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Structural behavior of rf magnetron sputtered cupric oxide (CuO) films

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ABSTRACT

In this paper CuO thin film, has been deposited using RF sputtering technique, then the thin film has been characterized by XRD, the results showed that the strongest peak was at 35.5966 degree, FWHM was at 0.64 degree, lattice constant was 2.52 Aº and the average grain size was 13.62 nm. AFM analysis showed that the increasing of temperature led to increase of roughness average from (3.77 to 15.7) nm, root mean square from (4.66 to 18.8) nm and ten points height from (22.6 to 52.6) at 250,300,350 and 400 ºC respectively. On the other hand, granularity accumulation distribution charts showed average diameter has varied from (57.42, to 135.41) nm with grain numbers per line (364, to 135) respectively.

1. Introduction

CuO or Cupric (tenorite) Oxide is one of the three main forms of Cooper Oxides[6]. It is a p-type could be produced from Cu metal oxidation[5], the band gap of CuO is ranged between 1.2-2.0 eV[11]. CuO has witnessed a variety of applications such as storage media, gas sensors, semiconductors, etc [9]. Due to high photoconductivity and photochemistry of CuO , it was using in solar cells industry[13], in addition of its high optical transmittance and low electrical resistivity[12] . Many techniques has been developed to fabricate CuO thin film , for example; electrochemical deposition[3] , DC magnetron sputtering[8] rf magnetron sputtering[1] , vacuum annealing[7], Pulsed Laser Deposition[2][14], chemical vapor

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deposition[4]. In this research, CuO thin film has been deposited by using RF magnetron sputtering which allow researcher to control sputtering power and pressure and substrate temperature, then to sputter more thin films under different substrate temperature to study surface morphology.

2. Materials and Methods

CuO thin film has been deposited as explained below: cleaned the pure cooper target 99.95% (5cm dia.) with soft sanding and ethanol, then placed cleaned substrate with deionized water and ethanol using ultrasonic on sample stage (18.5cm dia.) at the center of chamber (30*30)cm, closed the cover of the chamber and engaged the doorknob clock wise until tightened firmly, started the rotary vacuum, then turned on the pressure gauge, after reaching pressure to value of 1.5 $*10^{-3}$ mbar, the sputtering system (13.56Hz) has been turned on, the heat has been up to 150 ºC degree. Then let Argon gas (purity 99.99%) to flow through chamber, until the working pressure reached to 4.5$*10^{-2}$ mbar, after that turned on the water chiller, RF supply has been turned on(200W.Max) , loading the power up to 150 W. and reducing the reflected power until reached zero to generate most effective plasma. At this stage the deposition process has been started, the time of deposition lasted for 40 minutes. The system has been shut down and cooled to the room temperature and substrate has been taken outside of chamber. The deposited thin film has been characterized with XRD to study structural behavior. The same process has been repeated with different temperatures as four more thin films has been deposited at 250, 300, 350, 400 ºC. Finally deposited thin films has been analyzed with AFM to study morphology of thin films surfaces.

3. Results and Discussion

Structural Properties: thin film has been characterized by XRD(SHIMADZU Japan XRD 6000) , the results showed in Table (1) below.

Table (1) XRD structural parameters of CuO

Compound	2 θ (deg)	FWHM (deg)	Lattice Const.(Aº)	G.S (nm)
CuO	35.5966	0.64	2.52	13.62

Also the XRD test results (Card 00-002-1040) has stated that CuO which is polycrystalline (Monoclinic), the strongest peaks were (2θ= 35.5966, 38.7825, 48.6503), that is similar to [hkl= (002), (111), (-202)] respectively. Other peaks can be observed at (24.9453) degree for the glass and (2θ= 36.4352, 42.3220, 61.3967) degree for Cu_2O, as shown in Fig. (1).

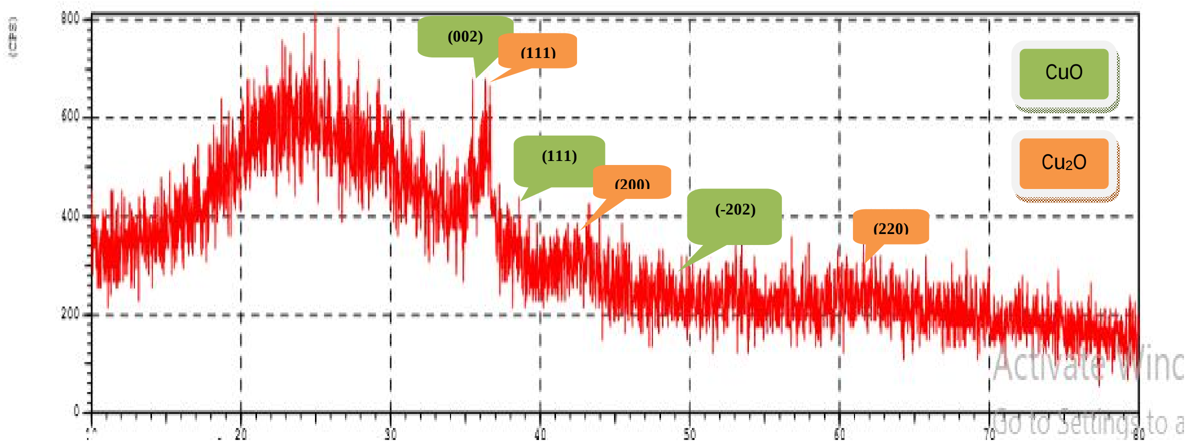

Fig. (1): XRD Patterns of CuO thin film

Average of Grain Size has be calculated by Scherer's Crystallite Size Formula[10].

$$G.S = \frac{k\lambda}{\beta\cos\theta} \quad (1)$$

where k is called shape factor which have value of 0.94, λ is the wavelength, ß is FWHM (full-width at half- maximum value) and θ is Bragg's diffraction angle.

Morphology: in order to study the effect of temperature change on structural behavior, four CuO thin films have been deposited on glass substrates with following parameters (working Pressure: 4.5*10-2 mbar, deposition Time: 40 min. , and applied Power: 150 Watt.), those films were deposited in different temperatures, (250, 300, 350, 400)ºC, then the samples have been analyzed with AFM (SPM AA3000) to analyze surface morphology of the films as shown in Table (2).

Table (2) AFM results of CuO thin films prepared under different temperatures

Sample #	Temp. ºC	Roughness Average nm	Root Mean Square nm	Ten Points Height nm
1	250	3.77	4.66	22.6
2	300	4.56	5.63	28.9
3	350	6.41	7.76	20.1

4	400	15.7	18.8	52.6

Also AFM images shows CuO films at different temperatures fig(2).

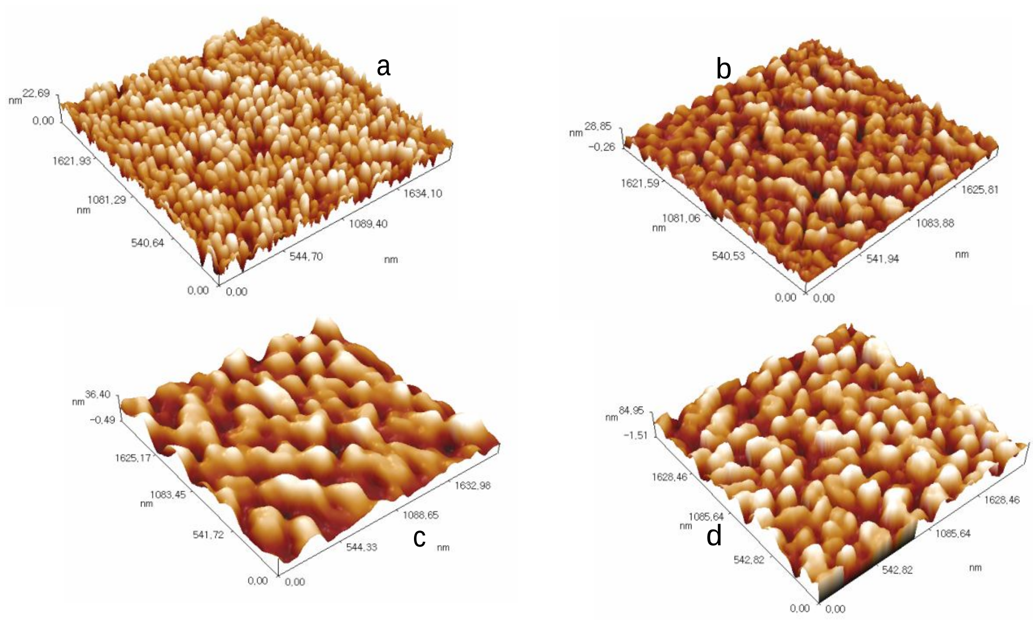

Fig.(2) AFM images of CuO prepared film at (a.250, b.300, c. 350, d.400)ºC

Also granularity accumulation distribution report has showed average grain diameter and number of grains per line for each sample . As shown in Table(3)and Fig(3).

Table (3) Average diameter and number of grains per line

Sample #	Temp. ºC	Average Diameter of grain nm	No. of grains per line
1	250	57.42	364
2	300	71.56	145
3	350	161.61	35
4	400	135.41	135

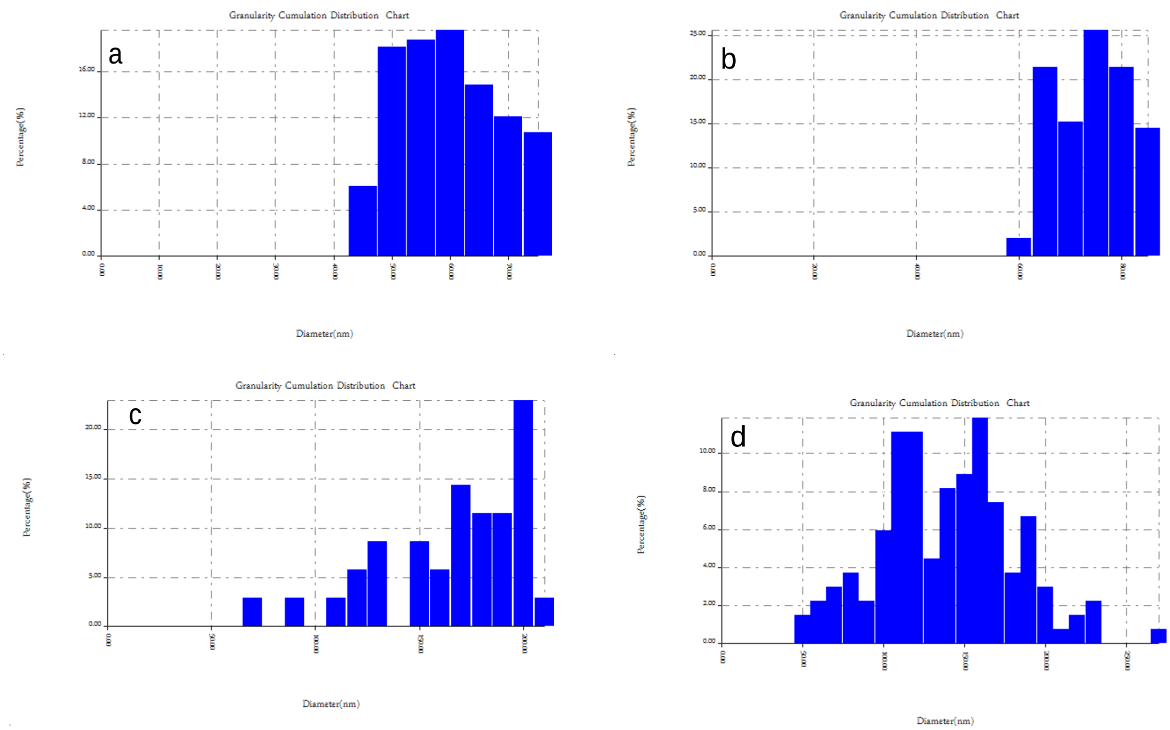

Fig. (3): Granularity Cumulation Distribution of CuO prepared thin film at (a.250, b.300, c.350, d.400) ºC

In general, it is clear from tables and images above that the roughness average and RMS have increased when the temperature of the sample is increased, also ten points height as well.

4. Conclusion

Firstly, CuO thin film, has been deposited on glass substrates using RF sputtering technique at applied power 150W, working pressure 4.5*10-2mbar , substrate temperature 150ºC for 40 minutes, then the thin film has been characterized by XRD, the results showed that strongest peak was at 35.5966 degree, and FWHM was 0.64 degree, lattice constant was 2.52 Aº, and the average grain size was 13.62 nm, then another four samples have been deposited with the same previous parameters, but in different temperatures. Then those samples have been analyzed with AFM to study the effect of increasing temperature on morphology of surface, results showed that the increasing of temperature led to increase of roughness average from (3.77 to 15.7) nm, root mean square from (4.66 to 18.8) nm and ten points height from (22.6 to 52.6) at 250,300,350 and 400 ºC respectively. On the other hand, granularity accumulation distribution charts showed average diameter has varied from (57.42, to 135.41) nm with grain numbers per line (364, to 135) respectively. Which means at higher temperature roughness of thin film surface will increase due to increasing of grains size.

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