

## Study of the effect of CdCl<sub>2</sub> introduction on the high temperature activation treatment of CdTe:As polycrystalline thin films

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V-doped CdTe polycrystalline films can achieve both doping activation and defect passivation by high-temperature CdCl<sub>2</sub> heat treatment, but this requires simultaneous modulation of the amount of CdCl<sub>2</sub> introduced to obtain high-quality films. It is found that increasing the CdCl<sub>2</sub> introduction does not change the physical phase structure and lattice constant of CdTe:As thin films, but promotes grain recrystallisation, and can promote the formation of A-center, and inhibit the formation of Cd vacancy (V<sub>Cd</sub>) defects, as well as the formation of deep energy level defects. The results provide guidance for the improvement of high-temperature CdCl<sub>2</sub> heat treatment of V-doped CdTe polycrystalline thin films.

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### 1. Introduction

CdTe polycrystalline thin films doped with group V elements (As, Sb, and P) can increase the hole concentration above  $10^{16} \text{ cm}^{-3}$  [1-3], which is expected to enhance the Voc and overall performance of CdTe solar cells [4]. Nevertheless, the current Voc of V-doped polycrystalline CdTe (CdTe:Gv) devices remains relatively low (~800 mV), and the overall performance has not been significantly enhanced [5]. The current study indicates that this phenomenon is related to the deterioration in the quality of the absorber layer following the introduction of V-doping into CdTe [6-8]. Therefore, it is essential to gain a comprehensive understanding of the primary factors influencing the properties of CdTe:Gv thin films in order to provide guidance for enhancing the quality of the absorber layer of CdTe:Gv and improving the overall device performance.

The doping of CdTe with group V elements necessitates an activation treatment to enhance the hole concentration. Density functional theory (DFT) calculations have revealed that a high-

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temperature activation treatment of CdTe:Gv thin films in Cd-rich environments facilitates the suppression of compensatory defect complexes (AX centres) generation, which can avoid the Fermi energy level pinning, and thus enhance the hole concentration[9]. In 2018, the University of Delaware, USA, reported that high-temperature rapid heat treatment of CdTe:Gv thin films in Cd-rich environments yielded hole concentrations in excess of  $10^{16} \text{ cm}^{-3}$ [3]. It was reported in 2019 that the CdCl<sub>2</sub> heat treatment of the CdTe absorber layer is required to passivate defects during device preparation. This is because the CdCl<sub>2</sub> treatment is carried out at a high temperature (400°C) in an air-ambient environment. The simultaneous achievement of thin film passivation and activation with higher carrier concentrations than those observed in samples treated by rapid thermal annealing in a Cd-rich ambient environment was reported. This greatly simplified the post-doping treatment process[10]. The majority of subsequent reports on CdTe:Gv films and devices employ this methodology, wherein passivation and activation are achieved concurrently through high-temperature CdCl<sub>2</sub> heat treatment[1, 11-13].

It is established that the application of heat treatment with CdCl<sub>2</sub> plays a significant role in the promotion of recrystallisation and defect passivation of CdTe thin films, as well as the enhancement of device performance[14-17]. Previously, the CdCl<sub>2</sub> heat treatment temperature of CdTe films and devices was typically low (~385°C). However, Bidaud T. and Amarasinghe M. et al. demonstrated that elevating the CdCl<sub>2</sub> heat treatment temperature to 400°C and above could promote the recrystallisation growth of CdTe grains and passivation of defects, and prolong the carrier lifetime[18, 19]. This provides guidance for the performance enhancement of the devices, such as Voc guidance. Nevertheless, our experiments revealed that high-temperature CdCl<sub>2</sub> heat treatment, when combined with other unchanged conditions, did not enhance the crystalline quality and device performance of CdTe films (unpublished). The studies were conducted by Hsu C. A. and Paul S. et al. have demonstrated that high-temperature CdCl<sub>2</sub> heat treatment does not significantly enhance the performance of the device, including Voc, and may even result in a reduction in performance[20, 21]. It can be observed that an increase in the CdCl<sub>2</sub> heat treatment temperature does not necessarily result in an improvement in the quality and device performance of CdTe films. Given that CdCl<sub>2</sub> is employed primarily to facilitate recrystallisation and grain boundary passivation during CdCl<sub>2</sub> heat treatment[14, 22, 23], it is also essential to consider the quantity of CdCl<sub>2</sub> introduced when preparing CdTe:Gv thin films that necessitate high-temperature activation in order to achieve highly efficient activation while enhancing the quality of the films. Nevertheless, there is a paucity of data regarding the impact of CdCl<sub>2</sub> introduction on the high-temperature activation treatment of CdTe:Gv thin films. This limits the ability to inform research aimed at enhancing the quality and device performance of CdTe:Gv thin films.

Currently, the most efficient method of doping group V elements is in-situ doping, which involves the use of As as the most promising group V element compared to the other group V elements (P, Sb), which are better doped[3, 5, 11, 13]. In this study, CdTe in-situ As-doped thin films were obtained by vacuum evaporation deposition, followed by high-temperature (400°C) heat treatment under different CdCl<sub>2</sub> introduction conditions. X-Ray Diffractometer (XRD) and scanning electron microscopy (SEM) were employed to assess the crystalline quality of the prepared CdTe:As films. Low-temperature photoluminescence (PL) analysis, coupled with DFT calculations, was utilized to elucidate the nature of the defects present in the CdTe:As films. It has been demonstrated that the introduction of CdCl<sub>2</sub> during the high-temperature heat treatment of CdTe:As thin films can significantly enhance the crystalline quality of the films without affecting the physical phase

structure or lattice constant. Furthermore, it can facilitate the formation of shallow host-affected defects in the A-centre ( $V_{\text{Cd-Cl}_{\text{Te}}}$ ) and inhibit the formation of Cd vacancy ( $V_{\text{Cd}}$ ) defects, as well as deep energy level defects, due to the insufficient introduction of  $\text{CdCl}_2$ .

## 2. Experiments

$\text{CdTe:As}$  films (1.5  $\mu\text{m}$ ) were deposited on corning glass substrates by vacuum thermal evaporation of As-containing  $\text{CdTe}$  source powder, which was obtained by high-temperature smelting of  $\text{CdTe}$  and 1 wt.%  $\text{Cd}_3\text{As}_2$  mixed powder, followed by breaking and grinding. The vacuum degree for the deposition process is  $10^{-4}$  Pa and the substrate temperature is maintained at  $400^\circ\text{C}$ . Then a 0.1 mol/L aqueous  $\text{CdCl}_2$  solution was uniformly sprayed on the surface of the as-deposited  $\text{CdTe:As}$  films by an ultrasonic nebulizer to form a thin layer of  $\text{CdCl}_2$ , and a high-temperature treatment was carried out at  $400^\circ\text{C}$  for 30 min. It is noted that the amount of  $\text{CdCl}_2$  sprayed on the surface of  $\text{CdTe:As}$  films is very small and difficult to quantify, hence the amount of  $\text{CdCl}_2$  was quantified by the spraying time of  $\text{CdCl}_2$  solution.

The As-content in the prepared  $\text{CdTe:As}$  films obtained by an inductively coupled plasma mass spectrometer (ICP-MS) was about  $4 \times 10^{15} \text{ cm}^{-3}$ . The phase structure of  $\text{CdTe:As}$  films was investigated by X-Ray Diffractometer (XRD), which was equipped with a  $\text{Cu-K}_\alpha$  radiation ( $\lambda = 1.5405 \text{ \AA}$ ). The scanning electron microscopy (SEM) was employed to characterize the surface morphology of the  $\text{CdTe:As}$  films. The excitation light used in the PL test is a quasi-continuous laser with a wavelength of 650 nm, a frequency of 25 MHz and an average power of 1.74 mW. And the excitation light is incident from the glass side. The minimum sample temperature for PL testing was controlled at 23 K by a closed-cycle liquid helium cryostat.

## 3. Results and discussion

### 3.1. Phase structure analysis

The XRD patterns of  $\text{CdTe:As}$  films with various  $\text{CdCl}_2$  spraying time are shown in Figure 1 (a), all films exhibit a typical cubic structure with a strong preferential orientation along the (111) plane. As shown in Figure 1 (b), the (111) orientation gradually enhanced with increasing the  $\text{CdCl}_2$  spraying time from 2 min to 4 min, and then slightly reduced with continuing to 5 min. It was also evident from Figure 1 (a) that a  $\text{CdTe}$  oxide phase appeared in the samples with  $\text{CdCl}_2$  spraying time of 4 min and 5 min.

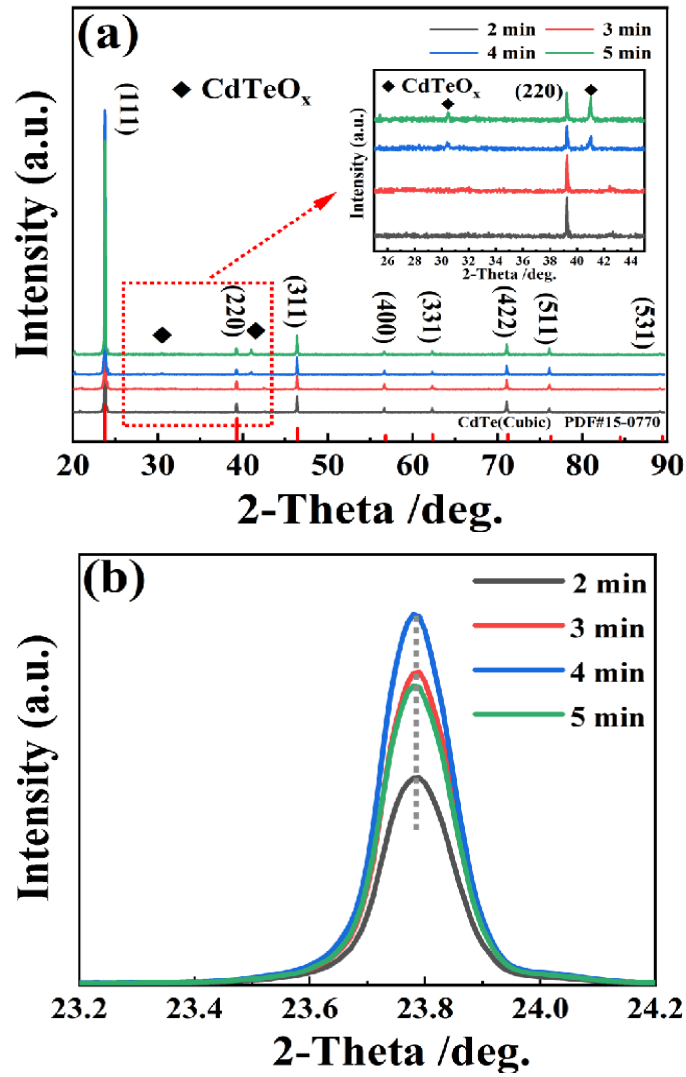


Fig. 1. XRD patterns at  $2\theta$  of (a)  $10\text{-}90^\circ$  and (b)  $23.2\text{-}24.2^\circ$  for CdTe:As films with different CdCl<sub>2</sub> spraying time.

The lattice constant  $a_0$  could be obtained from equation (1) for the cubic crystal structure, where (hkl) is the Miller index,  $\theta$  is the Bragg diffraction angle, and  $\lambda$  is the wavelength of emitted x-rays.

$$\sin^2(\theta) = \frac{\lambda^2}{4a_0^2} (h^2 + k^2 + l^2) \quad (1)$$

The microcrystalline size (D) of the film could be obtained by Scherrer formula (2), where K is a constant related to the crystal shape (usually 1) and  $\beta$  is the full width at half maximum (FWHM) of the diffraction peak.

$$D = \frac{K \cdot \lambda}{\beta \cdot \cos\theta} \quad (2)$$

The presence of micro-strain ( $\varepsilon$ ) in the crystalline alloy sample could cause a broadening of the diffraction peak, and the relationship between  $\varepsilon$  and  $\beta$  follows Eq. (3). Since the CdTe films are alloy materials, the micro-strain of the films could also be described by this equation[24].

$$\varepsilon = \frac{\beta}{4\tan\theta} \quad (3)$$

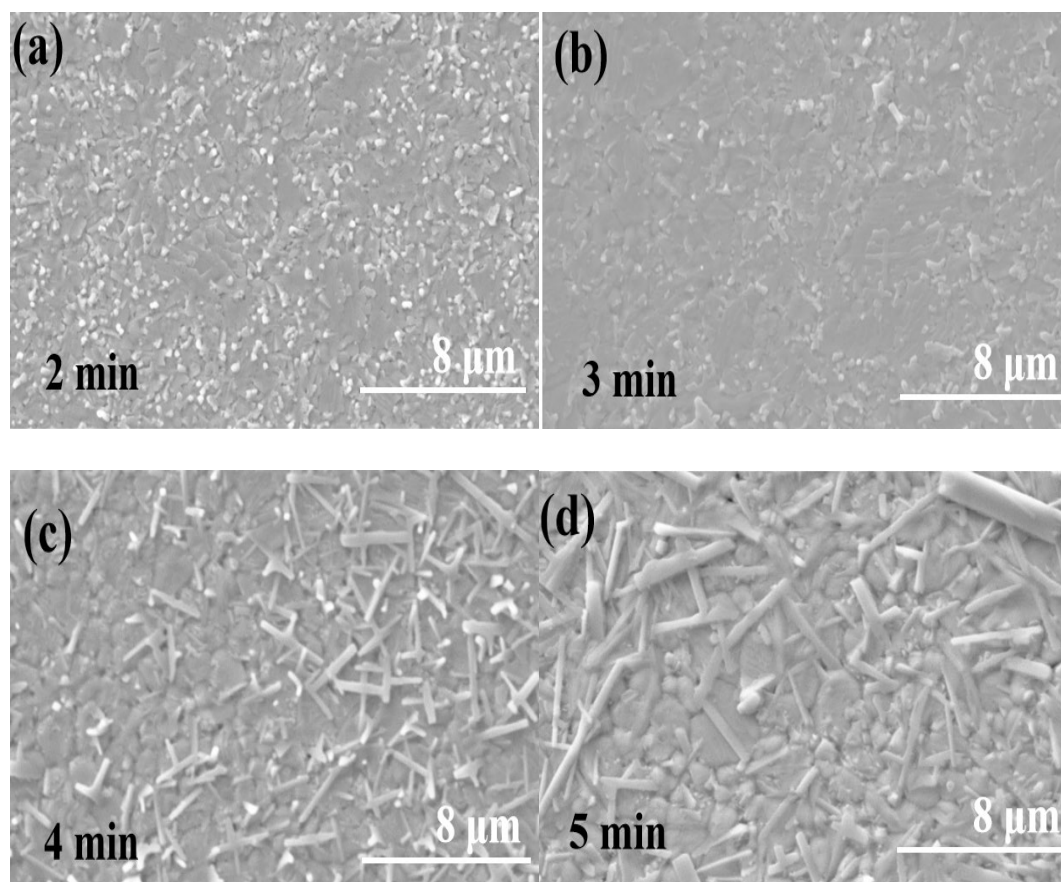
The values of  $a_0$ , D and  $\varepsilon$  for the (111) plane of CdTe:As films in Figure 1 (b) were obtained according to Eqs. (1), (2) and (3), respectively. And the results are listed in Table 1. the lattice constant of the CdTe:As films remains almost unchanged at 6.48 Å as the amount of CdCl<sub>2</sub> introduced is increased. The micro-strain  $\varepsilon$  exhibited a slight decrease from 0.09 to 0.08 as the spraying time exceeded 2 min. However, the prolongation of the spraying time did not result in a significant change. The spraying time was increased from 2 min to 4 min, and the microcrystalline size D gradually increased from 21.96 nm to 22.46 nm, and then decreased after continuing to extend the spraying time to 5 min, decreasing to 22.07 nm. These results indicated that an appropriate increase in the amount of CdCl<sub>2</sub> is beneficial to improve the recrystallization quality of the films, which has a positive effect on reducing the bulk defects in CdTe:As absorber and enhancing the device performance.

Table 1. Calculated structural parameters for the (111) plane of CdTe:As films with different CdCl<sub>2</sub> spraying time.

Time/min	2 $\theta$ (deg.)	$\beta$ (deg.)	$a_0$ (Å)	D (nm)	$\varepsilon$
2	23.79	0.14	6.48	21.96	0.09
3	23.79	0.14	6.48	22.35	0.08
4	23.78	0.14	6.48	22.46	0.08
5	23.79	0.14	6.48	22.07	0.08

### 3.2. Surface and section morphology analysis

The surface SEM images of CdTe:As films with different CdCl<sub>2</sub> spraying time are shown in Figure 2. The surface showed a blocky shape with CdCl<sub>2</sub> spraying time less than 4 min, and became a granular shape with spraying time reached 4 min. Continuing to prolong the spraying time to 5 min, the grain shape did not change, but some grains increased in size. It is noteworthy that some streaks appeared on the film surface when the spraying time exceeded 3 min. According to the x-ray energy dispersive spectroscopy (EDS) line-scan test (Supporting section 1), these streaks were the oxides of excess CdCl<sub>2</sub>, as they showed a higher content of Cl and O elements, but a lower content of Cd. Consequently, a 4-min spraying could introduce sufficient CdCl<sub>2</sub> into a CdTe:As film, and the excess CdCl<sub>2</sub> would attach on the film due to oxidation. It is also deduced that the oxide phase appeared in the film with a spraying time over 3 min in Figure 1 (a) is a result of the oxidation of excess CdCl<sub>2</sub> on the surface.



*Fig. 2. SEM images (20000x magnification) of the surface of CdTe:As films with various CdCl<sub>2</sub> spraying time.*

Cross-sectional SEM images of CdTe:As films with different CdCl<sub>2</sub> spray times are shown in Figure 3. When the spray time of the CdCl<sub>2</sub> solution was less than 4 min, the resulting grains were relatively small. Upon reaching a spray time of 4 min, the grains exhibited a notable increase, accompanied by the emergence of columnar crystals. It can be observed that an increase in the quantity of CdCl<sub>2</sub> introduced is conducive to the promotion of grain recrystallisation growth, which can serve to reduce the formation of defects. And the formation of columnar crystals is conducive to the longitudinal transport of photogenerated carriers in the absorber layer of the device, which plays an important role in enhancing the device performance.

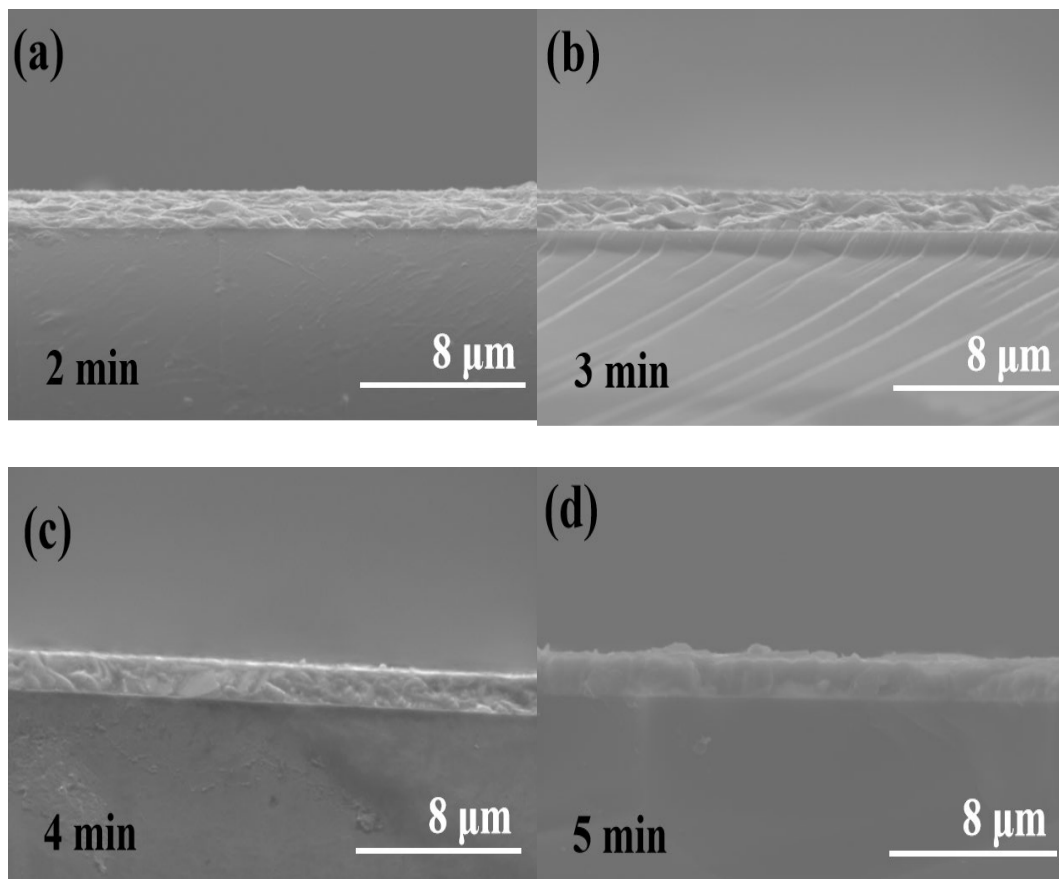


Fig. 3. Cross-sectional SEM images (20000x magnification) of CdTe:As films with different CdCl<sub>2</sub> spray times.

### 3.3. Low-temperature PL analysis

Low-temperature PL tests were performed at 23 K on CdTe:As films, as shown in Figure 4. From Figure 4 (a), all films showed a very strong and broad emission peak centered at 1.416 eV, which was gradually enhanced with increasing CdCl<sub>2</sub> spraying time. Shoulders were observed at 1.456 eV, 1.436 eV and 1.396 eV on the adjacent sides of this emission center, and a weak shoulder was present at ~1.50 eV. In addition, two very weak shoulders were also observed at ~1.56 eV and 1.26 eV for films with spraying time less than 4 min (Figure 4 c and d).

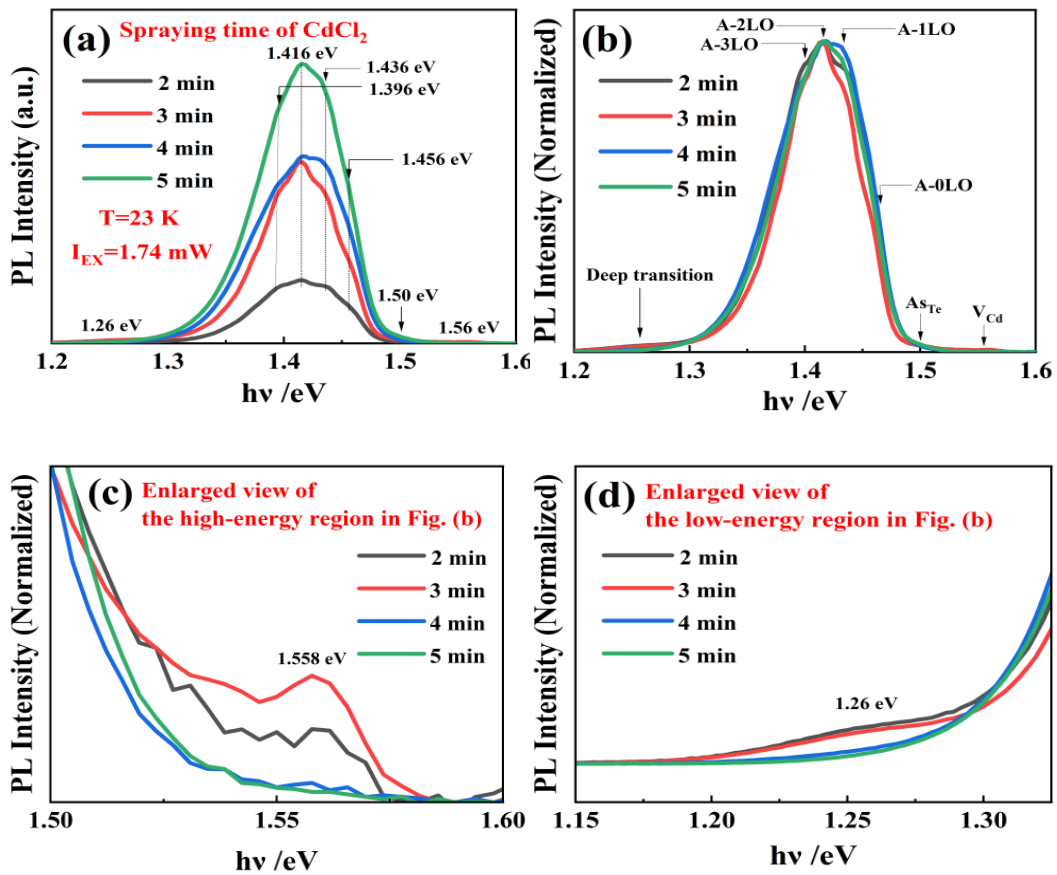


Fig. 4. (a) PL spectra and (b) normalised PL spectra of CdTe:As thin films at 23 K with different CdCl<sub>2</sub> spray times, where the excitation light wavelength is 650 nm and the excitation power is 1.74 mW, and (c) and (d) local magnification of Fig. (b) in the high-energy and low-energy regions, respectively.

The emission peaks in the low-temperature PL spectra of CdTe polycrystalline films are usually classified into four types according to their origin[25-31]: (1) band-edge exciton transition peak at  $\sim 1.59$  eV; (2) band-edge shallow defects transition peak near 1.55 eV; (3) extended defects (e.g., slip dislocation[32]) transition peak at  $\sim 1.48$  eV, also commonly referred to as Y-band; (4) the A-center (Cl-associated defect complexes[33], e.g., Cl<sub>Te</sub>-V<sub>Cd</sub>) transition peak at  $\sim 1.42$  eV. The exciton peak at  $\sim 1.59$  eV was not observed in the PL spectra of all CdTe:As films (Figure 4), probably because the test temperature was not low enough (23 K). In fact, the binding energy of band-edge exciton is so small that its emission is usually observed only at an extremely low temperature ( $\sim 5$  K).

The CdTe:As films with CdCl<sub>2</sub> spraying for 2 min and 3 min showed an emission peak at  $\sim 1.56$  eV, which disappeared with the spraying time prolonged to 4 min (Figure 4 c). We also observed this emission peak in the low-temperature PL spectrum (Figure 5) of a CdTe film prepared under the same conditions (CdCl<sub>2</sub> spraying for 3 min). It is evident that this peak did not originate from Cl-related and As-related shallow defects and increasing the amount of CdCl<sub>2</sub> would suppress these defects. According to Halliday D P and Shin H. Y. et al. we attribute the  $\sim 1.56$  eV peak to the Cd vacancy (V<sub>Cd</sub>), i.e., the electron bound to V<sub>Cd</sub> transition[29, 34]. The emission peak at  $\sim 1.42$  eV



is observed as a dominant peak in the PL spectra of all CdTe:As films and CdTe films. The intensity of this peak increased with increasing CdCl<sub>2</sub> spraying time (Figure 4 a), indicating that this peak is attributed to the A-center (V<sub>Cd</sub>-Cl<sub>Te</sub> complexes). The gap between the adjacent shoulders on either side of A center peak is ~20 meV, which is comparable to the LO phonon energy of CdTe (21 meV[35]). Apparently, these shoulders are attributed to the phonon replicas of A center, which is consistent with the reports[8, 29, 30, 32]. Notably, no significant Y-band emission peak was observed at ~1.48 eV in Figure 4 a. This may be attributed to the fact that the Y-band was covered by the A center emission signal, which often happens in the PL spectra of CdTe polycrystalline films[30, 36, 37].

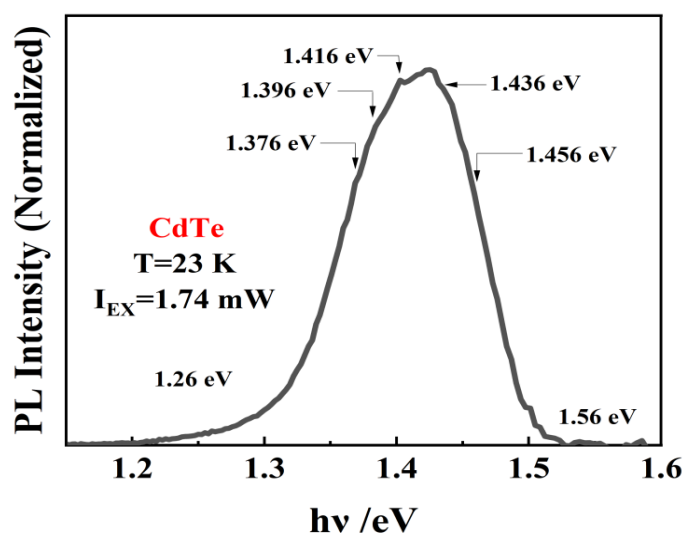


Fig. 5. PL spectrum of an undoped CdTe film prepared (CdCl<sub>2</sub> spraying for 3 min) and tested under the same condition as the CdTe:As films.

Besides the four types of emission peaks commonly found in CdTe polycrystalline films, all CdTe:As films also showed a weak shoulder at ~1.5 eV (Figure 4 a). We have not observed this shoulder in the PL spectrum of the CdTe film (Figure 5), and thus attributed this shoulder in Figure 4 (a) to As-related defects. Mccandless B et al. observed a distinct emission peak at 1.5 eV in the PL spectrum of CdTe:As polycrystalline films and attributed it to the A<sub>STe</sub> shallow acceptor [10]. Therefore, we also attributed the weak shoulder at ~1.5 eV here to the A<sub>STe</sub> defect. In addition, a shoulder showed at ~1.26 eV for samples with CdCl<sub>2</sub> spraying time less than 4 min, and it disappeared when spraying time reached 4 min (Figure 4 d). We also observed this low-energy shoulder in the PL spectrum of an undoped CdTe film (Figure 5). In addition, we found that this shoulder was still visible at 103 K for samples with CdCl<sub>2</sub> spraying time less than 4 min and was significantly enhanced compared to the intensity of the A center peak (Figure 6). Therefore, this shoulder may originate from a deeper defect unrelated to Cl and As, and the formation of this defect could be suppressed by increasing the amount of CdCl<sub>2</sub>. Finally, the origin of each emission peak is labeled in Figure 4 b.

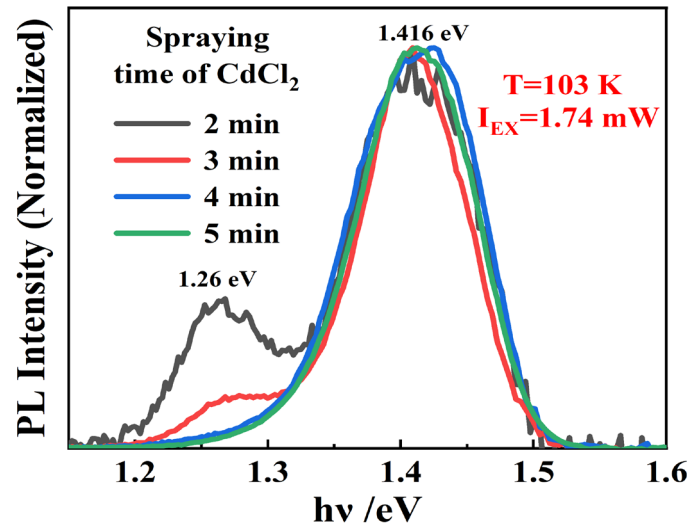


Fig. 6. Normalized PL spectra at 103 K for CdTe:As films treated with different CdCl<sub>2</sub> spraying time.

#### 4. Conclusion

The XRD and SEM tests of CdTe:As polycrystalline films heat-treated at high temperatures under different CdCl<sub>2</sub> introduction levels show that increasing the CdCl<sub>2</sub> introduction does not change the phase structure and lattice constants of the films, which remain at 6.48 Å, and promotes grain recrystallisation and growth to form columnar crystals. However, it increases the microcrystalline size and grain size of the CdTe:As films, up to 22.46 nm, and reduces the microstrain of the films from 0.09 to 0.08. The excess CdCl<sub>2</sub> is oxidised and adheres to the CdTe:As films when the spraying time exceeds 4 min.

Low-temperature PL tests revealed that increasing the amount of CdCl<sub>2</sub> introduction promotes the formation of shallow host-accepted defects in the A-centre ( $V_{Cd-Cl_{Te}}$ ) and suppresses the formation of Cd vacancy ( $V_{Cd}$ ) defects caused by the insufficient CdCl<sub>2</sub> introduction as well as the formation of deep-energy level defects. In summary, an appropriate increase in the amount of CdCl<sub>2</sub> introduced during the high-temperature CdCl<sub>2</sub> heat treatment of CdTe:As polycrystalline thin films is of great significance in enhancing the quality of the films, which would be essential to improve the transport properties of photocarriers in CdTe devices.

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