111        | 25.125 | 25.219 | 3.5414 | 3.5284 | 6.1113 | 0.1229 |
|                             | 200        | 29.092 | 29.184 | 3.0670 | 3.0575 | 6.1150 | 0.1101 |
|                             | 220        | 41.610 | 41.739 | 2.1687 | 2.1623 | 6.1159 | 0.1326 |
|                             | 311        | 49.227 | 49.360 | 1.8494 | 1.8448 | 6.1185 | 0.1300 |
|                             | 222        | 51.573 | 51.723 | 1.7707 | 1.7659 | 6.1172 | 0.1589 |
|                             | 400        | 60.307 | 60.448 | 1.5335 | 1.5302 | 6.1208 | 0.1160 |
|                             | 420        | 68.334 | 68.505 | 1.3716 | 1.3685 | 6.1201 | 0.1637 |
|                             | 422        | 75.934 | 76.135 | 1.2521 | 1.2492 | 6.1198 | 0.2100 |
| 80                          | 111        | 25.125 | 25.124 | 3.5414 | 3.5416 | 6.1342 | 0.1662 |
|                             | 200        | 29.092 | 29.093 | 3.0670 | 3.0668 | 6.1336 | 0.1606 |
|                             | 220        | 41.610 | 41.629 | 2.1687 | 2.1677 | 6.1311 | 0.2792 |
|                             | 311        | 49.227 | 49.248 | 1.8494 | 1.8487 | 6.1314 | 0.4310 |
|                             | 222        | 51.573 | 51.608 | 1.7707 | 1.7696 | 6.1300 | 0.3414 |
|                             | 400        | 60.307 | 60.336 | 1.5335 | 1.5328 | 6.1312 | 0.6570 |
|                             | 420        | 68.334 | 68.406 | 1.3716 | 1.3703 | 6.1281 | 0.5694 |
|                             | 422        | 75.934 | 76.055 | 1.2521 | 1.2503 | 6.1251 | 0.6369 |
Table 3. Structural parameters of PbSe thin films for different deposition temperatures

| Deposition Temperature (°C) | 20 values (degree) | Grain Size (D) (nm) | Strain $10^{-2}$ ($\varepsilon$) (Lin$^2$.m$^{-4}$) | Dislocation Density $10^{15}$ (Lines/m$^2$) |
|-----------------------------|-------------------|---------------------|-----------------------------------------------|---------------------------------------------|
| 50                          | 25.114            | 34.2632             | 1.0566                                        | 0.8518                                      |
|                             | 29.076            | 46.1844             | 0.7839                                        | 0.4688                                      |
|                             | 41.604            | 28.2945             | 2.1275                                        | 1.2490                                      |
|                             | 49.237            | 15.2447             | 2.3748                                        | 4.3029                                      |
|                             | 51.628            | 4.7250              | 7.6621                                        | 44.7915                                     |
|                             | 60.468            | 8.3257              | 4.3484                                        | 14.4264                                     |
| 60                          | 25.129            | 47.9197             | 0.7555                                        | 0.4354                                      |
|                             | 29.103            | 73.6410             | 0.4915                                        | 0.1843                                      |
|                             | 41.642            | 39.1357             | 0.9249                                        | 0.6529                                      |
|                             | 49.270            | 39.9956             | 0.9050                                        | 0.6251                                      |
|                             | 51.644            | 23.8107             | 1.5203                                        | 1.7638                                      |
|                             | 68.398            | 4.8259              | 7.1816                                        | 4.2938                                      |
| 70                          | 25.219            | 69.1818             | 0.5234                                        | 0.2089                                      |
|                             | 29.184            | 77.8740             | 0.4650                                        | 0.1648                                      |
|                             | 41.739            | 66.9679             | 0.5408                                        | 0.2229                                      |
|                             | 49.360            | 70.2422             | 0.5156                                        | 0.2026                                      |
|                             | 51.723            | 58.0291             | 0.6241                                        | 0.2969                                      |
|                             | 60.448            | 82.7821             | 0.4375                                        | 0.1459                                      |
|                             | 68.505            | 61.3220             | 0.5907                                        | 0.2659                                      |
|                             | 76.135            | 50.1867             | 0.7217                                        | 0.3970                                      |
| 80                          | 25.124            | 51.1484             | 0.7078                                        | 0.3822                                      |
|                             | 29.093            | 53.3758             | 0.6782                                        | 0.3510                                      |
|                             | 41.629            | 31.7933             | 1.1387                                        | 0.9893                                      |
|                             | 49.248            | 21.1772             | 1.7095                                        | 0.2297                                      |
|                             | 51.608            | 26.9957             | 1.3411                                        | 0.1372                                      |
|                             | 60.336            | 14.6077             | 2.5784                                        | 4.6863                                      |
|                             | 68.406            | 17.6193             | 2.0547                                        | 3.2212                                      |
|                             | 76.055            | 16.5387             | 2.1890                                        | 3.6559                                      |

The thinly deposited PbSe film SEM representation (60 ° C, 70 ° C & 80 ° C) is seen in Figure 2. From Figure 2(a), the SEM micrograph is studied at a resolution at a particular magnification with a film deposition temperature of 60 ° C. The vacuity between the molecules indicates that PbSe molecules are loosely bundled at a resolution.

For Figure 2, Figure b) the grain is distinctly outlined and the variation in the intergrain is decreased to a more crystalline film, the flat and consistent surface indicator. It could be the product of the (200) plane's extremely favorite concentration.

The secondary development on the surface is apparent from Figure 2(c). Due to the dislocation, density is decreased and the crystalline size is increased. It is necessary to remember the strong change of the crystallite size in the SEM micrograph due to the rise in the film deposition temperature.
Figure 2. SEM of PbSe thin films deposited for a different deposition temperature at 2hrs time period (a) 60°C X 25000 (b) 70°C X 25000 (c) 80°C X 25000

For the confirmation of the composition of fine films as shown in Figure 3 (a, b, c) an energy dispersive X-ray analysis (EDAX) is applied. The energy scattering X-ray analyses (EDAX) show the numerous peaks, which suit the present elements concurrently. The concentration of the elements found in the environment is therefore calculated. PbSe shows thin films with various deposition (60 °C, 70 °C and 80 °C), EDAX scale, 2 hours duration. The large peaks for BP and Se are observed in spectrum and the EDAX signal complies with the elevated pureness of the thin film. PbSe is not identified with any contaminations. Table 4 displays the atomic and weight percentage of Pb & Se components. It shows varying temperature cycles and elemental analysis. The average nuclear percentage of Pb is observed. Almost, the stoichimetric composition is present in every case without the inclusion of other impurities.
Figure 3. EDAX of PbSe thin film for different deposition temperature periods a) 60°C b) 70°C c) 80°C.

Table 4. Weight and Atomic percentage of Pb & Se for PbSe thin films prepared at 2hours for different deposition temperature

| Deposition Temperature (°C) | Element | Weight % | Atomic % |
|-----------------------------|---------|----------|----------|
| 60                          | PbL     | 75.16    | 53.56    |
|                             | SeK     | 24.84    | 46.44    |
| 70                          | PbL     | 74.87    | 53.16    |
|                             | SeK     | 25.13    | 46.84    |
| 80                          | PbL     | 74.29    | 52.41    |
|                             | SeK     | 25.71    | 47.59    |

4. Conclusion
The XRD design of PbSe films deposited at different time period reveals the polycrystalline structure. The thickness of the film varied in the range of 1400 Å to 5200Å. It is prominent that the rise of the film deposition is due to temperature and the crystallite element in the SEM micrograph which naturally increases. The composition of EDAX Spectrum is determined to have good picks for Pb and Se and the EDAX Spectrum which is no more impurities to validate the PbSe films that are extremely clean.
5. References

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