CHARACTERIZATION OF NICKEL LEAD SULPHIDE THIN FILMS:
X-RAY DIFFRACTION STUDIES

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ABSTRACT
In this work, nickel lead sulphide thin films were grown by inexpensive and simple method, namely chemical bath deposition technique. Nickel sulphate, sodium thiosulfate and lead nitrate as the sources of Ni$^{2+}$, S$^{2-}$ and Pb$^{2+}$ ions, respectively. The effect of deposition period will be studied from 8 to 34 hours at room temperature. The obtained films were characterized by X-ray diffraction. The results reveal that the number of peaks was increased from two to five peaks as the deposition time increased up to 34 hours. In other words, more materials will be deposited onto substrates for longer deposition time.

Keywords: thin films, x-ray diffraction, metal chalcogenide.

INTRODUCTION
Nowadays, in search of new semiconducting materials for solar energy conversion through photo electrochemical cells, and metal chalcogenide materials are increasingly studied by many researchers. These materials have been prepared by many deposition methods including chemical deposition method, physical deposition method and sputtering deposition technique. The obtained films were characterized by using various tools such as Rutherford backscattering spectrometry [1-6], photoluminescence technique [7-10], transmission electron microscopy [11-14], X-ray diffraction [15-18], Raman spectroscopy [19, 20], profilometer [21,22], atomic force microscopy [23-26], and UV-visible spectrophotometer [27-30] as reported by many scientists.

In this study, the influence of the deposition time (8-34 hours) on structural properties of chemical bath deposited nickel lead sulphide thin films was investigated by X-ray diffraction. These films were deposited onto soda lime glass in acidic medium at room temperature.

MATERIALS AND METHODS
In this experiment, the soda lime glass slides were employed to be the substrate to prepare thin films. Before deposition, the substrate was cleaned with distilled water and ultrasonically cleaned in ethanol solution for 10 minutes and finally dried in air. All reagents were purchased and used without further purification. Deionized water (Alpha-Q Millipore) was employed as solvent in all experiments. First, 25 mL of 0.6 M sodium thiosulfate solution was added into the beaker that contained 25 mL of 0.6 M nickel sulphate and 25 mL of 0.6 M lead nitrate, respectively. Then, these solutions were mixed together for few minutes. In order to control the pH at constant value of 1.6, hydrochloric acid solution was added to the solution. The deposition process was carried out for different deposition times (8-34 hours) at room temperature. After completion of film deposition, the glass substrate was removed from the beaker and cleaned with distilled water. Then the deposited films were dried in the desiccator and subjected to further analyses.

The structure of the films was studied by X-ray diffraction (XRD) with a PANalytical X-Pert PW 3040 diffractometer equipped with a CuK$\alpha$ ($\lambda=0.15418$ nm) radiation source. Data were collected by step scanning from 10° to 90° with a step size of 0.026° ($2\theta$).

RESULTS AND DISCUSSIONS
The X-ray diffraction (XRD) patterns obtained for thin films prepared at various deposition times were shown in Figures 1, 2, 3, and 4. Identification of the peaks on the X-ray diffraction patterns indicates that the obtained films are polycrystalline in nature. Also, the crystal structure was identified to be rhombohedral. Figure 1 shows the films prepared for 8 hours. The XRD pattern displays that the intensities of the signals are rather weak because of the thin film nature of the samples. The observed $d$-values of Ni$_3$Pb$_2$S$_2$ thin films were compared with standard $d$-values which obtained from Joint Committee on Powder Diffraction Standard (JCPDS) data (Reference code: 00-006-0459). Preferred orientation of Ni$_3$Pb$_2$S$_2$ thin films is observed to be along (110) and (300) planes as indicated in Figure-1.
Figure-1. XRD pattern of thin films prepared for 8 hours.

Figure-2. XRD pattern of thin films prepared for 11 hours.
Figure-2 shows the XRD pattern for the films prepared for 11 hours. The peaks corresponding to the (110), (024) and (300) planes have been detected. Comparison between the samples prepared for 8 and 11 hours show that the intensity of the peaks increased for the films prepared at longer time. Also, the number of peaks in the films prepared for 11 hours increased to three peaks with $d$-values successfully matching standard $d$-values (Table-1). Figure-3 shows the thin films prepared for 28 hours. The peaks obtained show the (012), (110) and (006) planes have been deposited. On the other hand, thin films prepared for 34 hours indicate five peaks corresponding to (012), (110), (024), (122) and (300) planes. The intensities of these peaks were much better as shown in Figure 4 as compared to other samples. The (110) peak indicates the highest intensity for all samples.
Table-1. Comparison between observed $d$-values of Ni$_3$Pb$_2$S$_2$ thin films prepared at different deposition times with JCPDS data.

<table>
<thead>
<tr>
<th>Deposition time (h)</th>
<th>Standard $d$-values (Å)</th>
<th>Observed $d$-values (Å)</th>
<th>Plane (hkl)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>2.80 1.61</td>
<td>2.81 1.61</td>
<td>110 300</td>
</tr>
<tr>
<td>11</td>
<td>2.80 1.97 1.61</td>
<td>2.82 1.98 1.62</td>
<td>024 300</td>
</tr>
<tr>
<td>28</td>
<td>3.97 2.80 2.27</td>
<td>3.95 2.80 2.26</td>
<td>012 110 006</td>
</tr>
<tr>
<td>34</td>
<td>3.97 2.80 1.97 1.76 1.61</td>
<td>3.95 2.79 1.98 1.75 1.62</td>
<td>012 110 122 300</td>
</tr>
</tbody>
</table>

CONCLUSIONS
Ni$_3$Pb$_2$S$_2$ thin films have been deposited onto soda lime glass substrate at room temperature. The effect of deposition period on the structure of films was investigated using X-ray diffraction. The observation of X-ray peaks showed that the obtained films were polycrystalline in nature. As the deposition time was increased to 34 hours, the number of peaks also increased.

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REFERENCES


