STUDIA UBB. CHEMIA, LV, 3, 2010

# X-RAY DIFFRACTION AND ATOMIC FORCE MICROSCOPY STUDIES OF CHEMICAL BATH DEPOSITED FeS THIN FILMS

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**ABSTRACT.** FeS thin films were deposited onto indium tin oxide glass substrates by chemical bath deposition method. The main objective of the paper was to investigate the influence of the bath temperature on the properties of thin films. The structural and morphological properties of the thin films were studied using X-ray diffraction and atomic force microscopy, respectively. According to XRD results, the number of FeS peaks increased to four peaks and the intensities of these peaks were improved for the films deposited at higher bath temperature. The AFM analysis showed that an increased in bath temperature allowed more materials to be deposited onto the substrate and thicker films to be formed.

***Keywords:*** *chemical bath deposition, thin films, iron sulphide, X-ray diffraction*

## INTRODUCTION

Iron sulphide thin films have been the subject of intensive research in the past decade. These thin films have found important applications such as photoelectrochemical and solar cell applications. Several preparative routes of synthesis of iron sulphide thin films were reported in the literature such as metal organic chemical vapour deposition [1], molecular beam deposition [2], reactive DC magnetron sputtering [3], flash evaporation [4], electrodeposition [5] and chemical bath deposition [6]. From the methods used for the preparation of iron sulphide thin films, the chemical bath deposition method is often preferred because it is simple, economic and offers a possibility for large area deposition. The chemical bath deposition method has frequently been used for the deposition of thin films such as PbS [7], PbSe [8], CdTe [9], $Ni_4S_3$ [10], $AgIn_5S_8$ [11] and $Cd_{0.5}Zn_{0.5}Se$ [12]. So far, to our knowledge, a study on the properties of the chemical bath deposited iron sulfide thin films has not been reported by other researchers except Anuar et al. [6]. They found that the pH played an important role in the process of deposition of $FeS_2$ thin films in the presence of triethanolamine solution.

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In this work, for the first time, the FeS thin films have been chemically deposited on indium tin oxide glass substrate in the presence of sodium tartrate as a complexing agent. The effect of bath temperature (50-80 ℃) on the properties of these films is investigated. These thin films have been characterized using X-ray diffraction and atomic force microscope for structural and surface morphological properties studies.

## RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction patterns of the iron sulphide thin films deposited under different bath temperatures. The XRD patterns from all the samples have shown a major diffraction peaks at 2θ equal to 29.9° and 43.6°, which are corresponding to (200) and (202) o rientation of hexagonal structure of FeS. For the films deposited at higher bath temperature (60-80 ℃), in addition to the (200) and (202) planes, we also observed other peaks such as (112) and (220) peaks which are corresponding to the FeS phase [13]. The observed *d*–spacing values are in good agreement with the standard *d*-spacing values (Reference code: 01-080-1028). The lattice parameter values for the dominant structure are: a=b=6.958 Å, c=5.824 Å. It is found that the intensities of FeS peaks enhanced with the increase of the bath temperature indicating the degree of crystallinity of the films increases. It is also observed that the films show a most preferred orientation along (202) plane.

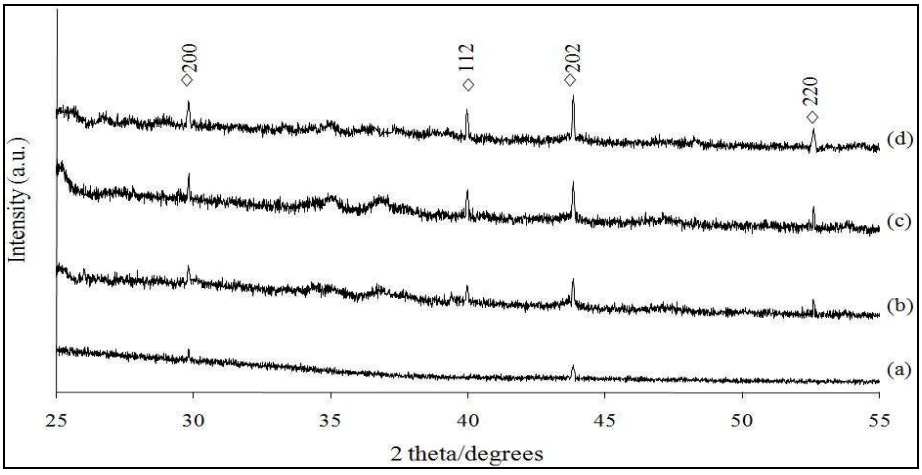


**Figure 1.** X-ray diffraction patterns for FeS thin films deposited at various bath temperatures. (a) 50 ℃ (b) 60 ℃ (c) 70 ℃ (d) 80 ℃

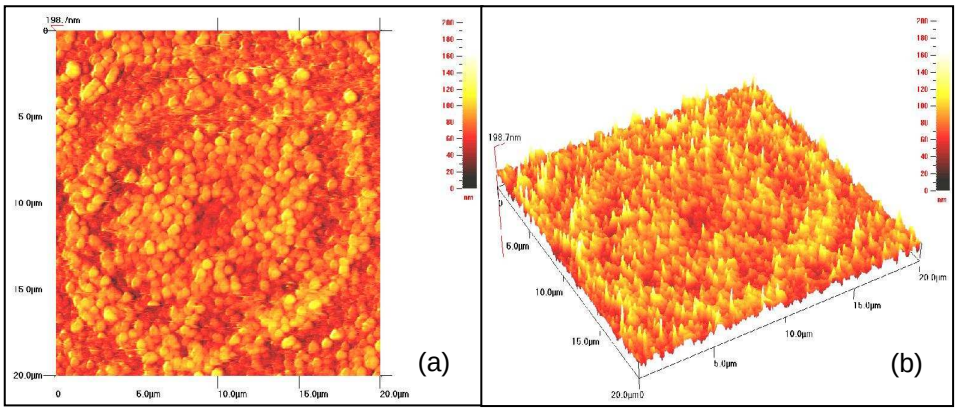


**Figure 2.** Atomic force microscopy images for FeS thin films deposited at 50 ℃ (a) 2-dimensional (b) 3-dimensional

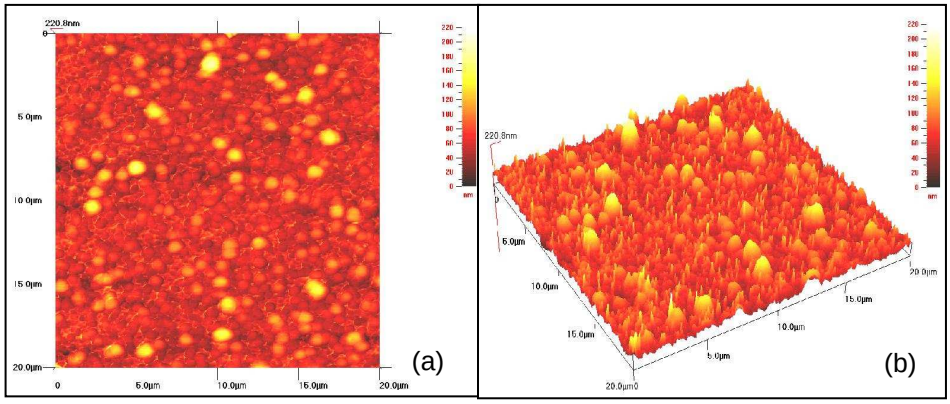


**Figure 3.** Atomic force microscopy images for FeS thin films deposited at 60 ℃ (a) 2-dimensional (b) 3-dimensional

The surface morphology of FeS thin films deposited under various bath temperatures was investigated using atomic force microscopy (AFM) technique. Figure 2 shows a typical two-dimensional (Figure 2a) and three-dimensional (Figure 2b) AFM images of the FeS thin films deposited at 50 ℃. These films revealed that grains were very small in size (0.4-0.5 μm) with no well-defined grain boundaries. It is known that the lower the bath temperature, smaller crystal size could be observed. From AFM analysis, it is observed that the FeS thin films are non-uniform when the bath temperature is increased to 60 ℃ (Figure 3a and 3b) and 70 ℃ (Figure 4a an d 4b), respectively. We

observed that the surface morphology of these films is more or less the same and the average grain size is around 0.5-1.0 µm. When the deposition temperature is further increased to 80 ℃, the grow th morphology is relatively better with more homogeneous spherical FeS grains with their size ranging from 1.8-2.0 µm as shown in Figure 5 (Figure 5a and 5b).

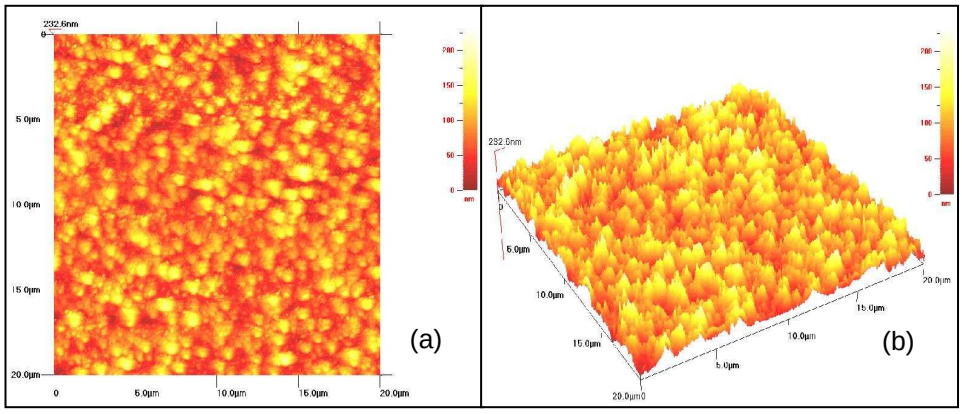


**Figure 4.** Atomic force microscopy images for FeS thin films deposited at 70 ℃ (a) 2-dimensional (b) 3-dimensional

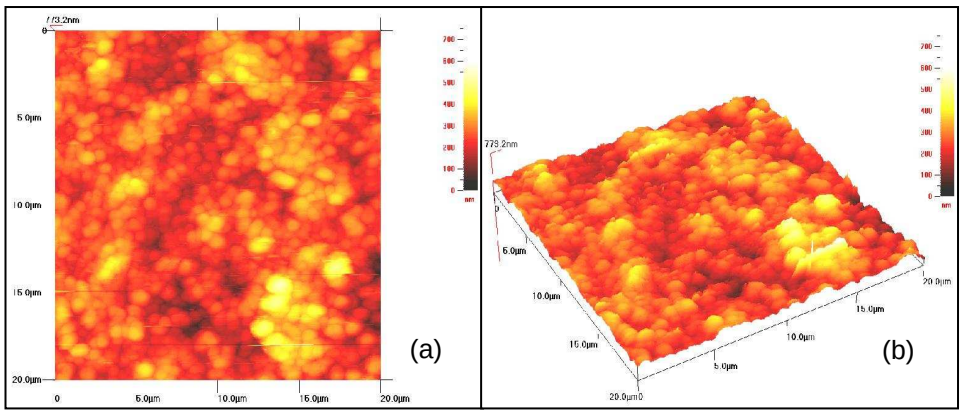


**Figure 5.** Atomic force microscopy images for FeS thin films deposited at 80 ℃ (a) 2-dimensional (b) 3-dimensional

The investigation on the surface roughness and the thickness of thin films from AFM images was carried out by many researchers [14-16]. Root mean square (RMS) roughness is defined as the standard deviation of the

surface height profile from the average height, is the most commonly reported measurement of surface roughness [17]. The surface roughness is 16, 20, 30 and 63 nm for the films deposited at 50, 60, 70 and 80 ℃, respectively. The surface roughness of the film is unavoidable since grains are grown with different sizes and spherical in shapes.

On the other hand, at the right side of the AFM images, an intensity strip is shown, which indicates the depth and height along the *z*-axis. The thickness values of 199, 221, 233 and 773 nm have been observed for samples deposited at 50, 60, 70 and 80 ℃, respecti vely. This result shows that an increase in bath temperature allows more materials to be deposited onto indium tin oxide substrate and thicker films to be formed. The AFM results suggested that the influence of bath temperature on the surface morphology of thin films is significant.

## CONCLUSIONS

The FeS thin films were successfully deposited on indium tin oxide glass substrates using chemical bath deposition technique from acidic medium in the presence of sodium tartrate solution. Structural studies showed polycrystalline nature of deposited films with hexagonal structure for all samples. According to XRD results, the number of FeS peaks increased and the intensities of these peaks were much better for the films deposited at higher bath temperature. The smaller grain was observed for lower bath temperature while a larger grain was obtained for higher bath temperature based on AFM analysis. Uniform morphology with spherical shaped grains was seen through AFM images for the films deposited at 80 ℃. Therefore, deposition at 80 ℃ was found to be the best bath t emperature to prepare good quality thin films under the current conditions.

## EXPERIMENTAL SECTION

Iron sulphide thin films were deposited on indium tin oxide (ITO) glass substrates using chemical bath deposition method. Prior to deposition, the substrate was degreased in ethanol for 10 min, followed by ultrasonically cleaned with distilled water for another 15 min and finally dried in air. During the deposition process, an aqueous solution of iron nitrate was used as iron source, sodium thiosulfate as sulfide source and sodium tartrate as complexing agent. All these chemicals used for the deposition were analytical grade. All the solutions were prepared in deionised water (Alpha-Q Millipore). For deposition, 25 ml of 0.2 M iron nitrate was complexed with 25 ml of 0.25 M sodium tartrate. To this, 25 mL of 0.2 M sodium thiosulphate was added slowly

to the reaction mixture. The pH was adjusted to 1.5 by addition of hydrochloric acid with constant stirring. The clean glass substrate was vertically immersed into the chemical bath solution with the temperatures of 50, 60, 70 and 80 ℃, respectively. After the deposition time of 120 min, the glass substrate was taken out of the bath, washed with distilled water and dried in desiccators for further characterization.

X-ray diffraction (XRD) analysis was carried out using a Philips PM 11730 diffractometer for the $2\theta$ ranging from 25° to 55° with CuK α (λ=1.5418 Å) radiation. The surface morphology, thickness and roughness were examined by recording atomic force microscopy (AFM) images in contact mode (commercial $Si_3N_4$ cantilever) with Q-Scope 250. Contact mode imaging employed a soft cantilevered beam that had a sharp tip at its end which was brought in contact with the surface of the sample. Values of root mean square (RMS) roughness were calculated from the height values in the atomic force microscopy images using the commercial software.

**ACKNOWLEDGMENTS**

The authors would like to thank the Department of Chemistry, University Putra Malaysia (UPM) for the provision of laboratory facilities and Ministry of Science, Technology and Innovation (MOSTI) for the National Science Fellowship.

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