



SEM ANALYSIS OF $\text{Ni}_3\text{Pb}_2\text{S}_2$ THIN FILMS PRODUCED BY CHEMICAL BATH DEPOSITION TECHNIQUE IN THE PRESENCE OF THE Na_2EDTA

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ABSTRACT

The $\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films have been deposited on to substrate (soda lime glass slide) under different deposition times by using chemical bath deposition technique. In this work, disodium ethylenediaminetetraacetate dihydrate (Na_2EDTA) was used as complexing agent. So far, this deposition technique is known as the cheapest and easiest method as reported by many researchers. In this work, the scanning electron microscopy (SEM) was used to study the morphologies of $\text{Ni}_3\text{Pb}_2\text{S}_2$ films. The best result could be observed for the films prepared at 1 hour. These films exhibited uniform, compact with diameters of 0.4 to 2 μm .

KEYWORDS: Semiconductor, $\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films, ternary, scanning electron microscopy, chemical bath deposition

INTRODUCTION

Thin films have been synthesized by using various deposition techniques. Generally, these deposition techniques could be divided into two groups such as physical and chemical deposition method. For example, spray pyrolysis¹, chemical bath deposition method², electro deposition method³, thermal evaporation method⁴, hydrothermal method⁵, SILAR technique⁶ and chemical vapor deposition⁷ have been reported by many researchers. They choose chemical bath deposition method because of simple equipment⁸, large scale production⁹, low material wastage and low cost¹⁰. The obtained films could be used in wide applications including solar cell¹¹, photoconductor, holographic recording media¹², infrared detector¹³, photocatalysis¹⁴, light emitting diode¹⁵, hydrogen generation¹⁶, laser screen, sensor device¹⁷ and thin film transistors¹⁸.

The main objective in this work is to produce ternary thin films via chemical bath deposition in the presence of complexing agent. This is the first time, $\text{Ni}_3\text{Pb}_2\text{S}_2$ thin films were synthesized using Na_2EDTA . Scanning electron microscopy was employed to investigate the morphologies of obtained films.

METHODOLOGY

Thin films were produced by using aqueous solutions of nickel (II) sulfate, lead (II) nitrate, sodium thiosulfate and Na_2EDTA . 25 mL of 0.1 M Na_2EDTA was placed into separate beakers (25 mL of 0.12 M of nickel (II) sulfate and 25 mL of 0.12 M lead (II) nitrate). The components of a mixture were physically mixed together and at the same time, 25 mL of 0.4 M of sodium thiosulfate was added. In this work, films were grown onto substrate under various deposition times such as 20, 40, 60 and 80 minutes. The soda lime glass slide was used as substrate during the deposition process at temperature of 63 °C, pH 1.2. The substrate was placed vertically into the bottom of the beaker. Before use, glass slides were treated for 30 min with ultrasonic wave in the bath of ethanol and then rinsed with deionized water. After complete deposition process, the obtained films were rinsed with distilled water and finally dried at 85 °C for 24 hours. Surface morphology was studied by JEOL (JSM-6400) scanning electron microscopy operating at an accelerating voltage of 20 kV under 5000X magnification.



RESULTS AND DISCUSSIONS

Based on the literature survey, characterization of thin films has been done by using different tools. For example, X-ray diffraction¹⁹, field emission scanning electron microscope²⁰, energy dispersive X-ray analysis²¹, atomic force microscopy, UV-visible spectrophotometer²², photoluminescence spectroscopy²³, Raman spectroscopy²⁴ and Fourier transform infrared spectroscopy²⁵.

The scanning electron microscope (SEM) was used to study the morphology of samples. The advantages of scanning electron microscopy (SEM) and limitations of transmission electron microscopy (TEM) & scanning probe microscopy (SPM) were highlighted in Table 1. SEM scans a focused electron beam over a surface in order to create an image. The electrons (in the beam) will interact with thin films, producing different signals in order to obtain information about the surface morphology of sample.

Table 1: Advantages of SEM, disadvantages of TEM and SPM

Advantages of scanning electron microscopy
Simple to use ²⁶ Magnifies objects more than 500000X Can provide accurate measuring and detailed images ²⁷ Most samples require minimal preparation action ²⁸ Can give high resolution and depth of focus ²⁹
Disadvantages of transmission electron microscope
Large, expensive ³⁰ Laborious sample preparation ³¹ Special maintenance is needed ³²
Disadvantages of scanning probe microscopy
The maximum image size is smaller ³³ Slower in acquiring images ³⁴

The morphologies of the Ni₃Pb₂S₂ films prepared for 20 minutes and 40 minutes were indicated in figure 1 and figure 2(See Appendix), respectively. The SEM analysis revealed that a mixture of particles of various sizes [0.2 μm to 2 μm] in these samples. The films produced for 1 hour showed smooth, dense and uniform with average diameter of 0.5 μm as indicated in figure 3. Figure 4(See Appendix) showed the films prepared for 80 minutes. The grains became larger, showed irregular shape, diameters were 0.5-2.5 μm.

CONCLUSION

Nanostructured Ni₃Pb₂S₂ films have been successfully produced by using chemical bath deposition method in the presence of Na₂EDTA. Deposition was carried out under various deposition times. The scanning electron microscopy was used to study the morphology of films. Experimental results showed that uniform and compact surface could be observed for the films prepared for 1 hour.

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APPENDIX

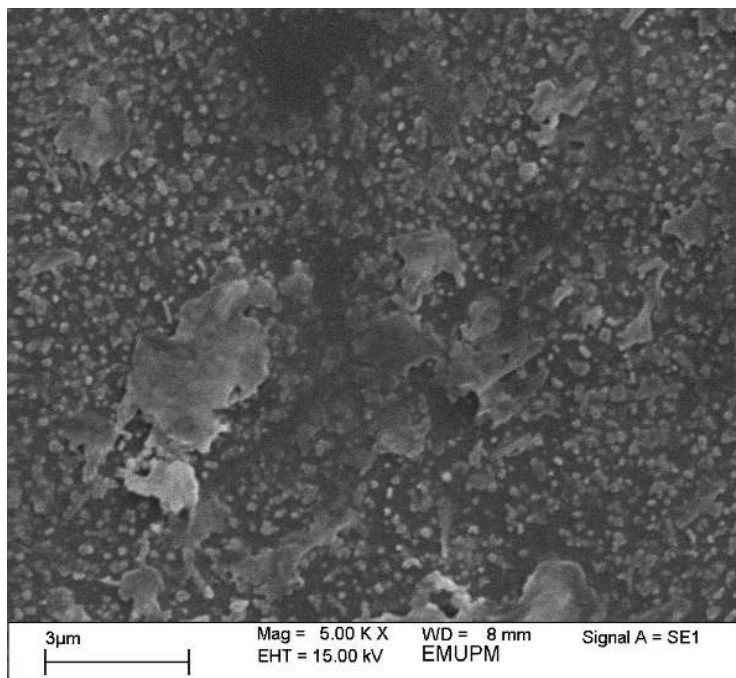


Figure 1: SEM of Ni₃Pb₂S₂ films prepared for 20 minutes

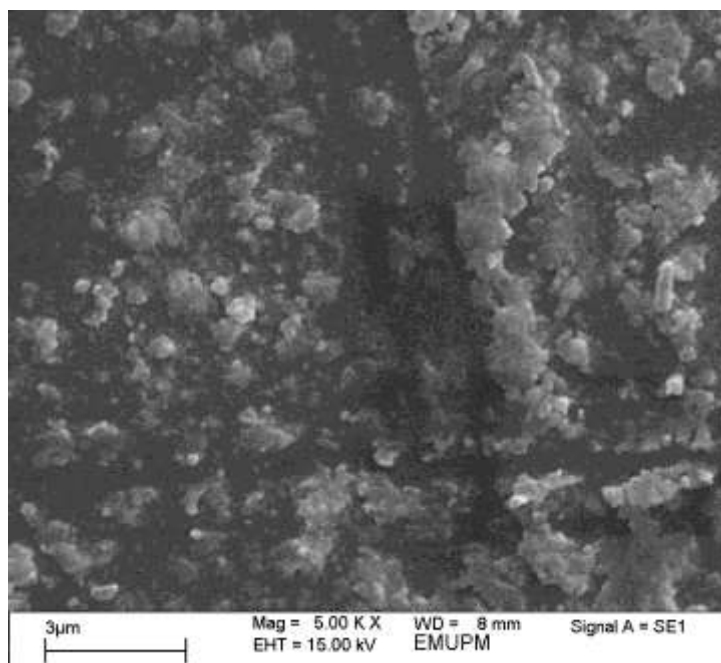


Figure 2: SEM of Ni₃Pb₂S₂ films prepared for 40 minutes

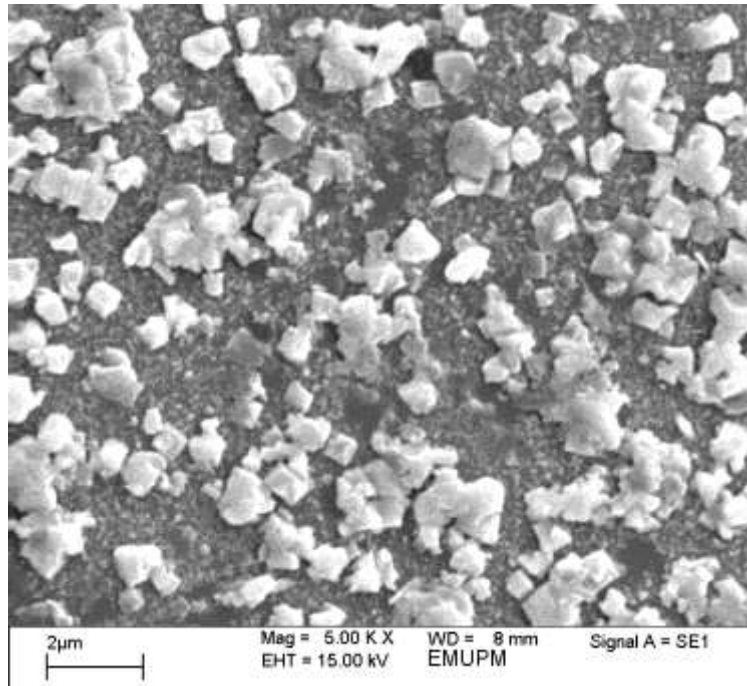


Figure 3: SEM of $\text{Ni}_3\text{Pb}_2\text{S}_2$ films prepared for 60 minutes

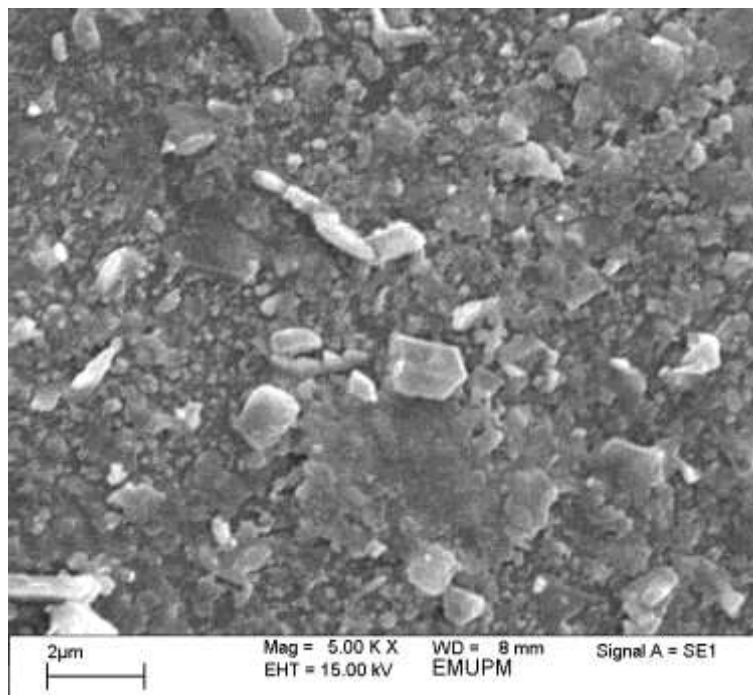


Figure 4: SEM of $\text{Ni}_3\text{Pb}_2\text{S}_2$ films prepared for 80 minutes



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