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Air Annealing Effects on Spray-Deposited CuO Thin Films and p-CuO/n-Si Heterojunction Photo Response

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Abstract: CuO thin films were deposited on quartz and Si substrates by spray pyrolysis using a 0.1 M copper acetate precursor at a substrate temperature of 350 °C for 4 min and subsequently air-annealed at 450 and 500 °C for 1 h. The influence of annealing temperature on the structural, optical, electrical, and photo response properties of the films was investigated. XRD analysis confirmed the formation of predominantly single-phase monoclinic CuO, with improved crystallinity after annealing. The crystallite size increased from 5.22 nm for the as-deposited film to 8.13 nm for the C4-A500 film, while the crystallinity increased from 61.9% to 85.6%. Optical studies revealed enhanced absorption after annealing, with C4-A500 showing the highest absorption. Hall measurements confirmed p-type conductivity for all films, with a significant reduction in resistivity from 8.362 Ω m to 0.265 Ω m after annealing at 450 °C and a maximum carrier mobility of 161 × 10^{-13} m² V^{-1} s^{-1} for C4-A500. Furthermore, p-CuO/n-Si heterojunction devices fabricated using the annealed films exhibited clear rectifying and photovoltaic behaviour. At a reverse bias of −1 V, the responsivity increased from approximately 4.7 mA/W for C4-A450 to 17 mA/W for C4-A500. The results demonstrate that air annealing, particularly at 500 °C, significantly improves the structural, optical, and photo response characteristics of spray-deposited CuO films, making them promising for photodetector and optoelectronic applications.

Keywords: *Copper Oxide, Spray Pyrolysis, Heterojunction, Responsivity.*

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I. INTRODUCTION

Copper oxide (CuO) has attracted considerable attention as a p-type semiconductor because of its direct band gap, high optical absorption coefficient, environmental compatibility, and low fabrication cost. Owing to these properties, CuO has been extensively investigated for applications in photovoltaic devices, photodetectors, gas sensors, lithium-ion batteries, and supercapacitors.

Among various deposition techniques, spray pyrolysis is a simple, scalable, and cost-effective method for depositing uniform CuO thin films over large areas [1, 2]. The structural and electrical properties of spray-deposited CuO films strongly depend on deposition parameters and post-deposition heat treatment [3].

Although CuO/n-Si heterojunctions have been investigated primarily as solar cells, their relatively low photovoltaic conversion efficiency limits their practical use in energy harvesting applications. However, these heterojunctions exhibit excellent photo response under reverse bias, suggesting their potential as visible-light photodiodes [4].

The present work investigates the photo response characteristics of 4-minute spray deposited p-CuO/n-Si heterojunctions fabricated under different annealing conditions. Particular emphasis is placed on evaluating the responsivity and external quantum efficiency to assess their suitability for photodetection applications.

II. MATERIALS AND METHODS

A 0.1 M copper acetate precursor solution was prepared by dissolving the required amount of copper acetate monohydrate (Sigma-Aldrich) in double-distilled water and magnetically stirring it at room temperature for about 10 min to obtain a homogeneous solution. The precursor was then spray-deposited onto pre-cleaned quartz and Si substrates of area maintained at 350 °C. Quartz substrates were cleaned sequentially with DI water, chromic acid, and acetone, whereas Si substrates were cleaned using standard RCA-1 and RCA-2 procedures. The deposition was performed for 48 cycles, with each cycle consisting of a 5 s spray period followed by a 60 s stop period**.** After deposition, the CuO films were air-annealed at 450 and 500 °C for 1 hour**.**

The 4-min spray deposited CuO thin film was designated as C4-ASD in the as-deposited state, while the films air-annealed at 450°C and 500°C for 1 hour were designated as C4-A450 and C4-A500, respectively.

III. RESULTS AND DISCUSSION

- *Structural Properties*

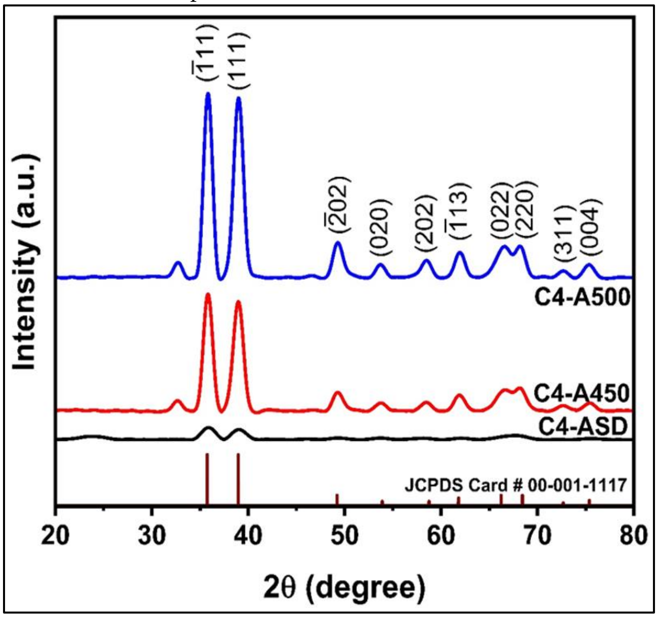

Fig 1 XRD Patterns of C4-ASD, C4-A450 and C4-A500 CuO Thin Films.

The XRD patterns of the C4-ASD, C4-A450 and C4-A500 CuO thin films are shown in Fig. 1. The as-deposited film (C4-ASD) exhibits relatively weak diffraction peaks, indicating poor crystallinity. After air annealing, the diffraction peaks become sharper and more intense, particularly for C4-A500, confirming an improvement in crystallinity with increasing annealing temperature.

The prominent peaks at and are indexed to the and (111) planes, respectively. The additional reflections at approximately 49.2°, 53.8°, 58.5°, 61.8°, 66.2°, 68.0°, 72.5° and 75.3° correspond to the, (020), (202), (022), (220), (311) and (004) planes of monoclinic CuO. These reflections are consistent with JCPDS card No. 00-001-1117. The absence of additional peaks indicates the formation of predominantly single-phase monoclinic CuO. The enhancement in peak intensity and sharpness after annealing can be attributed to improved atomic rearrangement and reduction of structural disorder. Thus, air annealing, especially at 500 °C, enhances the crystalline quality of the CuO thin films [5].

Table 1 Structural Parameters of C4-ASD, C4-A450 and C4-A500 CuO Thin Films.

Sample	2θ degree	β	D nm	d nm	δ nm	ε	Crystallinity %
C4-ASD	35.88	1.6699	5.222	0.2501	0.0153	0.0225	61.9
C4-A450	35.82	1.1306	7.713	0.2505	0.0065	0.0153	82.1
C4-A500	35.82	1.0731	8.126	0.2505	0.0058	0.0145	85.6

The structural parameters calculated from the most intense peak ($\bar{1}11$) are presented in Table 1. The FWHM (β) decreases from 1.6699° to 1.0731°**,** while the crystallite size increases from 5.22 to 8.13 nm with annealing. The nearly constant -spacing (~0.250 nm) confirms that the CuO crystal structure is retained. The decrease in strain and defect-related parameters, together with the increase in crystallinity from 61.9% to 85.6%**,** indicates improved structural ordering and crystallite growth upon air annealing. Thus, C4-A500 exhibits the best structural quality among the studied films**.**

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➢ *Optical Properties*

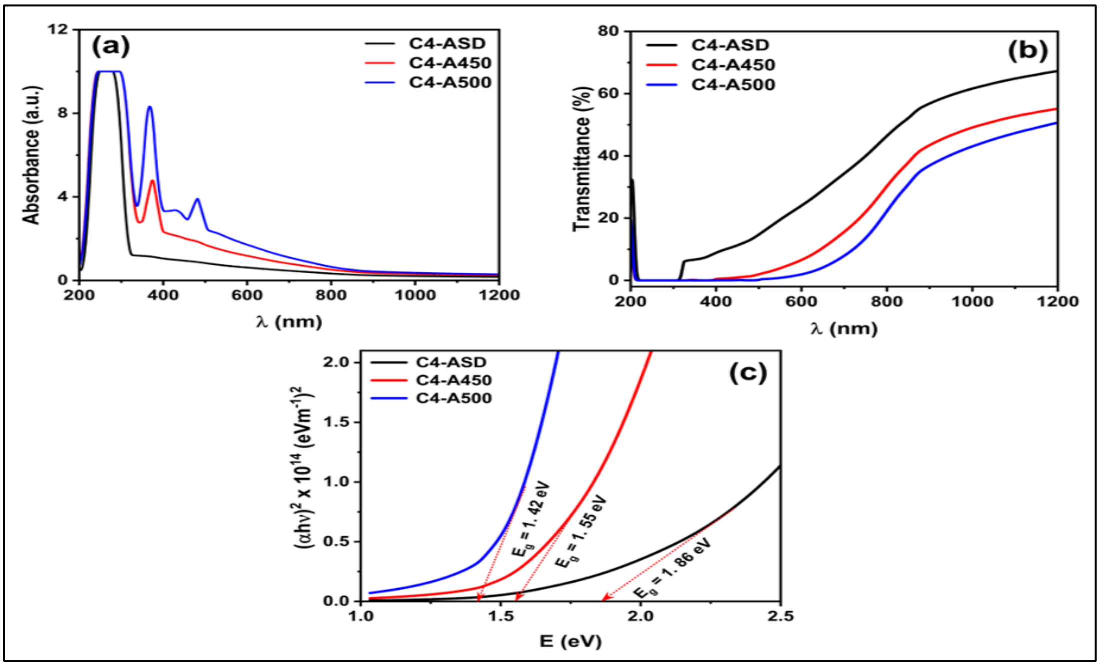

Fig 2 UV–Vis–NIR Spectra of CuO Thin Films: (a) Absorbance, (b) Transmittance and (c) Tauc Plots.

Fig 2(a) shows the absorbance spectra and Fig. 2(b) the transmittance spectra of the as deposited and air annealed Cuo thin films. The C4-ASD film exhibits relatively lower absorption in the visible region, which can be attributed to its lower crystallinity and higher structural disorder. Air annealing at 450 and 500 °C enhances the optical absorption due to improved crystallinity, grain growth, and reduction of defect states. A slight red shift of the absorption edge is also observed after annealing, indicating a marginal reduction in the optical band gap, possibly due to strain relaxation and changes in the electronic structure. Among the studied films, C4-A500 exhibits the highest absorption, indicating improved optical quality and enhanced light-harvesting capability.

The Tauc plots in Fig. 2(c) show that the optical band gap of the SPD CuO films changes with air annealing. The estimated band-gap energies are 1.86 eV for C4-ASD, 1.55 eV for C4-A450, and 1.42 eV for C4-A500**.** Thus, the band gap decreases progressively with increasing annealing temperature [5]. This reduction can be associated with improved crystallinity, grain growth, strain relaxation, and changes in defect states and the electronic structure of the CuO films. The reduced band gap after annealing also agrees with the observed red shift of the absorption edge**.** Among the samples, C4-A500 exhibits the lowest band gap **(**1.42 eV**),** indicating enhanced visible-light absorption and improved optical response.

The electrical parameters of the CuO thin films are presented in Table 2. All the samples exhibit p-type conductivity, confirming CuO as a p-type semiconductor. The as-deposited C4-ASD film shows a high resistivity of 8.362 Ω m, which decreases significantly to 0.265 Ω m after annealing at 450 °C. At 500 °C, the resistivity slightly increases to 0.401 Ω m. The carrier concentration decreases markedly from to m with annealing, while the Hall mobility increases from 0.658 to 161 ×10 m² V s. The increase in mobility can be attributed to improved crystallinity and reduced structural defects following annealing. The relatively low resistivity of C4-A450 indicates better electrical conductivity, whereas C4-A500 exhibits the highest carrier mobility. Overall, air annealing significantly modifies [5] the electrical properties of the CuO films by improving carrier transport.

Table 2 Hall effect Parameters of C4-ASD, C4-A450 and C4-A500 CuO Thin Films.

Sample	R_s **MΩ**	**T nm**	ρ Ωm	V_H **mV**	R_H **x10⁻⁸** m^3C^{-1}	p_s **x10²⁵** m^{-2}	**P x10³²** m^{-3}	μ **x10⁻¹³** $m^2V^{-1}s^{-1}$	**Material Type**
C4-ASD	18.398	454.5	8.362	0.15	0.55	51.6	11.36	0.658	*p*
C4-A450	0.713	371.6	0.265	2.22	7.34	3.16	0.85	27.7	*p*
C4-A500	1.520	263.9	0.401	9.81	64.70	0.254	0.096	161	*p*

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IV. FABRICATION OF P-CUO/N-SI HETEROJUNCTION DEVICES

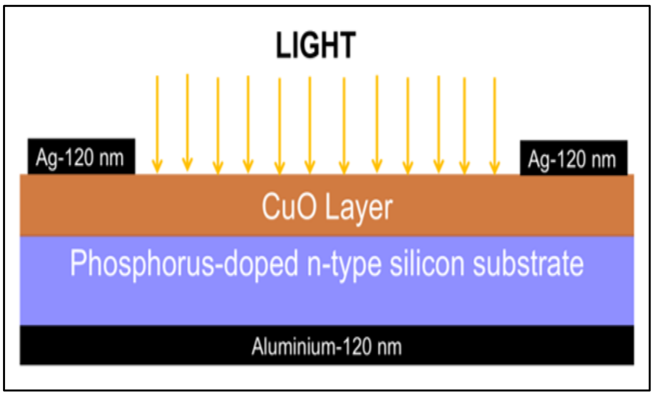

Fig 3 Schematic Structure of the Fabricated p-CuO/n-Si Heterojunction Device.

Fig 3 represents the schematic structure of the fabricated p-CuO/n-Si heterojunction device with Ag and Al ohmic contacts. Spray-deposited CuO thin films prepared for 4 min and air-annealed at 450°C and 500°C were used to fabricate p-CuO/n-Si heterojunction devices. The CuO films were deposited on polished phosphorus-doped n-type Si wafers by spray pyrolysis, forming a p–n heterojunction at the CuO/Si interface. A circular Ag top contact of 2 mm diameter and 120 nm thickness was deposited on the CuO surface by RF sputtering, while a 120 nm thick Al layer was deposited on the backside of the Si substrate as the rear ohmic contact. The built-in depletion region at the p-CuO/n-Si interface provides the rectifying and photovoltaic characteristics of the fabricated devices [6].

V. PHOTO RESPONSE CHARACTERISTICS OF P-CUO/N-SI HETEROJUNCTIONS

➢ *Current–Voltage Characteristics*

Current-voltage characteristics were measured under dark and illuminated conditions using a Class AAA solar simulator equipped with a xenon lamp.

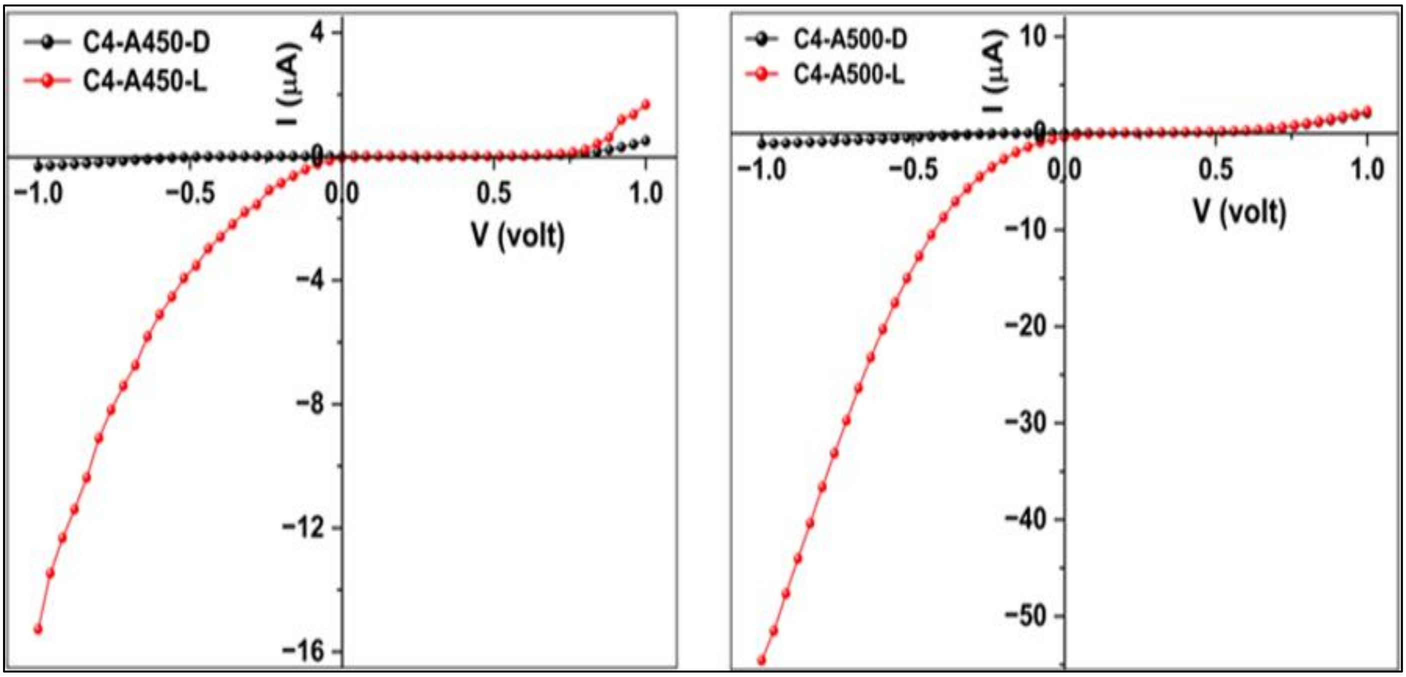

Fig 4 Dark and Illuminated I–V Characteristics of p-CuO/n-Si Heterojunction Devices Annealed at 450 and 500 °C.

The characteristics of the fabricated p-CuO/n-Si heterojunction devices annealed at 450 °C and 500 °C are shown in Fig. 4. Both devices exhibit clear rectifying behaviour, confirming the formation of a p–n heterojunction [4]. Under illumination, the current increases significantly, particularly in the reverse-bias region, demonstrating a clear photovoltaic response. The C4-A500 device shows a higher photocurrent than C4-A450, indicating improved photo response with increasing annealing temperature.

➢ *Photo Responsivity*

The photocurrent (I_{photo}) was calculated from the difference between the current measured under illumination (I_L) and the dark current (I_D):

$$I_{photo} = I_L - I_D \tag{1}$$

The photoresponsivity (R), which represents the photocurrent generated per unit incident optical power, was determined using:

$$R = \frac{I_{photo}}{P_{in}} \tag{2}$$

where P_{in} is the incident optical power falling on the active area of the device. The responsivity is expressed in mA/W. A higher responsivity indicates more efficient conversion of incident optical radiation into electrical current.

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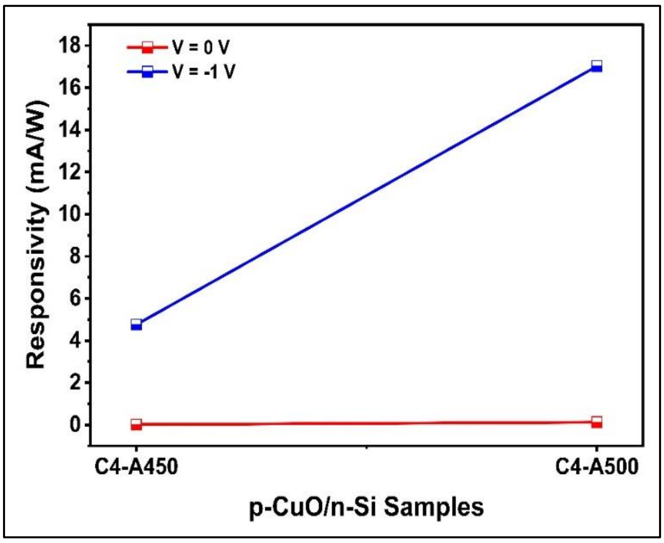

Fig 5 Responsivity characteristics of p-CuO/n-Si heterojunction devices at different bias voltages.

The responsivity of the 4-minute spray deposited p-CuO/n-Si heterojunction devices were investigated under zero bias and reverse bias conditions.

The responsivity of the p-CuO/n-Si heterojunction devices are shown in Fig. 5. At 0 V bias**,** the responsivity is very low for both C4-A450 and C4-A500. In contrast, at a reverse bias of −1 V, the responsivity increases significantly from approximately 4.7 mA/W for C4-A450 to 17 mA/W for C4-A500**.** The enhanced responsivity at reverse bias can be attributed to the increased electric field across the depletion region, which promotes efficient separation and collection of photogenerated charge carriers. The higher responsivity of C4-A500 indicates improved photo response and carrier collection following annealing at 500ºC [6].

VI. CONCLUSIONS

CuO thin films were successfully deposited by spray pyrolysis and subsequently air-annealed at 450°C and 500 °C. XRD results confirmed monoclinic CuO with enhanced crystallinity upon annealing; the crystallinity increased from 61.9% to 85.6%, accompanied by an increase in crystallite size from 5.22 to 8.13 nm and reduced strain and defect-related parameters. Annealing also enhanced the optical absorption and improved the electrical transport properties of the films. All samples exhibited p-type conductivity, with C4-A450 showing the lowest resistivity, while C4-A500 exhibited the highest carrier mobility. The fabricated p-CuO/n-Si heterojunctions showed clear rectifying and photovoltaic behaviour. Under −1 V reverse bias, the responsivity reached 17 mA/W for C4-A500, compared with 4.7 mA/W for C4-A450. Overall, air annealing at 500 °C produced the best combination of structural quality, optical absorption, carrier mobility, and photo response, demonstrating the potential of spray-deposited CuO for heterojunction photodetector and optoelectronic applications.

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