

Systematic Approach to Higher Fe Doping in CuO Materials

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Abstract: In the present work, pure and Fe-doped CuO thin films were successfully deposited onto glass substrates using the spray pyrolysis technique. The influence of Fe incorporation on the structural and morphological properties of CuO thin films was systematically investigated. X-ray diffraction (XRD) analysis confirmed the formation of monoclinic CuO with good crystallinity. The diffraction peaks observed in the patterns correspond to the characteristic reflections of CuO, and their intensity and sharpness were noticeably enhanced after Fe doping, indicating improved crystalline quality of the films without the presence of secondary phases.

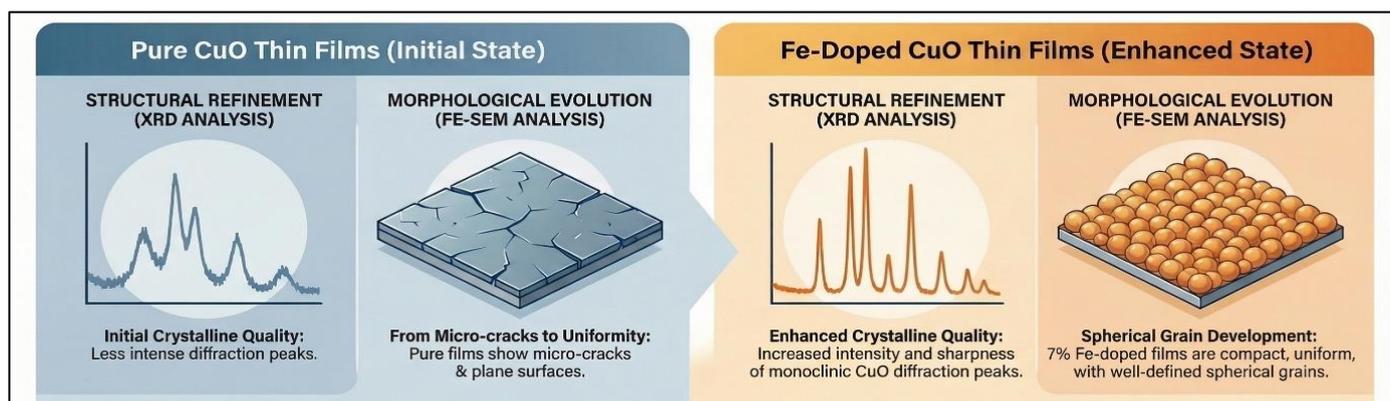


Fig 1 Graphical Abstract.

Field emission scanning electron microscopy (FE-SEM) was employed to examine the surface morphology of the deposited films. Fe-doped CuO film (7%) showed a more uniform and compact morphology consisting of well-defined spherical grains. The incorporation of Fe significantly modified the growth behavior of CuO, resulting in improved surface uniformity. These results show the doping importance of Fe the potential applications in electronic and sensing devices.

Keywords: CuO, Fe, Spray Pyrolysis, Thin Film, XRD, FE-SEM.

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I. INTRODUCTION

Transition metal oxide semiconductors have attracted considerable attention due to their remarkable structural stability, tunable electrical properties, and wide range of technological applications [1-3]. Among these materials, copper oxide (CuO) has emerged as an important p-type semiconductor owing to its narrow band gap (1.2–1.9 eV), good chemical stability, non-toxicity, and low cost [4-7]. Because of these properties, CuO has been extensively

investigated for applications in gas sensors, catalysis, lithium-ion batteries, solar cells, antimicrobial coatings, and optoelectronic devices [8].

The physical and functional properties of CuO can be significantly modified by incorporating suitable dopant elements. In particular, transition metal doping has proven to be an effective strategy to tailor the structural, electrical, optical, and sensing characteristics of CuO thin films. Iron (Fe) is considered an attractive dopant because its ionic radius

is comparable to that of Cu^{2+} ions, which allows it to substitute into the CuO lattice without causing major structural distortion [9]. Among the various deposition methods, spray pyrolysis is widely used due to its simplicity, cost-effectiveness, scalability, and suitability for large-area thin film preparation. This technique enables precise control over film thickness, composition, and surface morphology by adjusting deposition parameters such as precursor concentration, substrate temperature, and spray rate [10].

In the present work, Fe-doped CuO thin films were synthesized using the spray pyrolysis technique with varying iron concentrations. The influence of Fe incorporation on the structural and morphological properties of CuO thin films was systematically investigated. The obtained results provide valuable insight into the role of cobalt doping in modifying the microstructure and functional characteristics of CuO thin films, which may be beneficial for advanced sensing and optoelectronic applications [11, 12].

II. EXPERIMENTAL AND METHODS

➤ Materials

Copper (II) acetate monohydrate ($\text{CH}_3\text{OOH})_2\text{Cu}\cdot\text{H}_2\text{O}$ and ferrous acetate tetrahydrate ($\text{Fe}(\text{CH}_3\text{COO})_2\cdot 4\text{H}_2\text{O}$) ~99.00% purity, AR grade were treated as host and dopant precursors, procured from SD fine-chem. Ltd. India. Glass slides with dimension 75*25*1.35 mm from blue-star was used. For cleaning purpose, chromic trioxide and acetone were purchased from Global Nanotech, India.

Before deposition, glass slides were cleaned with labolene and washed under running tap water. After drying, slides were dipped in chromic trioxide solution which is

boiled at 175°C for 2 hours. Then slides are washed with Distilled water and kept for ultrasonic cleaning for 30 mins in the solution of acetone and Deionized water in 1:3 ratio. After using a drayer, slides become ready to use.

➤ Synthesis of CuO and 7% Fe Doped CuO

Pure and Fe doped CuO (7 %) thin film preparation is carried out on soda lime glass substrate using spray pyrolysis setup. For host precursor, 0.1 m of copper acetate was added to 20 ml of DDW and stirred until a homogeneous solution was formed. Then this precursor solution is sprayed onto a glass substrate. Similarly, Dopant precursor solution is formed by mixing Cobalt acetate 0.1 molar into 20 ml DDW and added drop-wise into host precursor solution, to achieve desired doping concentrations.

➤ Material analysis

Characterization techniques were employed to investigate the structural, morphological, optical, and functional properties of the synthesized samples. The structural properties were analyzed using X-ray diffraction (Ultima IV, Rigaku, Japan) with Cu-K α radiation ($\lambda = 1.54 \text{ \AA}$) in the 2θ range of 20–80°. Morphological features such as grain size and surface texture were examined using Field Emission Scanning Electron Microscopy (FE-SEM, SUPRA55, Zeiss).

III. RESULTS AND DISCUSSION

➤ Result

• XRD Analysis

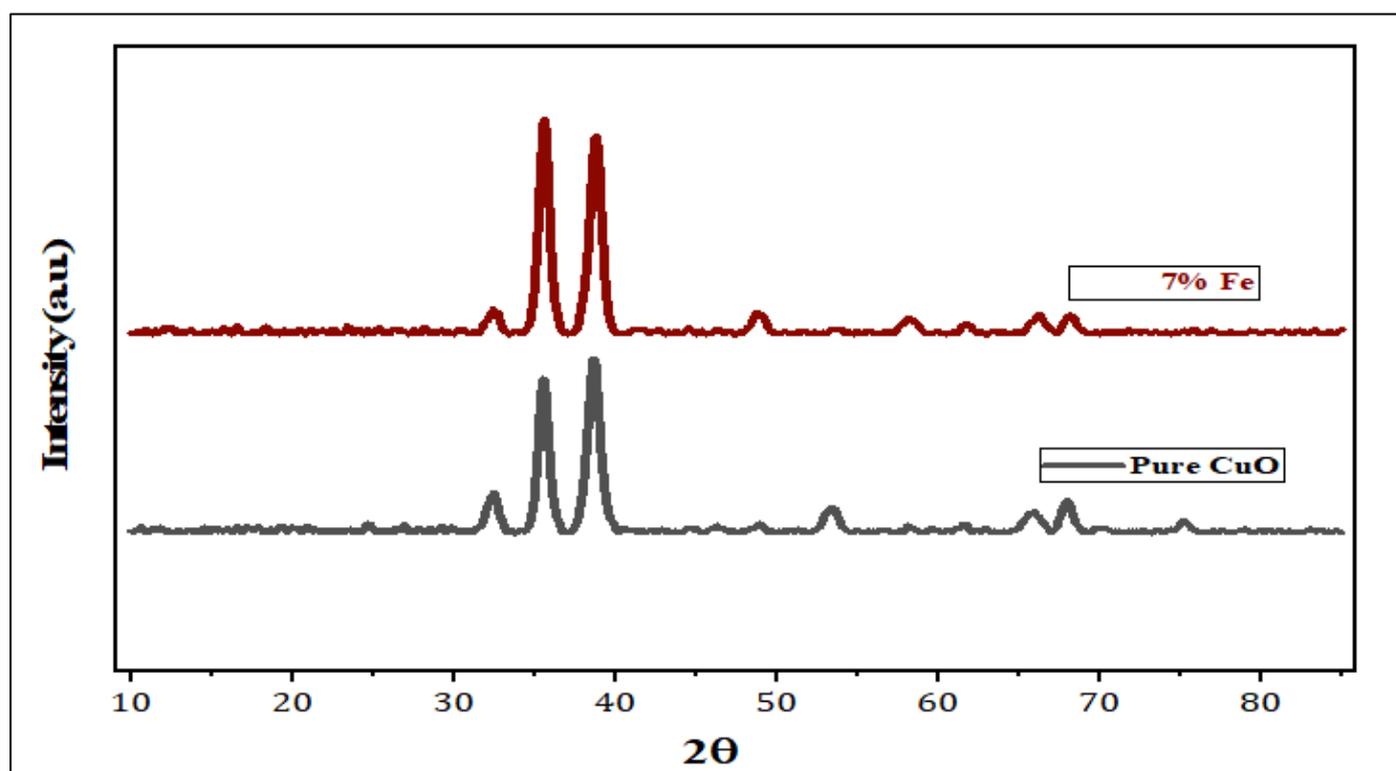


Fig 2 XRD pattern of CuO and Fe Doped CuO Thin Films.

The structural characteristics of Fe-doped CuO thin films were analyzed using X-ray diffraction (XRD) with CuK α radiation over a diffraction angle (2θ) range of 20° – 80° . Figure 2 shows the XRD patterns of pure and 7% Fe-doped CuO thin films deposited by the spray pyrolysis technique. The diffraction patterns reveal several well-defined peaks appearing at approximately 32.2° , 35.5° , 38.7° , 49.2° , and 61.6° , indicating the formation of crystalline CuO [13-16].

The sharp and intense nature of the diffraction peaks suggests that the deposited films possess good crystallinity and phase purity. All observed peaks are

consistent with the standard diffraction data of monoclinic CuO, confirming the successful formation of CuO thin films without the appearance of secondary impurity phases. The obtained diffraction patterns are in good agreement with the standard JCPDS card No. 48-1548, which corresponds to monoclinic CuO structure [17]. These results indicate that Fe incorporation does not significantly disturb the crystal structure of CuO but slightly influences the crystallinity of the films.

- *FE-SEM Analysis*

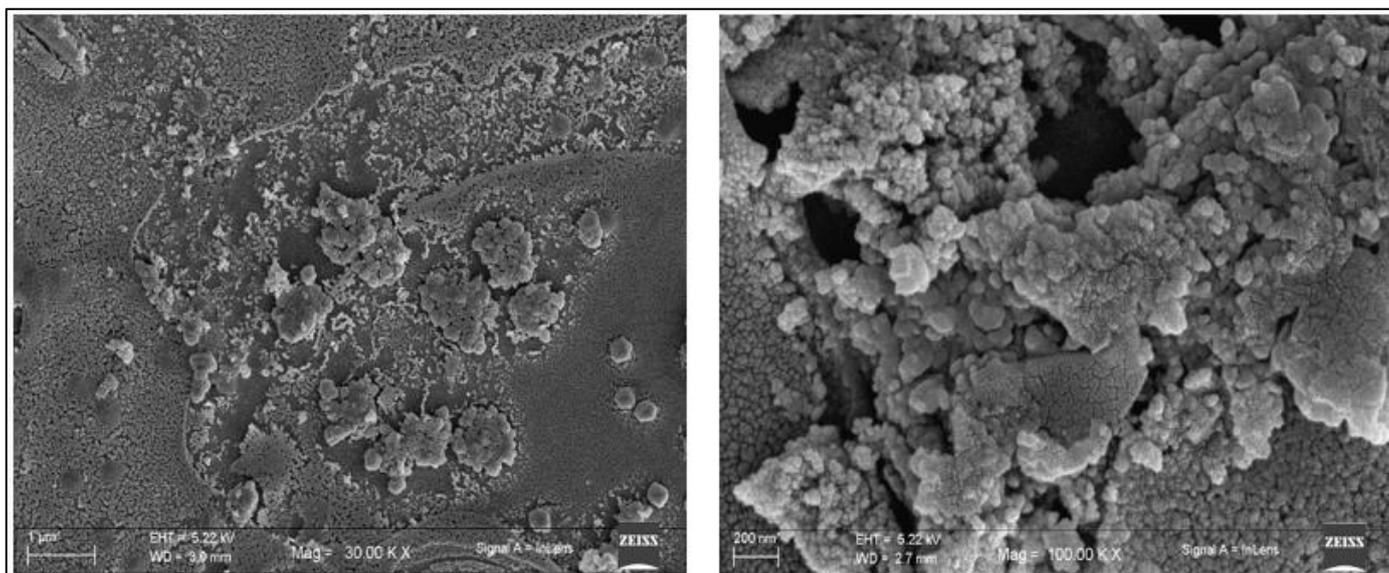


Fig 3 FE-SEM of CuO and Fe Doped CuO Thin Films.

The surface morphology of the prepared thin films was investigated using field emission scanning electron microscopy (FE-SEM). Figure 3 presents the FE-SEM images of pure CuO and 7% Fe-doped CuO thin films deposited by the spray pyrolysis technique. The micrographs reveal that the morphology of the films is strongly influenced by the incorporation of Fe dopant.

The pure CuO thin film exhibits a comparatively plane surface with the presence of micro-cracks and irregular grain distribution, indicating a relatively non-uniform growth pattern. Such cracks may arise due to internal stress developed during the film formation and subsequent cooling process [18]. In contrast, the Fe-doped CuO film with 7% Fe concentration shows a significant modification in surface morphology. The microstructure transforms into a well-defined spherical grain morphology with improved uniformity and better grain distribution over the substrate surface [19].

Furthermore, the 7% Fe-doped CuO film appears compact and crack-free, suggesting that Fe incorporation promotes uniform nucleation and growth of CuO crystallites [20]. The formation of spherical grains indicates enhanced crystallite aggregation and improved surface coverage [21]. This morphological transformation confirms that Fe doping

plays a crucial role in modifying the microstructure of CuO thin films. The observed results are also in good agreement with the structural analysis obtained from XRD, indicating that Fe incorporation effectively alters the growth characteristics of CuO without introducing secondary phases.

- *Discussion*

In the present study, Fe-doped CuO thin films were successfully deposited by the spray pyrolysis technique. The structural and morphological properties of the prepared films were systematically investigated. The XRD analysis confirmed the formation of monoclinic CuO with good crystallinity. After Fe incorporation, the diffraction peaks became sharper and more intense, indicating an improvement in the crystalline quality of the films without the formation of secondary phases. The FE-SEM analysis revealed a significant change in the surface morphology with Fe doping. The pure CuO film exhibited a relatively plane surface with the presence of micro-cracks, whereas the 7% Fe-doped CuO film showed a more uniform and compact surface with well-defined spherical grains. The improved grain distribution and crack-free morphology suggest that Fe doping promotes better nucleation and growth of CuO crystallites during the spray pyrolysis process. Overall, the incorporation of Fe plays a vital role in enhancing the structural quality and surface morphology of CuO thin films. The improvement in

crystallinity along with the formation of uniform spherical grains indicates that Fe doping is an effective approach for tailoring the microstructural properties of CuO. These modifications may further contribute to improved performance of CuO-based materials in various electronic, sensing, and catalytic applications.

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