



**INVESTIGATION OF MORPHOLOGICAL PROPERTIES OF THE COPPER
SULFIDE FILMS IN ACIDIC MEDIA BASED ON ATOMIC FORCE
MICROSCOPY**

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ABSTRACT:

CuS thin films were grown at various pH values by chemical bath deposition method. The surface morphologies such as roughness, thickness and grain size of the obtained films were characterized using atomic force microscopy. Based on atomic force microscopy images, the films deposited at pH 1 was generally uniform. Meanwhile, the films deposited at higher pH such as pH 2 and 2.5, were observed to cover the surface of the substrate incompletely. On the other hand, we have observed a very strong dependence of the surface roughness and thickness of these films on the various pH solutions.

KEY WORD: *Steroids, Withania somnifera, TLC, HPT Chemical bath deposition, Roughness, Thickness, Copper sulphide LC.*

INTRODUCTION:

Nowadays, nanomaterial has attracted a great deal of attention in researches and some technical uses. Copper sulfide, as an example of nanomaterial, can play an important role in optoelectronic applications, solar cells, photoconductors, laser materials and sensor materials. Many methods such as spray pyrolysis (Nascu et al., 1997), successive ion layer adsorption and reaction method (Zhuge et al., 2009), photochemical deposition (Podder et al., 2005), electrodeposition (Anuar et al., 2002) and chemical bath deposition (Gadave and Lokhande, 1993) have been developed to prepare copper sulphide thin films. Among these techniques, chemical bath deposition method is said to be commonly used to deposit metal thin films onto glass substrates. This is because of this method is cost effective,

time saving and precise control of the deposition process. Some researchers have reported on the chemical bath deposition of ZnSe (Anuar et al, 2011), FeSe (Ubale et al, 2013), SnS (Jayasree et al, 2013), CdSe (Zhao et al, 2013), MnS (Ulutas et al, 2013), CdS (Zhou et al, 2013), CdS_{1-x}Se_x (Muthukumaran and Muthusamy, 2012), ZnS_xSe_{1-x} (Liu et al., 2013), Cd_{0.7}Zn_{0.3}S (Al-Jubory, 2012) thin films from aqueous solution.

In this work, we investigate the use of chemical bath deposition technique for the growth of CuS films on glass substrate under various pH values. The copper sulfide films characterization using atomic force microscopy is presented.

MATERIALS AND METHODS:

Preparation of thin films

All chemicals were from commercial sources and were the highest purity available (analytical reagent grade). Therefore, these chemicals were used without further purification. All solutions in this study were deionized water (Alpha-Q Millipore) with a resistivity of 18.4 MΩcm. The microscope glass slide was employed as substrate in this study. The substrate was cleaned in ethanol, then ultrasonically cleaned with distilled water and finally they were dried in an oven at 90 °C. Aqueous solutions of copper sulfate, thiourea, tartaric acid and hydrochloric acid were used to deposit copper sulphide thin films. First, 25 mL of tartaric acid (0.10 M) solution was mixed with 25 mL of copper sulfate (0.15 M) in a beaker. Then, under continuous stirring, 25 mL of thiourea (0.15 M) was added in solution. The bath pH was carefully adjusted between pH 1 to 2.5 by controlled addition of hydrochloric acid. Finally, the substrate was immersed vertically into the same beaker. In order to produce thin films onto substrate, the deposition was carried out at temperature of 70 °C. The beaker was not stirred during the thin films deposition. After completion of films deposition (100 min), the deposited films were washed with distilled water and dried in air for further characterization.

Characterization of thin films

The surface morphologies such as thickness, grain size and surface roughness were studied by recording atomic force microscopy (Q-Scope 250) images in contact mode with a commercial Si₃N₄ cantilever. Values of root mean square (RMS) roughness were calculated from the height values in the atomic force microscopy images using the commercial software.

RESULTS AND DISCUSSION:

The atomic force microscopy (AFM) images of CuS films deposited on glass substrate, which scanned in an area of 20 μm X 20 μm are shown in Figure 1, 2, 3 and 4, respectively. The observed dependence of

the morphology of films under various pH values as indicated in figures. The AFM images indicate that there are many pinholes on the CuS films which deposited at higher pH such as 2 (Figure 3) and 2.5 (Figure 4). In addition, the AFM images reveal that these samples are found to cover the surface of the substrate incompletely. Also, these samples are less dense when compared with other samples. As shown in our studies, we can diminish the pinhole on the CuS films by decreasing the pH value. From the Figure 1, the films can be grown uniformly onto glass substrate without cracks. These films showed the grain size of 2-2.5 μm . On the other hand, the grain size for the films deposited at pH 1.5 (Figure 2) is bigger (4-4.5 μm) than that of the films deposited at pH 1 (Figure 1). Furthermore, we detected that the non-uniformity surface and grain sizes are dissimilar to each another for these films.

Experiment results indicated that the film thickness is significantly influenced by the pH. The thickest films obtained for the films prepared at pH 2 (685.9 nm) while the thinnest films observed at pH 1 (5.81 nm). Based on AFM images, the thickness values for films deposited at pH 1.5 and 2.5 are found to be 13.85 and 11.19 nm, respectively.

On the other hand, we found that, the surface roughness is also sensitive to changes in pH. The roughest films obtained for the films prepared at pH 2 (47.02 nm) while the smoothest films observed at pH 2.5 (0.78 nm). Based on AFM images, the surface roughness values are found to be 0.80 and 2.18 nm for the films deposited at pH 1 and 1.5, respectively.

CONCLUSION:

The morphology of the CuS films which produced using chemical bath deposition method was mainly affected by the deposition parameter such as pH. The uniform films were obtained at lower pH such as pH 1. While, the films formation were found to be irregular as the pH further increased to 2.5. On the other hand, the thickest and roughest films could be prepared at pH 2 based on atomic force microscopy images.

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REFERENCES:

Al-Jubory, A.A. (2012). Study of the effect of copper doping on the structural and optical properties of $\text{Cd}_{0.7}\text{Zn}_{0.3}\text{S}$ nanocrystalline thin films prepared by chemical bath deposition. International Journal of Science and Technology, 2, pp. 707-712.

- Anuar, K., Zainal, Z., Hussein, M.Z., Saravanan, N. and Haslina, I. (2002). Cathodic electrodeposition of Cu_2S thin film for solar energy conversion. *Solar Energy Materials and Solar Cells*, 73, pp. 351-365.
- Anuar, K., Ho, S.M., Tan, W.T., Atan, S., Kelvin and Saravanan, N. (2011). Chemical bath deposition of ZnSe thin films: SEM and XRD characterization. *European Journal of Applied Sciences*, 3, pp. 113-116.
- Gadave, K.M. and Lokhande, C.D. (1993). Formation of Cu_xS films through a chemical bath deposition process. *Thin Solid Films*, 229, pp. 1-4.
- Jayasree, Y., Chalapathi, U. and Sundara Raja, V. (2013). Growth and characterization of tin sulphide thin films by chemical bath deposition using ethylene diamine tetra-acetic acid as the complexing agent. *Thin Solid Films*, 537, pp. 149-155.
- Liu, J., Wei, A.X., Zhuang, M.X. and Zhao, Y. (2013). Investigation of the $\text{ZnS}_x\text{Se}_{1-x}$ thin films prepared by chemical bath deposition. *Journal of Materials Science: Materials in Electronics*, 24, pp. 1348-1353.
- Muthukumar, S. and Muthusamy, M. (2012). Structural, optical, FTIR and photoluminescence studies of $\text{CdS}_{1-x}\text{Se}_x$ thin films by chemical bath deposition method. *Journal of Materials Science: Materials in Electronics*, 23, pp. 1647-1656.
- Nascu, C., Pop, I., Ionescu, V., Indrea, E. and Bratu, I. (1997). Spray pyrolysis deposition of CuS thin films. *Materials Letters*, 32, pp. 73-77.
- Podder, J., Kobayashi, R. and Ichimura, M. (2005). Photochemical deposition of Cu_xS thin films from aqueous solutions. *Thin Solid Films*, 472, pp. 71-75.
- Ubale, A.U., Sakhare, Y.S., Bhute, M.V., Belkhedkar, M.R. and Singh, A. (2013). Size-dependent structural, electrical and optical properties of nanostructured iron selenide thin films deposited by chemical bath deposition method. *Solid State Sciences*, 16, pp. 134-142.
- Ulutas, C., Guneri, E., Kirmizigul, F., Altindemir, G., Gode, F. and Gumus, C. (2013). $\gamma - \text{MnS}$ thin films prepared by chemical bath deposition: Effect of bath temperature on their physical properties. *Materials Chemistry and Physics*, 138, pp. 817-822.

Zhao, Y., Yan, Z.Q., Liu, J. and Wei, A.X. (2013). Synthesis and characterization of CdSe nanocrystalline thin films deposited by chemical bath deposition. *Materials Science in Semiconductor Processing*, 16, pp. 1592-1598.

Zhou, L.M., Hu, X.F. and Wu, S.M. (2013). Effects of deposition temperature on the performance of CdS films with chemical bath deposition. *Surface and Coatings Technology*, 228, pp. S171-S174.

Zhuge, F.W., Li, X.M., Gao, X.D., Gan, X.Y. and Zhou, F.L. (2009). Synthesis of stable amorphous Cu₂S thin film by successive ion layer adsorption and reaction method. *Materials Letters*, 63, pp. 652-654.

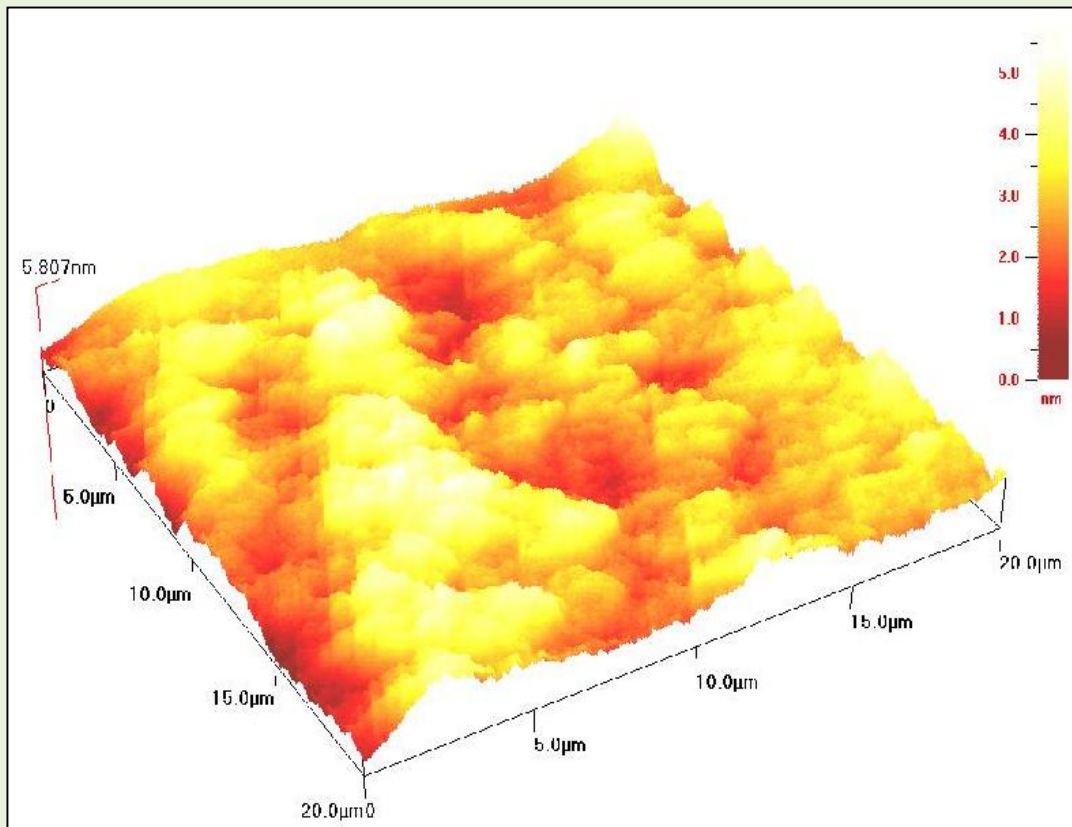


Figure 1: Atomic force microscopy image of CuS thin films prepared at pH 1

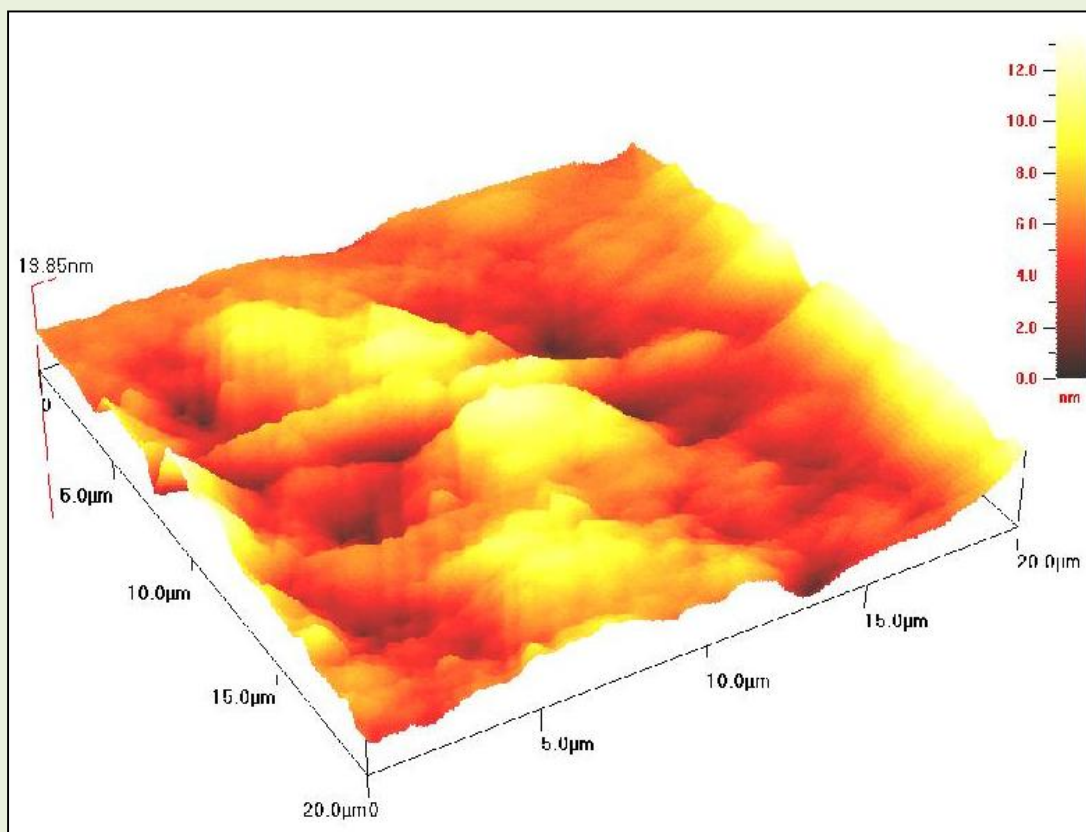


Figure 2: Atomic force microscopy image of CuS thin films prepared at pH 1.5

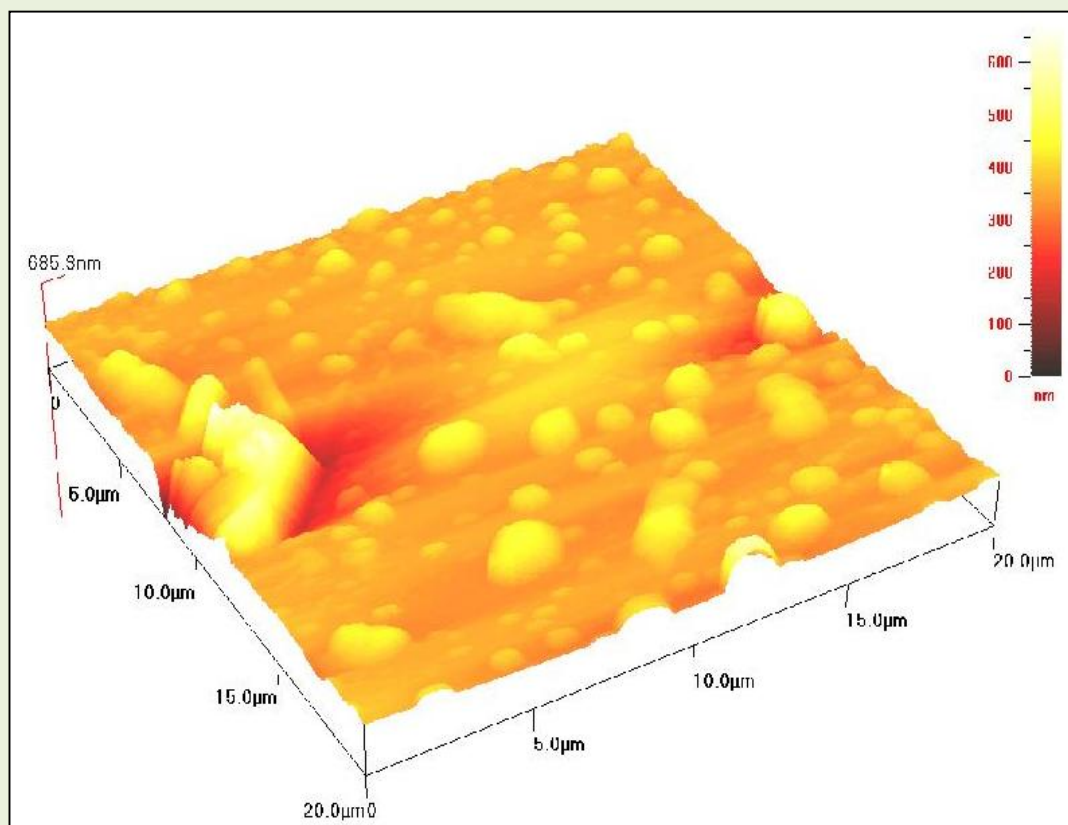


Figure 3: Atomic force microscopy image of CuS thin films prepared at pH 2

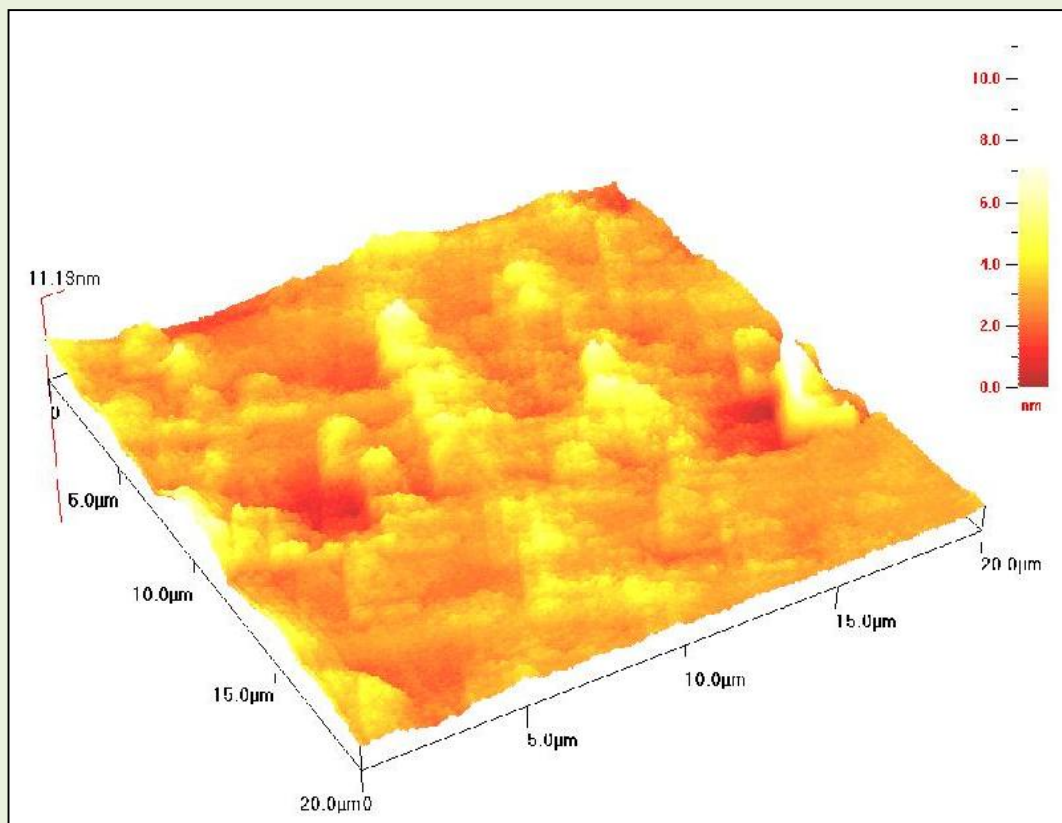


Figure 4: Atomic force microscopy image of CuS thin films prepared at pH 2.5