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Scanning Electron Microscopy Study of Surface Morphology of $\text{Ni}_3\text{Pb}_2\text{S}_2$ Thin Films

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$\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films have been prepared using a simple and economical chemical bath deposition method. These films were produced from aqueous solutions in the presence of complexing agent. The morphologies of $\text{Ni}_3\text{Pb}_2\text{S}_2$ films were characterized using scanning electron microscopy. Scanning electron microscopy micrographs revealed that there are many various morphologies were obtained on the films deposited under various deposition conditions such as pH, deposition time and bath temperature.

Keywords: Thin films, Chemical bath deposition, Scanning electron microscopy.

INTRODUCTION

Recently, ternary chalcogenide thin films have received a great deal of attention due to their potential application in the area of thermoelectric, electronic, semiconductor technology, photovoltaic and optoelectronic devices. Up to date, ternary chalcogenide thin films have been prepared by many deposition techniques including arrested precipitation technique¹, electro deposition²⁻⁵, thermal evaporation⁶, successive ionic layer adsorption and reaction⁷, spray pyrolysis⁸⁻¹⁰, radio-frequency reactive sputtering technique¹¹ and chemical bath deposition method¹²⁻¹⁹. In this work, we produce thin films by using low cost and easy technique, namely chemical bath deposition method if compared with other deposition techniques.

In this work, the scanning electron microscopy is used to investigate the morphology of $\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films. There are many deposition conditions such as deposition time, bath temperature and pH that need to be studied in order to obtain good quality of thin films.

EXPERIMENTAL

Nickel(II) sulfate, lead(II) nitrate, sodium thiosulfate and tartaric acid were used in this work were of analytical reagent grade. Water is obtained from a Millipore Alpha-Q water purification system (18.2 M Ω cm, Millipore Corporation). For the preparation of $\text{Ni}_3\text{Pb}_2\text{S}_2$ films, 20 mL of 0.1 M tartaric acid was placed into separate beakers which contained 20 mL of 0.1 M of nickel(II) sulfate and 20 mL of 0.1 M lead(II) nitrate, respectively. Then, under continuous stirring, the components of a mixture were physically mixed together. In

this experiment, the bath temperature (55, 70, 75 and 80 °C), pH (pH 1.1, 1.3 and 1.5) of the solution and deposition time (25, 45, 60 and 85 min) were varied to optimize the $\text{Ni}_3\text{Pb}_2\text{S}_2$ films formation. The microscope glass slide was placed vertically to the bottom of the beaker during the deposition process. Before use, glass slides were treated for 0.5 h with ultrasonic wave in the bath of ethanol and then rinsed with deionized water. After complete deposition, the obtained films were rinsed with distilled water and finally dried at 80 °C for 8 h.

Surface morphology was studied by JEOL (JSM-6400) scanning electron microscopy operating at an accelerating voltage of 20 kV under 1000X magnification.

RESULTS AND DISCUSSION

Nowadays, the applications of scanning electron microscopy (SEM) are very common in materials science. Generally, it is used by researchers to determine the surface morphology and to measure the grain size. The SEM systems are much more user friendly and permit the measurement in macro and submicron ranges.

Fig. 1 shows the SEM micrographs of $\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films deposited under various bath temperatures range of 55 to 80 °C. Fig. 1b indicates that the films prepared at 70 °C, it is possible to observe that the surface of these films are smooth, dense and uniform with average diameter of 2.5 μm . On the other hand, at higher bath temperature such as 75 °C (Fig. 1c) and 80 °C (Fig. 1d), the grains get bigger with an irregular shape. Diameters of these films are distributed from 4 to 12 μm .

Fig. 2 indicates the SEM micrograph of $\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films prepared at different deposition times such as 25, 45, 60 and 85 min. Based on the SEM micrographs, the films prepared at longer deposition time are always thicker and larger than shorter time as shown in Fig. 2d. The films prepared

at 25 min were very small in size with no well-defined grain boundaries as indicated in Fig. 2a. However, scanning electron microscopy analysis reveals that the most homogeneous and regular surface could be obtained at the deposition time of 60 min.

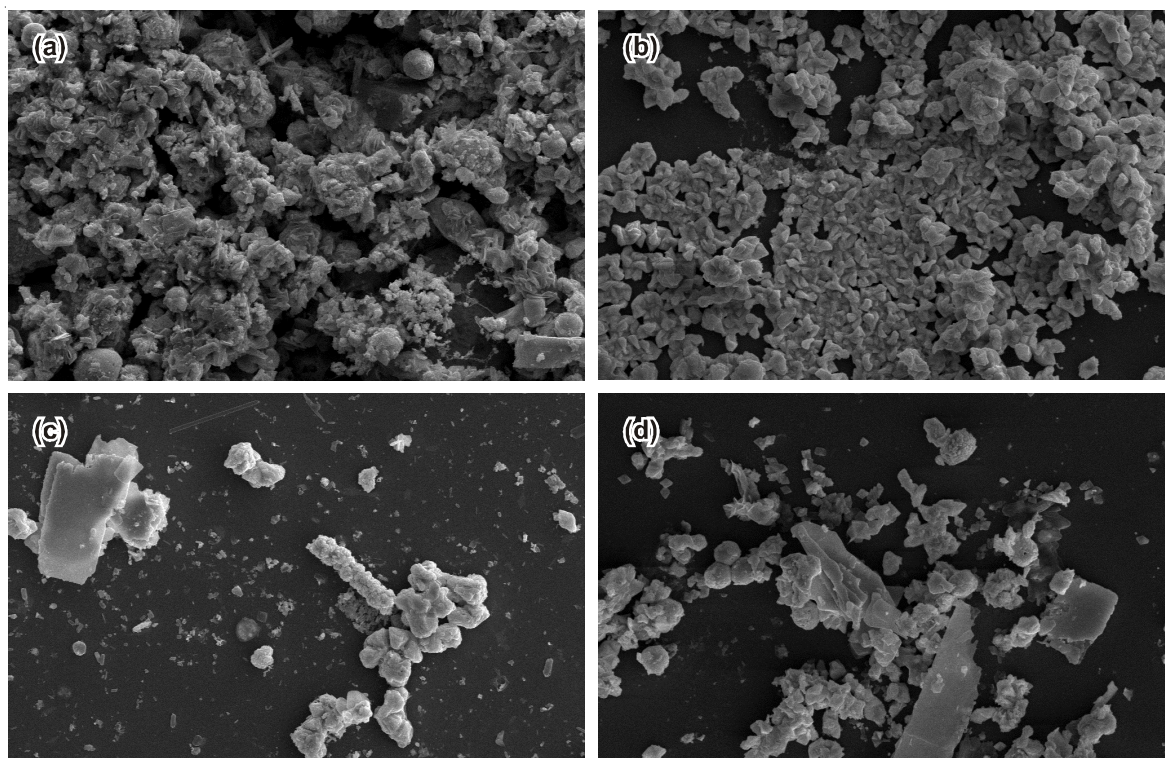


Fig. 1. Scanning electron microscopy micrographs of $\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films deposited at different bath temperatures for 60 min, pH 1.5 (a) 55 °C (b) 70 °C (c) 75 °C (d) 80 °C

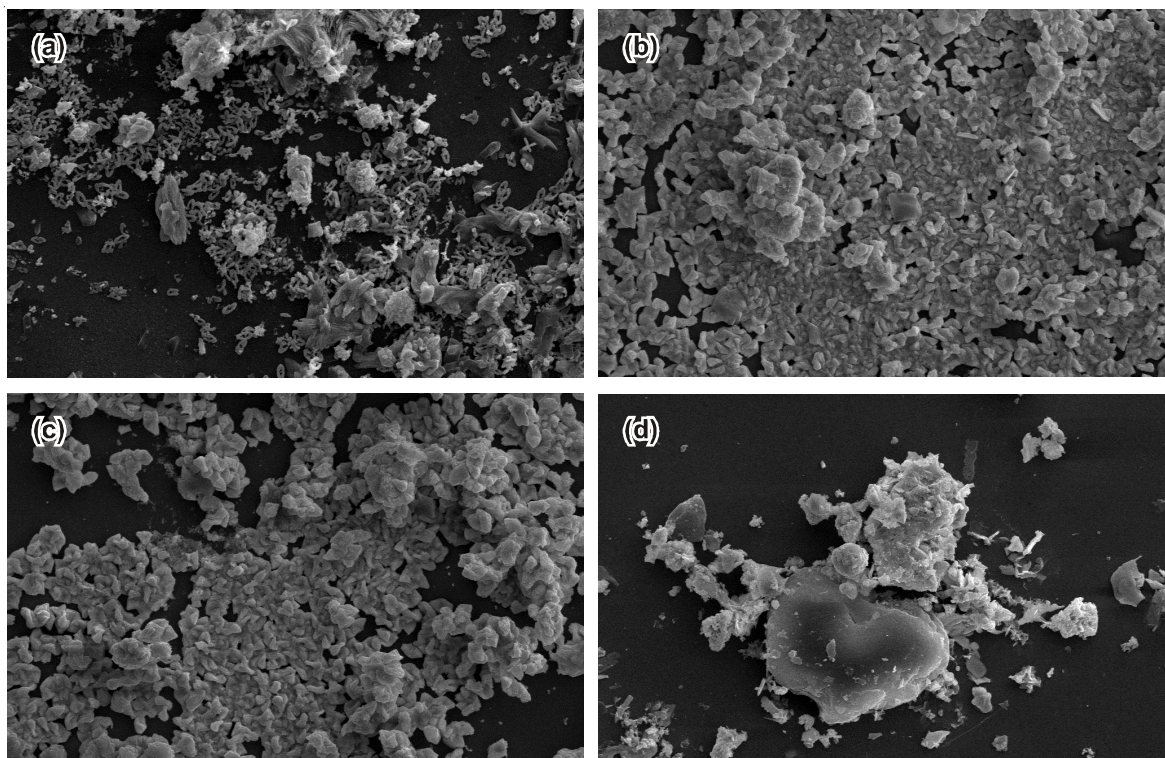


Fig. 2. Scanning electron microscopy micrographs of $\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films deposited at different deposition times for 70 °C, pH 1.5 (a) 25 min (b) 45 min (c) 60 min (d) 85 min

Fig. 3 displays the SEM micrograph of $\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films produced under various pH conditions of 1.1, 1.3 and 1.5. The average particle size is around 8-10 μm and the surface morphology was more or less the same for the films deposited at pH of 1.1 and 1.3. However, as pH was increased to 1.5, the SEM micrograph indicated a uniform structure if compared to other samples.

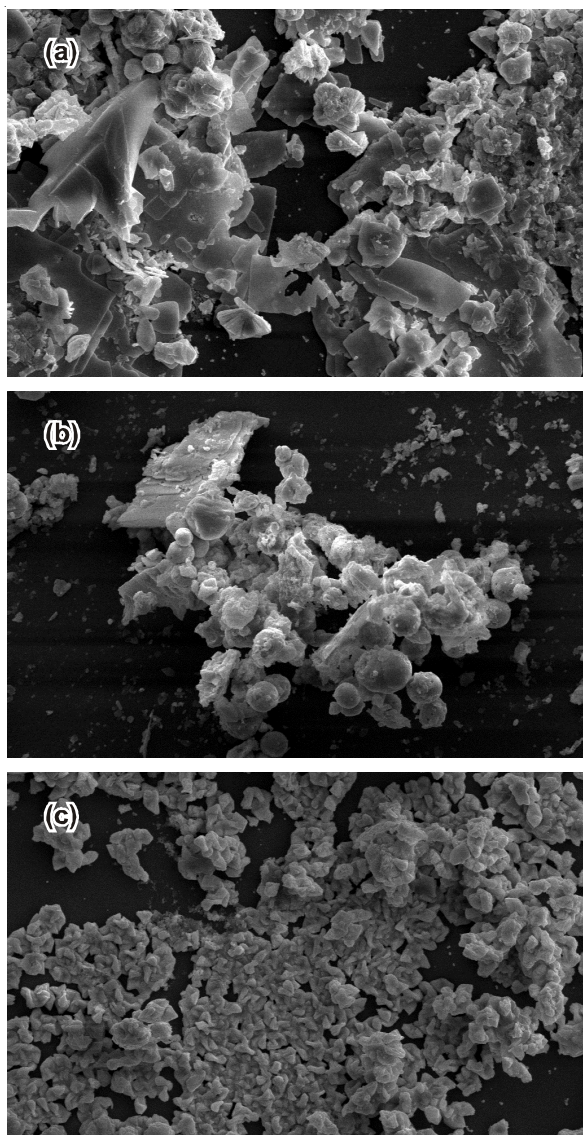


Fig. 3. Scanning electron microscopy micrographs of $\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films deposited at different pH values for 70 °C, 60 min (a) pH 1.1 (b) pH 1.3 (c) pH 1.5

Conclusion

Thin films of $\text{Ni}_3\text{Pb}_2\text{S}_2$ were deposited on glass slides from aqueous solutions in which the tartaric acid was used as complexing agent. The morphologies of films which prepared under various deposition parameters were investigated using scanning electron microscopy. The scanning electron microscopy micrographs revealed that the most homogeneous and regular surface could be obtained at the deposition time of 60 min, pH 1.5 and bath temperature of 70 °C.

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