Facilely Fabricated Bright Lead-Free Perovskite Cs$_3$Cu$_2$I$_5$ Scintillator Film for High-Definition X-Ray Imaging

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1. Introduction

Since X-ray was discovered by Rontgen in 1895, the X-ray imaging technology has been quickly developed and applied in numerous fields, including nuclear physics, scientific research, medical, security, and nondestructive inspection.[1] An essential component for X-ray imaging is a scintillator, which converts X-rays with high energy into ultraviolet/visible photons with low energy to be detected by photodetectors (e.g., amorphous Si photodiodes, photomultiplier tubes) for imaging.[2] At present, thallium-doped cesium iodide (CsI:Tl), cerium-doped yttrium lutetium silicate ((YAG:Ce), and Ce:LuAG) are widely recognized as commercial scintillators.[3] However, these traditional commercial scintillators mostly contain toxic elements or costly rare earth elements. For instance, YAG:Ce and Ce:LuAG scintillators are limited by their low light yield and high fabrication cost, and CsI:Tl crystals are restricted by their poor stability and extreme toxicity of thallium. Therefore, the development of scintillator materials with environmental stability, low cost, simple synthesis, high light yield, and nontoxicity is urgently needed for the commercial application of X-ray detectors.

Nowadays, lead halide perovskite nanocrystals have been widely reported as promising scintillators due to their flexible preparation process, high stopping power, low detection limit, and multicolor radioluminescence (RL).[4] However, the small Stokes shift and self-absorption severely suppress the light emission efficiency and make it difficult to meet general commercial requirements. Meanwhile, the toxicity of lead is harmful to human health and environment. Moreover, the instability of lead...
halide perovskite itself and the difficulty of spin coating large-area sizes of high-quality nanocrystal films also hinder its commercial applications seriously.

Here, a compact and uniform lead-free perovskite Cs$_3$Cu$_2$I$_5$ film was synthesized by traditional chemical vapor deposition (CVD). The Cs$_3$Cu$_2$I$_5$ film shows a broad band emission peak at 445 nm with a large Stokes shift and a high photoluminescence quantum yield (PLQY) of about 85.14%. Meanwhile, the film exhibits brightness of RL and long-term stability under natural storage conditions. More important, the film illustrates a light yield of 39 251 photons MeV$^{-1}$ and sensitive scintillation response to X-ray signals. For X-ray imaging, Cs$_3$Cu$_2$I$_5$ scintillator can produce clear images of insect specimens and can distinguish line pairs of nanowire diameters with spacing of 5 μm, with a spatial resolution of ≈100 lp mm$^{-1}$. Meanwhile, it also provides high-quality cross sections for the different parts of ant specimens and then a 3D image reconstructed by CT technology. All these excellent properties show the broad prospect of Cs$_3$Cu$_2$I$_5$ film as high-definition, sensitive, and stable X-ray detectors.

2. Results and Discussion

2.1. Phase Purity and Morphology

To determine the crystal structure properties of the Cs$_3$Cu$_2$I$_5$, the film was examined by X-ray diffraction (XRD) and is shown in Figure 1a. The XRD patterns of Cs$_3$Cu$_2$I$_5$ film agreed very well with the (JCPDS no. 45-0077) standard card, confirming that the as-synthesized Cs$_3$Cu$_2$I$_5$ film is pure phase. Meanwhile, all peaks are well matched with the orthorhombic structural Cs$_3$Cu$_2$I$_5$ in space group, in agreement with a previous report.[5] Scanning electron microscopy (SEM) equipped with energy dispersive spectrometer (EDS) was used to observe and analyze the morphologies of the surface and the cross section of the film. The SEM image of the surface Cs$_3$Cu$_2$I$_5$ film and the corresponding Cs, Cu, and I mappings inferred via SEM are provided in Figure 1b. The average content of the Cs, Cu, and I elements was 29.53%, 18.69%, and 51.78%, respectively, which agrees with the stoichiometry of Cs$_3$Cu$_2$I$_5$. An image of the cross section of Cs$_3$Cu$_2$I$_5$ film and the corresponding Cs, Cu, and I mappings inferred via SEM are provided in Figure 1c. The average contents of Cs, Cu, and I elements are 28.11%, 20.16%, and 51.73%, respectively, indicating that pure-phase Cs$_3$Cu$_2$I$_5$ has been successfully formed. EDS elemental mapping showed that the Cs, Cu, and I elements were homogeneously distributed in the entire Cs$_3$Cu$_2$I$_5$ film, indicating that no secondary phase formed. All characterization results show that we have successfully synthesized phase-pure Cs$_3$Cu$_2$I$_5$ film.

To have convincing results, X-ray photoelectron spectroscopy (XPS) measurements were employed to analyze the surface chemical composition and the valence state of Cs$_3$Cu$_2$I$_5$. The XPS survey (Figure 2a) confirmed the presence of cesium (Cs), copper (Cu), and iodine (I) in the crystals, and peaks assigned to Cs 1s, Cs 3d, Cu 2p, and I 3d are observed, which were in good agreement with literature.[6] Moreover, XPS measurements were performed to identify the valence state of Cu ions. Therefore, high-resolution XPS spectra of the Cu, Cs, and I elements are given in Figure 2b–d. As shown in Figure 2c, the measured binding energies of Cu 2p$_{1/2}$ and Cu 2p$_{3/2}$ are 931.98 and 951.88 eV, respectively, which are consistent with Cu$^{+}$ and 932.0 eV, suggesting the presence of Cu$^{+}$ in the resulting sample.[7] However, the characteristic satellite peak of Cu$^{2+}$ is reported at ≈943 eV,[8] which is absent in our results. Therefore, the valence state of Cu in the products is Cu$^{+}$, not divalent copper. In addition, peaks centered at 723.81 and 737.79 eV are ascribed to Cs 3d$_{3/2}$ and Cs 3d$_{5/2}$. Similarly, the 1 d$_{5/2}$ doublet with binding energies of 618.64 and 630.15 eV confirms the presence of I$^{-}$.[9]

As a result, the above results provide further evidence of the successful synthesis of our target sample.

2.2. Photophysical Properties

Figure 3a shows the optical properties of the Cs$_3$Cu$_2$I$_5$ perovskite film. It was characterized using a photoluminescence excitation (PLE), PL, and UV/vis absorption spectrometer. The Cs$_3$Cu$_2$I$_5$ film displays a strong ultraviolet absorption peak at ≈275 nm,
a broad PLE spectrum with a peak wavelength of ≈300 nm, and a broad blue PL emission with a peak wavelength of ≈445 nm. It is obvious that there is a large Stokes shift (170 nm). PL spectra (Figure 3b) measured at different excitation wavelengths ranging from 250 to 320 nm give the same peak shape but different intensities. It suggests that the observed blue emission arises from the radiative recombination from the same excited state. The Cs₃Cu₂I₅ film exhibits an absolute PLQY of 85.14% at the excitation wavelength of 300 nm, as shown in Figure 3c. The PL spectra of Cs₃Cu₂I₅ measured at 293–373 K are shown in Figure 3d. The PL spectra show only one broadband emission ranging from 300 to 700 nm with its peak at around 445 nm. The broadband emission of the PL peak positioned at 293 K (inset Figure 3d) can be fit to a single peak with a Gaussian function. Figure 3e displays the excitation power-dependent PL spectra of Cs₃Cu₂I₅. It can be clearly observed that the PL intensity increases with an increase in power. The relationship between the integral PL intensity and power density can be correlated with the equation \( I = nL^k \), where \( I \) is the integral PL intensity, \( L \) is the excitation power, \( n \) is the emission efficiency, and \( K \) is the fitting value, which represents the emission mechanism. The exponent \( K = 1–2 \) can be ascribed to free exciton and self-trapped exciton (STE) emission, and the exponent \( K < 1 \) can be attributed to defect-related emission. To determine the origin of the broad emission, we fit the integral PL intensity versus power in Figure 3f. The fit value of \( K \) is 1.05 ± 0.03. Therefore, we can infer that the broad emission that originated from exciton emission. To investigate the luminescence kinetics of Cs₃Cu₂I₅, time resolved photoluminescence spectrum measurements were carried out. Figure 3g shows the PL decay curve of the Cs₃Cu₂I₅ sample collected at the emission maximum of 445 nm that was fitted with a single-exponential function to calculate PL lifetime \( \tau = 1.12 \mu s \). Combining the large Stokes shift of 170 nm and microsecond PL lifetime, it can be speculated that Cs₃Cu₂I₅ has a phosphorescent feature and the PL emission originates from the emission of STEs. This emission behavior is indeed similar to what has been observed in the other phosphorescent perovskites and was attributed to STEs that arose from the Jahn–Teller distortion of the polyhedron in the excited state. The formation of efficient STEs is highly dependent on the deformable lattice (or crystal structure) under photoexcitation. Hence, the 0D structure of Cs₃Cu₂I₅ is shown in Figure 3h. Cs₃Cu₂I₅ crystallizes in the orthorhombic Pnma space group and the Cu¹⁺ cations occupy two kinds of sites: in the first Cu¹⁺ site, Cu¹⁺ is bonded in a distorted trigonal planar geometry to three I⁻ atoms. In the second Cu¹⁺ site, Cu¹⁺ is bonded in a
distorted tetrahedral geometry to four I\(^{1-}\) atoms. Two Cu\(^{+}\) ions are linked by five I\(^{-}\) ions to form [Cu\(_2\)I\(_5\)]\(^{3-}\) clusters and the Cs cations surrounded isolated [Cu\(_2\)I\(_5\)]\(^{3-}\) clusters. Such a structure is similar to the previously reported 0D Cs\(_4\)PbBr\(_6\), which also has spatially isolated [PbBr\(_6\)]\(^{4-}\) octahedra surrounded by Cs\(^+\) ions. The excitons are strongly confined to each [PbBr\(_6\)]\(^{4-}\) octahedron, enabling a high exciton binding energy of 353 meV and a high PLQY between 42\% and 45\%.\(^{[15]}\) This is because when the dimension of perovskite is reduced, the quantum confinement of the material itself is more obvious, which leads to the increase in exciton binding energy and PLQY. It also gives the reason for the high PLQY of the prepared 0D Cs\(_3\)Cu\(_2\)I\(_5\) sample. In general, the STE luminescence is accompanied by another emission peak attributed to direct transition. However, only one emission peak was observed for Cs\(_3\)Cu\(_2\)I\(_5\). It is possible that in such materials where excitons are strongly confined, excited-state structural reorganization dominates the PL mechanism of Cs\(_3\)Cu\(_2\)I\(_5\). The time-resolved PL is fit by a single exponent, which further proves the type of exciton radiation recombination. The excitation and recombination processes for Cs\(_3\)Cu\(_2\)I\(_5\) are schematically depicted in Figure 3i. Upon excitation, the soft crystal lattices dissipate a large portion of energy due to Jahn–Teller distortion and then undergo self-trapped states originating from the excited-state structural reorganization.\(^{[16]}\) Finally, the strongly Stokes shifted broadband blue emission with microsecond decay time comes from the radiative transition from the STE state down to the ground state.

2.3. Air Stability

The stability of traditional perovskite materials has always restricted its development, so we investigate the air stability of the as-prepared Cs\(_3\)Cu\(_2\)I\(_5\) film. It was placed in air for 60 days.
and their purity and PL properties with its initial-state sample were compared. Figure 4a compares the XRD patterns of the Cs$_3$Cu$_2$I$_5$ fresh film (0 day) and Cs$_3$Cu$_2$I$_5$ film exposed in air after 60 days. As shown, all these patterns can be indexed to Cs$_3$Cu$_2$I$_5$ compound; no change in the XRD pattern was observed after being exposed to air for 60 days, which clearly indicated the excellent air stability of the as-prepared Cs$_3$Cu$_2$I$_5$ film. Figure 4b shows the PL spectra of the Cs$_3$Cu$_2$I$_5$ fresh sample and sample exposed in air for 60 days. It can be clearly seen that the profiles of these emission curves are the same, but the emission intensity is different. In addition, the PLQY value of the Cs$_3$Cu$_2$I$_5$ sample exposed to air for 60 days was measured and the results are shown in Figure 4c. Amazingly, the PLQY of the sample exposed to air for 60 days remained above 50% and as high as 58.32%. At the same time, we also measured the PL decay curve of the Cs$_3$Cu$_2$I$_5$ sample (in air for 60 days) and calculated PL lifetime $\tau = 1.05 \mu$s, as shown in Figure 4d. It indicates that samples exposed to air for 60 days have some defects. All these results together demonstrated the outstanding air stability of the as-prepared blue-emitting Cs$_3$Cu$_2$I$_5$ film.

2.4. X-Ray Scintillation Properties

For X-ray imaging, an indispensable characteristic of an excellent scintillator is the X-ray stopping power. The absorption coefficient curves of Cs$_3$Cu$_2$I$_5$ and several typical scintillators versus photon energy from 1 keV to 100 000 MeV based on the photon cross section database (XCOM web-tool) are plotted in Figure 5a. The X-ray attenuation ability can be determined by the absorption coefficient ($\alpha$), which mainly depends on the atomic number ($Z$) and density ($\rho$), $\alpha = \rho Z^4/4\pi^3$, where $A$ is the atomic mass, and $E$ is the radiation energy. Here, the atomic mass $A$ is 55 and the effective atomic number of Cs$_3$Cu$_2$I$_5$ was calculated to be 51.1 and the density is 4.5 g cm$^{-3}$. The high effective atomic number and large density guarantee the good absorption capacity of Cs$_3$Cu$_2$I$_5$ film. Compared with other scintillators such as CsI:TI, LuAG:Ce, CsPbBr$_3$, and Si, the Cs$_3$Cu$_2$I$_5$ absorption of X-ray is comparable and slightly higher than commercial scintillators (CsI:TI and LuAG:Ce) in some photon energy ranges. Figure 5b shows the RL spectra of our Cs$_3$Cu$_2$I$_5$ and the commercial LuAG:Ce; Cs$_3$Cu$_2$I$_5$ film exhibits higher RL intensity than the LuAG:Ce commercial scintillator with the same thickness under 50 keV X-ray radiation conditions. Remarkably, the light yield of Cs$_3$Cu$_2$I$_5$ (39251 photons MeV$^{-1}$) is almost twice that of commercial LuAG:Ce (22 000 photons MeV$^{-1}$). As is well known, the low-dose indirect detection and imaging require the RL yield of scintillators to be higher than 20 000–25 000 photons MeV$^{-1}$. The Cs$_3$Cu$_2$I$_5$ scintillator film is also better than that of recently reported lead halide perovskite CsPbBr$_3$ NC scintillators.

Figure 4. a) A comparison of the XRD patterns of Cs$_3$Cu$_2$I$_5$ fresh sample and Cs$_3$Cu$_2$I$_5$ sample exposed to air after 60 days. b) PL spectra of Cs$_3$Cu$_2$I$_5$ fresh sample and Cs$_3$Cu$_2$I$_5$ exposed to air after 60 days. c) PLQY measurement of the Cs$_3$Cu$_2$I$_5$ perovskite film exposed to air after 60 days. d) Time-resolved PL decay and fitting curve of the Cs$_3$Cu$_2$I$_5$ perovskite film exposed to air after 60 days.
Figure 5. a) Calculated absorption coefficients of Cs$_3$Cu$_2$I$_5$, CsI:Tl, LuAG:Ce, CsPbBr$_3$, and Si as a function of the photon energy in the range of $10^{-2}$–$10^2$ MeV. b) RL spectra of Cs$_3$Cu$_2$I$_5$ perovskite film and the LuAG:Ce single-crystal scintillator. c) RL spectra of Cs$_3$Cu$_2$I$_5$ under different radiation dose rates. d) Dose dependence of the RL intensity shows a linear relationship. e) Stability of the RL intensity of Cs$_3$Cu$_2$I$_5$ under continuous X-ray irradiation at a dose rate of 0.2 mGy s$^{-1}$. f) Stability of the RL intensity of Cs$_3$Cu$_2$I$_5$ that is stored in air for 15 days.

($\approx$21 000 photons MeV$^{-1}$)$^{[19]}$, the correlative CsPbBr$_3@$Cs$_x$Pb$_{3-x}$Br$_6$ ($6000$ photons MeV$^{-1}$)$^{[20]}$ and the lead-free halide scintillators like K$_2$CuBr$_3$ (23 806 photons MeV$^{-1}$)$^{[21]}$ Figure 5c shows the RL spectra of Cs$_3$Cu$_2$I$_5$ under X-ray irradiation operated at 50 keV with different radiation doses. No obvious difference was observed between the RL and the PL spectra, suggesting the same recombination mechanism under X-ray and UV light irradiation. The integrated RL intensity shows an exceptional linear relationship in a broad range from 25 to 188 μGy s$^{-1}$, as shown in Figure 5d. The lowest detectable dose rate of the scintillator is 0.815 μGy s$^{-1}$, which is derived from the fitted line intercept with dose rate. This value is much lower than that of the current medical diagnostic (5.5 μGy s$^{-1}$)$^{[22]}$. The inherent stability of the scintillator
material, also known as radiation hardness, is a key parameter to describe the resistance to radiation. It is worth noting that Cs$_3$Cu$_2$I$_5$ exhibits excellent stability under continuous X-ray irradiation at a dose rate of 0.2 mGyair s$^{-1}$, as shown in Figure 5e. To evaluate the air stability, the RL-related measurement is shown in Figure 5f. It still shows good performance under the same radiation conditions after 15 days of storage in air, indicating its outstanding air stability.

2.5. X-Ray Imaging and CT

Based on excellent stability, high light yield, and favorable X-ray response, we used the Cs$_3$Cu$_2$I$_5$ film as the scintillator and analyzed its imaging properties on the X-ray imaging system. The imaging process of the X-ray instrument is described by the simple schematic diagram, as shown in Figure 6a. The target was placed between the X-ray source and the detecting component. The X-ray images containing a small bamboo stick, a steel ball, a needle, an ant, and a mosquito are obtained under same energy at same radiation conditions, as shown in Figure 6b-i. The internal structure and external profiles of the bamboo stick can be clearly observed, as well as the skeletal structure of the ant and mosquito. In particular, the pinholes inside the bamboo rods and the eyes of the ants can be clearly seen in the X-ray images without any destruction. The inset of Figure 6a shows the X-ray imager detector response function and the best response of the instrument is at the peak wavelength of 580 nm. However, the emission peak of our sample is at 445 nm and coupling to the detector is only 75%. Therefore, a clearer image will be obtained if the detector is replaced with more efficient coupling.

Conventional X-ray imaging can only produce one image at most, which is not very discriminative and thus limits its application in medical diagnosis. To solve this problem, researchers have designed the CT technique. It can reconstruct the structure of the various layers of an object at any depth or at any angles.[23] Therefore, the ability to reconstruct images clearly is another important indicator of whether scintillators can be used for medical diagnosis. Accordingly, we have evaluated the resolution of

![Figure 6. a) Schematic of the prototype projection system for X-ray imaging. Inset shows the detector response function. The photograph of the b) bamboo sticks, c) needle, d) ant, and e) mosquito and the corresponding X-ray image of f) bamboo sticks, g) needle h) ant, and i) mosquito.](image-url)
The standard test pattern plate is composed of a lead bar with different wide slits, based on which the spatial resolution of Cs₃Cu₂I₅ film can be obtained from the minimum separable interval width on the imaging picture. As shown in Figure 7a, the minimum separable interval width on the imaging picture is 5 μm, and the resolution of Cs₃Cu₂I₅ film is calculated to be as high as 100 lp mm⁻¹. To further explore the application of the prepared scintillators in practice medical diagnosis, we explored the application of Cs₃Cu₂I₅ film in CT. Figure 7b–d shows the CT images of different parts of ants at different angles. The ant’s head, chest, and abdomen can be clearly seen. Due to the higher attenuation coefficient of high-density eyes compared with other parts, the eyes are as bright gray. It is proved that the ants’ eyes are rich in tissue and high in density. Figure 7e shows a reconstructed image of the ant in 3D, where the profile of the ant can be seen clearly. It demonstrated the outstanding scintillator performance of the as-prepared Cs₃Cu₂I₅ film. To sum up, a high linear relationship between luminescence and the incident dose rate, together with the high-definition X-ray image, demonstrates that Cs₃Cu₂I₅ film can be serviced as ideal X-ray imaging materials for the applications of security, specifically medical diagnostics.

3. Conclusion

In summary, we have successfully prepared the air-stabilized Cs₃Cu₂I₅ as a practical scintillator film for X-ray imaging using the conventional CVD method. To elucidate its practical application, it was evaluated from a commercial concept, including material structure, stability, optical properties, light yield, and imaging resolution. The Cs₃Cu₂I₅ scintillator film obtained a light yield of 39 251 photons MeV⁻¹, which is better than commercial LuAG:Ce scintillator. X-ray imaging was also successfully demonstrated with a resolution of up to 100 lp mm⁻¹. Finally, we use this durable Cs₃Cu₂I₅ scintillator film as X-ray imaging screens for bamboo stick, needle, and insect specimens; it presents high-quality images. In particular, the pinhole inside the bamboo stick and the ant’s eye can be clearly seen in the X-ray images without any damage. More importantly, the CT images of different parts of the ant specimen can be reconstructed, further demonstrating the application of the Cs₃Cu₂I₅ film in practical medical diagnosis. All these excellent properties show the broad prospect of the Cs₃Cu₂I₅ film as high-definition, sensitive, and stable X-ray detectors.

4. Experimental Section

Cs₃Cu₂I₅ Perovskite Film Preparation: CuI (98.0%) and CsI (99.5%) were purchased from Aladdin. All chemicals were used as received without further purification. Quartz boat containing raw materials CsI and CuI in a 3:2 molar ratio was put into the tube furnace and then the glass substrate was sent into the quartz tube and was 20 cm away from the center temperature. Finally, the tubular furnace was pumped down to 50 Pa and 30 sccm of nitrogen was introduced. After that, it was heated up to 540 °C at 10 °C min⁻¹ and the reaction temperature was kept for 1 h, and then it was slowly cooled down to room temperature. A white crack-free perovskite thin film was obtained.

Characterization: The XRD analysis was carried out on Ultima IV with Cu Ka radiation (λ = 1.54 Å), and diffraction patterns were scanned within an angular range of 10°–60°. SEM and elemental analysis were performed on a Carl Zeiss Ultra Plus scanning electron microscope equipped with X-ray EDS. The XPS spectra measurements were conducted using a ThermoFisher (Nexsa). PL and PLE spectra were recorded utilizing a spectrofluorometer (F4600) with a continuous-wave Xenon lamp as the excitation source. The PLQY values of the Cs₃Cu₂I₅ samples were measured using Edinburgh 980 equipped with an integrating sphere. The luminescence decay lifetime was recorded on a time-correlated single-photon counting system of Edinburgh 980. The RL spectra were recorded by combining the X-ray tube with the fluorescence spectrophotometer. X-ray images were recorded in a high-resolution micro-CT equipped with X-ray source (L9181-02).
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Conflict of Interest
The authors declare no conflict of interest.

Author Contributions

C.X.X., Z.L.S., and S.H.L. conceived the project. H.G. and Q.X.Z designed and performed the experiments. P.R., J.P.C., Y.Z.Z., Y.L.T., Z.H.Z., and L.S.C.X.X., Z.L.S., and S.H.L. contributed to writing the article. All authors discussed and reviewed the manuscript.

Data Availability Statement
The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords
perovskite films, quantum yield, scintillators, stabilities, X-ray imaging

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