

衬底温度对热蒸发溴化亚铜纳米薄膜结构及光电性能的影响

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Effect of substrate temperature on structural and optoelectronic properties of thermally evaporated CuBr nanofilms

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衬底温度对热蒸发溴化亚铜纳米薄膜结构 及光电性能的影响

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摘 要: 采用热蒸发法沉积溴化亚铜 (CuBr) 纳米薄膜, 系统研究了衬底温度对其光学和电学特性的影响, 以评估其在光电子器件中的集成潜力。传统研究多将热蒸发 CuBr 薄膜用作光电子器件中的空穴传输层, 然而 CuBr 在约 410 nm 处存在尖锐的蓝光激子发射峰, 该独特光致发光 (photoluminescence, PL) 特性说明其可作为发光器件的发光层。所制备的 CuBr 薄膜具有多晶闪锌矿结构, 表面光滑且形貌良好。值得注意的是, 该薄膜在可见光区域展现出超过 80% 的高透射率, PL 光谱表明, 所制备的薄膜在 412 nm 处呈现显著的发光峰, 与 CuBr 的本征发光特性一致。随着衬底温度升高, 薄膜结晶度提高, PL 强度进一步增强, 同时电阻率降低。上述结果表明, 通过热蒸发法制备的 CuBr 纳米薄膜, 为后续 CuBr 基电致发光器件的构建提供了材料与工艺基础。

关键词: 溴化亚铜; 纳米薄膜; 热蒸发; 衬底温度; 光电性能

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Effect of substrate temperature on structural and optoelectronic properties of thermally evaporated CuBr nanofilms

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Abstract: The cuprous bromide (CuBr) nanofilms were deposited via thermal evaporation, and the influence of substrate temperature on their optical and electrical properties was systematically investigated to evaluate their potential for integration into optoelectronic devices. Current research on thermally evaporated CuBr thin films has primarily employed them as hole transport layers in optoelectronic devices. However, the CuBr exhibits a sharp blue excitonic emission peak at approximately 410 nm, a unique photoluminescence (PL) characteristic that suggests its viability as an emissive layer for light-emitting devices. The as-deposited CuBr thin films prepared in this work exhibit a polycrystalline zinc-blende structure with smooth surface morphology and good uniformity. Notably, the films show a high transmittance exceeding 80% in the visible spectral range, and the PL spectra reveal a prominent emission peak at approximately 412 nm for the as-deposited films, consistent with the intrinsic emission features of CuBr. With the increase of substrate temperature, the crystallinity of the films was improved, accompanied by enhanced PL intensity and reduced resistivity. These results demonstrate that the CuBr nanofilms deposited by thermal evaporation provide a material and processing foundation for the subsequent construction of CuBr-based electroluminescent devices.

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Key words: CuBr; nanofilms; thermal evaporation; substrate temperature; optoelectronic properties

Introduction

Over the past decades, transparent conductors (TCs) have attracted extensive attention for applications in optoelectronic devices, such as transparent electrodes for solar cells, light-emitting diodes and photodetectors^[1-7]. To achieve the high transparency (transmittance > 80%) in the visible region, the optical bandgap of semiconductors should be below 3 eV. Both p-type and n-type TCs with excellent performance are essential for these devices. In contrast, n-type TCs have already reached a high level of development, whereas the progress of p-type TCs has been hampered by self-compensation in wide-bandgap semiconductors^[8-10]. Consequently, only a limited number of p-type semiconductors, such as CuAlO₂, SnO₂, and MgGa₂N₃, have been well developed and commonly reported^[11-16]. In this context, CuBr with high p-type conductivity has emerged as a promising candidate. CuBr possesses a direct bandgap of approximately 3 eV and exhibits excellent transparency (> 80%) in the visible region^[17, 18]. It also features a favorable crystal structure and good lattice compatibility with Si or GaAs substrates^[19]. Moreover, due to its large excitonic binding energy (~108 meV)^[20, 21], CuBr can generate intense excitonic emission, making it a promising emitter for light-emitting diodes.

CuBr, as a wide-bandgap p-type semiconductor, has attracted considerable attention due to its sharp blue excitonic emission peak at approximately 410 nm^[17-19]. This unique photoluminescence characteristic endows it with potential application value in the field of light-emitting devices. However, the luminescent performance of CuBr thin films is highly dependent on their crystalline quality and microstructure, both of which are largely governed by the fabrication methods and processing conditions. To date, the preparation routes for CuBr thin films can be broadly categorized into wet chemical deposition techniques and physical vapor deposition methods. Wet chemical approaches, including spin-coating processes^[19], hydrothermal synthesis^[22], and solution immersion^[23], have been demonstrated to enable controllable preparation of CuBr thin

films or nanostructures. Nevertheless, these methods typically involve solution-based processing steps, which inevitably introduce surface contamination, interfacial defects, and residual impurities, thereby exerting adverse effects on the luminescence efficiency of the films. Thermal evaporation, as a clean, low-cost, and facile physical vapor deposition technique, allows for the deposition of thin films under vacuum conditions with reduced contamination, thus holding promise to circumvent the aforementioned issues inherent to wet chemical routes. GANCHEV M et al.^[24] have preliminarily validated the feasibility of fabricating CuBr thin films via thermal evaporation. However, their work was primarily oriented toward the application of CuBr as a hole transport layer in perovskite solar cells, and did not address the luminescence properties of CuBr, nor did it systematically investigate the effects of deposition process parameters on the crystalline quality and luminescent performance of the films.

In this work, we systematically investigated the crystal structure, morphology, optical and electrical properties of CuBr thin films deposited by thermal evaporation. To optimize the film performance, the substrate temperature was varied from 50 °C to 200 °C during deposition, and its influence on the film properties was analyzed. The results demonstrate that CuBr thin films deposited at elevated substrate temperatures exhibit improved crystallinity and reduced resistivity. This study provides quantitative mechanism analysis and practical engineering processing guidance for CuBr emissive layers, laying a material and processing foundation for the potential application of CuBr in light-emitting devices.

1 Experimental details

CuBr thin films were deposited on glass substrates via thermal evaporation using a vacuum coating system (SD400M, Shenyang Qihui Vacuum Technology Co., Ltd.). Glass substrates were cleaned using alcohol and deionized water before deposition. After that, these were dried with pure nitrogen gas flow to remove any dust or particles. CuBr powder (99%, purchased from

Aladdin company) was used as initial materials, heated up to 520 °C for 1 h in a vacuum before being cooled down to room temperature to form CuBr crystal. CuBr crystal was the core material of thermal evaporation. The deposition chamber was pumped down to a base pressure of 3×10^{-3} Pa. During evaporation, the deposition rate was maintained at 2 Å/s, as monitored by the quartz crystal microbalance (SI-TM216D). The substrate temperature was varied from 50 °C to 200 °C. After deposition, the film thickness was measured at multiple positions on each sample using a surface profilometer (XP-2, KLA-Tencor), and the average thickness of the CuBr films was determined to be approximately 120 nm.

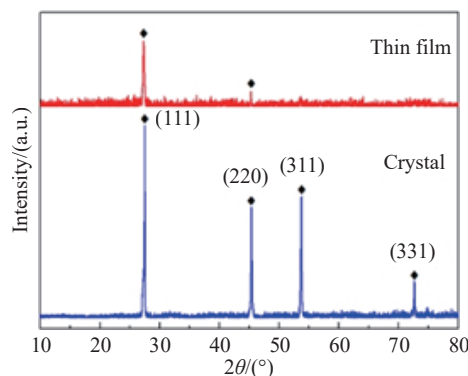
The structural characterization of deposited CuBr thin films was carried out by a Bruker D8 advance X-ray diffractometer (XRD) in the span of angle $10^\circ \sim 80^\circ$, using monochromatic Cu-K α radiation. The surface morphological analysis was done using scanning electron microscopy (SEM) JSM model 7800. The chemical state and composition of films were investigated by X-ray photoelectron spectroscopy (XPS) Thermo ESCALAB 250Xi. The optical properties of these films were recorded by UV-vis-NIR scanning spectrophotometer (UV-3101 PC). Photoluminescence spectra were measured by a fluorescence spectrometer (FLS920) with a 325 nm He-Cd laser as the excitation source. A four-point probe measurer was used to estimate the sheet resistivity value of a layer of an electronic material through Keithley 2410.

To ensure the reliability of the experimental data, for SEM grain size analysis, at least five randomly selected regions were imaged for each sample, and more than 100 grains were counted to obtain statistically meaningful average grain sizes. XRD patterns and PL spectra were collected from multiple different positions on each sample to verify the crystallinity uniformity and the representativeness of the luminescence data. Quantitative results for grain size were presented as mean $\pm$ standard deviation.

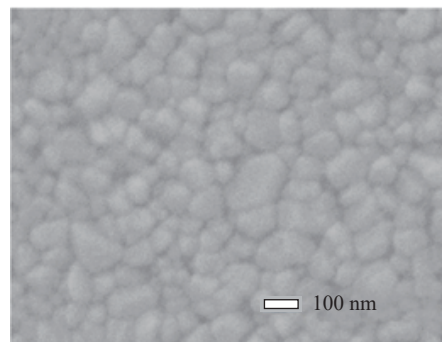
2 Results and discussions

The structural characterization of CuBr crystal and thin films deposited at 150 °C is shown in Figure 1.

Figure 1(a) depicts the XRD patterns of CuBr crystals and CuBr thin films deposited at a substrate temperature of 150 °C. Both are characteristic of γ -CuBr in the zinc-blende configuration (JCPDS: 06-0292), and no detectable impurities are observed. The diffraction peaks of the CuBr crystals are centered at $2\theta \approx 27.1^\circ$, 45.0° , 53.3° , and 72.3° , corresponding to the (111), (220), (311), and (331) reflections, respectively. The presence of a large number of crystallographic orientations reveals the polycrystalline nature of the CuBr crystals. In contrast, the CuBr thin films exhibit lower crystallinity after thermal evaporation, as evidenced by the fewer and broader diffraction peaks. The most prominent peak is identified as the (111) plane, indicating preferential growth along the (111) direction. This preferential orientation is attributed to the low surface energy of the (111) plane, which promotes faster nucleation and growth along this direction, while peaks corresponding to other planes diminish at higher diffraction angles^[25].



(a) XRD diagram of CuBr crystal and thin film



(b) SEM image of CuBr thin film surface

Fig. 1 Structural characterization of CuBr crystal and thin films deposited at 150 °C

Figure 1(b) presents the surface morphology of the CuBr thin film deposited at 150 °C. The film exhibits

compact grain growth on the glass substrate, with a smooth and homogeneous surface.

Figure 2(a) presents the PL spectra of the CuBr crystals and CuBr thin films deposited at the substrate temperature of 150 °C, measured under 325 nm excitation. All samples exhibit emission peaks at approximately 412 nm. The PL intensity of the crystals is significantly higher than that of the thin films. Figure 2(b) shows the transmission and absorption spectra of the CuBr thin films. The absorption band edge is observed at approximately 417 nm, corresponding to a small Stokes shift relative to the PL peak. The films exhibit high transmittance in the visible region (400 nm~750 nm), with the transmittance rising significantly above approximately 420 nm, which is beneficial for light extraction in luminescent device applications.

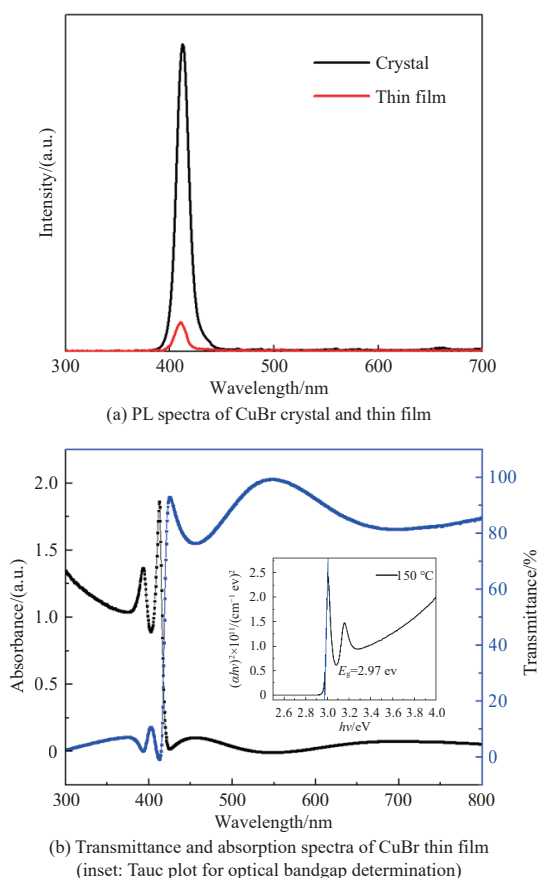


Fig. 2 Optical properties of CuBr crystal and thin films deposited at 150 °C

The optical bandgap energy is estimated using Tauc's equation^[26-28]:

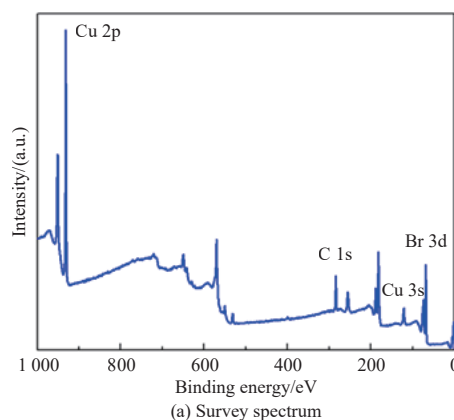
$$\alpha h\nu = B(E_g - h\nu)^n \quad (1)$$

where α is the absorption coefficient, E_g is the

bandgap, ν is the frequency of incident light, h is the Planck's constant, B is a constant that depends on the transition probability, and n is equal to 1/2 for direct transition.

The inset of Figure 2(b) shows the Tauc plot of $(\alpha h\nu)^2$ as a function of photon energy ($h\nu$). The linear behavior observed above the optical bandgap confirms the occurrence of a direct optical transition in the CuBr thin films. Extrapolating the linear portion of the curve to $(\alpha h\nu)^2=0$ yields an optical bandgap of approximately 2.97 eV, which is consistent with previous reports^[29]. This relatively wide bandgap confirms that the thermally evaporated CuBr thin films are wide-bandgap semiconductors. In addition, the transmission spectra reveal that the average transmittance exceeds 80% in the visible region, demonstrating the high transparency of the CuBr films in this spectral range.

Figure 3 presents the XPS spectra of CuBr thin films deposited at a substrate temperature of 150 °C. The survey spectrum, along with the high-resolution core-level spectra of Cu 2p and Br 3d, is shown in Figure 3(a)~3(c). In the Cu 2p core-level spectrum, two strong peaks are observed at 932.38 eV and 952.28 eV, corresponding to the Cu 2p_{3/2} and Cu 2p_{1/2} spin-orbit split states, respectively. The Br 3d spectrum (Figure 3(c)) can be deconvoluted into two peaks with binding energies of 69.08 eV and 70.08 eV, which are assigned to Br 3d_{5/2} and Br 3d_{3/2}, respectively. No additional peaks are detected, indicating the high chemical purity of the thermally evaporated CuBr films. The Cu-to-Br peak area ratio is approximately 1 : 1, confirming that the films maintain the stoichiometric composition of CuBr.



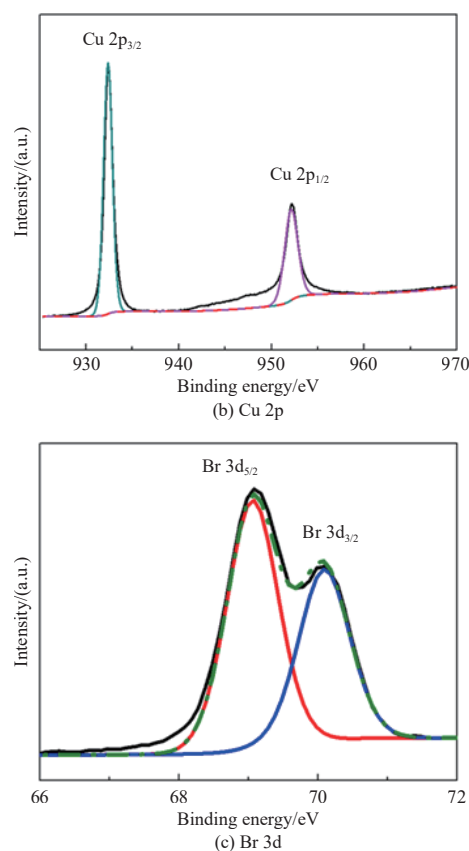
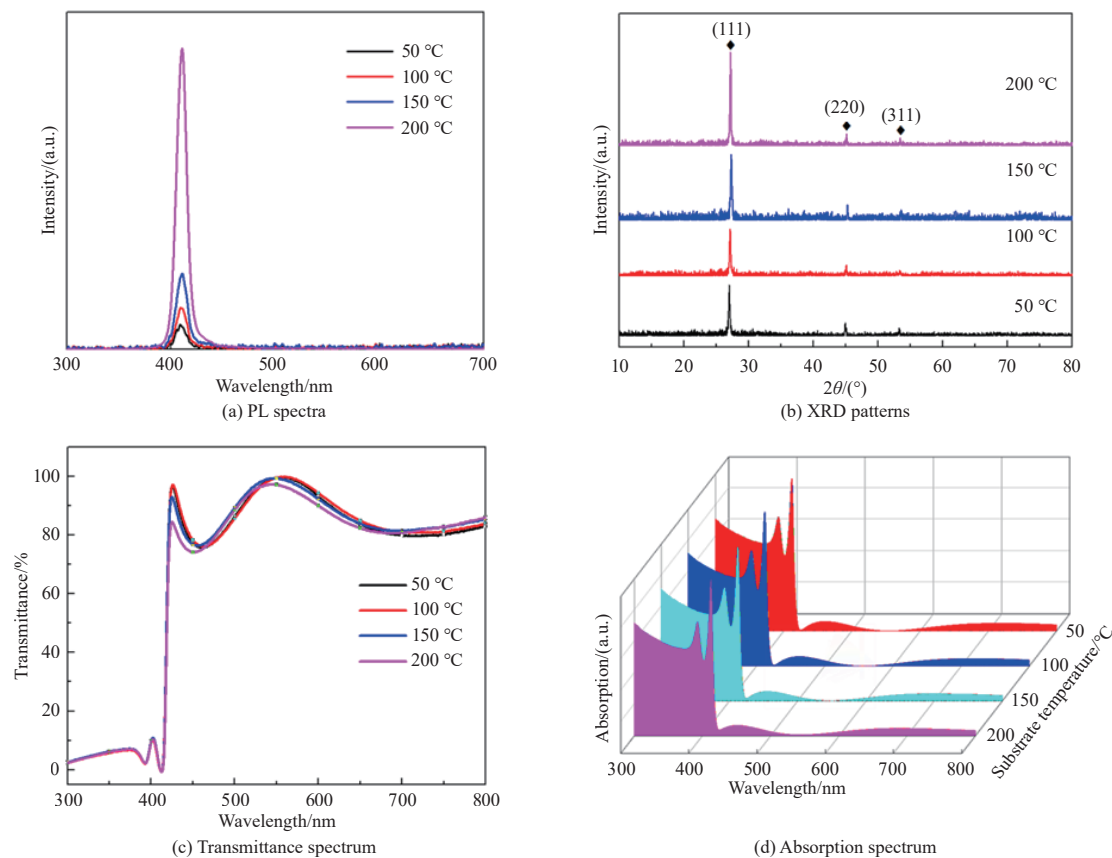


Fig. 3 XPS spectra of CuBr thin films at substrate temperature of 150 °C

As reported, the substrate temperature during deposition significantly influences the electrical conductivity and optical transmittance of CuBr thin films^[30]. In this work, films are deposited at various substrate temperatures ranging from 50 °C to 200 °C. Figure 4(a) presents the PL spectra of the films deposited at different substrate temperatures. All films exhibit a sharp emission peak at approximately 412 nm, corresponding to the characteristic excitonic emission of CuBr. The PL intensity increases with increasing substrate temperature, consistent with the improved crystallinity revealed by the XRD analysis in Figure 4(b). As shown in Figure 4(c), the transmission spectra of the films deposited at various substrate temperatures reveal that the films are highly transparent in the visible region, with a steep decline observed at approximately 417 nm. In addition, the transmittance in the longer-wavelength region exhibits a slight increase as the substrate temperature rises. All films exhibit a high transmittance exceeding 80% in the visible range, making them suitable candidates for transparent luminescent layers. Figure 4(d) shows the absorption spectra of the films deposited at various substrate temperatures. All the intense absorption peaks occur at about 417 nm.



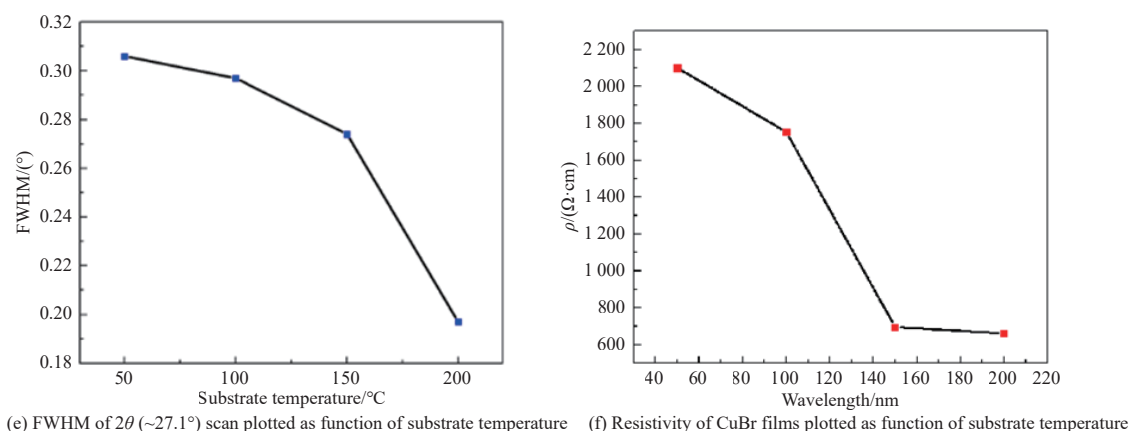


Fig. 4 CuBr thin films at various substrate temperatures (50 °C~200 °C)

The optical bandgap (E_g) of thin films was calculated using Tauc's equation, shown as below:

$$\alpha h\nu = c(h\nu - E_g)^m \quad (2)$$

where α is the absorption coefficient, $h\nu$ is the photon energy, c is a constant related to the edge width, and m depends on the nature of the electronic transition ($m=1/2$ for direct allowed, 2 for indirect allowed, 3/2 for direct forbidden, and 3 for indirect forbidden transitions). The calculated optical bandgap remains approximately 2.97 eV as the substrate temperature increases from 50 °C to 200 °C, indicating that the substrate temperature does not alter the bandgap of the CuBr films.

Figure 4(b) shows the XRD patterns of the films deposited at various substrate temperatures. Three diffraction peaks corresponding to (111), (220) and (311) reflections are observed, which belong to cubic zinc-blende structure (γ -CuBr). All the films exhibit a single-phase polycrystalline nature. With increasing substrate temperature, the intensity of the (111) peak increases significantly, the (111) plane is identified as the preferential orientation for all temperatures. Figure 4(e) presents the full width at half maximum (FWHM) of the (111) diffraction peak for CuBr thin films deposited at various substrate temperatures. The FWHM values decrease from 0.306° to 0.197° as the substrate temperature increases, indicating that films deposited at higher temperatures possess improved crystallinity. This trend suggests that small grains coalesce effectively to form larger grains with increasing substrate temperature. On this basis, the

lattice spacing d , the lattice parameter a and the internal strain ε are calculated by using the following formula:

$$d = \frac{\lambda}{2 \sin \theta} \quad (3)$$

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} \quad (4)$$

$$\varepsilon = \frac{\beta}{4 \tan \theta} \quad (5)$$

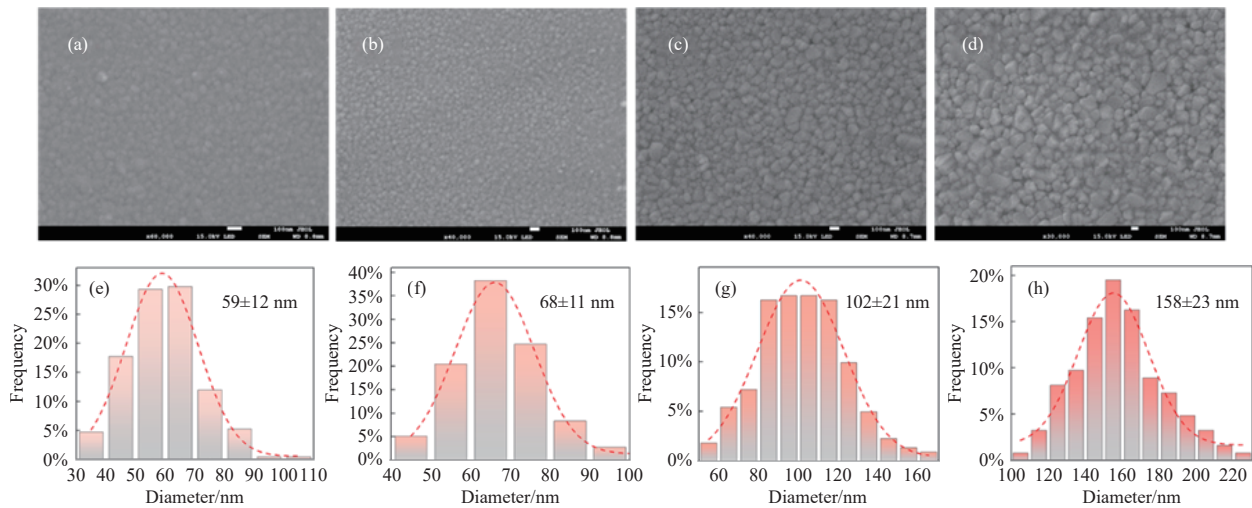
Where h , k and l are the Miller indices, and β is the structural broadening, shown in Figure 4(e).

The values of lattice parameters calculated from the above relation are close to 5.69 Å, which is in good agreement with the standard value taken from the powder diffraction file card (JCPDS: 06-0292). The internal strain decreases with the increasing substrate temperature, further supporting the better crystallization through higher substrate temperature.

Figure 5(a)~(d) shows the surface morphologies of CuBr thin films deposited at various substrate temperatures. All films exhibit homogeneous and compact surfaces. It is evident that as the substrate temperature increases, the grain size increases and the crystalline quality improves, which is consistent with the XRD results presented in Figure 4(b). As shown in Figure 5(e)~(h), the average grain diameter increases from 59 nm to 158 nm with increasing substrate temperature, and the grains exhibit a relatively broad size distribution.

The number of crystallites per unit area N , dislocation density δ and of CuBr thin films are evaluated as mention below^[31-32]:

$$N = \frac{t}{D^3} \quad (6)$$



(a)~(d) Surface morphologies by SEM images ; (e)~(h) Grain size distribution

Fig. 5 CuBr thin films at different substrate temperatures from 50 °C to 200 °C

$$\delta = \frac{1}{D^2} \quad (7)$$

Where t and D represent the thickness of thin films and average crystallite size, respectively.

The number of crystallites per unit area N and the dislocation density δ are inversely proportional to average crystallite size D . With the higher substrate temperature, CuBr thin films have more crystallites per unit area and lower dislocation density. These results show that raising the substrate temperature can optimize the crystallization process of CuBr thin films.

Figure 4(f) illustrates the resistivity of CuBr thin films deposited at various substrate temperatures. The resistivity decreases from approximately $2101 \Omega \cdot \text{cm}$ to $660 \Omega \cdot \text{cm}$ as the substrate temperature increases from 50 °C to 200 °C. It is well established that grain boundaries play an important role in influencing electrical resistivity through electron scattering^[33-34]. Films with higher crystallinity possess larger grain sizes and fewer grain boundaries, which reduce the electron scattering effect. Consequently, CuBr thin films deposited at 200 °C exhibit the lowest resistivity and the highest crystallinity among the investigated temperatures.

3 Conclusion

This work presents a process for depositing p-type CuBr thin films via thermal evaporation. To investigate the structural, morphological, optical, and electrical

properties, CuBr films with a thickness of approximately 120 nm were fabricated on glass substrates. The films exhibit a polycrystalline cubic structure with a pronounced (111) preferential orientation. At a substrate temperature of 200 °C, the films show the highest crystallinity and the smoothest surface morphology. As the substrate temperature increases from 50 °C to 200 °C, the average grain size increases from 59 nm to 158 nm. Consequently, the PL intensity at approximately 412 nm increases with improving crystallinity. The optical bandgap remains constant at approximately 2.97 eV, while the films maintain high transparency exceeding 80% in the visible region. A sharp absorption peak is observed at approximately 417 nm, corresponding to a small Stokes shift. The resistivity decreases with increasing substrate temperature, and the films deposited at 200 °C exhibit the lowest resistivity. These results provide valuable insights into the preparation of CuBr thin films by thermal evaporation and demonstrate their potential for future optoelectronic applications.

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