

FABRICATION OF Cs₂TeI₆ FILM BY ANNEALING CsI FILM IN TeI₄ VAPOUR: EFFECT OF ANNEALING TEMPERATURE

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Abstract

Halide perovskites are now considered promising materials in the field of optoelectronic devices. Recently, the double halide-perovskite cesium tellurium iodine Cs₂TeI₆ has shown its potential application due to its strong stability, high absorption coefficient, suitable bandgap and composition of non-toxic elements. However, studies on its experimental fabrications are still limited. In previous works, we reported a method for fabricating Cs₂TeI₆ thin films by a synthesis of a CsI film followed by annealing in TeI₄ vapor. Those studies focused more on application orientation rather than in-depth analysis of the influence of technical parameters on the film formation. In this study, we make CsI films by using the chemical vapor deposition method and then investigate the effect of TeI₄-annealing temperature in the range of 120-330°C on the Cs₂TeI₆ film formation. After that, the optical property and long-term stability are examined. At 240°C, the film is black, tightly packed and pin-hole free with a good adhesion with the substrate. It has high absorbance in the visible region with an optical bandgap of 1.61 eV and the film is found to be very stable in humid air for 20 weeks.

Keywords: Halide perovskite; cesium tellurium iodide; chemical vapor deposition method; absorbing layer; thin film.

1. Introduction

Recently, halide perovskites have emerged as promising materials in the optoelectronics field due to their significant optical and electrical properties like a proper and tunable bandgap, high optical absorption coefficients, long diffusion lengths, a low recombination rate and compositional flexibility [1-5]. Despite their extensive advantages, the commercial development of halide perovskite optoelectronics has been limited by their toxicity and instability [6-8]. Beside the conventional ABX₃ perovskite structure, some of the compositional and structural derivatives of the perovskite family

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have been studied and explored so far [9-11]. One of these derivatives is the vacancy-ordered double-perovskite Cs_2TeI_6 .

Cs_2TeI_6 material was reported to have strong optical absorption with all coefficient values greater than 10^5 cm^{-1} in the entire visible-light spectrum [12]. It has an indirect bandgap with experimental values ranging between 1.57 and 1.59 eV [13-14]. One of the advantages of the Cs_2TeI_6 material over other perovskites for practical applications is its good structural stability in ambient air. Maughan et al. [14] suggested that it was due to the strong covalent Te-I bonding. Liu et al. [12] also demonstrated that the formation energy ΔH of Cs_2TeI_6 material is - 0.71 eV [12], which is significantly higher than that of other perovskites [12, 15]. Therefore, this material is suggested as a viable option for an absorbing layer in optoelectronic applications.

To the best of our knowledge, only a few experimental studies on this material have been published over the last ten years. As a result, its experimental fabrications and characterizations are still limited. Xu et al. [13] and Guo et al. [16] used electrospray to fabricate Cs_2TeI_6 films with tens of μm thickness. They focus on its X-ray detecting application without looking into the impact of any fabrication conditions. In 2020, Isabel et al. [17] reported the fabrication of Cs_2TeI_6 thin films by the solution method. They applied a thermal annealing after a spin-coating of mixed CsI- TeI_4 solution, however, many large Cs_2TeI_6 crystals developed on the surface of the film. Therefore, additional research is necessary to improve the quality as well as the understanding and application of this material. In previous works, we reported a method for fabricating Cs_2TeI_6 thin films by synthesizing CsI films followed by annealing them in TeI_4 vapor. These Cs_2TeI_6 films were investigated for NO_2 -sensing for gas sensors and photo-sensing for photosensors. Those studies focused more on application orientation than an in-depth analysis of the influence of technical parameters on the film formation.

In this study, we present the fabrication of the Cs_2TeI_6 film using a two-step dry process, similar to that described in our studies [18, 19]. The first step is the deposition of the CsI film by chemical vapor deposition (CVD) method and the second step is the annealing of the CsI film in TeI_4 vapor. In the second step, the effect of annealing temperature on Cs_2TeI_6 film formation was investigated in the range of 120-330°C. Following that, the optical property and long-term stability were investigated. At 240°C, the film is black, tightly packed and pin-hole free with good adhesion to the substrate. It has high absorbance in the visible region with an optical bandgap of 1.61 eV and the film was found to be very stable in humid air for 20 weeks.

2. Experimental procedure

2.1. Material synthesis

CsI (120 mg, 99.9%, Alfa Aesar) and TeI₄ (120 mg, 99.9%, Alfa Aesar) powders were used as precursors and the Cs₂TeI₆ film was grown on a Pyrex glass substrate. The experimental setup for this experiment is depicted in Fig. 1.

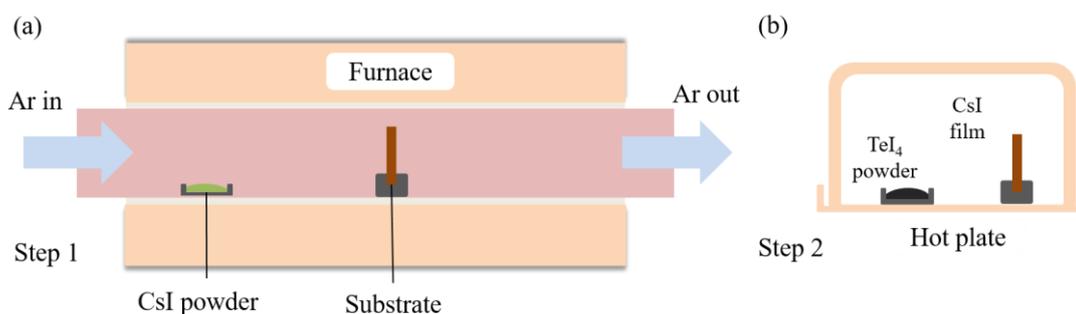


Fig. 1. Experimental setup for synthesis of Cs₂TeI₆ film: (a) Chemical vapor deposition for fabrication of CsI film; (b) annealing system for conversion of CsI film to Cs₂TeI₆ film.

In Step 1, a Pyrex glass substrate (20 mm × 20 mm) was washed with trichloroethylene (TCE), acetone, and deionized water in an ultrasonic bath and dried by nitrogen flow. CsI powder and substrate were put into a quartz tube of CVD system at fixed positions. The temperatures of the CsI powder and substrate were heated to 425°C and 300°C, respectively. The system was heated at a rate of 8 °C/min and the deposition time was 10 min. During the evaporation process, 0.4 sccm of Ar gas was introduced into the quartz tube to maintain a pressure of 0.03 Torr. In Step 2, the CsI film was combined with TeI₄ powder in an alumina chamber and put into an insulation system. The Cs₂TeI₆ fabrication took place at atmospheric pressure in ambient air without any gas flow. The annealing temperature was varied from 120°C to 300°C to examine the change of morphologies and phase structures on the film; others properties such as the optical property, the long-term stability of the Cs₂TeI₆ film were investigated. A hot plate was used as a thermal source and the annealing time is 1 h. After each step, the system was naturally cooled down to room temperature.

2.2. Material characterization

The structural properties of the deposited materials at different annealing temperatures were investigated by X-ray diffraction (XRD) from 10° to 50° on a Panalytical X'pert Pro X-ray diffractometer in ambient air using Cu-Kα₁ radiation (1.540598 Å) at 40 kV potential voltage and 30 mA current. The morphological

property was characterized using a JSM-6701F FESEM equipment with an acceleration voltage of 10 kV. The optical property was exhibited in absorption spectrum ranging from 300 nm to 900 nm using a Cary 5000 UV-Vis-NIR Spectrophotometer.

To aid in the investigation of the effect of the annealing temperature on the perovskite formation, we also synthesized Cs_2TeI_6 powder through a wet ball-milling method and performed a thermal gravimetric analysis (TGA) on it. The CsI, TeI_4 powders, and HI acid were weighed at a mol ratio of $\text{CsI} : \text{TeI}_4 : \text{HI} = 2 : 1 : 0.6$ and put together into the diethyl ether (DEE) solvent. Zirconia balls and a Nalgene bottle were used as the milling medium and it was milled for 18 h. The DEE was removed after the milling process had been completed by drying at 70°C for 5 h. Subsequently, the structure was checked through XRD. The TGA data were recorded from 0°C to 500°C in N_2 ambient at a rate of $10^\circ\text{C}/\text{min}$ using a SDT 650 System from TA instruments.

3. Results and discussion

Figure 2a presents the morphology of the CsI film after the first step, the film is composed of crystalline grains with dimensions ranging from 100 to 900 nm and has a thickness of $\sim 0.7 \mu\text{m}$. In Fig. 2b, the film exhibits the cubic phase (Pm-3m) of CsI, the main peaks at $2\theta = 27.62, 39.45, 48.83^\circ$ corresponding to the atomic plane of (110), (200), and (211), respectively.

The second step is depicted in Fig. 3a, which shows the XRD patterns of the film after annealing the CsI film in TeI_4 vapor for 1 h at different temperatures of 120, 180, 240, 300 and 330°C . In this figure, it was found that:

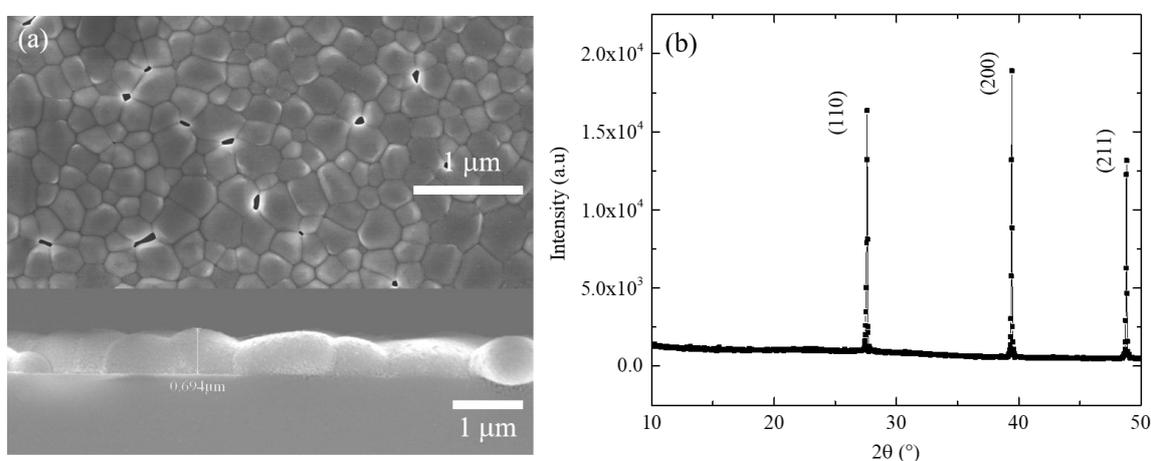


Fig. 2. (a) Top-view and cross-sectional SEM images and (b) XRD pattern of the CsI film.

After 1 h of annealing at 120°C, the pattern contained only CsI peaks, with no further peaks of Cs₂TeI₆ (Fig. 3a - black line); 120°C is too low temperature for the formation of Cs₂TeI₆. At 180°C, the peaks of Cs₂TeI₆ can be seen clearly, even though, the peaks of CsI at 27.5°, 39.4° and 48.8° still appeared in the pattern (Fig. 3a - red line); the conversion to Cs₂TeI₆ was not completed.

After annealing in TeI₄ vapor at 240°C or 300°C for 1 h, all peaks in the patterns can be assigned to the cubic phase of Cs₂TeI₆ (Fm-3m) (JCPDS 01-073-0330) (Fig. 3a - green and blue lines). Fig. 3b shows the XRD pattern of the 240°C-annealed film as representative, where the major peaks at 26.37°, 30.58° and 43.74° are consistent with the corresponding (222), (020), and (044) diffraction planes. The characteristic peaks associated with CsI, TeI₄, or other impurities are not present in the film.

At 330°C, all peaks belong to CsI, with no Cs₂TeI₆ peak being visible in the pattern (Fig. 3a - cyan line).

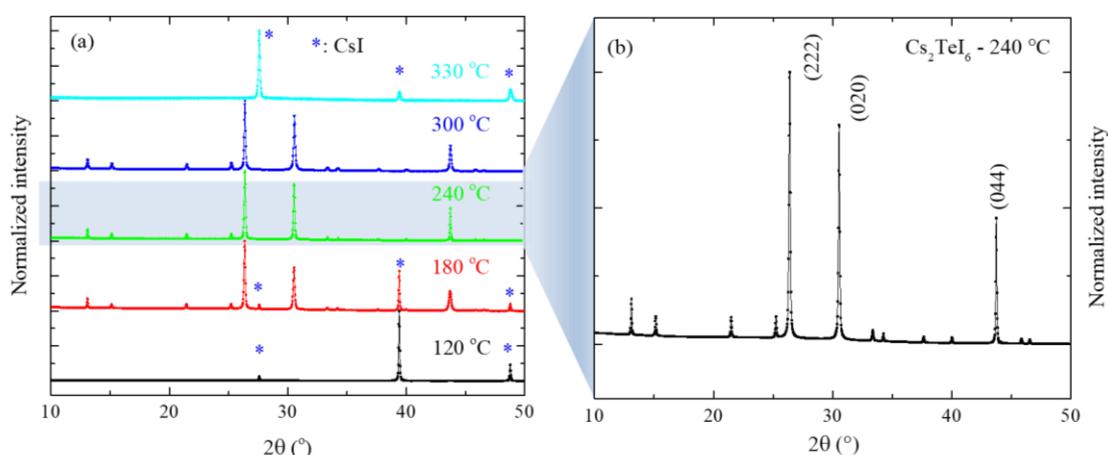


Fig. 3. (a) XRD patterns of the films after annealing the CsI film in TeI₄ vapor at 120, 180, 240, 300, and 330°C for 1 h; (b) The detailed XRD pattern of Cs₂TeI₆ fabricated at 240°C for 1 h with the main peaks marked with atomic planes.

The thermal characteristic of the ball-milled Cs₂TeI₆ powder will help us understand the effect of temperature on the phase formation of the Cs₂TeI₆ film. The cubic structure of the powder is shown in Fig. 4a while the TGA curve is shown in Fig. 4b. The TGA curve shows that the thermal decomposition of Cs₂TeI₆ powder started at ~ 300°C and the extrapolated onset temperature was ~ 350°C. After the decomposition, the leftover powder corresponds to ~ 48% of the initial sample weight, indirectly suggesting that the white remaining powder is CsI, which theoretically constitutes ~ 46% of the total weight in Cs₂TeI₆. This data explain the result that after annealing for 1 h at 330°C, the Cs₂TeI₆ film is decomposed into the CsI film.

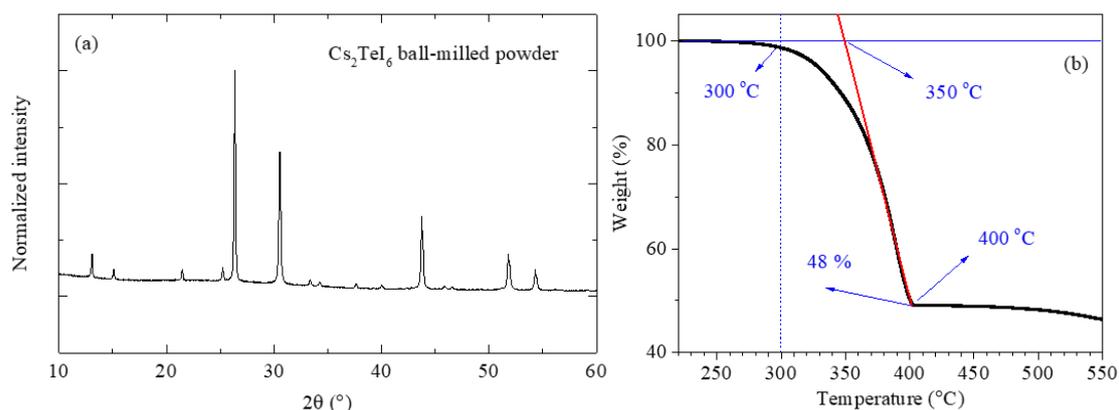


Fig. 4. (a) XRD pattern and (b) TGA curve of ball-milled Cs_2TeI_6 powder.

Based on the XRD results above, the morphologies of the Cs_2TeI_6 films synthesized at 240 and 300°C for 1 h were further investigated by SEM measurements. At 240°C, the film was homogeneous, dense, without pinholes and adhered well to the substrate; its thickness is 1.22 μm (Fig. 5a). Meanwhile, at 300°C, visible micrometer-scale fractures on the surface indicate the discontinuity of the film (Fig. 5b). This effect may be produced by the difference in thermal stress between the glass substrate and the Cs_2TeI_6 film at high temperature; the film can not support the volume expansion at that temperature, resulting in the crack development. The Cs_2TeI_6 film, fabricated at 240°C, was chosen for investigation of the other characteristics.

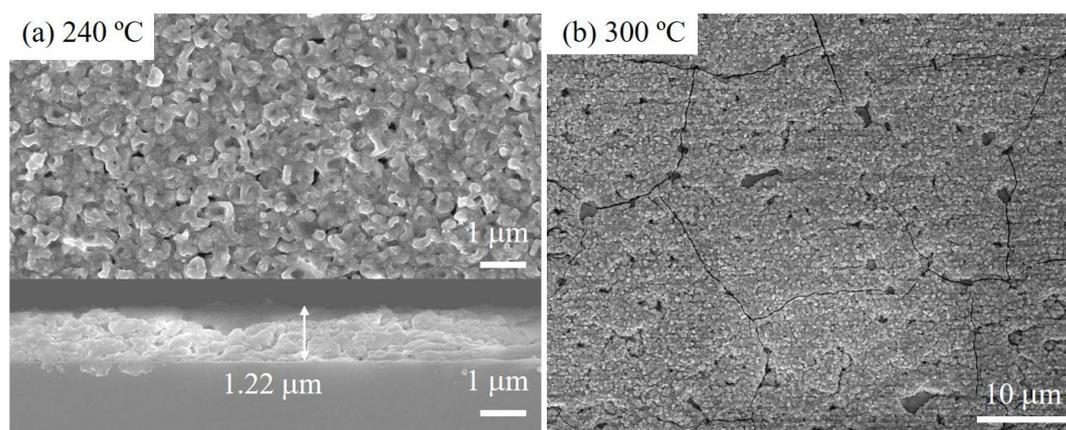


Fig. 5. SEM pictures of Cs_2TeI_6 film synthesized at 240°C (a) and 300°C (b) for 1 h.

The absorbance spectrum of the Cs_2TeI_6 film in the wavelength range of 300-1100 nm is shown in Fig. 6a. The absorbance is approximately 1.5 in the infrared region ($\lambda \geq 800$ nm) but rapidly increases in the visible region, especially, it exceeds 5

in the 300-635 nm range. As an indirect bandgap [9, 14, 20], in Fig. 6b, the optical bandgap of 1.61 eV was determined by the Tauc method, which is close to the values of previous experimental studies. Its value is a little higher than that of its counterpart Cs_2SnI_6 (1.58 eV), which is consistent with the observation that: stronger covalent Te-I bonding makes the lowest conduction band less dispersive and moves the conduction band minimum to a higher energy, resulting in an increase in the bandgap when replacing Sn by Te in the B site of A_2BX_6 halide perovskites [9, 14].

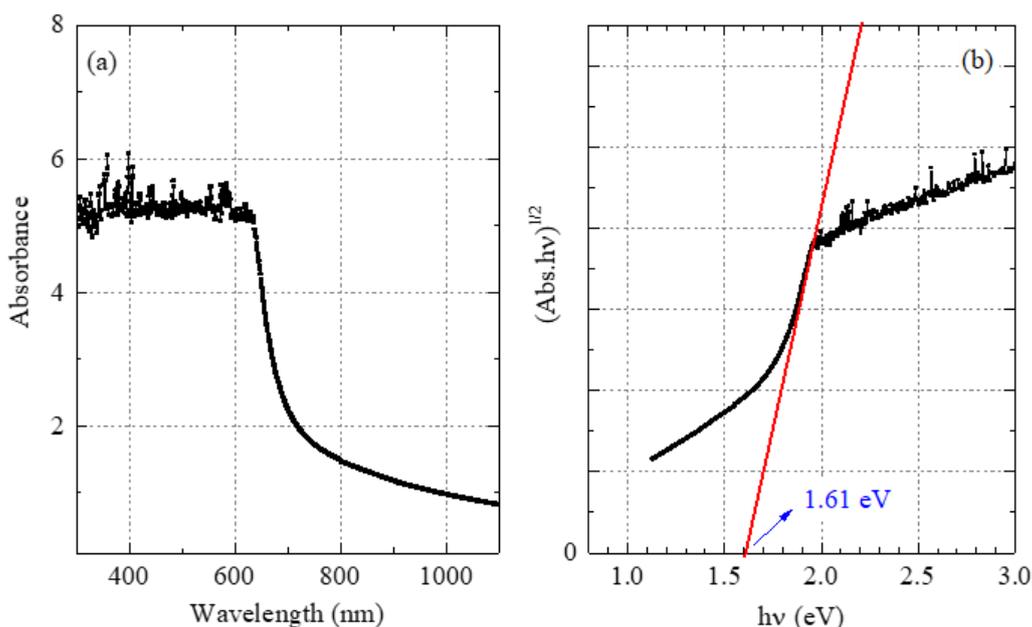


Fig. 6. (a) Absorbance spectrum of the Cs_2TeI_6 film and (b) Tauc plot $((\text{abs.} \cdot h\nu)^{1/2}$ versus $h\nu$) of absorbance spectrum assuming an indirect Cs_2TeI_6 film bandgap.

The long-term phase stability in ambient air is one of the factors that need to be considered when using halide perovskites in practical applications. Some well-known perovskites such as $\text{MA}(\text{FA})\text{PbI}_3$ or $\text{MA}(\text{FA})\text{PbBr}_3$ have to be made in an inert gas (glove box) and often decompose rapidly in ambient air after a few days [21, 22]. In this work, the phase of the Cs_2TeI_6 film was checked after 15 and 20 weeks in humid air (40-60% RH) at 20-25°C. After 15 weeks, there are no other impurity XRD peaks observed (Fig. 7 - red line), indicating the good long-term stability in humid air. After 20 weeks, only a minor peak of CsI impurity appeared at $2\theta \sim 27.5^\circ$ (Fig. 7 - green line), which can be attributed to a slow decomposition of the film. Its good stability can be assigned to the strong covalent bonding of Te-I [14].

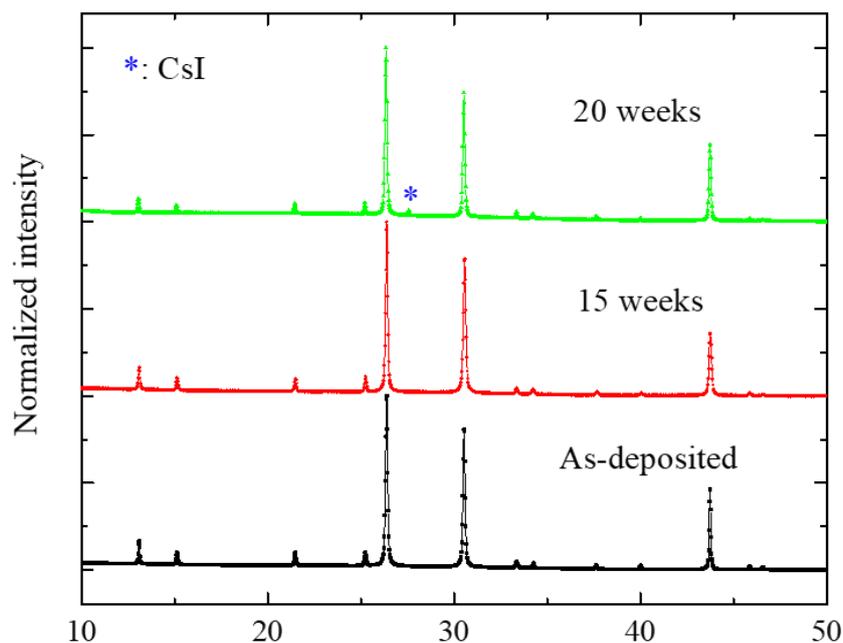


Fig. 7. XRD patterns of Cs_2TeI_6 film annealed in TeI_4 at 240°C for 1 h when being kept at room temperature in the 40-60% humid air for 0, 15 and 20 weeks.

4. Conclusion

In summary, a Cs_2TeI_6 perovskite film was synthesized by a process of fabrication of CsI film by the CVD method and followed by an annealing treatment in TeI_4 vapor. The effect of annealing temperature on the Cs_2TeI_6 film formation was investigated in the range of $120\text{-}330^\circ\text{C}$. When annealing the CsI film in TeI_4 vapor at 240°C for 1 h, the film is continuous, tightly packed with a good adhesion with the substrate. It also showed high absorbance in the visible region and good long-term stability in humid air. The Cs_2TeI_6 thin film has potential for use as an absorbing layer in optoelectronic devices.

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CHẾ TẠO MÀNG Cs₂TeI₆ BẰNG PHƯƠNG PHÁP XỬ LÝ NHIỆT MÀNG CsI TRONG HƠI TeI₄: ẢNH HƯỞNG CỦA NHIỆT ĐỘ XỬ LÝ

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Tóm tắt: Trong lĩnh vực quang điện tử, halide perovskite được xem là những vật liệu có nhiều tiềm năng ứng dụng. Gần đây, vật liệu double-perovskite Cs₂TeI₆ đã cho thấy độ bền cao trong không khí, khả năng hấp thụ quang rất mạnh với bề rộng vùng cấm phù hợp, ngoài ra nó không chứa các thành phần hữu cơ cũng như các nguyên tố độc hại. Tuy nhiên, số lượng những nghiên cứu về quá trình thực nghiệm chế tạo màng còn tương đối ít. Trong các nghiên cứu trước, chúng tôi đã tổng hợp màng Cs₂TeI₆ bằng cách ủ màng CsI trong hơi TeI₄ nhưng tập trung khảo sát khả năng ứng dụng hơn là phân tích ảnh hưởng các thông số kỹ thuật trong quá trình chế tạo màng. Trong nghiên cứu này, chúng tôi nghiên cứu ảnh hưởng của nhiệt độ ủ tới tính chất của màng Cs₂TeI₆ trong dải 120-330°C. Ở 240°C, màng có màu đen, không có lỗ rỗng và bám dính tốt với đế; màng Cs₂TeI₆ có độ hấp thụ cao với E_g = 1,61 eV, và màng rất bền trong không khí ẩm trong 20 tuần.

Từ khóa: Halide perovskite; cesium tellurium iodide; lắng đọng hơi hóa học; màng hấp thụ; màng mỏng.

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