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Design of D.C. planer magnetron sputter for preparing copper-oxide thin films

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Abstract

A vacuum chamber (made up of stainless steel) and magnetron target (made up of copper) have been designed, manufactured and assembled. The sputtering target of pure copper make (dimensions 13 cm) involves cooling with (dimension 6.5 cm, with depth 3 cm) and it contains a circular magnet (6 x 3cm, an outer and inner diameter respectively). The chisel radius of target sputtering was about 9-11 cm. The systems have been tested with the pressure of $6x10^{-5}$ mbar. The effective parameters of magnetron sputtering system operation have been collected including the pressure (75, 150, 300 and 450 mTorr), distance (3, 4, 5 and 6 cm) and voltage difference (218 to 310 Volt) between the target and the substrate. Finally, we have got maximum thickness on pressure point 300 mTorr, 3 cm distance and 220 Volt. An oxide copper thin film was deposited on different substrate including glass and optical the characteristics *viz*. optical energy gap, refractive index, extinction coefficient and real and imaginary dielectric constants were calculated.

Keywords: Optical properties, sputtering, thin films, DC magnetron sputtering.

Introduction

The phenomenon of sputtering has several advantages in film deposition. There is no direct heating of the material as in evaporation methods. Therefore, there is no reaction between the source and crucible place. The deposition rate is linearly dependent upon the bombarding ion flux, whereas, in evaporation there is an exponential dependence of rate on source temperature. The average arrival energy at the substrate is higher for sputtered atoms (about 10 eV) than for evaporated atoms (about 0.25 eV at 300 K) and this is usually the reason for enhanced adhesion (Poate *et al*., 1978; Vesson & Kern, 1985; Proud, 1991; Suhail, 1992).

When sputtering is used with a transverse magnetic field, several important modifications of the basic processes should be produced. Target generated secondary electrons do not bombard the substrate because they are trapped in cyclonical trajectories near the target and thus do not contribute to increase substrate temperature and radiation damage. This allows the use of low temperature substrate (for example, plastic) and surface sensitive substrate (for example, metal oxide semiconductor devices), without adverse effects. In addition, it produces higher deposition rates than conventional sources and tends itself to economic, large area industrial application. There are cylindrical, conical and planar magnetron sources, all with particular advantages and disadvantages in specific applications. As in the case of the other forms of sputtering, magnetron sources can also be used in a sputtering mode (Thornton, 1978; Masissel & Glang 1979; Lieberman & Allan Lichtenberg 1994; Smith, 1995). The properties of a planar magnetron source can be summarized as follows (Hass, 1966):

A high rate of sputtering is obtained due to the confinement of the plasma close to the target surface. The heating of the substrate by stray electrons is low. In many cases this is the limiting factor for high rate conventional sputtering. Sputtering can take place at a low gas pressure because of the increased path length of electrons within the plasma and prevention of their escape. When this sputtering source is used in the presence of a reactive gas to deposit a compound film, further advantages ensue: 1) The rate of deposition can easily be controlled to allow balanced processes to be maintained. 2) The gas pressures required for sputtering and for producing reactive processes are compatible. 3) Alloy targets can easily be sputtered to give films of similar metal ratios.

The first practical magnetron sputtering cathode was developed by Chapin in 1974. Latter, Nyaiesh (1981) studied the glow discharge characteristics and sputtering behavior of a variety of metals. In 1985 Nyaiesh and Elphich designed a planar magnetron using electromagnetic coils. Rastogi *et al.* (1987) used U shaped demountable magnets such that the magnetic field lines run parallel to the target. Spencer *et al.* (1987) and Spencer and Howson (1988) showed that increasing field up to around 250 Gauss gave sharp improvement in the operating characteristics. In 1992 Suhail studied the reactive magnetron sputtering-plasma and oxide films. They have analyzed the reactive sputtering process by glow discharge characteristics, the optical properties and structure of titanium and zirconium.

Panwar *et al*. (1997) studied the design and performance of a scanning magnetron sputtering target. The magnetic field on the target surface was scanned by electromechanical movement of magnet array along the length using a step motor and studied the voltage-current characteristics of stainless steel and Cu. Posadowski (1999) studied the self-sputtering with DC magnetron source. Veldeman *et al*. (1999) studied the influence of magnetron configuration on CoCr films sputtered on flexible substrates for different pressures (10-65 mTorr).

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Seranni *et al*. (2000) studied the plasma characterization of a DC closed field magnetron sputtering device. Berger (2001) studied tribological characterization of magnetron sputtering TiB_2 and Cr/CrN coatings. Yamagishi *et al*. (2003) reported the effect of Oxygen partial pressure on the optical properties of DC magnetron sputtered TiO_2 films by using different oxygen partial pressures in the range ($1x10^{-4}$-$5x10^{-3}$ mbar) and at constant temperature of 573 ^{o}K. Bhuvaneswari *et al.* (2003) studied Zirconium Nitride films deposited by reactive magnetron sputtering. They showed that the current-voltage characteristics affect pressure, sputtering rate and the structures. Knizikevicis (2010) studied the reactive sputter deposition of titanium dioxide films in Ar + O_2 atmosphere. The processes of sputtering, adsorption and heterogeneous reactions are included in the model. Singh *et al*. (2010) made thin film of tantalum and titanium deposited on glass substrate using reactive magnetron sputtering; they showed some of optical properties affect the discharge voltage on the transmittance of the films.

The aim of our work is to design, build and constrict a vacuum chamber and magnetron target with water cooling, that is suitable to prepare thin film by DC magnetron sputtering. The main parameter which affect the plasma (as we studied) like gas pressure , distance between the target and the substrate, the voltage and the current were studied for some properties of Cu thin film which is prepared by DC planar magnetron sputtering technique.

Experimental technique

The system designed was similar to the Edward's system and made by the Heavy Engineering Equipments State Company (H.E.E.S.Co.). The system has several parts, which are:

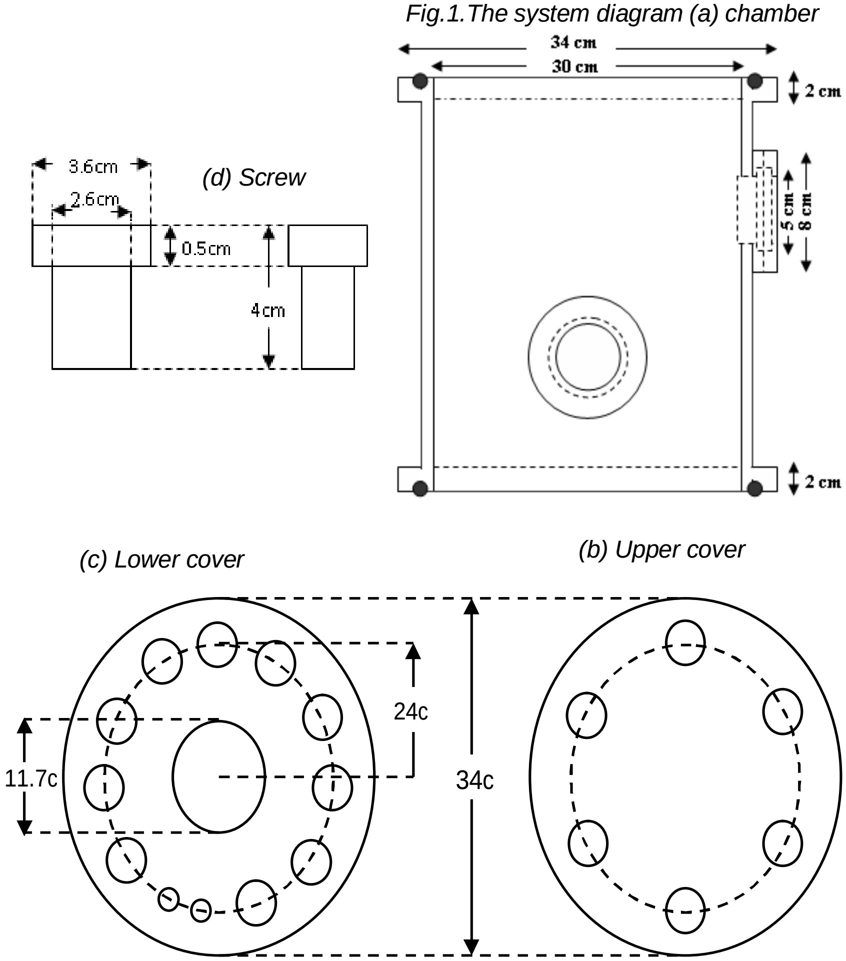
Vacuum chamber

The chamber is made up of stainless-steel designed in a cylindrical shape with two open ends. It has 30 cm in diameter, 36 cm in height and 0.6 cm thickness in the body but the ends are 2 cm, which have a groove in the medial of dimension 0.5 cm width and 0.3 cm depth for O-rang. It contained two Pyrex windows with thickness of about 2 cm and diameter of about 5 cm in different levels (Fig.1a).

Upper cover

It is designed as a flange shape with 2 cm thickness and 34 cm diameter which contains 6 holes (3.6 cm the first diameter and 2.7 cm the second diameter). They lay about 12 cm from the centre of the flange as shown in Fig (1b).

Fig.1.The system diagram (a) chamber



Lower cover

The dimensions of lower cover are similar to that the upper cover but it contain 10 holes with dimensions similar to that of the upper and have 2 holes (with the first diameter 1.4 cm and the second diameter 1.1 cm). The flange has hole in the center with diameter of 11.7 cm to high vacuum (oil pump) (Fig.1c).

The screw

Designed two kinds of screw from Aluminum (Al). One with the 3.6 cm upper diameter; thickness 0.5 cm and the lower diameter of 2.6 cm. In another type, the upper diameter was 1.4 cm with thickness 0.5 cm and the lower diameter 1 cm. All the screws were of 4 cm in length (Fig.1d).

The target

We designed and constructed a target from copper which contains two parts; the first part as a flange used as a cover with thickness of 1 cm and 13 cm diameter. The second part has the same diameter as the first part but it contains cooling pond with 4 cm height and 6.5 cm diameter. A groove with diameter 7.5 cm, depth of 0.1 cm and width of 0.15 cm which has used for O-ring. The cooling pond contains a magnet with circular shape of 6 cm as outer diameter, 4 cm as inner diameter and 3 cm height used for producing magnetic field. The cooling pond has 2 pipes one for inlet and another for outlet water (Fig.2). The chamber was designed and used with Edward's vacuum

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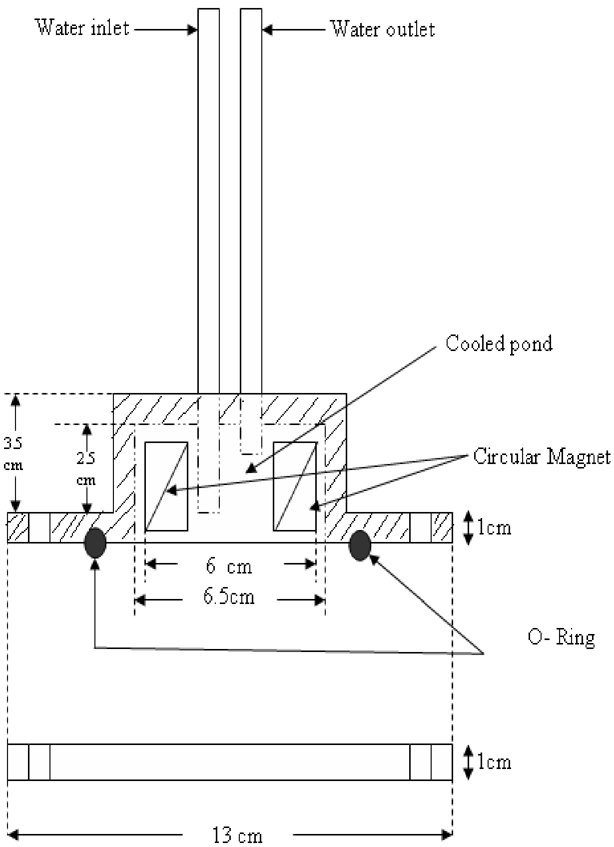
system pumped by a mechanical pump to a base pressure of 10^{-3} mbar, after which, it was pumped by oil diffusion pump to a base pressure of 10^{-5} mbar.

Fig.2.The target diagram



The films were coated using dc magnetron sputtering system, with a 300 lit /sec diffusion pump and 200 lit /min rotary vacuum pump combination. The system gives an ultimate vacuum of 5 x 10^{-5} mbar. The pressure was monitored with a pirani-penning gauge combination. The target disk was mechanically clamped to a water cooled copper cathode and the assembly, was used as a sputtering target. The variable power supply for magnetron is of high voltage type with a capacity to draw 0.6 Amps current at 8 kV. IOLAR2 grade argon (99 %) has been used as sputtering gas. Pre sputtering was done for 15 min for copper targets in pure argon to remove any surface oxide from the sputtering track of the target that had been formed on the target surface during exposure to air.

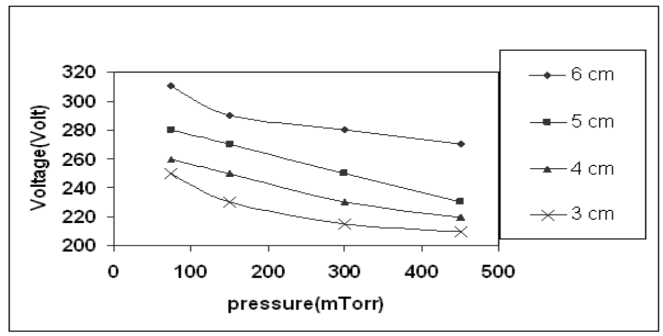
During sputtering,the system was evacuated to $5x10^{-5}$ mbar. Then argon gas was admitted into the chamber through the needle valve and the required partial pressure was set and allowed to stabilize. We used different pressure of Argon which is about $1x10^{-1}$ to $6x10^{-1}$ mbar and with varied distance between the target and the substrate.

Optical interference fringes" have been used to determine the film thickness. X-ray diffract meter analysis was used to determine the crystalline structure of material thin films sample. The optical transmission spectra of films prepared on glass substrate was measured using Cintra5 spectrophotometer over the wavelength range (180-1100 nm). The optical band gap was determined using Tauc's formula. The transmittance and absorption spectra curves were used to calculate the refractive index, extinction coefficient and dielectric constant of (CuO) thin film.

Result and discussion

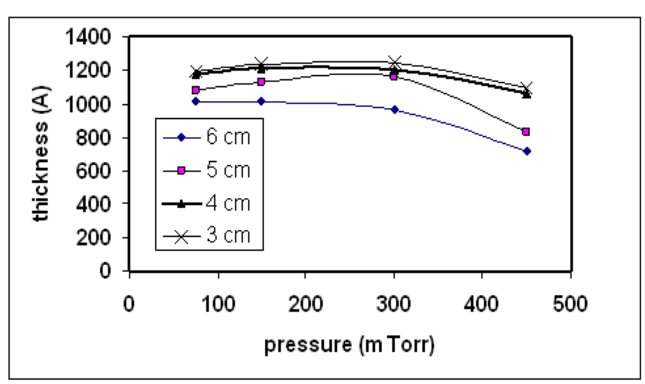
Magnetron sputtering parameters

Fig.3. The variation of target voltage as a function of pressure of different distance



There are several parameters that influence magnetron sputtering process such as distance, pressure and thickness. Fig.3 shows this variation of target voltage as a function of argon pressure for different distances. The target voltage decreases progressively with increasing argon pressure which can be attributed to the target voltage decreases when argon pressure increases. Pressure for a given amount of applied power, higher sputtering rates will usually be obtained under conditions which favor high current and low voltage. This is because the number of ions striking the target is directly proportional to the current. With increasing pressure the current in the discharge increases while the voltage decreases so it is not surprising the deposition rate will usually increase with pressure.

Fig.4.The variation of film thickness as a function of pressure



The erosion track which depends on the distribution of the magnetic field on the target is a ring with an internal diameter of 9 cm and an external diameter of 11 cm. Maximum erosion is seen in a track of about 3 cm width. Fig.4 shows the thickness of film as a function of pressure. The film thickness increases with increasing the argon pressure until it reaches the maximum value of thickness at 300 mTorr then decreases. This may be attributed to increasing the number of collision by increasing the pressure.

At pressure 300 mTorr, according to Pastern's law, the atoms sputtered will be collided with each other, so the atoms will not be going to the substrate. As the pressure increases, an increasing fraction of the material that leaves the target does not reach the substrate but returns to the former through back diffusion.

Most of the above observations lead us to believe that any understanding of the sputtering process would require the analysis of plasma in such processes. This would not only help in improving the control over process parameters but also further improve the technique to obtain quality films.

X-ray diffraction analysis

The investigation of X-ray diffraction (XRD) has been studied of the copper (Cu) thin films with different parameter like the distances and pressures.

The (XRD) of copper films deposited on glass substrate at different distance (d= 6, 5, 4, 3 cm) and pressure (P = 75, 150, 300, 450 mTorr) (Fig.5,6,7,8)respectively. The Copper oxide (CuO) nature of the film may be attributed to its ability to oxidation at normal condition. The oxidation increased where the pressure increased and the distance decreased.

The Optical energy gap (E_g^{opt}) measurement

We examined $(\alpha h\nu)^{1/r}$ versus photon energy and selected the optimum straight line fitting consistent with the dependence of $(\alpha h\nu)^2$. Taking a straight line relationship at high absorption data ($\alpha \geq 10^3$) cm^{-1} the intercept at $[(\alpha h\nu)^2 = 0]$ gives the value of energy gap for direct transition.

Fig.5.Structure of CuO at distance 6 cm at different pressure (a)75mTorr (b)150 mTorr (c) 300 mTorr & (d)450 mTorr

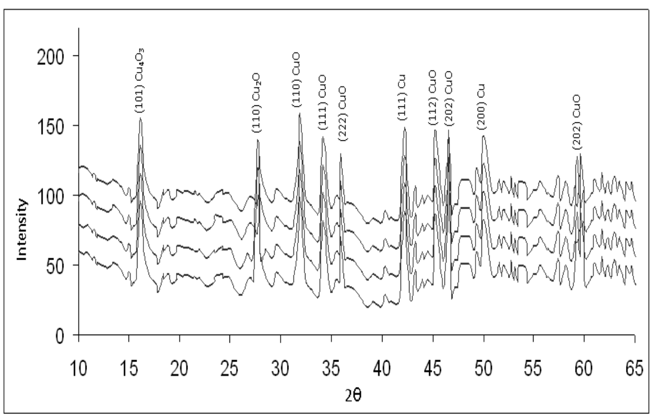


Fig.6.Structure of CuO at distance 5 cm at different pressure (a) 75mTorr (b)150 mTorr (c)300 mTorr & (d) 450 mTorr

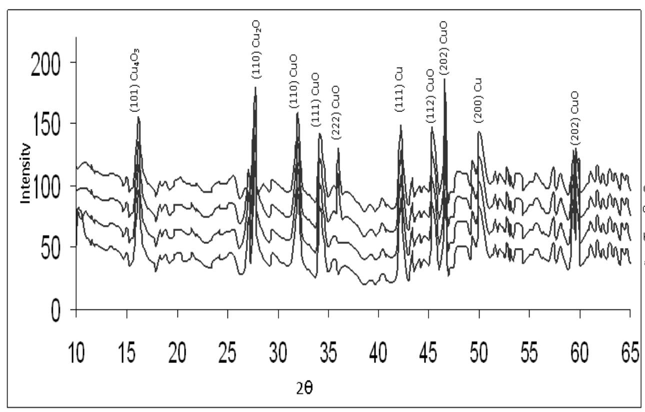


Fig.7.The structure of CuO at distance 4 cm at different pressure (a)75mTorr (b)150 mTorr (c)300 mTorr & (d) 450 mTorr

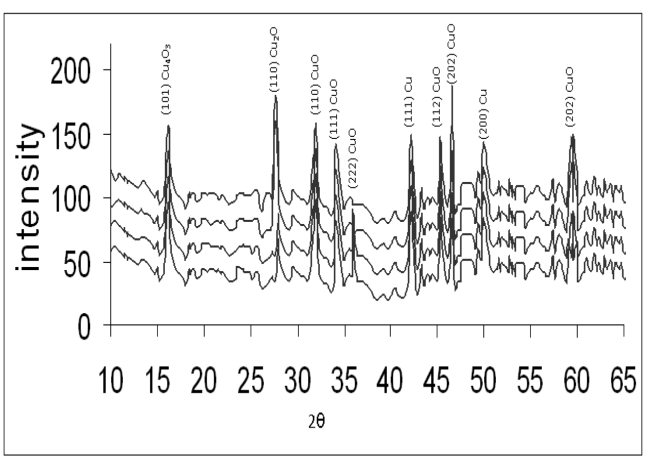
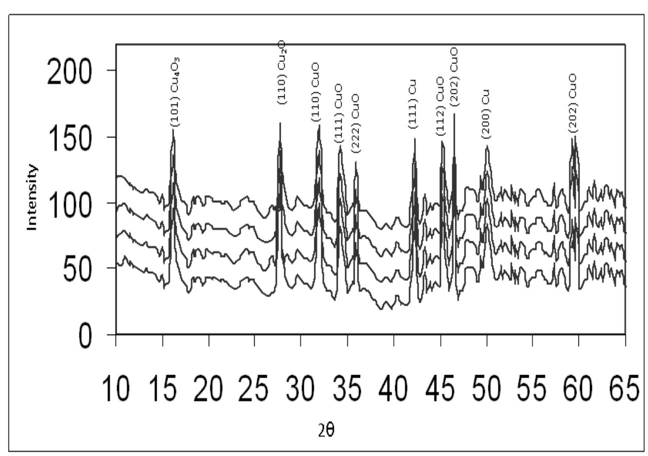


Fig.8. Structure of CuO at distance 3 cm at different pressure (a)75mTorr (b)150 mTorr (c) 300 mTorr & (d)450mTorr



The value of the energy gap was about 2.5 -2.85 eV. Table 1 shows that the optical energy gap decreased with increasing pressure while it increased with decreasing distance. This shift in the optical energy gap is due to the oxidation surfaces of copper films.

Table1.The values of distance d, pressures P, optical energy gap E_g, refractive index n, extinction coefficient k, real parts of dielectric constant ε_r and imaginary parts of dielectric constant ε_i for CuO films

	P (mTorr)	E_g (eV)	n	k× 10^{-4}	ε_r	ε_i× 10^{-4}
6	75	2.7	1.511	0.65	2.3	1.2
	150	2.65	1.513	3.9	2.3	8
	300	2.6	1.515	2.7	2.3	6
	450	2.5	1.514	1	2.3	1.2
5	75	2.8	1.513	6.1	2.3	12
	150	2.65	1.514	19	2.3	44
	300	2.5	1.516	6.2	2.3	12
	450	2.7	1.514	8	2.3	15
4	75	2.85	1.602	11	2.4	24
	150	2.55	1.604	2,7	2.4	30
	300	2.6	1.605	5.1	2.4	6
	450	2.7	1.604	7.5	2.4	3
3	75	2.75	1.620	5.5	2.4	12
	150	2.6	1.632	5.1	2.4	12
	300	2.85	1.650	3.8	2.5	14
	450	2.6	1.552	6	2.1	12

Optical constant

Refractive index

The refractive index (n) is one of the fundamental properties of materials, because it is closely related to the electronic polarizability of ions inside the material and depends on the thickness and structure of the films. The reflectance (R) was calculated from transmittance and absorbance spectra and then used to evaluate the refractive index (n). The dispersion refractive index (n_λ) can be determined using a suitable computer program in the region of fundamental absorption edge.

The refractive index are shown in Table 1 at varied distance (d = 6, 5, 4, 3 cm) pressure of argon gas (P = 75, 150, 300, 450 mTorr) for each distance as a function of wavelength at λ=550 nm.

Extinction coefficient

The behavior of the extinction coefficient (k) of oxide copper thin films as a function of wavelength for different distances and pressures have similar behavior to the corresponding absorption coefficient. From the Table (1) we can see the value of extinction coefficient decreased with the increased in the pressure at the distance d = 6 cm while at another distances we see the k decreased when the pressure =150 mTorr and increased with the increased the pressure.

Dielectric constant

The real (ε_r) part and the imaginary part (ε_i) of the dielectric constant for as-deposited oxide copper films for different value of distance (d = 6, 5, 4, 3 cm) and different argon gas pressure are shown in table (1). One can observe that (ε_r) has a similar behavior to n and (ε_i) has a similar behavior to k.

Conclusions

We design the vacuum chamber (30 cm diameter, 36 cm heights and 0.6 cm thickness) with target dimensions of 13 cm diameter and 7 cm heights. We used circular magnets inside the target. Thin films were prepared at different argon gas pressures and different distances on glass substrate. The results deduced from this work include: Sputtering of oxide copper in argon gas resulted in interesting changes in the glow discharge characteristics. As the argon pressure increased, the target voltage decreased. The thickness decreased after 300 mTorr for all distances. The conditions to prepare thin films were 300 mTorr argon pressure, 220 V target voltage and 3 cm the distance between target and substrate. X-ray diffraction confirms the polycrystalline structure of copper deposited on glass prepared at different pressure and distance. The optical energy gap decreased with increasing pressure while it increased with decreasing distance. The refractive index and extinction coefficient increased with increasing argon pressure and decreasing distance. The real and imaginary dielectric constants depend on refractive index and extinction coefficient respectively.

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