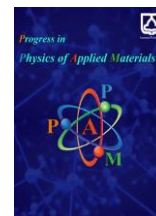




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Annealing-Temperature-Dependent Formation of Formamidinium Lead Bromide Perovskite Thin Films for Light-Emitting Diode Applications

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ABSTRACT

Formamidinium lead bromide (FAPbBr₃) perovskite thin films are attractive emissive layers for green perovskite light-emitting diodes because of their narrow photoluminescence emission and favorable optoelectronic properties. In this study, FAPbBr₃ thin films were prepared through a two-step route combining thermal evaporation and solution processing. First, PbBr₂ layers were deposited by thermal evaporation, followed by spin coating of a formamidinium-based solution to promote the conversion of the precursor layer into FAPbBr₃. The resulting films were then thermally treated at different annealing temperatures ranging from 75 °C to 150 °C in order to evaluate the influence of post-deposition heating on film formation, morphology, crystallinity, optical response, and device performance. The results show that annealing temperature strongly affects the structural and optoelectronic quality of the perovskite layer. Among the investigated conditions, the film annealed at 125 °C exhibited favorable surface coverage and crystallographic characteristics and produced the best overall PeLED performance. The optimized PeLED device based on this film produced green emission with a peak wavelength of 531 nm and a full width at half maximum of 32 nm. A maximum luminance of 100 cd m⁻² and a current efficiency of 1 cd A⁻¹ were obtained. These findings indicate that careful control of the annealing step is an effective strategy for improving FAPbBr₃ thin-film quality and the performance of corresponding perovskite light-emitting diodes.

1. Introduction

Metal-halide perovskites constitute a broad class of solution- and vacuum-processable semiconductors whose optical and electronic properties can be tuned through compositional and structural design. Their strong radiative recombination, composition-tunable emission, and compatibility with relatively low-temperature processing have made them attractive for light-emitting devices. Early demonstrations established electroluminescence from organometal-halide

perovskites, while subsequent studies have emphasized the roles of emitter composition, film morphology, interfaces, and charge management in determining PeLED performance [1–4].

Within this family of materials, perovskite-based LEDs are especially appealing for display and lighting systems because they can deliver high color purity through narrow-band emission while remaining compatible with relatively low-temperature fabrication.

Progress in perovskite electroluminescent devices has relied on simultaneous advances in active-layer

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composition, interfacial engineering, and control of thin-film microstructure [1,3,4]. Efficient emission is governed not only by the band structure of the emitting material: film continuity, crystallization quality, trap density, and the balance between injected electrons and holes also determine how effectively excitons are formed and converted into photons. At a broader LED-engineering level, recent work published in PPAM has likewise highlighted the importance of nanoscale emitter and optical-layer design in controlling emission intensity and spectral response [5].

The bromide perovskite FAPbBr_3 is one of the key candidates for green PeLED emitters. Its emission typically lies in the green spectral region with a comparatively narrow profile, making it attractive for color-pure display applications [2,6,7]. Recent studies further show that the performance of FAPbBr_3 films is strongly processing- and interface-dependent: crystallization, surface coverage, trap density, and interfacial reactions can markedly influence radiative efficiency and device operation [8,9]. These findings reinforce the need for careful control of phase formation and microstructure when FAPbBr_3 is used as the emissive layer.

Most reported FAPbBr_3 emissive layers are prepared by solution-based methods because these routes are experimentally simple and enable direct control of precursor chemistry [6,8,9]. Nevertheless, solution processing can present challenges in thickness control, large-area uniformity, and batch-to-batch reproducibility. Thermal evaporation offers precise thickness control and compact precursor layers, and vacuum-based PeLED fabrication has therefore attracted increasing attention [10–15]. More recently, sequential layer-by-layer thermal evaporation followed by post-annealing has been shown to provide controllable crystallization and high film uniformity in PeLED emissive layers [16]. A sequential strategy in which an evaporated PbBr_2 film is converted by spin coating a formamidinium precursor solution can similarly combine the advantages of vacuum deposition and wet processing. In this configuration, thermal treatment after spin coating is particularly important because it affects interdiffusion of the precursors, solvent removal, conversion to the perovskite phase, nucleation and crystal growth, and the formation of optically active or defective sites.

Previous annealing and vacuum-deposition studies provide important context for the present work, although their material systems and processing routes differ substantially. Liang et al. [17] investigated solution-processed MAPbBr_3 PeLEDs and reported an optimum annealing condition of 80 °C for 20 min, yielding a maximum luminance of 13,700 cd m^{-2} and a current efficiency of 8.22 cd A^{-1} . Vacuum-based approaches have also been demonstrated for inorganic and mixed-composition perovskite emitters [10–16]. For example, all-vacuum CsPbBr_3 PeLEDs with an EQE of 8.0% were reported by Du et al. [11], while Bai et al. [15] investigated co-evaporated CsPbBr_3 films and subsequent high-temperature annealing. Li et al. [16] further demonstrated that layer-by-layer thermal evaporation combined with controlled post-annealing can regulate precursor

diffusion, reaction, and crystallization. These studies establish the strong processing dependence of halide-perovskite emitters; however, they involve either different perovskite compositions or fundamentally different deposition schemes from the sequential evaporated- PbBr_2 /solution-FA conversion route employed here.

Previous studies have demonstrated that thermal annealing can substantially influence the morphology, crystallization, and electroluminescent characteristics of bromide-based perovskite emitting layers [16,17]. However, the processing dependence observed in solution-processed or fully vacuum-deposited perovskites cannot necessarily be transferred directly to a sequential hybrid route in which a thermally evaporated PbBr_2 precursor layer is subsequently converted to FAPbBr_3 by solution deposition of the formamidinium precursor. In our previous study [18], precursor-solution concentration was investigated as the principal variable in this two-step FAPbBr_3 fabrication approach. In that concentration-optimization study, the optimized PeLED exhibited a maximum luminance of approximately 100 cd m^{-2} , an electroluminescence peak near 526 nm, and an emission FWHM of approximately 32 nm [18]. In the present study, the precursor concentration is held constant while post-deposition annealing temperature is varied independently. The optimized 125 °C device exhibits a maximum luminance of 100 cd m^{-2} , a current efficiency of 1 cd A^{-1} , an EL peak at 531 nm, and an FWHM of 32 nm. Therefore, the advancement of the present work lies not in claiming a higher peak luminance than Ref. [18], but in isolating annealing temperature as an independent processing variable and establishing its relationship with the structural, optical, and device-level response of films produced by the same sequential fabrication route.

By systematically varying the annealing temperature from 75 to 150 °C, we examine how thermal treatment affects phase formation, crystallite dimensions, surface morphology, photoluminescence behavior, and the resulting electroluminescent device characteristics. The purpose of this study is therefore not merely to identify an optimum annealing temperature, but to establish the relationship between post-deposition thermal treatment and the structure–optical–device response of FAPbBr_3 films produced by this sequential evaporation/spin-coating route.

2. Experimental

The device architecture used in this work is schematically presented in Figure 1. The PeLED structure was designed as ITO/PEDOT:PSS/TFB/ FAPbBr_3 /TPBi/LiF/Al, with an active area of 0.1 cm^2 defined by the patterned electrodes. Patterned ITO-coated glass substrates were first cleaned sequentially in deionized water, acetone, ethanol, and isopropanol using an ultrasonic bath. The cleaned substrates were then dried in an oven at 110 °C for 40 min. Prior to film deposition, the ITO substrates were treated with oxygen plasma and immediately transferred into a nitrogen-filled glovebox. PEDOT:PSS was deposited on the ITO surface by spin coating at 3500 rpm for 45 s

and subsequently annealed at 130 °C for 20 min in air. A TFB layer was then spin-coated on top of PEDOT:PSS at 3500 rpm for 30 s, followed by thermal treatment at 120 °C for 30 min.

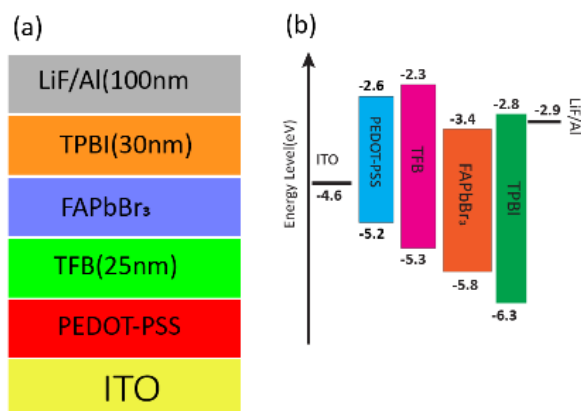


Fig. 1. Schematic illustration of the FAPbBr₃-based PeLED structure: (a) device configuration and (b) energy-level alignment of the functional layers.

After preparation of the hole-transport layers, the samples were placed in a vacuum chamber for PbBr₂ deposition. PbBr₂ powder with a purity of 99.9% was used as the evaporation source. The chamber pressure was maintained at approximately 3×10^{-3} Pa during deposition, and the deposition rate and film thickness were monitored using a quartz crystal microbalance. The evaporated PbBr₂ films were then transferred for the second step of perovskite formation. A formamidinium precursor solution with a concentration of 10 mg mL⁻¹ was spin-coated onto the PbBr₂-coated substrates. The spin-coating procedure consisted of an initial rotation at 1500 rpm for 30 s to spread the solution uniformly over the precursor layer, followed by a final rotation at 2000 rpm to remove excess solution from the substrate edges. Thermal annealing was applied after spin coating to complete the conversion process and to tune the microstructure of the FAPbBr₃ emissive layer. Since annealing can influence precursor diffusion, solvent removal, crystallization behavior, surface coverage, and film microstructure, four processing temperatures were investigated. The samples were annealed on a hot plate at 75, 100, 125, and 150 °C and are denoted hereafter as FAPbBr₃-75 °C, FAPbBr₃-100 °C, FAPbBr₃-125 °C, and FAPbBr₃-150 °C, respectively. After optimization of the perovskite layer, the upper functional layers were deposited by thermal evaporation. TPBi, LiF, and Al layers with thicknesses of 20, 2, and 80 nm, respectively, were deposited sequentially to complete the PeLED devices.

The energy-level arrangement of the device components is shown in Figure 1(b). PEDOT:PSS was employed to modify the ITO surface and facilitate hole injection into the device. TFB served as the hole-transport layer and also helped confine electrons within the emissive region. On the opposite side of the perovskite layer, TPBi acted as the electron-transport and hole-

blocking layer, promoting charge recombination inside the FAPbBr₃ active layer. This multilayer configuration was selected to improve carrier balance and enhance radiative recombination in the perovskite emitting film.

The morphology of the FAPbBr₃ thin films was examined by scanning electron microscopy using a Carl Zeiss SEM system. Photoluminescence spectra were collected with a Hitachi F-4600 fluorescence spectrophotometer. The PL spectra were analyzed without intensity normalization, while UV-visible absorption spectra were recorded using a Shimadzu UV-3101PC spectrophotometer equipped with 10 mm quartz cells. The crystal structure of the films was evaluated by X-ray diffraction using a PANalytical Empyrean diffractometer with Cu K α radiation. For quantitative peak-width analysis, the (100) diffraction reflection in the 2 θ range of 14.3–15.3° was fitted using a pseudo-Voigt profile with a linear background. The apparent crystallite size was calculated from the fitted full width at half maximum (FWHM) using the Scherrer equation, $D = K\lambda / (\beta \cos\theta)$, where D is the apparent crystallite size, $K = 0.9$ is the shape factor, $\lambda = 0.15406$ nm is the wavelength of Cu K α radiation, β is the fitted FWHM expressed in radians, and θ is the Bragg angle. The uncertainties correspond to one standard error obtained from the nonlinear peak fitting and were propagated through the Scherrer calculation. Because an instrumental-broadening reference measurement was not available, instrumental broadening was not subtracted; therefore, the calculated values are reported as apparent Scherrer crystallite sizes.

Atomic force microscopy images were obtained using a Shimadzu SPM-9600 instrument to analyze surface topography and roughness. The fabricated PeLED devices were characterized under ambient conditions. Current density-voltage-luminance characteristics were measured using the electrical-optical measurement setup described for the devices, and electroluminescence spectra were recorded using a USB 2000 spectrometer together with a Keithley 2400 source meter. The active device area was fixed at 0.1 cm² by the patterned ITO and Al electrodes.

3. Results and Discussion

The influence of annealing temperature on the optical behavior of the FAPbBr₃ thin films was first examined by UV-visible absorption and steady-state photoluminescence measurements. Figure 2 presents the absorption and PL spectra of the films annealed at 75, 100, 125, and 150 °C. As shown in Figure 2(a), all samples exhibit an absorption onset near 540 nm, which is consistent with the optical response characteristics of bromide-based FAPbBr₃ perovskites. The absorption intensity increases with increasing annealing temperature, while the absorption onset remains close to 540 nm. The nearly unchanged absorption edge indicates that the fundamental optical transition remains similar across the investigated samples, whereas the variation in

absorption magnitude reflects annealing-dependent changes in the optical response of the films. Because film thickness and absolute conversion yield were not independently quantified, the increase in absorption intensity is not assigned to a single microscopic mechanism.

The photoluminescence spectra in Figure 2(b) further confirm the strong dependence of the emissive properties on annealing temperature. All samples show green emission with peak positions located between 524 and 538 nm, while the corresponding FWHM values lie in the range of 38–54 nm. The PL peak intensity increases with increasing annealing temperature, and the film annealed at 150 °C exhibits the highest peak intensity among the investigated samples. However, the highest PL peak intensity does not coincide with the optimum PeLED performance, which is obtained for the film annealed at 125 °C. This observation indicates that steady-state PL intensity alone cannot be used as the criterion for identifying the optimum annealing condition. Rather, device performance is governed by the combined influence of film morphology, crystallographic characteristics, surface quality, interfacial contact, and charge injection and transport within the complete device architecture.

A shift in the emission peak and variation in the spectral width are also observed with annealing temperature, confirming that post-deposition thermal treatment modifies the optical response of the FAPbBr₃ films. Importantly, the optical response does not follow the same trend as the device performance. The PL peak intensity reaches its maximum for the film annealed at 150 °C, whereas the highest electroluminescent output is obtained for the 125 °C device. This decoupling indicates that the steady-state PL response of the isolated film cannot, by itself, predict the behavior of the complete PeLED, in which charge injection, transport, interfaces, and film continuity also contribute to the measured electroluminescent performance.

X-ray diffraction analysis was carried out to evaluate the crystallographic evolution of the FAPbBr₃ films as a function of annealing temperature. The XRD patterns shown in Figure 3 display the main diffraction reflections at approximately 14.77°, 20.95°, 29.79°, and 33.41°, which are assigned to the (100), (110), (200), and (210) planes of FAPbBr₃, respectively. The fitted peak positions show only minor variations among the four annealing conditions; for example, the (100) reflection remains within approximately 14.759–14.770°. This limited shift indicates that no major change in the FAPbBr₃ diffraction pattern occurs over the investigated annealing-temperature range.

Moreover, no obvious residual PbBr₂ peak is detected, suggesting that the sequential evaporation/spin-coating process effectively converts the PbBr₂ precursor layer into the perovskite phase. The (100) reflection becomes progressively narrower with increasing annealing temperature. Because diffraction intensity can also be influenced by factors other than crystallite size, the structural evolution was evaluated quantitatively using the FWHM of the fitted (100) reflection rather than by peak intensity alone.

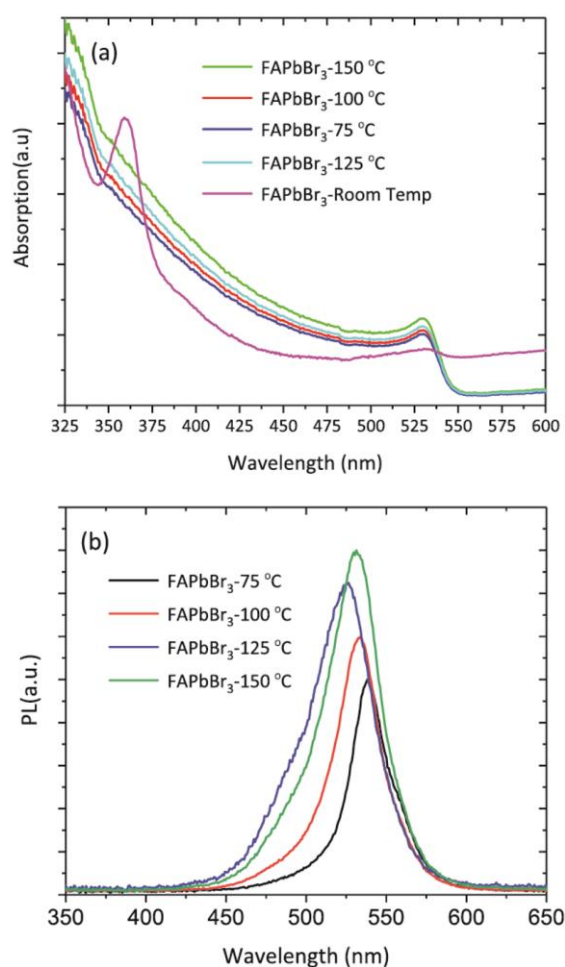


Fig. 2. Optical characteristics of FAPbBr₃ thin films annealed at different temperatures: (a) UV-visible absorption spectra and (b) steady-state photoluminescence spectra. The PL spectra are presented without intensity normalization.

Quantitative fitting of the (100) diffraction reflection reveals a systematic narrowing of the peak with increasing annealing temperature. The fitted FWHM values are $0.152 \pm 0.006^\circ$, $0.121 \pm 0.002^\circ$, $0.104 \pm 0.004^\circ$, and $0.0918 \pm 0.0009^\circ$ for the films annealed at 75, 100, 125, and 150 °C, respectively. Using these values in the Scherrer equation yields apparent crystallite sizes of 52.6 ± 1.9 , 66.2 ± 1.4 , 76.9 ± 2.9 , and 87.3 ± 0.9 nm, respectively. Thus, the coherently diffracting domain size increases progressively with increasing annealing temperature. These Scherrer-derived values describe coherent crystalline-domain dimensions and should not be directly equated with the surface grain dimensions observed by SEM. Notably, the largest apparent crystallite size is obtained at 150 °C, whereas the optimum PeLED performance occurs at 125 °C. This difference demonstrates that crystallite size alone does not determine device performance and that the overall device response depends on the combined structural, morphological, interfacial, and charge-transport characteristics. Such a coupled dependence of PeLED performance on film morphology, structural characteristics, and device architecture has also been reported in previous studies [3,17].

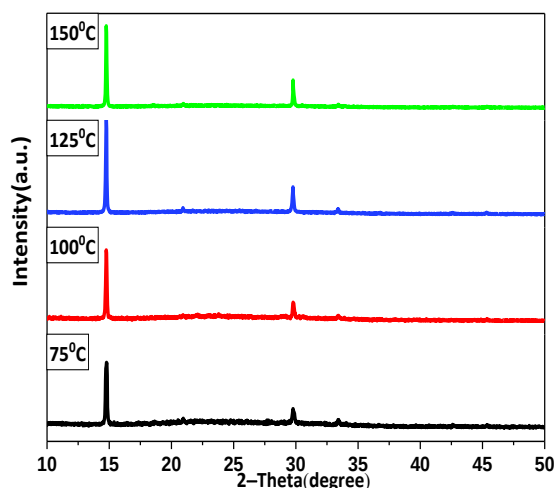


Fig. 3. X-ray diffraction patterns of FAPbBr₃ thin films prepared under different annealing temperatures.

The fitted parameters of the FAPbBr₃ (100) diffraction reflection and apparent Scherrer crystallite sizes are presented in Table 1.

Table 1. Fitted parameters of the FAPbBr₃ (100) diffraction reflection and apparent Scherrer crystallite sizes.

Annealing temperature (°C)	2θ (°)	FWHM (°)	Apparent crystallite size* (nm)
75	14.770	0.152 ± 0.006	52.6 ± 1.9
100	14.765	0.121 ± 0.002	66.2 ± 1.4
125	14.759	0.104 ± 0.004	76.9 ± 2.9
150	14.766	0.0918 ± 0.0009	87.3 ± 0.9

*The uncertainties correspond to one standard error obtained from nonlinear peak fitting and propagated through the Scherrer calculation. Instrumental broadening was not subtracted; therefore, the values are reported as apparent Scherrer crystallite sizes.

The surface morphology of the films was investigated by top-view SEM imaging, as shown in Figure 4. The films annealed at 75 and 100 °C display relatively dense coverage, but their surfaces contain less distinct crystal boundaries and less developed grain features. Compared with the 125 °C film, the samples annealed at 75 and 100 °C exhibit less clearly developed surface domains, indicating that the film morphology remains sensitive to the post-deposition annealing condition. When the annealing temperature is increased to 125 °C, the surface becomes more uniform and compact, with clearly defined perovskite domains and reduced morphological irregularities. The film annealed at 150 °C also exhibits a largely continuous surface. Despite its higher PL peak intensity, however, the corresponding PeLED does not exhibit the best device performance. This observation indicates that electroluminescent performance cannot be inferred directly from steady-state PL intensity and is also influenced by film microstructure, interfacial properties, and charge injection and transport within the complete device. Taken together with the PL and XRD results, the SEM observations demonstrate that annealing temperature simultaneously modifies the morphological, structural, and optical characteristics of the FAPbBr₃ films.

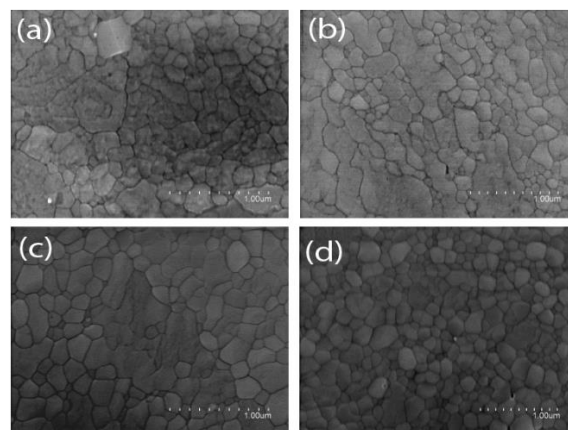


Fig. 4. Top-view SEM images of FAPbBr₃ thin films annealed at: (a) 75 °C, (b) 100 °C, (c) 125 °C, and (d) 150 °C.

Atomic force microscopy was used to further examine the surface topography of the optimized film. Figure 5 shows the AFM image of the FAPbBr₃-125 °C sample. The film exhibits continuous surface coverage with an RMS roughness of approximately 3.3 nm, confirming the relatively smooth surface of the 125 °C film. Such morphology is favorable for the formation of continuous interfaces with adjacent transport layers. Previous PeLED studies have shown that film morphology and surface coverage can strongly affect charge leakage and device efficiency [3]. However, because AFM measurements were not performed for all annealing temperatures in the present study, no quantitative roughness-temperature correlation is inferred here.

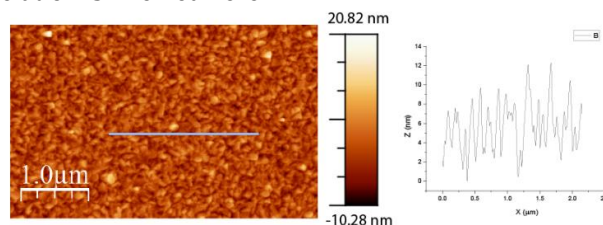


Fig. 5. AFM surface topography of the optimized FAPbBr₃ film annealed at 125 °C.

The electroluminescent performance of the PeLEDs was then evaluated through current density-voltage-luminance measurements. Figure 6 shows the J-V-L characteristics of devices fabricated with FAPbBr₃ layers annealed at different temperatures. All devices exhibit an increase in current density after the turn-on region, confirming charge injection through the multilayer structure. However, the device based on the FAPbBr₃-125 °C film shows the most favorable electrical and optical response. This device reaches a current density of 10 A m⁻² at 4.5 V and delivers a maximum current efficiency of 1 cd A⁻¹. The maximum luminance reaches 100 cd m⁻² at a driving voltage of 8 V. These measurements establish 125 °C as the annealing condition that provides the highest overall electroluminescent performance among the investigated devices. However, the J-V-L data alone do not allow the individual contributions of charge injection, transport, and recombination kinetics to be separated.

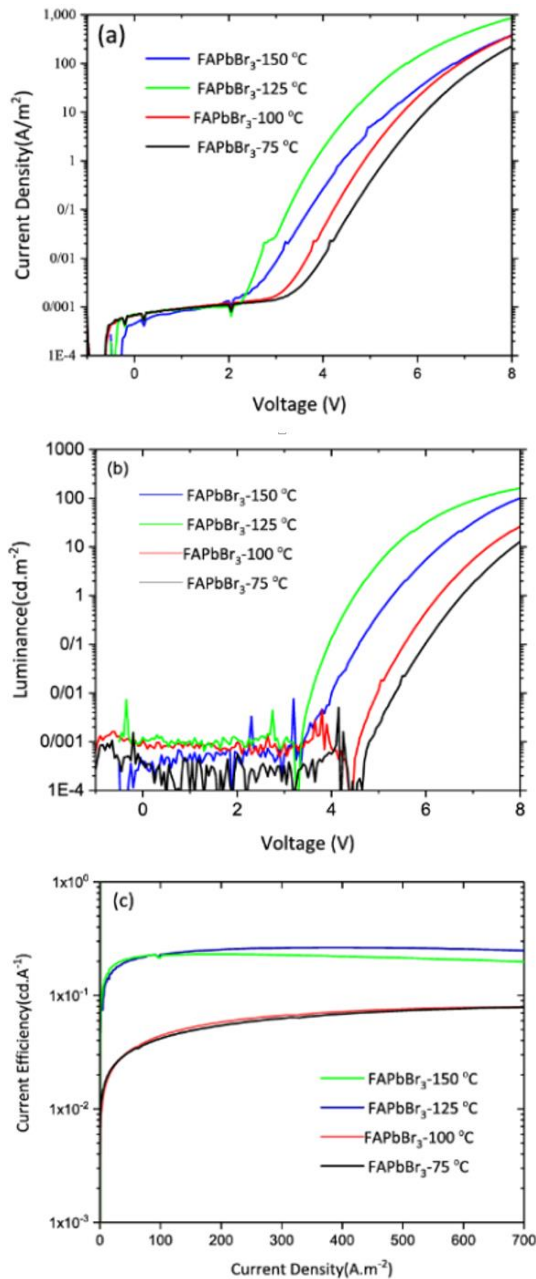


Fig. 6. Current density–voltage–luminance characteristics of PeLED devices fabricated with FAPbBr₃ layers annealed at different temperatures.

The improved device performance of the FAPbBr₃-125 °C sample coincides with the favorable combination of its crystallographic characteristics, uniform surface coverage, and low surface roughness. Although the film annealed at 150 °C exhibits the highest steady-state PL peak intensity, it does not produce the highest electroluminescent device output. This difference demonstrates that PL peak intensity alone is not a sufficient predictor of PeLED performance and that film microstructure, interfacial properties, and charge injection and transport must also be considered.

A quantitative comparison of the structural, optical, and device results reveals that the different measured parameters do not evolve monotonically toward the device optimum. As the annealing temperature increases from 75 to 150 °C, the FWHM of the FAPbBr₃ (100)

diffraction reflection decreases from $0.152 \pm 0.006^\circ$ to $0.0918 \pm 0.0009^\circ$, corresponding to an increase in apparent crystallite size from 52.6 ± 1.9 to 87.3 ± 0.9 nm. The PL peak intensity also reaches its maximum at 150 °C. Nevertheless, the highest device performance is obtained at 125 °C, where the PeLED reaches a maximum luminance of 100 cd m^{-2} and a current efficiency of 1 cd A^{-1} . Therefore, neither the largest coherent crystalline-domain size nor the highest steady-state PL peak intensity corresponds to the optimum electroluminescent device. Instead, the results indicate that the 125 °C condition provides the most favorable overall balance among the measured structural and morphological characteristics and the device-level electrical and optical response.

The present measurements do not directly resolve defect density or carrier-recombination kinetics. Therefore, the reduced device output at 150 °C is not attributed here to a specific defect-mediated or non-radiative mechanism. Measurements such as time-resolved photoluminescence, absolute photoluminescence quantum yield, or direct trap-density characterization would be required to establish such mechanisms quantitatively. Overall, the annealing temperature after spin coating is a critical processing parameter for controlling FAPbBr₃ thin-film quality and optimizing the performance of PeLED devices.

4. Conclusions

In this work, FAPbBr₃ perovskite thin films were fabricated using a sequential process consisting of thermal evaporation of PbBr₂ followed by spin coating of a formamidinium precursor solution. The effect of post-deposition annealing temperature on film formation and PeLED performance was systematically investigated by applying thermal treatments at 75, 100, 125, and 150 °C. The results show that annealing temperature is an important processing parameter that influences the morphology, crystallographic characteristics, optical emission, and electroluminescent behavior of the FAPbBr₃ active layer.

Among the investigated conditions, annealing at 125 °C produced the most suitable film quality for light-emitting diode operation. This sample exhibited favorable surface coverage and crystallographic characteristics together with a smooth surface morphology having a root mean square roughness of 3.3 nm. Although the film annealed at 150 °C exhibited both the highest steady-state PL peak intensity and the largest apparent crystallite size, the 125 °C sample provided the most favorable overall PeLED performance. This finding demonstrates that neither PL peak intensity nor crystallite size alone is sufficient to determine the optimum annealing condition.

The corresponding PeLED device generated green emission with a peak wavelength of 531 nm and a full width at half maximum of 32 nm. A maximum luminance of 100 cd m^{-2} and a current efficiency of 1 cd A^{-1} were obtained for the optimized device.

These findings demonstrate that careful adjustment of the annealing step after spin coating can effectively improve the structural and optoelectronic quality of FAPbBr₃ thin films. The combined thermal

evaporation/spin-coating approach therefore provides a practical route for preparing uniform FAPbBr₃ emissive layers and optimizing green PeLED devices.

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Conflicts of Interest

The authors declare that they have no competing financial interests or personal relationships that could have influenced the work reported in this manuscript.

Authors Contribution Statement

Saleh Younes: manuscript drafting, writing, revision, and editing.

Vahid Fallah Hamidabadi: methodology, formal analysis, review, and editing.

Ali Bahari: supervision, conceptualization, methodology, data curation, investigation, validation, and formal analysis.

Behrooz Eftekharinia: data curation, investigation, validation, conceptualization, and formal analysis.

Hasan Sliman: validation, review, and editing.

All authors reviewed and approved the final version of the manuscript.

Data Availability Statement

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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