# Chapter 5 Structural characterisation of copper oxide by X-ray diffraction

# Capítulo 5 Caracterización estructural de óxido de cobre por difracción de rayos-X

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**DOI:** 10.35429/H.2021.9.1.70.96

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A. Ledesma (Coord.). Engineering Science and Technology. Handbooks-©ECORFAN-México, Estado de México, 2021.

**Abstract**

In this work, the study of the structural characterization of copper oxide by the X-ray diffraction technique is presented. To obtain layers of copper oxide, sputtering and thermal oxidation techniques were combined. The average crystal size was calculated for the sputtered copper samples. For the copper oxide films obtained by thermal oxidation, both the crystal size and the texture coefficient were calculated. The crystalline quality was poor for layers obtained by sputtering. Thermal oxidation carried out on these films transformed its structure to the copper oxide phase known as cupric oxide.

**Copper oxide, X-ray diffraction, Average crystal size**

**Resumen**

En este trabajo se presenta el estudio de la caracterización estructural del óxido de cobre mediante la técnica de difracción de rayos X. Para la obtención de capas de óxido de cobre se combinaron las técnicas de sputtering y oxidación térmica. Se calculó el tamaño medio de los cristales para las muestras de cobre pulverizadas. Para las películas de óxido de cobre obtenidas por oxidación térmica, se calculó tanto el tamaño de los cristales como el coeficiente de textura. La calidad cristalina era pobre para las capas obtenidas por sputtering. La oxidación térmica realizada en estas películas transformó su estructura en la fase de óxido de cobre conocida como óxido cúprico.

**Óxido de cobre, Difracción de rayos-X, Tamaño promedio de cristal**

## 5.1 Introduction

Nowadays, technology has presented great advances in conjunction with research and experimentation, covering needs by exploiting natural resources to the maximum. Copper has been used since antiquity for its physical and chemical properties that enhance it taking countless technological applications. Together with its oxides (CuO) and ($Cu_2O$), it maintains a more sophisticated development in its use in areas such as electronics, optoelectronics, etc. Among its advantages is the cost-effective reusability at the end of its useful life. The alloys are generally hard, strong and corrosion resistant. Thin films have achieved great specialisation in their study. They are formed by the growth of atoms or molecules that impinge on the surface of a substrate, with a thickness ranging from 1-100 nm. They can be manufactured by different methods depending on the material to be deposited and are used in optoelectronic devices, wear-resistant coatings, insulation, conductivity of electronic circuits, microelectronics such as semiconductor heterostructures and electrodes, among others. The production of CuO films is possible through a combination of methods such as sputtering and thermal oxidation. Because sputtering is a technique capable of producing thin films of almost any material and any type of substrate capable of withstanding high temperatures, it has been one of the most widely used. There are two types of sputtering (reactive and non-reactive). Reactive sputtering uses a mixture of gases, usually a reactive gas (hydrogen) and an inert gas (argon), which react with the target used in the deposition inside the chamber. In contrast to the non-reactive method, only the inert gas is used or the vacuum is simply evaporated and the target is deposited on the undisturbed substrate. The present work focuses specifically on non-reactive sputtering performed under vacuum without the use of an inert gas. When a material exhibits a number of properties useful for performing a certain function, analysis is carried out by a technique or method. Numerous structural characterisation techniques have been used in the field of materials and nanomaterials. X-ray diffraction is the most widely used analytical technique for the structural characterisation of samples, whereby it is possible to observe crystalline structures. This technique has excelled in different areas such as chemistry, mineralogy, biology and areas related to materials science, resulting in a positive impact on their study. The phenomenon occurs when a wave encounters an obstacle or an opening in its propagation of a size comparable to its wavelength. This tool has a wide range of data of relevant interest to analyse and study the determination of the geometry of each cell. The technique offers different utilities such as studying the deterioration, restoration and treatment of materials, as well as their properties and composition. The properties of materials are conditioned by the nature of the atoms that compose them and the way in which they bond with others, seeking maximum stability and a configuration with the lowest possible energy. Thus, many materials have a certain arrangement and organisation of atoms.

The so-called crystalline materials are composed of a spatial network of atoms distributed periodically and without any gaps, there are 14 possible types of Bravais networks. From the following work it is expected to show the importance of the experimental conditions used to obtain CuO. This is done by the combination of two techniques used in the synthesis due to the efficiency they offer together. In addition to optimising the production process in order to obtain samples analysed by means of a non-destructive structural characterisation technique.

## 5.2 Experimental methodology

The production of CuO deposits needs to fulfil certain criteria in order to offer high optimisation and excellent properties when used in different areas of study. This work focuses on the two aforementioned techniques to obtain CuO. The process of obtaining CuO is shown below.

*Deposition of Cu using the sputtering technique*

In this process, the deposition is carried out using the sputtering technique with the equipment (Agar auto sputter coater model 108A), using a Cu target with a purity of 99.9%. The Cu is deposited on the substrate forming nanostructures and subsequently thin films. Vacuum was performed in the chamber with a vacuum pump at 0.12 mb. Depending on the target-substrate distance and the deposition time the sample turns different shades.

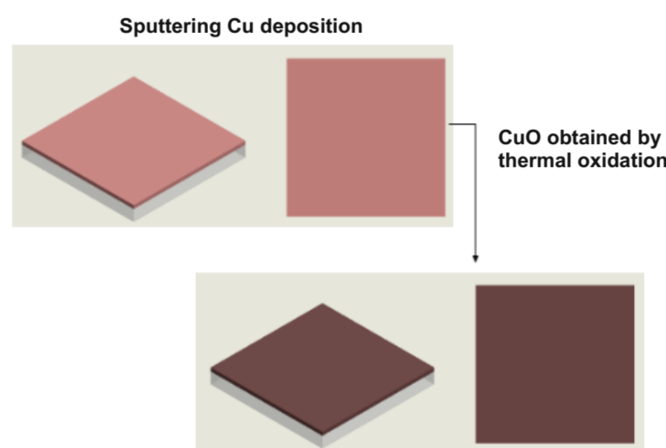
*Thermal oxidation of Cu*

This technique is complementary to sputtering. In this stage, the Cu deposit is placed inside a muffle at a high temperature (the temperatures used in oxidation are 500ºC and 900ºC). The equipment is composed of a closed chamber covered by a refractory material that allows a uniform heat delivery to the interior. The Cu undergoes an oxidation process as a result of the temperature used.

*Obtaining CuO by the combination of two techniques sputtering and thermal oxidation for samples with short deposition time at 30 mm target-substrate distance*

Deposition was carried out at a 30 mm target-substrate distance in a short deposition time, the sample takes a light colour even if it goes through the oxidation process using a muffle. The Cu deposit does not show much difference compared to the unoxidised sample. The diagram in figure 5.1 shows the process of obtaining CuO.

**Figure 5.1** Production of CuO films by sputtering and thermal oxidation with 30 mm target-substrate spacing in a short deposition period



## 5.3 Results

*XRD Cu diffractograms*

Figure 5.2 shows the diffractograms obtained by XRD. The measurement time and the number of steps that have been executed during the analysis define the clarity and accuracy of the diffraction peaks. The number of steps executed was 3001 steps. In the four diffractograms, a curve appears between 2θ= 22º and 32º indicating the reading of an amorphous "substrate" material where the copper was deposited.

Copper was sputtered for 10 minutes, deposited on a glass substrate by means of a copper target at a target-substrate distance of 30 mm. As a result, a film was obtained which at first sight is not continuous. It presents the phenomenon of transmission, so it is possible to observe the background where it is positioned (M1, M2 and M3). In M1 the analysis was carried out at 0.1 s for each step, this diffractogram does not show any representative peak. The analysis was carried out again increasing the measurement time to 0.5 s for each step represented in the M2 diffractogram where an improvement is noticeable, however, it does not show outstanding peaks, so it was decided to carry out a new XRD analysis at 1 s representative of the M3 diffractogram where the copper peaks are not present.

Normally the standard time in the equipment is 0.1 s for each step; sufficient time for a crystalline material to show its representative peaks. In the diffractograms they do not stand out in comparison to the noise, which is why they were not followed up, and possible influencing factors are presented below:

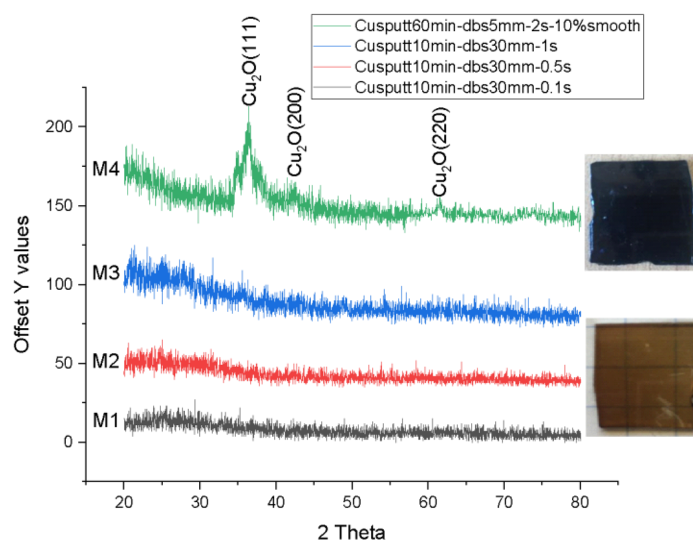
- The layer is not continuous, so the peaks are not distinguishable in the diffractogram.

- The layer may be very thin (the copper film shows the transmission phenomenon).

- The material (copper) may have low crystallinity.

M4 is a comparative analysis of a Cu deposit for 60 minutes, using a Cu target at a target-substrate distance of 5 mm analysed by XRD for 2s. Smooth at 10% was performed to smooth the excess noise presented in the diffractogram, improving the definition of the peaks. The deposited layer is dark due to the increased deposition time, decreased white-substrate distance and longer XRD analysis time. The analysis shows three peaks at 2θ= 36.38º, 42.42º and 61.48 corresponding to (111), (200) and (220) planes of the Cu2O structure (PDF 00-005-0667). The presence of Cu2O in the Cu deposit is due to the possible O-Cu reaction inside the chamber even after vacuum pumping, being an easily oxidised metal, the reaction is evident. The average crystal size taken from the most intense peak (111) corresponding to 2θ= 36.38º is 8.25 nm. Having a small crystal size, Cu nanostructures are considered where thin films are not yet formed.

**Figure 5.2** XRD diffractograms obtained from Sputtering; M1; Cu deposition for 10 min using a Cu blank at a 30 mm blank-substrate distance analysed at 0.1 s, M2; Cu deposition for 10 min using a Cu blank at a 30 mm blank-substrate distance analysed at 0.5 s, M3; Cu deposition for 10 min using a Cu blank at a 30 mm blank-substrate distance analysed at 1 s. M4 Cu deposition for 60 min using a Cu blank at a 5 mm blank-substrate distance analysed at 2 s. Each running 3001 steps.



Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (111) plane of the diffractogram of the M4 Cu deposit.

*T*= Glass size to be calculated

*K*= 0.94

$\lambda$= 0.15402 nm

*B*= 1.04656

$\theta_B$ = 16.18

- Conversion of units

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(1.04656\ )(\pi)}{180} = 0.01826591782 \quad (1)$$

- Replacement

$$T = \frac{(0.94)(0.15402\ nm)}{(0.01826591782\ )\ coscos\ (16.18)} \quad (2)$$

$$T = \frac{0.1447788\ nm}{0.01754242333} \quad (3)$$

$T$ = 8.2530 nm

The crystal size for the (111) plane is 8.2530 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (200) plane of the diffractogram of the Cu M4 deposit.

- Data

T= Crystal size to be calculated

*K*= 0.94

$\lambda$= 0.15402 nm

*B*= 1.48975

$\theta_B$ = 21.21

Conversion of units

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(1.48975\ )(\pi)}{180} = 0.02600104253 \quad (4)$$

Replacement

$$T = \frac{(0.94)(0.15402\ nm)}{(0.02600104253\ )\ coscos\ (21.21)} \tag{5}$$

$$T = \frac{0.1447788\ nm}{0.02423974939} \tag{6}$$

$T\ = 5.9727$ nm

The crystal size for the (200) plane is 5.9727 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (220) plane of the diffractogram of the Cu deposit M4

Data

T= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.98597

$\theta_B$ = 30.74

Conversion of units

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.98597)(\pi)}{180} = 0.01720842283 \tag{7}$$

- Replacement

$$T = \frac{(0.94)(0.15402\ nm)}{(0.01720842283\ )\ coscos\ (30.74)} \tag{8}$$

$$T = \frac{0.1447788\ nm}{0.01479056432} \tag{9}$$

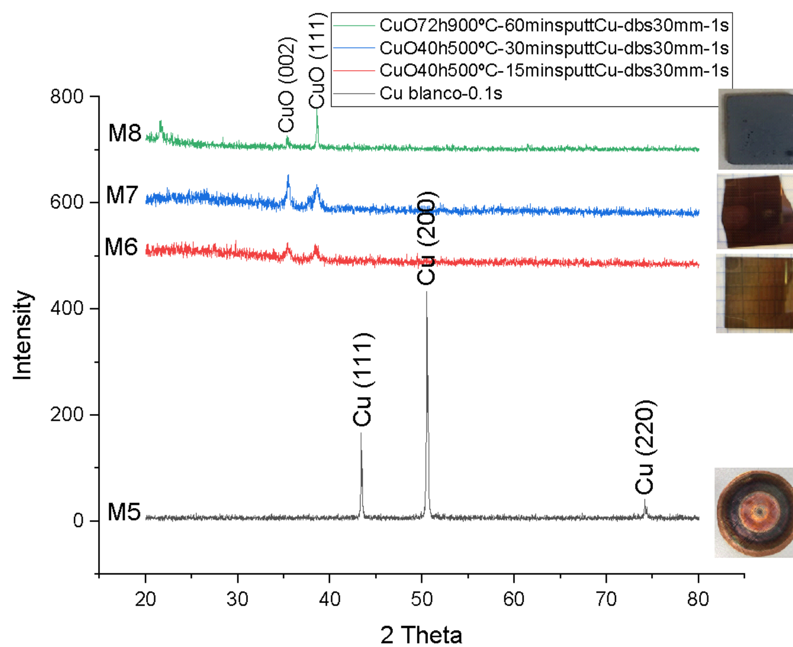
$T\ = 9.7885$ nm

The crystal size for the (220) plane is 9.7885 nm.

*XRD diffractogram of CuO*

Figure 5.3 shows the diffractograms obtained by XRD, the number of steps executed for each diffractogram was 3001 steps. The M5 diffractogram is representative of the copper target (a target is the material to be evaporated by ion bombardment and then deposited on a substrate). It is possible to observe an erosion pattern due to the sputtering process it has undergone during its lifetime. Diffractogram M6 is a deposit of copper by sputtering for 15 minutes and then oxidised for 40 h at 500ºC, the film obtained is thin, presents low crystallinity and shows the transmission phenomenon. The diffractogram M7 represents the deposit by sputtering for 30 minutes and then oxidised for 40 h at 500ºC, the deposit obtained is not very translucent due to the increase in mass (Cu) which influences the definition and width of the peaks. The M8 diffractogram is a sputtering deposit for 60 minutes on a quartz substrate and then oxidised at 900ºC in a muffle. The sample obtained takes on a grey colour due to the significant increase in deposition. The 4 samples analysed were obtained at a target-substrate distance of 30 mm.

**Figure 5.3** Diffractograms obtained by XRD. M5; representative of the copper blank analysed by XRD for 0.1s, M6; Cu deposited for 15 min at a blank-substrate distance of 30mm and subsequently oxidised for 40 h at 500ºC analysed by XRD for 1s (CuO). M7; Cu deposition for 15 min at a target-substrate distance of 30 mm and subsequently oxidised for 40 h at 500ºC analysed by 1s XRD (CuO). M8; Cu deposit for 60 min at a target-substrate distance of 5 mm and subsequently oxidised for 72 h at 900ºC analysed by XRD for 1 s (CuO)




The M5 diffractogram shows the Cu target used in the sputtering in which the characteristic Cu peaks located at 2θ= 43.38º, 50.50º and 74.18º respective to the (111), (200) and (220) planes stand out. The calculated TC value of the most intense peak is 2.1994 which indicates that it is preferentially oriented towards the (200) plane. The average crystal size taken from the highest peak (200) is 39.8764 nm.

The M6 diffractogram is the result of a CuO deposit. The Cu was deposited by sputtering for 15 min on a glass substrate, then subjected to a thermal process (muffle) for 40 h at 500ºC resulting in copper oxidation. XRD analysis was used to determine the phases present in the film in which there are two peaks at 2θ= 35.38º and 38.42º corresponding to (002) and (111) planes of the CuO structure (PDF 00-045-0937). The diffractogram does not show a great difference in the intensity of the peaks, so it does not have a preferential orientation, so it was checked by estimating the texture coefficient, which gave a result of 0.97393, less than 1. The average size of the crystal taken from the most intense peak (002) is 13.9357 nm, which indicates the presence of nanostructures. The M7 diffractogram is the result of a CuO deposit (the sample turns a darker colour due to a higher amount of mass (Cu) being oxidised compared to the sample obtained for 15 min). Cu was deposited by sputtering for 30 min on a glass substrate, then subjected to a thermal process (muffle) for 40 h at 500ºC, resulting in copper oxidation. XRD analysis was used to determine the phases present in the deposit where it shows two peaks at 2θ= 35.38º and 38.42º corresponding to (002) and (111) planes of the CuO structure (PDF 00-045-0937). The peaks are at the same 2θ position of the M4 diffractogram but this time the peaks are more intense and their width decreases. There is a little noticeable difference in the intensity of the (111) plane at 2θ= 38.42º with respect to the other peak, that is why the preferential orientation was estimated and the result was 1.0807 greater than 1, which corresponds to the preferential orientation towards the (111) plane. The average crystal size taken from the most intense peak (002) is 21.1260 nm which indicates the formation of a thin film. The M8 diffractogram represents the CuO deposit. The Cu was deposited by Sputtering for 60 minutes on a quartz substrate, then it was subjected to a thermal process (muffle) for 72 h at 900ºC as a result the oxidation is obtained. XRD analysis at 1 s for each step was used to determine the phases present in the deposit. Three peaks are presented in which only two were identified at 2θ=35.36º and 38.60º corresponding to (002) and (111) planes of the CuO structure (PDF 00-045-0937).

*Texture coefficient comparison*

Table 5.1 shows the results obtained by calculating Eq. (2) Texture coefficient. The CuO M6 (15 min deposit by Sputtering with a thermal process "oxidation" by a muffle for 40 h at 500ºC) does not show preferential orientation as a tiny amount of Cu is oxidised whereas the diffractogram M7 (copper deposit for 30 min by Sputtering with a thermal process "oxidation" by a muffle for 40 h at 500ºC) doubles the Cu deposit i.e., more mass (Cu) is oxidised, so the peaks are better defined and start to have a preferential orientation towards the (002) plane. The diffractogram of the M8 (deposit for 60 minutes oxidised for 72 h at 900ºC) already presents a preferential orientation towards the plane (002) due to the improvement of obtaining, on the other hand they are not compared with the diffractogram M5 where the texture coefficient is higher because it is the Cu target having an excellent crystalline structure due to the definition of the peaks.

**Table 5.1** Preferential orientation calculated using Eq. (2) Texture Coefficient

| Diffractogram | Texture coefficient | Preferential orientation |
|---|---|---|
| M5 (white de Cu-0.1s) | 2.1994 | Preferentially oriented towards the plane (200) |
| M6 (CuO40h500ºC-15minSputtCu-dbs30mm-1s) | 0.9739 | No preferential orientation |
| M7 (CuO40h500ºC-30minSputtCu-dbs30mm-1s) | 1.08076 | Preferentially oriented towards the plane (002) |
| M8 (CuO72h900ºC-60minsputt-dbs30mm-1s) | 1.5135 | Preferably oriented towards the plane (002) |

*Crystal size comparison*

Table 5.2 shows the average crystal size of the diffraction peaks, which is inversely proportional to the width of the diffraction peaks (the wider the peak, the smaller the crystal size). The diffractogram M6 represents the smallest crystal size, because the peaks are too wide and not very intense, the sample contains a combination of CuO nanostructures without forming a thin film, whereas in the diffractogram M8, because of the longer Cu deposition time, more particles were deposited. The deposit already forms a CuO thin film.

**Table 5.2** Average crystal size calculated by Eq. (2) Scherrer

| Diffractogram | Map | Average crystal size (nm) |
|---|---|---|
| M5 (white de Cu-1s) | (111) | 49.6264 |
| | (200) | 39.8764 |
| | (220) | 31.8092 |
| M6 (CuO40h500ºC-15minSputtCu-dbs30mm-1s) | (002) | 13.9357 |
| | (111) | 15.2728 |
| M7 (CuO40h500ºC-30minSputtCu-dbs30mm-1s) | (002) | 21.1260 |
| | (111) | 19.0288 |
| M8 (CuO72h900ºC-60minsputt-bds30mm-1s) | (002) | 38.9619 |
| | (111) | 53.0491 |

Calculations of the texture coefficient and crystal size for the diffractograms in figure 8

Calculation of Eq. (2) Texture coefficient (R, Lopez, & Leyva Porras, 2021) for copper target M5

- Data:

TC= texture coefficient

I= measured relative intensities of each plane (hkl) in the XRD pattern of the characterised sample.

| Map (111) | Map (200) | Map (220) |
|---|---|---|
| 30.03 | 100 | 8.22 |

$I_0$= intensity of the same plane taken from the standard reference data (PDF 00-004-0836 for CuO)

| Map (111) | Map (200) | Map (220) |
|---|---|---|
| 100 | 46 | 20 |

$n$= number of diffraction peaks: 3

- Replacement

$$= \frac{100}{46}\left\{\left(\frac{1}{3}\right)\left(\frac{38.03}{100}+\frac{100}{46}+\frac{8.22}{20}\right)\right\}^{-1} \quad (10)$$

$$TC = \frac{100}{46}\left(\frac{1}{\left(\frac{38.03}{100}+\frac{100}{46}+\frac{8.22}{20}\right)}\right) \quad (11)$$

$$TC = \frac{100}{46}\left(\frac{1}{(2.9652)}\right) \quad (12)$$

$$TC = \frac{50}{23}\left(\frac{1}{0.9884}\right) \quad (13)$$

$$TC = \left(\frac{50}{22.7332}\right) \quad (14)$$

$$TC = 2.1994 \quad (15)$$

TC >1, the calculated value of the strongest peak is 2.1994, which indicates that the CuO film is preferentially oriented in the (200) plane.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (100) plane of the diffractogram of copper target *M5*

Data

T= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.17989

$\theta_B$ = 21.6903

Conversion of units:

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.17989)(\pi)}{180} = 3.139672791x10^{-3} \quad (16)$$

- Replacement

$$T = \frac{(0.94)(0.15402\ nm)}{(3.139672791x10^{-3})\ coscos\ (21.6903)} \quad (17)$$

$$T = \frac{0.1447788\ nm}{2.917368746x10^{-3}} \quad (18)$$

$T$ = 49.6264 nm

The crystal size for the (100) plane is 46.6264 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (200) plane of the diffractogram of the copper blank *M5*

Data:

T= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.23001

$\theta_B$ = 25.2564

Conversion of units:

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.23001\,)(\pi)}{180} = 4.014431813x10^{-3} \quad (19)$$

- Replacement

$$T = \frac{(0.94)(0.15402\,nm)}{(4.014431813x10^{-3}\,)\,coscos\,(21.6903)} \quad (20)$$

$$T = \frac{0.1447788\,nm}{3.630682206x10^{-3}} \quad (21)$$

$T\,=$ 39.8764 nm

The crystal size for the (200) plane is 39.8764 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (220) plane of the diffractogram of the copper blank *M5*

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.32691

$\theta_B$ = 37.0882

Conversion of units

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.32691\,)(\pi)}{180} = 5.705655858x10^{-3} \quad (22)$$

- Replacement

$$T = \frac{(0.94)(0.15402\,nm)}{(5.705655858x10^{-3}\,)\,coscos\,(37.0882)} \quad (23)$$

$$T = \frac{0.1447788\ nm}{4.551448133x10^{-3}} \tag{24}$$

$T = 31.8092$ nm

The crystal size for the (220) plane is 31.8072 nm.

Calculation of Eq. (2) Texture coefficient (R, Lopez, & Leyva Porras, 2021) for CuO *M6*.

- Data

*TC*= texture coefficient

I= measured relative intensities of each plane (hkl) in the XRD pattern of the characterised sample.

| Map (002) | Map (100) |
|---|---|
| 100 | 95.87 |

$I_0$= intensity of the same plane taken from the standard reference data (PDF 00-045-0937 for CuO)

| Map (002) | Map (100) |
|---|---|
| 100 | 91 |

*n*= number of diffraction peaks: 2

- Replacement.

$$= \frac{100}{100}\left\{\left(\frac{1}{2}\right)\left(\frac{100}{100} + \frac{95.87}{91}\right)\right\}^{-1} \tag{25}$$

$$TC = \frac{100}{100}\left(\frac{1}{\left(\frac{100}{100}+\frac{95.87}{91}\right)}\right) \tag{26}$$

$$TC = 1\left(\frac{1}{\left(1+\frac{95.87}{91}\right)}\right) \tag{27}$$

$$TC = 1\left(\frac{1}{(2.0535)}\right) \tag{28}$$

$$TC = 1\left(\frac{1}{1.0267}\right) \tag{29}$$

$$TC = \left(\frac{1}{1.0267}\right) \tag{30}$$

$$TC = 0.97393 \tag{31}$$

TC <1, is not a textured film because it does not have a preferential orientation.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (002) plane for CuO *M6*

Data

*T*= Crystal size to be calculated

*K*= 0.94

$\lambda$= 0.15402 nm

$B$= 0.62479

$\theta_B$ = 17.69

Conversion of units:

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.62479\,)(\pi)}{180} = 0.01090464263 \quad (32)$$

- Replacement

$$T = \frac{(0.94)(0.15402\,nm)}{(0.01090464263)\,coscos\,(17.69)} \quad (33)$$

$$T = \frac{0.1447788\,nm}{0.01038901148} \quad (34)$$

$T\,=\,13.9357$ nm

The crystal size for the (002) plane is 13.9357 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (111) plane for CuO *M6*

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.57516

$\theta_B$ = 19.21

Conversion of units

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.57516\,)(\pi)}{180} = 0.01003843573 \quad (35)$$

- Replacement.

$$T = \frac{(0.94)(0.15402\,nm)}{(0.01003843573\,)\,coscos\,(19.21)} \quad (36)$$

$$T = \frac{0.1447788\,nm}{9.479485166x10^{-3}} \quad (37)$$

$T\,=\,15.2728$ nm

The crystal size for the (100) plane is 15.2728 nm.

Calculation of Eq. (2) Texture coefficient (R, Lopez, & Leyva Porras, 2021) for CuO *M7*

Data

*TC*= coeficiente de textura

*I*= measured relative intensities of each plane (hkl) in the XRD pattern of the characterised sample.

| Map (002) | Map (111) |
|---|---|
| 100 | 77.4 |

$I_0$= intensity of the same plane taken from the standard reference data (PDF 00-045-0937 for CuO).

| Map (002) | Map (111) |
|---|---|
| 100 | 91 |

*n*= number of diffraction peaks: 2.

- Replacement.

$$= \frac{100}{100}\left\{\left(\frac{1}{2}\right)\left(\frac{100}{100}+\frac{74.4}{91}\right)\right\}^{-1} \quad (38)$$

$$TC = \frac{100}{100}\left(\frac{1}{\left(\frac{100}{100}+\frac{74.4}{91}\right)}\right) \quad (39)$$

$$TC = 1\left(\frac{1}{\left(1+\frac{74.4}{91}\right)}\right) \quad (40)$$

$$TC = 1\left(\frac{1}{(1.8505)}\right) \quad (41)$$

$$TC = 1\left(\frac{1}{0.925274}\right) \quad (42)$$

$$TC = \left(\frac{1}{0.925274}\right) \quad (43)$$

$$TC = 1.0807 \quad (44)$$

TC >1The calculated value of the strongest peak is 1.0807, which indicates that the CuO film is preferentially oriented in the (111) plane.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (002) plane for CuO *M7*

Data

*T*= Crystal size to be calculated

*K*= 0.94

$\lambda$= 0.15402 nm

*B*= 0.41228

$\theta_B$ = 17.75

Conversion of units:

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.41228\,)(\pi)}{180} = 7.19564344x10^{-3} \quad (45)$$

- Replacement.

$$T = \frac{(0.94)(0.15402\;nm)}{(7.19564344x10^{-3}\,)\;coscos\;(17.75)} \quad (46)$$

$$T = \frac{0.1447788\;nm}{6.853100588x10^{-3}} \quad (47)$$

$T\; = 21.1260$ nm

The crystal size for the (002) plane is 21.1260 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (111) plane for CuO *M7*

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.46183

$\theta_B$ = 19.27

Conversion of units:

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.46183\,)(\pi)}{180} = 8.060454084x10^{-3} \quad 48)$$

- Replacement.

$$T = \frac{(0.94)(0.15402\;nm)}{(8.060454084x10^{-3}\,)\;coscos\;(19.27)} \quad (49)$$

$$T = \frac{0.1447788\;nm}{7.608858109x10^{-3}} \quad (50)$$

$T\; = 19.0288$ nm

The crystal size for the (002) plane is 19.0288 nm.

Calculation of Eq. (2) Texture Coefficient (R, Lopez, & Leyva Porras, 2021) for the M8

Data

$TC$= texture coefficient

$I$= measured relative intensities of each plane (hkl) in the XRD pattern of the characterised sample

| Map (002) | Map (111) |
|---|---|
| 35.32 | 100 |

$I_0$= intensity of the same plane taken from the standard reference data (PDF 00-004-0836 for CuO).

| Map (002) | Map (111) |
|---|---|
| 100 | 91 |

$n$= number of diffraction peaks: 2

- Replacement.

$$= \frac{100}{91}\left\{\left(\frac{1}{2}\right)\left(\frac{100}{91}+\frac{35.32}{100}\right)\right\}^{-1} \tag{51}$$

$$TC = \frac{100}{91}\left(\frac{1}{\left(\frac{100}{91}+\frac{35.32}{100}\right)}\right) \tag{52}$$

$$TC = \frac{100}{91}\left(\frac{1}{(1.452101099)}\right) \tag{53}$$

$$TC = \frac{100}{91}\left(\frac{1}{0.7260505495}\right) \tag{54}$$

$$TC = \left(\frac{100}{66.0706091}\right) \tag{55}$$

$$TC = 1.5135 \tag{56}$$

TC >1, the calculated value of the strongest peak is 1.5135, which indicates that the CuO film is preferentially oriented in the (111) plane.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (002) plane of the diffractogram M8

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.22346

$\theta_B$ = 17.6800

Conversion of units:

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.22346)(\pi)}{180} = 3.900112747x10^{-3} \tag{57}$$

- Replacement.

$$T = \frac{(0.94)(0.15402\ nm)}{(3.900112747x10^{-3})\ coscos\ (17.6800)} \tag{58}$$

$$T = \frac{0.1447788\ nm}{3.71590087x10^{-3}} \quad (59)$$

$T = 38.9619$ nm

The crystal size for the (002) plane is 38.9619 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (111) plane of the diffractogram M8

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.16568

$\theta_B$ = 19.3005

Conversion of units:

(°) gradians to (rad) radians.

1° = 0.0174533 rad.

$$\frac{(0.16568\ )(\pi)}{180} = 2.891661505x10^{-3} \quad (60)$$

- Replacement.

$$T = \frac{(0.94)(0.15402\ nm)}{(\ 2.891661505x10^{-3})\ coscos\ (19.3005)} \quad (61)$$

$$T = \frac{0.1447788\ nm}{2.72914454x10^{-3}} \quad (62)$$

$T = 53.0491$ nm

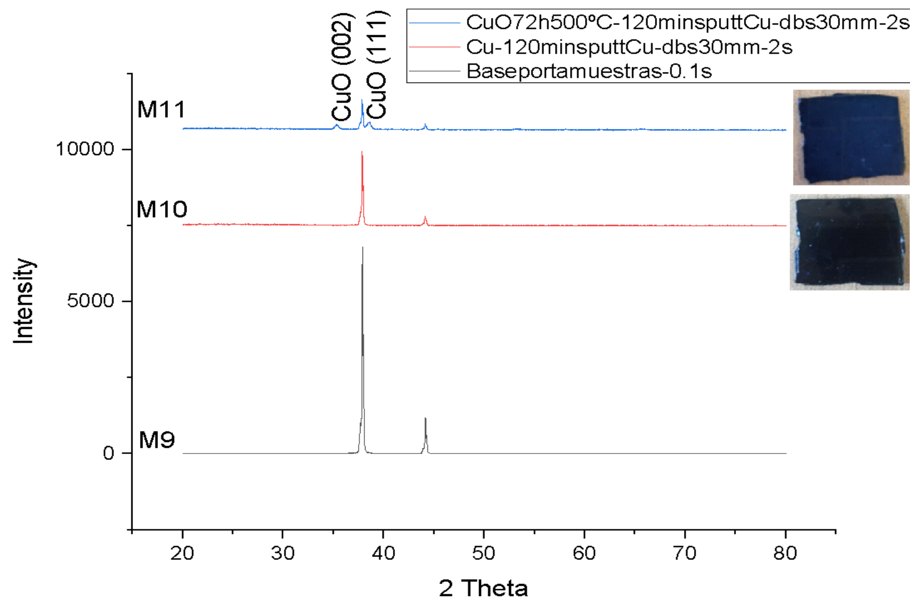
The crystal size for the (111) plane is 53.0491 nm.

Diffractogram of the sample holder, Cu and CuO deposit by XRD

Figure 5.4, M9 represents an XRD analysis of the base of the sample holder for 0.1 s. M10 is a Cu deposit by means of a Cu target deposited on a glass substrate by sputtering for 2 h, then analysed by XRD for 2 s. The sample takes a dark colour due to the CuO. The sample takes a dark colour due to the long deposition time. M11 is a Cu deposit by sputtering Cu target for 120 min deposited on a glass substrate, then oxidised in a flask for 72 h at 500ºC and analysed by XRD for 2 s. The sample obtained from the Cu sputtering was oxidised in a flask for 72 h at 500ºC and analysed by XRD for 2 s. The sample obtained from the Cu sputtering was then oxidised in a flask for 72 h at 500ºC.

The sample obtained from the oxidation has similarity compared before oxidisation.

**Figure 5.4** Diffractogram obtained by XRD. M9; sample holder base analysed for 0.1 sec. M10; Cu deposition by Sputtering for 120 min, analysed for 2s. M11; (CuO) Cu deposition by Sputtering for 120 min, with 72 hours of thermal oxidation by muffle at 500ºC and analysed for 2s (CuO)




Cu was sputtered for 120 min by sputtering deposited on a glass substrate and then XRD analysis was performed for 2s (M10), the intense peaks are found at 2θ= 37.88º and 44.14º. When characterising the diffraction peaks from standard reference data for Cu no similarity in the positions was found. Subsequently, an analysis of the M9 sample holder base was carried out for 0.1 s, presenting similarity in the diffraction peaks found at 2θ= 37.86º and 44.12º. The peaks present higher intensity compared to the M9 diffractogram considering that the analysis time was shorter. Due to the similarity it is possible that both diffractograms present the measurement of the sample holder base, when having interference to the Cu deposit the peaks decrease their intensity. Possible reasons why the sample was not read by XRD:

- The Cu target has low or no crystallinity.
- The Cu layer is not continuous.

Some authors previously reported that the deposition rate should not exceed a limit such that the overgrowth layer is deposited before atomic hopping to an equilibrium position is possible, resulting in non-crystalline nuclei formed on the substrate surface. The diffractogram of M11 is representative of Cu deposition by sputtering for 120 min deposited on a glass substrate and subsequently oxidised in a flask for 72 h at 500°C and analysed by XRD for 2 s. The increased analysis time was possible due to the increase of analysis time. The increase in analysis time it was possible to observe four peaks at 2θ= 35.34º, 37.84º, 38.56º and 44.12º. Only two peaks 2θ= 35.34º and 38.56º corresponding to (002) and (111) planes of the CuO structure (PDF 00-045-0937) could be identified. Due to the thermal process that the Cu deposit underwent, CuO was visible in the M11 diffractogram. The two remaining peaks 2θ= 37.84º and 44.12 are attributed to the measurement of the base of the sample holder because it presents the same peaks seen previously in M9 and M10. The presence of the sample holder peaks meant that texture coefficient and crystal size calculations were not performed because it is not of interest to study the material (aluminium) of the sample holder.

*XRD diffractogram of CuO*

Figure 5.5 shows the different diffractograms obtained by XRD, M12 is a Cu deposit using a Cu target (at a target-substrate distance of 30 mm) by the sputtering technique for 120 min deposited on a glass substrate, subsequently oxidised in a flask for 72 h at 500ºC and analysed by XRD for 1 s (with pink base). M13 is a Cu deposit by sputtering Cu target for 120 min deposited on a glass substrate, then oxidised in a flask for 72 h at 500ºC and analysed by XRD for 3 s (pink based). M14 is a Cu deposit using a Cu target (at a target-substrate distance of 5 mm) by the sputtering technique for 45 min deposited on a glass substrate, subsequently oxidised in a fume cupboard for 72 h and analysed by XRD for 0.1 s.

**Figure 5.5**

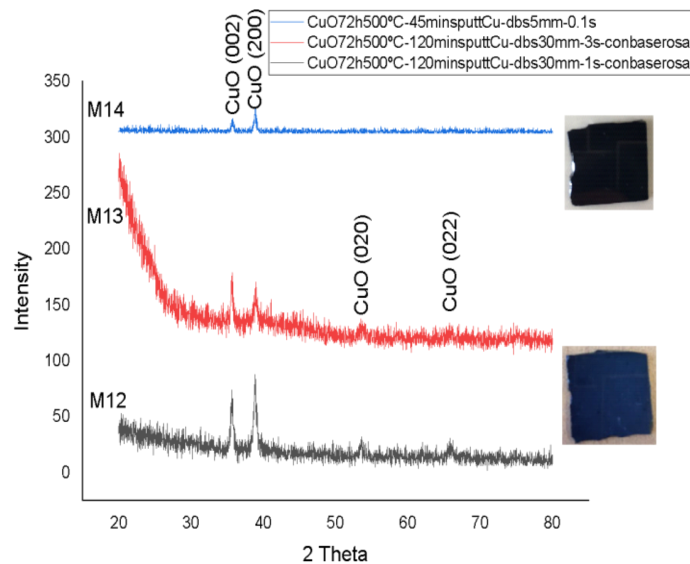



Figure 5.5 Diffractogram obtained by XRD. M12; Cu deposit sputtered for 120 min (30 mm blank-substrate distance), with 72 hours of thermal oxidation by muffle at 500ºC and analysed for 1 s (with pink base) (CuO). M13; Cu deposition by sputtering for 120 min (30 mm blank-substrate distance), with 72 h thermal oxidation by flask at 500°C and analysed for 3 s (pink basis) (CuO). M14; Cu deposit by sputtering for 45 min, (target-substrate distance 5 mm), subsequently oxidised at 72 h by a muffle at 500ºC and analysed for 0.1 s (CuO). Due to the diffractograms obtained with sample holder reading, a pink base was included between the sample holder and the deposit in M12 and M13 in order to obtain only the CuO analysis. The diffractogram of M12 is representative of Cu deposition by sputtering for 120 min deposited on a glass substrate at a target-substrate distance of 30 mm, then oxidised in a flask for 72 h at 500°C and analysed by XRD for 1 s (for this analysis a pink base was included to avoid reading the sample holder). Four peaks are shown at 2θ= 35.64º, 38.90, 53.62 and 65.86 low intensity corresponding to (002), (200), (020) and (022) planes of the CuO structure (PDF 00-045-0937). The texture coefficient calculated for the M12 diffractogram is 1.0310, because it is greater than 1 it is considered a textured film oriented towards the (200) plane. The average crystal size taken for the most intense peak (200) is 20.7663, due to the crystal size it is already considered a thin film. The diffractogram of M13 is representative of Cu deposition by sputtering for 120 min deposited on a glass substrate at a white-substrate distance of 30 mm, then oxidised in a flask for 72 h at 500°C and analysed by XRD for 3 s (for this analysis a pink base was included to avoid reading the sample holder). Four peaks are shown at 2θ= 35.62º, 38.84º, 53.48º and 65.72º with a small intensity, these peaks are the same as those seen in M12 corresponding to (002), (200), (020) and (022) planes of the CuO structure (PDF 00-045-0937). The texture coefficient calculated for the diffractogram of M13 is 0.2416, because it is less than 1 it does not have a preferential orientation. The average crystal size taken for the most intense peak (002) is 22.7857, due to the crystal size it is already considered a thin film. The diffractogram of M14 is representative of Cu deposition by sputtering for 45 minutes on a glass substrate at a target-substrate distance of 5 mm to decrease the deposition time. Subsequently it is oxidised for 72 h at 500ºC and by XRD analysed for 0.1 s. The deposit is similar to the one obtained during 120 min at 30 mm with the only difference that the last two peaks (020) and (022) are not found in this diffractogram. Two peaks were found at 2θ= 35.78 and 38.92 corresponding to (002) and (200) planes of the CuO structure (PDF 00-045-0937) similar to those found in M12 and M13. The texture coefficient calculated for the diffractogram of M14 is 1.7174, because it is greater than 1 it is considered a textured film oriented towards the (200) plane. The average crystal size taken for the most intense peak (200) is 25.5054, due to the crystal size it is already considered a thin film. The largest average crystal size belongs to the diffractogram of M14 due to the white-substrate arrangement in the sputtering deposition.
Calculation of Eq. (2) Texture Coefficient (R, Lopez, & Leyva Porras, 2021) for the M12

Data

*TC*= texture coefficient

*I*= measured relative intensities of each plane (hkl) in the XRD pattern of the characterised sample

| Map (002) | Map (200) | Map (020) | Map (022) |
|---|---|---|---|
| 81.22 | 100 | 31.53 | 33.73 |

$I_0$= intensity of the same plane taken from the standard reference data (PDF 00-004-0836 for CuO)

| Map (002) | Map (200) | Map (020) | Map (022) |
|---|---|---|---|
| 100 | 28 | 6 | 8 |

$n$= number of diffraction peaks: 4

- Replacement.

$$= \frac{100}{28}\left\{\left(\frac{1}{4}\right)\left(\frac{100}{28}+\frac{81.22}{100}+\frac{31.53}{6}+\frac{33.73}{8}\right)\right\}^{-1} \quad (63)$$

$$TC = \frac{100}{28}\left(\frac{1}{\frac{1}{4}\left(\frac{100}{28}+\frac{81.22}{100}+\frac{31.53}{6}+\frac{33.73}{8}\right)}\right) \quad (64)$$

$$TC = \frac{100}{28}\left(\frac{1}{\frac{1}{4}(13.85487857)}\right) \quad (65)$$

$$TC = \frac{100}{28}\left(\frac{1}{3.463719643}\right) \quad (66)$$

$$TC = \left(\frac{(100)(1)}{(28)(3.463719643)}\right) \quad (67)$$

$$TC = \left(\frac{100}{96.9841499}\right) \quad (68)$$

$$TC = 1.0310 \quad (69)$$

TC >1, the calculated value of the most intense peak is 1.0310, which indicates that the CuO film is preferentially oriented in the (200) plane.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (002) plane of the diffractogram M12

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.46062

$\theta_B$ = 17.8127

Conversion of units:

(°) gradians to (rad) radians.

1° = 0.0174533 rad.

$$\frac{(0.46062\,)(\pi)}{180} = 8.039335601x10^{-3} \quad (70)$$

- Replacement.

$$T = \frac{(0.94)(0.15402\ nm)}{(\ 8.039335601x10^{-3})\ coscos\ (17.8127)} \quad (71)$$

$$T = \frac{0.1447788\ nm}{7.653942795x10^{-3}} \quad (72)$$

$T\ = 18.9155$ nm

The crystal size for the (002) plane is 18.9155 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (200) plane of the diffractogram M12

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.42355

$\theta_B$ = 19.4191

Conversion of units:

(°) gradians to (rad) radians.

1° = 0.0174533 rad.

$$\frac{(0.42355\ )(\pi)}{180} = 7.392342047x10^{-3} \quad (73)$$

- Replacement.

$$T = \frac{(0.94)(0.15402\ nm)}{(\ 7.392342047x10^{-3})\ coscos\ (19.4191)} \quad (74)$$

$$T = \frac{0.1447788\ nm}{6.971805583x10^{-3}} \quad (75)$$

$T\ = 20.7663$ nm

The crystal size for the (200) plane is 20.7663 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (020) plane of the diffractogram M12

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.57

$\theta_B$ = 26.81

Conversion of units:

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.57)(\pi)}{180} = 9.948276736x10^{-3} \tag{76}$$

- Replacement.

$$T = \frac{(0.94)(0.15402\ nm)}{(9.948276736x10^{-3})\cos(26.81)} \tag{77}$$

$$T = \frac{0.1447788\ nm}{8.878996989x10^{-3}} \tag{78}$$

$T = 16.3057$ nm

The crystal size for the (020) plane is 16.3057 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (022) plane of the diffractogram M12

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.8921

$\theta_B$ = 32.93

Conversion of units:

(°) gradians to (rad) radians.

1° = 0.0174533 rad.

$$\frac{(0.8921)(\pi)}{180} = 0.01557025679 \tag{79}$$

- Replacement.

$$T = \frac{(0.94)(0.15402\ nm)}{(0.01557025679)\cos(32.93)} \tag{80}$$

$$T = \frac{0.1447788\ nm}{0.01306866684} \tag{81}$$

$T = 11.0783$ nm

The crystal size for the (022) plane is 11.0783 nm.

Calculation of Eq. (2) Texture coefficient (R, Lopez, & Leyva Porras, 2021) for M13

Data:

$TC$= texture coefficient

$I$= measured relative intensities of each plane (hkl) in the XRD pattern of the characterised sample.

| Map (002) | Map (200) | Map (020) | Map (022) |
|---|---|---|---|
| 100 | 81.23 | 45.26 | 40.85 |

$I_0$= intensity of the same plane taken from the standard reference data (PDF 00-004-0836 for CuO).

| Map (002) | Map (200) | Map (020) | Map (022) |
|---|---|---|---|
| 100 | 28 | 6 | 8 |

$n$= number of diffraction peaks: 4.

- Replacement.

$$= \frac{100}{100}\left\{\left(\frac{1}{4}\right)\left(\frac{100}{100}+\frac{81.23}{28}+\frac{45.26}{6}+\frac{40.85}{8}\right)\right\}^{-1} \tag{82}$$

$$TC = \frac{100}{100}\left(\frac{1}{\frac{1}{4}\left(\frac{100}{100}+\frac{81.23}{100}+\frac{45.26}{6}+\frac{40.85}{8}\right)}\right) \tag{83}$$

$$TC = \frac{100}{100}\left(\frac{1}{\frac{1}{4}(16.55065476)}\right) \tag{84}$$

$$TC = \frac{100}{100}\left(\frac{1}{4.13766369}\right) \tag{85}$$

$$TC = 1\left(\frac{1}{4.13766369}\right) \tag{86}$$

$$TC = 1(0.2416) \tag{87}$$

$$TC = 0.2416 \tag{88}$$

TC<1, the calculated value of the most intense peak is 0.2416, which indicates that the CuO film has no preferential orientation towards any plane.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (002) plane of the diffractogram M13

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.38244

$\theta_B$ = 17.8387

Conversion of units:

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.38244)(\pi)}{180} = 6.674837191x10^{-3} \tag{89}$$

- Replacement.

$$T = \frac{(0.94)(0.15402\ nm)}{(\ 6.674837191x10^{-3})\ coscos\ (17.8387)} \tag{90}$$

$$T = \frac{0.1447788\ nm}{6.353929014x10^{-3}} \tag{91}$$

$T\ = 22.7857$ nm

The crystal size for the (002) plane is 22.7857 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (200) plane of the diffractogram M13

Data

*T*= Crystal size to be calculated

*K*= 0.94

$\lambda$= 0.15402 nm

*B*= 0.55168

$\theta_B$ = 19.4787

Conversion of units:

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.55168\ )(\pi)}{180} = 9.628632417x10^{-3} \tag{92}$$

- Replacement.

$$T = \frac{(0.94)(0.15402\ nm)}{(\ 9.628632417x10^{-3})\ coscos\ (19.4787)} \tag{93}$$

$$T = \frac{0.1447788\ nm}{9.077542651x10^{-3}} \tag{94}$$

$T\ = 15.9491$ nm

The crystal size for the (200) plane is 15.9491 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (020) plane of the diffractogram M13

Data

*T*= Crystal size to be calculated

*K*= 0.94

$\lambda$= 0.15402 nm

*B*= 0.89178

$\theta_B$ = 26.74

Conversion of units:

(°) gradians to (rad) radians

1° = 0.0174533 rad.

$$\frac{(0.89178\,)(\pi)}{180} = 0.0155644972 \quad (95)$$

- Replacement.

$$T = \frac{(0.94)(0.15402\,nm)}{(\,0.0155644972)\,cos\,(26.74)} \quad (96)$$

$$T = \frac{0.1447788\,nm}{0.01389999075} \quad (97)$$

$T = 10.4157$ nm

The crystal size for the (020) plane is 10.4157 nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (022) plane of the diffractogram M13

Data

*T*= Crystal size to be calculated

*K*= 0.94

$\lambda$= 0.15402 nm

*B*= 0.45722

$\theta_B$ = 32.86

Conversion of units:

(°) gradians to (rad) radians.

1° = 0.0174533 rad.

$$\frac{(0.45722\,)(\pi)}{180} = 7.979994406x10^{-3} \quad (98)$$

- Replacement.

$$T = \frac{(0.94)(0.15402\,nm)}{(7.979994406x10^{-3})\,cos\,(32.86)} \quad (99)$$

$$T = \frac{0.1447788\,nm}{6.703186261x10^{-3}} \quad (100)$$

$T = 21.5985$ nm

The crystal size for the (022) plane is 21.5985 nm.

Calculation of Eq. (2) Texture coefficient (R, Lopez, & Leyva Porras, 2021) for M14

Data

*TC*= texture coefficient

*I*= measured relative intensities of each plane (hkl) in the XRD pattern of the characterised sample

| Map (002) | Map (200) |
|---|---|
| 58.75 | 100 |

$I_0$= intensity of the same plane taken from the standard reference data (PDF 00-004-0836 for CuO)

| Map (002) | Map (200) |
|---|---|
| 100 | 28 |

$n$= number of diffraction peaks: 2

- Replacement.

$$= \frac{100}{28}\left\{\left(\frac{1}{2}\right)\left(\frac{100}{100}+\frac{81.23}{28}\right)\right\}^{-1} \quad (101)$$

$$TC = \frac{100}{28}\left(\frac{1}{\left(\frac{100}{28}+\frac{58.75}{100}\right)}\right) \quad (102)$$

$$TC = \frac{100}{28}\left(\frac{1}{(4.158928571)}\right) \quad (103)$$

$$TC = \frac{100}{28}\left(\frac{1}{2.079464286}\right) \quad (104)$$

$$TC = \left(\frac{(100)(1)}{(28)(2.079464286)}\right) \quad (105)$$

$$TC = \left(\frac{100}{58.225}\right) \quad (106)$$

$$TC = 1.7174 \quad (107)$$

TC >1, the calculated value of the strongest peak is 1.7174, indicating that the textured CuO film is preferentially oriented in the (200) plane.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (002) plane of the diffractogram M14

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.36913

$\theta_B$ = 17.8899

Conversion of units:

(°) gradians to (rad) radians.

1° = 0.0174533 rad.

$$\frac{(0.36913)(\pi)}{180} = 6.442533868x10^{-3} \quad (108)$$

- Replacement.

$$T = \frac{(0.94)(0.15402\ nm)}{(\ 6.442533868x10^{-3})\ coscos\ (17.8889)} \quad (109)$$

$$T = \frac{0.1447788\ nm}{6.131028139x10^{-3}} \quad (110)$$

$T = 23.6141$ nm

The crystal size for the (002) plane is 23.6141nm.

Calculation of Scherrer's Eq. (1) (Patterson, 1939), for the (200) plane of the diffractogram M14

Data

$T$= Crystal size to be calculated

$K$= 0.94

$\lambda$= 0.15402 nm

$B$= 0.34485

$\theta_B$ = 19.4189

Conversion of units:

(°) gradians to (rad) radians.

1° = 0.0174533 rad.

$$\frac{(0.34485\ )(\pi)}{180} = 6.018767926x10^{-3} \quad (111)$$

- Replacement.

$$T = \frac{(0.94)(0.15402\ nm)}{(6.018767926x10^{-3})\ coscos\ (19.4189)} \quad (112)$$

$$T = \frac{0.1447788\ nm}{5.676378501x10^{-3}} \quad (113)$$

$T = 25.5054$ nm

The crystal size for the (200) plane is 25.5054 nm.

## 5.4 Conclusions

By combining two techniques, sputtering and thermal oxidation, it was possible to obtain samples with different thicknesses according to the time employed in the deposition. After the arrangement of the system with a target-substrate distance of 5 mm, deposits similar to those of 120 minutes were obtained in a shorter time, optimising the process. According to the diffractograms, it was observed that the material to be deposited, called "white", determines the characteristics of the deposits made. If this material does not have a crystalline structure, the diffraction peaks corresponding to this material will be null. If the deposit is not well placed in the sample holder of the XRD diffractometer, it will not be read at depth, in addition to including in the diffractograms the reading of the material of which the sample holder is made, interfering with the analysis. The calculations made for the texture coefficient and average crystal size present the parameters of the crystalline structure of each XRD diffractogram analysed. Confirming if it is a film according to the average crystal size and if it is textured towards a preferential plane. In conclusion, controlling the experimental conditions in the synthesis of CuO determines the properties of the thin films, as well as the future use in a device.

**5.5 References**

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