Enhanced photodetection performance of sputtered cupric oxide thin film through annealing process

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Abstract
This study demonstrates an improvement in the photodetection response of a Cupric Oxide (CuO) thin film through the annealing process. The CuO thin film was deposited on silicon substrate using DC magnetron sputtering system. Annealing of the as-deposited film was carried out in a muffle furnace at 400 and 500 °C for two hours. X-ray diffraction patterns revealed the formation of a single phase CuO film whose crystallinity was improved with increase of the annealing temperature. The field emission scanning electron microscopy indicated a compact and fine granular structure of the as-deposited film whereas the segregation and agglomeration of grains was observed after the film’s annealing. The photodetection performance of CuO film with Al contacts was investigated under the exposure of visible light. The current–voltage graphs of as-deposited and annealed films displayed Schottky contact between the metal and semiconductor, owing to a lower work function of Al than that of the CuO. The photo-to-dark current ratio of the device was significantly enhanced after the film’s annealing. The increase in photocurrent became more pronounced upon increasing the light intensity from 58 to 511 µW/cm². The maximum current gain and sensitivity values were found to be 66 and 6500% respectively at 10 V bias for the film annealed at 500 °C. The rise and fall time of the Al/CuO/Al photodetector was decreased after the film’s annealing.

Keywords Metal oxide semiconductor · Annealing · Copper oxide · Photodetection · Band gap

1 Introduction
Copper oxide is a II–VI semiconductor that is often obtained in two phases, named as cupric oxide (CuO) and cuprous oxide (Cu₂O). The CuO has a lower band gap (1.2 to 2.1 eV) as compared to the cuprous oxide phase (2.1 to 2.7 eV). Both of these phases find applications in various electronic and opto-electronic devices such as in solar cells, lithium batteries, light emitting diodes, photodiodes and optical fiber (Gulen et al. 2013; Bayansal et al. 2014; Oku et al. 2011; Hendi and Rashad 2018; Sullivan et al. 2016). Copper oxide
thin films have been synthesized by using various techniques including thermal evaporation, spray pyrolysis, sol–gel, chemical vapor deposition, molecular beam epitaxy, pulsed laser deposition, direct current/radiofrequency magnetron sputtering (Al-Kuhaili 2008; Moumen et al. 2019; Shariffudin 2015; Valtierra et al. 2003; Muthe et al. 1998; Farhad et al. 2020; Al-Ghamdi et al. 2016).

Post-deposition annealing of a metal oxide film in air is an effective way to enhance its crystalline quality. In the case of copper oxide, it has been realized that it is quite difficult to form cupric oxide phase without annealing. The as-prepared copper oxide films often contain cuprous oxide phase that can be changed into the cupric oxide at a certain annealing temperature (Johan et al. 2011; Figueiredo et al. 2008). Johan and co-workers (Johan et al. 2011) prepared copper oxide film on glass substrate by chemical deposition method. The films were annealed in air between 200 to 400 °C. The results showed that the films annealed at 300 °C contained mixed phases, whereas the cuprous oxide phase was transformed into the cupric oxide phase at 400 °C. Figueiredo et al. (Figueiredo et al. 2008) synthesized copper oxide film through the thermal oxidation of metallic copper and annealed it in the temperature range of 100 to 450 °C. The films at 100 °C exhibited mixed Cu and Cu$_2$O phases, whereas between 200 to 300 °C, the Cu$_2$O phase was obtained. The Cu$_2$O phase was transformed into CuO phase with further increase of the annealing temperature. Similarly, few other studies were also conducted to investigate the changes in the phases of copper oxide film through annealing process (Hojabri et al. 2014; Raship et al. 2017).

In recent times, there has been a significant interest in developing various types of photodetectors using metal oxide semiconductors (Abbas et al. 2018; Zhu et al. 2010; Zhai et al. 2009; Dushaq et al. 2019). For this purpose, a lot of work has been done to fabricate ultraviolet photodetectors based on the wide band gap semiconductors (Patel et al. 2015; Chu et al. 2020; Zhou et al. 2019; Tian et al. 2015). However, for the visible photodetectors, often silicon was utilized (Esteves et al. 2021). It has been found that the silicon based visible photodetectors are undesirable in flexible and transparent electronic applications due to their bulky and brittle properties (Song et al. 2019; Lin et al. 2015; Xie et al. 2017). Moreover, these photodetectors show lower current gain in the visible region as compared to the infrared region. Copper oxide is a p-type semiconductor that exhibits band gap corresponding to the visible region. Therefore, it can be effectively used for the photodetector application. Previous literature shows a limited work on the copper oxide based photodetectors (Song et al. 2019; Raghavendra et al. 2018; Zhang et al. 2014; Farrukh et al. 2021). Particulaly, improvement in the performance of CuO photodetectors through annealing has been rearly investigated as compared to other photodetectors (Raghavendra et al. 2018). In this work, thin film of CuO was deposited on silicon by DC magnetron sputtering, characterized for the fabrication of a metal–semiconductor-metal photodetector and its performance was improved through the annealing process.

2 Experimental work

Copper oxide (CuO) thin films were deposited on Si (100) by using direct current (DC) magnetron sputtering system. As-received silicon wafer was cut by the diamond cutter in different pieces (2×2 cm$^2$). The samples were cleaned using acetone to remove any contamination. The DC magnetron sputtering system was used for the deposition of CuO films on Si substrates. The samples were mounted on the substrate holder inside the sputtering
system. The sputtering target of pure copper was held inside the chamber. The chamber was evacuated using rotatory and turbo molecular pumps. Afterwards, argon and oxygen gasses were introduced into the chamber (Ar:O₂ = 90:10). In the absence of gasses, the base pressure was ~ 10⁻⁵ mbar. However, when gasses were entered inside the chamber, the working pressure was noted to be ~ 10⁻³ mbar. The target was sputtered at 30 W in Ar-O₂ atmosphere to deposit copper oxide film on Si substrate (Farrukh et al. 2021). The thickness of the deposited film (~ 400 nm) was estimated from the thickness profilometer fixed on the sputtering system. The average deposition rate was 0.8 Å/sec. The as-deposited films were annealed (in air) at 400 and 500 °C for 2 h in a muffle furnace. The annealed films were characterized by various techniques such as x-ray diffraction, field emission scanning electron microscope and UV–vis reflectance spectroscopy for structural, morphological, and optical measurements respectively. For the fabrication of metal–semiconductor-metal (MSM) CuO photodetector, aluminium (Al) contacts were deposited on the film by RF sputtering system using a metal mask. The schematic diagram of the metal mask is shown in Fig. 1a. The length and width of the metal mask were 0.12 mm and 3 mm respectively. The activation area for the incident light was 0.036 cm². The current–voltage system Keithly (SCS-4200) was used for the determination of photodetection characteristics of the copper oxide film. The schematic diagram of the photodetection set up is shown in Fig. 2b.

Fig. 1  a Schematic diagram of metal mask. b Schematic diagram of CuO based MSM photodetector
3 Results and discussion

3.1 X-ray diffraction results

Grazing incident x-ray diffraction (GIXRD) patterns of as-deposited and annealed CuO films in the 2θ range of 30–80° are shown in Fig. 2. These patterns show a well-defined CuO diffraction peak along (−111) plane (Farrukh et al. 2021). When the film is annealed at 400 °C, crystallinity of the film improves as the width of the diffraction peak decreases which is followed by an increase in its intensity. By increasing the annealing temperature to 500 °C, the width of CuO peak is further reduced. In addition, the diffraction peaks of CuO also appear along (200) and (202) planes. This indicates further improvement in the crystalline quality of the film. The CuO peaks along (200) and (202) planes below 500 °C could not be detected due to their low structural quality. For the observation of crystallite size (D) before and after the annealing, the Scherer formula was used;

\[ D = \frac{k\lambda}{\beta\cos\theta} \]  

Here, ‘λ’ is the wavelength of x-rays, the shape constant ‘k’ has a value of about 0.9, ‘β’ is the FWHM (in radian), ‘θ’ is the Bragg’s angle (in radian) (Hojbari et al. 2015). The values of crystallite size for as-deposited CuO film is found to be 16 nm whereas after annealing at 400 and 500 °C, the crystallite size was increased to 18 and 26 nm respectively. The increase of crystallite size after annealing is due to removal of strain/stress in the film by the heat treatment process (Malek et al. 2014). Annealing the CuO film in air relaxes the internal strains among the atoms of the film thus promoting crystallites of larger size with reduced grain boundaries. The small crystallite size shows nanocrystalline nature of the film. The microstrain in the film was evaluated from the following equation (Imran et al. 2019);
The microstrain before annealing was calculated to be 0.0021 whereas after annealing at 400 and 500 °C, the strain was reduced to 0.0018 and 0.0013 respectively. The dislocation density inside the pre- and post annealed films was found using the following formula:

\[ \delta = \frac{1}{D^2} \]  

The values of dislocation density came out to be \(38 \times 10^{14}\), \(29 \times 10^{14}\) and \(14 \times 10^{14}\) / m² for the as-deposited and annealed films at 400 and 500 °C respectively. These results depict that the CuO shows enhanced crystallinity when it is annealed at 500 °C. Annealing of CuO film facilitates the re-arrangement of atoms in the crystal and thus improves its crystalline quality.

### 3.2 FESEM analysis

The surface analysis is an important property of a thin film which may directly affect its electrical and optical properties. Therefore, investigation of the surface morphology of CuO film is of critical importance. In this work, the surface morphology of the CuO film was investigated through FESEM and the images obtained are presented in Fig. 3a–c. All these images were captured at 500 nm along with a magnification of 100,000 X. The micrograph of the as-deposited film is shown in Fig. 3a. The figure reveals a compact and smooth surface of the film containing fine granular structure with homogeneous distribution of grains over the surface. The average grain size of CuO was estimated to be 25 nm. The surface features of CuO film annealed at 400 °C and at 500 °C are shown in Fig. 3b, c respectively. The film annealed at 400 °C indicates that the grains are not as inter-connected to each other as observed in the case of as-deposited film. However, the average grain size becomes larger than that of the as-deposited film (30 nm). A few cracks on the film’s surface are also seen due to the thermal stresses generated during the annealing process. The thermal energy not only facilitates in the re-arrangement of atoms, but it also produces thermal stresses that may result in the generation of cracks inside the film. When the annealing temperature increases to 500 °C, then small grains merge together to form larger grains due to increased thermal energy. The average grain size at 500 °C is found to be 42 nm. The grains are slightly elongated and the film structure becomes denser as shown in the Fig. 3c. The surface also shows small cracks due to thermal stresses as explained above. These cracks normally occur in the CuO film when it is given a heat treatment and it is in accordance with the literature (Akgul et al. 2014). These results are consistent with the XRD results in which crystallite size was increased with increase of the annealing temperature.

### 3.3 Optical characterization

The optical characterization of copper oxide film of optimum crystallinity (annealed at 500 °C) was performed by using ultraviolet–visible reflectance spectroscopy to obtain the film’s band gap. The spectroscopy was performed in the reflectance mode and the reflectance spectrum of the film is shown in Fig. 4. The spectrum shows constructive and destructive interference fringes due to light reflections from the CuO film as well as...
from the silicon substrate. The reflected light waves interfere with each other and produce fringes. At a certain wavelength, the copper film absorbs energy and these fringes vanish. The point where the fringes disappear is referred to as cut-off wavelength point. The optical band gap of the CuO film was calculated by finding the cut-off wavelength through the following equation (Farrukh et al. 2021; Younas et al. 2019):

\[ E = \frac{hc}{\lambda} \]  

Here, \( h \) = Planck’s constant, \( c \) = speed of light, \( \lambda \) = wavelength of light

The cut-off wavelength of the CuO film was 470 nm whereas the corresponding band gap energy was calculated to be 2.63 eV. The value of band gap energy of CuO film in this work is higher than its normal value range. This may be due to quantum size effect that occurs in the film due to its small crystallite size which results into a splitting of energy.
levels of the film (Farrukh et al. 2021; Rehman et al. 2011). In this way, the distance between the energy levels/bands of the film increases, which increases the band gap.

### 3.4 Photodetection characteristics

#### 3.4.1 Current–voltage characteristics

Current–voltage (I–V) curves of the metal–semiconductor-metal (MSM) photodetectors based on the as-deposited and annealed CuO films are shown in Fig. 5a–c. These curves were taken out at a bias voltage of 10 V. The curve without light illumination represents dark I-V characteristics, whereas the curve under visible light ($\lambda = 460$ nm, $I = 58$ µW/cm$^2$) represents photo I-V characteristics. As we see, that the I-V characteristics of the as-deposited film do not display any appreciable difference between the dark and photocurrents (Fig. 5a). These curves show inconsistent behavior in the variation of current with voltage. The insignificant increase in photocurrent from the as-deposited film is due to the presence of structural defects (such as vacancies and interstitials) in it (Ghicov et al. 2006). These defects may act as trapping centers which reduce the flow of charge carriers, causing insignificant photocurrent. Moreover, the inhomogeneous metal–semiconductor interface in this film may be responsible for producing the uneven I-V curves.

The I-V curves of the MSM photodetector at 400 °C are shown in Fig. 5b. The figure shows an increase in the photocurrent when the device is exposed to visible light. The increase in photocurrent at 400 °C is more significant as compared to the as-deposited film. This may be due to reduction in the structural defects inside the film due to its annealing. The increase in photocurrent with respect to the dark current is ascribed to the generation of free electron–hole pairs by the incident light. The Fig. 5b shows the formation of Schottky contact between the metal (Al) and semiconductor (CuO film). The Schottky contact is formed due to lower work function of Al (4.1 eV) as compared to the p-type CuO film (4.7 eV). Consequently, holes from p-CuO move towards Al and cause the Schottky barrier between the metal and semiconductor. The I-V curves of the device at 500 °C are shown in Fig. 5c. The figure shows that with increase of the annealing temperature from 400 to 500 °C, the photo to dark current ratio increases which indicates higher current
The annealing process reduces the structural defects inside the film and allows an easy movement of the free charge carriers generated by the incident light. Therefore, the device performance is improved (Hajimazdarani et al. 2020).

### 3.4.2 Current–voltage characteristic at variable light intensities

The I-V characteristics of CuO film annealed at 400 °C and at 500 °C are shown in Fig. 6a-b respectively. The Fig. 6a shows that with increase in the light intensity, the photocurrent increases. The increase in photocurrent is attributed to the generation of more electron–hole pairs by the incident light (Gunasekran et al. 2020). Greater the number of incident photons, more will be the generated charge carriers and vice versa. Therefore, the photocurrent increases consistently with increasing the light intensity. The Schottky contact between the metal and semiconductor is also noticeable from the figure. Similarly, the I-V curves of CuO film annealed at 500 °C show increase in the photocurrent both in forward and reverse directions with increase of the light intensity (Fig. 6b). However, as compared to the film annealed at lower temperature (400 °C), the values of photocurrent
are higher at 500 °C, which is due to the better crystalline quality of the film (Chen et al. 2013).

3.4.3 Current–time (I-t) calculations at 10 V

The I-t measurements of the CuO photodetector were made to observe the repeatability performance of the fabricated device. These measurements were recorded by switching ON and OFF the visible light after regular intervals. Figure 7 shows I-t graphs of as-deposited and annealed films (400 °C). We see that the current increases when the light is incident on the film and once the light is turned OFF, the current falls again to its initial level. This means that the photodetector works consistently under the exposure of light. Both the dark and photocurrents of the as-deposited film are quite low, therefore, the pulses are not clearly visible on this larger graph scale. However, the photocurrent increases with increase
of the annealing temperature. The difference between dark and photocurrents is larger in the case of CuO film annealed at 500 °C as compared to that annealed at 400 °C (Fig. 7). Similarly, when the intensity of light is changed, then the I-t graphs show a consistent increase in the heights of the I-t pulses, indicating an increase in the photocurrent. These results show that by increasing the annealing temperature as well as the light intensities, the device shows better performance. In order to evaluate the current gain (G) and sensitivity (S), the following equations were used (Yusoff et al. 2015; Ahmad et al. 2020):

\[ G = \frac{I_p}{I_d} \]  \hspace{1cm} (5)

\[ S = \frac{I_p - I_d}{I_d} \times 100 \]  \hspace{1cm} (6)

Here, \( I_p \) is the photocurrent and \( I_d \) is the dark current. The values of current gain and sensitivity of the device at a fixed light intensity (58 µW/cm²) are shown in Table 1. The current gain and sensitivity increase with increase of the annealing temperature to 500 °C. The Table 1 shows that the current gain and sensitivity of the CuO photodetector increase after the film’s annealing. The maximum value of current gain and sensitivity at 58 µW/cm²

| Annealing temperature (°C) | Dark current (A) | Photocurrent (A) | Current gain | Sensitivity (%) |
|----------------------------|-----------------|-----------------|--------------|----------------|
| As-deposited               | 2.76 × 10⁻⁶     | 3.83 × 10⁻⁶     | 1.38         | 38.76          |
| 400                        | 3.53 × 10⁻⁵     | 5.93 × 10⁻⁴     | 16.8         | 1579           |
| 500                        | 9.9 × 10⁻⁶      | 4.73 × 10⁻⁴     | 47.7         | 4677           |
are found to be 47.7 and 4677% respectively for the film annealed at 500 °C. Similarly, the current gain and sensitivity values of CuO device at different light intensities are given in Tables 2, Table 3 and Table 4. The Table 2 presents the photodetection parameters of the as-deposited film at various light intensities. Similarly, Tables 3 and Table 4 show the photodetection results of the annealed films at 400 and 500 °C respectively. The photocurrent, current gain and sensitivity increase with increase of the light intensity from 87 to 511 µW/cm² at 10 V bias. The maximum current gain and sensitivity values are found to be 66 and 6500% respectively at 500 °C. By increasing the light intensity, more free charge carriers (electrons and holes) are generated that result in an increase of the photocurrent. Hence, the current increases by varying the light intensities, resulting in an increase in the sensitivity and current gain of the device. These results show that the photodetection characteristics of the film under visible light exposure are improved after the films’ annealing particularly at 500 °C.

3.4.4 Rise time and fall time

Table 2  Current gain and sensitivity of as-deposited CuO film at different light intensities

| Light intensities (µW/cm²) | Dark current (A) | Photocurrent (A) | Current gain | Sensitivity (%) |
|-----------------------------|------------------|------------------|--------------|----------------|
| 87                          | 2.21×10⁻⁶        | 3.01×10⁻⁶        | 1.36         | 36.2           |
| 146                         | 2.21×10⁻⁶        | 3.35×10⁻⁶        | 1.51         | 51.6           |
| 438                         | 2.21×10⁻⁶        | 3.54×10⁻⁶        | 1.60         | 60.1           |
| 470                         | 2.21×10⁻⁶        | 3.94×10⁻⁶        | 1.78         | 78.28          |
| 511                         | 2.21×10⁻⁶        | 4.18×10⁻⁶        | 1.9          | 89.1           |

Table 3  Current gain/sensitivity of CuO film annealed at 400 °C at various light intensities

| Light intensities (µW/cm²) | Dark current (A) | Photocurrent (A) | Current gain | Sensitivity (%) |
|-----------------------------|------------------|------------------|--------------|----------------|
| 87                          | 4.22×10⁻⁵        | 6.08×10⁻⁴        | 14.40        | 1340           |
| 146                         | 4.22×10⁻⁵        | 6.69×10⁻⁴        | 15.85        | 1485.3         |
| 438                         | 4.22×10⁻⁵        | 7.43×10⁻⁴        | 17.60        | 1660           |
| 470                         | 4.22×10⁻⁵        | 8×10⁻⁴           | 18.95        | 1795           |
| 511                         | 4.22×10⁻⁵        | 8.9×10⁻⁴         | 22           | 2009           |

Table 4  Current gain/sensitivity of CuO film annealed at 500 °C at various light intensities

| Light intensities (µW/cm²) | Dark current (A) | Photocurrent (A) | Current gain | Sensitivity (%) |
|-----------------------------|------------------|------------------|--------------|----------------|
| 87                          | 9.91×10⁻⁶        | 4.86×10⁻⁴        | 49           | 4804           |
| 146                         | 9.91×10⁻⁶        | 5.35×10⁻⁴        | 54           | 5298           |
| 438                         | 9.91×10⁻⁶        | 5.70×10⁻⁴        | 58           | 5651           |
| 470                         | 9.91×10⁻⁶        | 6×10⁻⁴           | 61           | 5954           |
| 511                         | 9.91×10⁻⁶        | 6.54×10⁻⁴        | 66           | 6500           |
In order to further evaluate the performance of the device, the rise and fall time of the device was calculated by magnifying the I-t pulses. The values of rise and fall time at 511 µW/cm² are given in Table 5. The rise and fall time decrease after the film’s annealing. The shortest values of rise time (0.16 s) and fall time (0.26 s) were obtained for the CuO film annealed at 500 °C. This shows that the reduction in structural defects allows the free charge carriers to move faster inside the film, allowing them to take shorter time to reach their respective electrodes. A comparison of the photodetection parameters of different types of photodetectors is given in Table 6.

| Table 5 Rise and fall time CuO film at 511 µW/cm² |
|-----------------------------------------------|
| Annealing temperature (°C) | Rise time (sec) | Fall time (sec) |
| As-deposited | 0.39 | 0.99 |
| 400 | 0.36 | 0.42 |
| 500 | 0.16 | 0.26 |

4 Conclusions

Annealing of DC sputtered CuO film on silicon substrate improves its photodetection performance. The MSM device based on the as-deposited film exhibits low values of current gain and sensitivity under the exposure of visible light of intensity 58 µW/cm², whereas after the film’s anealing up to 500 °C, both sensitivity and current gain of the device are considerably increased. Similarly, by increasing the incident light intensity from 58 to 511 µW/cm² at 10 V bias, the current gain and sensitivity are further increased. The maximum values of current gain and sensitivity in this work are found to be 66 and 6500% respectively at 500 °C. The increase in photodetection performance of the device is attributed to the reduction in structural defects inside the CuO film due to its annealing. The results of present work show that the high performance visible light sensitive CuO photodetectors can be prepared by optimizing the CuO film’s properties through the annealing treatment.
Table 6 Comparison of photodetection parameters of different types of photodetectors

| Type of materials | Type of detector | Fabrication technique | Wavelength $\lambda$ (nm) | Bias voltage (V) | Sensitivity (%) | Rise time (sec) | Reference |
|-------------------|------------------|-----------------------|---------------------------|-----------------|----------------|----------------|-----------|
| CuO               | MSM              | DC Sputtering         | 460                       | 10              | $6.5 \times 10^3$ | 0.16           | Present Work |
| CuO               | MSM              | Electron beam evaporation | 450                       | 5               | 40             | 53.8           | Raghavendra et al. (2018) |
| Cu$_2$O           | Heterojunction   | Hydrothermal method   | 425                       | 0               | -              | 0.0058         | Bai and Zhang (2016) |
| MoS$_2$           | MSM              | Chemical              | 500                       | 10              | -              | 0.00011        | Tsai et al. (2013) |
| CdS               | MSM              | CVD                   | 500                       | 1               | $9.7 \times 10^3$ | 0.009          | Husham et al. (2016) |
| V$_2$O$_5$        | MSM              | Spray pyrolysis       | 540                       | 5               | $2.6 \times 10^3$ | 0.78           | Abd-Alghafoor et al. (2016) |
| InAlN             | MSM              | Co-sputtering         | 520                       | 5               | $4.8 \times 10^3$ | 0.62           | Afzal and Devarajan (2016) |
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Declarations

Conflict of interest There is no conflict of interest/competing interest for this work.

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