Effect of Cd on cation redistribution and order-disorder transition in Cu$_2$(Zn,Cd)SnS$_4$†

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Cation substitution has been extensively used to improve the fundamental optoelectronic properties and the photovoltaic performance of kesterite solar cells, and some of the most promising results have been obtained by substituting zinc with cadmium. Structurally, the positive effects of Cd have been attributed to the expected increase in the formation energy of defects such as Cu$_{Zn}$ + Zn$_{Cu}$ due to the larger ionic radius of Cd$^{2+}$ as compared to Zn$^{2+}$. However, ab initio calculations using density functional theory (DFT) showed similar formation energies for Cu$_{Zn}$ + Zn$_{Cu}$ in Cu$_2$ZnSnS$_4$ and Cu$_{Cd}$ + Cd$_{Cu}$ in Cu$_2$CdSnS$_4$. Further, in this report, it is shown that Cd does not directly substitute the zinc lattice sites (2d Wyckoff positions) in the Cu$_2$ZnSnS$_4$ structure, but rather, a two-way cation restructuring due to the continuous transformation of the structure from kesterite to stannite leads to Cu replacing Zn, and Cd occupying the Cu sites (2a Wyckoff positions) in the partially Cd-substituted Cu$_2$Zn$_{1-x}$Cd$_x$SnS$_4$. Hence, the structural reasons for the beneficial effects of Cd need to be reinterpreted. Here, using computational model based on cluster expansion (fitted on DFT data), Monte-Carlo simulations, and differential scanning calorimetry, it is shown that Cu$_{Cd}$SnS$_4$ has less structural disorder than Cu$_2$ZnSnS$_4$ even if the thermodynamic point defect formation energy calculated using dilute point-defect models for disorder-inducing Cu$_{Zn}$ + Zn$_{Cu}$ and Cu$_{Cd}$ + Cd$_{Cu}$ defects in these two materials is predicted to be similar. This difference in the structural disorder is due to a sharp order-disorder transformation in Cu$_2$ZnSnS$_4$ at about 530 K, and a continuous order-disorder transformation in Cu$_2$CdSnS$_4$ throughout the range of processing temperatures.

1. Introduction

The photovoltaic performance of copper zinc tin sulfo-selenide (Cu$_2$ZnSn(S,Se)$_4$), a promising candidate for low-cost thin-film solar cells, is currently limited by point defects. Many of these point defects have a low formation energy, which is attributed to the similar ionic radii of Cu and Zn. Various approaches have been employed to tackle the formation of point defects. Cation substitution – that is, the substitution of one of the cations in Cu$_2$ZnSn(S,Se)$_4$ with an isostructural cation – has recently shown promise in altering the point defect characteristics. The substitution of Zn with Cd has been especially promising, with the highest reported efficiency of 11.5% for a partial substitution of Zn with Cd in copper zinc tin sulphide (Cu$_2$ZnSnS$_4$). The rationale behind the introduction of Cd in the Cu$_2$ZnSnS$_4$ structure is that the larger ionic radius of Cd$^{2+}$ as compared to Zn$^{2+}$ would increase the formation energy of the Cu$_{Zn}$ + Zn$_{Cu}$ antisite defect. However, DFT calculations predict that the formation energy of Cu$_{Zn/Cd}$ + Zn/Cd$_{Cu}$ is unaffected by the substitution of Zn with Cd. Hence, the structural characteristics that lead to the improved performance are unclear. Moreover, a detailed structural characterization is still missing for the partially Cd substituted Cu$_2$Zn$_{1-x}$Cd$_x$SnS$_4$ absorbers at compositions where the high efficiency is obtained. One of the reasons for this is that neutron diffraction, which has been used extensively to study the structure of kesterite, cannot be used for cadmium containing samples because of the high neutron absorption cross section of Cd. Hence, alternative approaches are needed to study the structure of Cu$_2$Zn$_{1-x}$Cd$_x$SnS$_4$.

In this paper, we study the structural characteristics of copper zinc cadmium tin sulphide (Cu$_2$Zn$_{1-x}$Cd$_x$SnS$_4$) using X-
ray diffraction (XRD), Raman spectroscopy, density functional theory (DFT) and differential scanning calorimetry (DSC). We show that the intuitively expected substitution of Zn with Cd on 2d Wyckoff sites does not occur in the Cu2ZnSnS4 structure, but rather, due to the continuous transformation of the structure from kesterite to stannite with increasing Cd substitution, Cd replaces Cu at 2a sites, while Cu moves to the Zn 2d positions.

Using DFT and Monte-Carlo calculations supported by DSC experiments, we show that Cu2CdSnS4 can have less disorder inducing CuCd + CdCu antisite defects compared to the CuZn + ZnCu defects in Cu2ZnSnS4, even if the formation energy of these defects calculated using dilute point-defect models is predicted to be similar.* Since these disorder-inducing defects are not expected to directly affect the optoelectronic properties, it is proposed that the reduced CuCd + CdCu antisites can destabilize other compensated deleterious defects containing CuCd, such as 2CuCd + SnCd, thereby contributing to the improvement in optoelectronic properties.

2. Experimental methods

2.1. Deposition of Cu2(Zn,Cd)SnS4 thin films

The thin films were deposited by spin-coating a solution of copper acetate hydrate (0.52 mol L⁻¹), zinc acetate dihydrate and cadmium acetate dihydrate (0.33 mol L⁻¹), tin chloride dihydrate (0.27 mol L⁻¹), and thiourea (2.06 mol L⁻¹), at 4000 rpm on Mo-glass substrates. The spin-coating process was repeated 13 times, and the sample was annealed in air at 280 °C for 2 minutes after spin-coating. These films were then sulfurized at 580–600 °C for 40 minutes in Ar + S atmosphere and were cooled naturally in the furnace. The Cu/[Cd + Sn + Zn] ratio was 0.86 and Sn/[Cd + Zn] ratio was 0.8 for all the samples.

2.2. Experimental characterization

XRD measurements were performed using a Bruker D8 Advance system using a Cu Kα radiation source (40 kV, 40 mA) in the grazing incidence mode, with a grazing angle of 4°. The use of GIXRD makes the results less sensitive to texture effects, and hence, we did not perform pole figure measurements to determine the preferred orientation of the thin films before analysing the XRD data in Section 3.1. XRD simulations were performed using the software VESTA.11 Raman and photoluminescence measurements were performed at room temperature on bare Cu2ZnSnS4, CdSnS4 absorbers with 633 nm laser excitation in a back-scattering configuration using iHR-320 monochromator coupled with a CCD and InGaAs detectors, and FHR-640 monochromator coupled with a CCD detector. All spectra were calibrated by imposing the position of the main peak of monocrystalline Si to 520 cm⁻¹. Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were performed using Labsys EVO 1150 from SETARAM Instrumentation, having a resolution of 0.2 μg and 10 μW, with a temperature accuracy of 1 °C. About 20 mg of powder samples for DSC-TGA were obtained by scraping sulfurized Cu2ZnSnS4 and Cu2CdSnS4 thin films. The samples are equilibrated at 30 °C for 10 min before heating to 350 °C at a rate of 3 °C min⁻¹ under vacuum.

2.3. Computational details

To understand the order-disorder phase transition of Cu2ZnSnS4 and Cu2CdSnS4, we performed canonical Monte-Carlo (MC) simulations with effective cluster interactions (ECIs) obtained through cluster expansion (CE) using the CASM code.12 The ECIs were obtained by fitting to the density functional theory (DFT) total energies within a ternary CE formula in which the cationic sites can be occupied by either Cu, Zn (Cd), or Sn for Cu2ZnSnS4 (Cu2CdSnS4). We considered pair interactions up to the fourth nearest neighbour (4NN) and triplet interactions up to the second nearest neighbour (2NN). For each material, about 700 stoichiometric supercells comprising up to 32 atoms were enumerated, for which the total energies were calculated with the Vienna ab initio package (VASP)15–16 adopting the Perdew–Burke–Ernzerhof (PBE) exchange-correlation functional17–20 and including a Hubbard U correction for the Cu 3d states [U = 6 eV].21 We used a kinetic cut-off energy of 500 eV for the projector-augmented wave basis sets and k-point meshes corresponding to a density of 1600 k-points per atom in the first Brillouin zone. The atomic positions and lattice vectors were allowed to relax until the forces were converged within 20 meV Å⁻¹. Based on a genetic algorithm, the cluster expanded total energies achieve a 10-fold cross-validation score better than 10 meV per atom. The subsequent MC simulations were performed with a 30 × 30 × 30 supercell amounting to a total of 216 000 atoms, among which half of the sites were active. Each MC run at a given temperature comprised 20 000 equilibration passes followed by 50 000 production passes with a sampling interval of 10.

3. Results and discussion

3.1. X-ray diffraction

3.1.1. Structural differences between Cu2ZnSnS4 and Cu2CdSnS4. It is known that Cu2ZnSnS4 thin films crystallize in the kesterite-type structure, while Cu2CdSnS4 films crystallize in the stannite-type structure (Fig. 1).22,23 Before studying the structural trends due to the partial substitution of Zn with Cd, we outline the differences between Cu2ZnSnS4 and Cu2CdSnS4: (i) The 2a sites in kesterite Cu2ZnSnS4 are occupied by Cu while the 2a sites in Cu2CdSnS4 are occupied by Cd, and (ii) the 2d sites in kesterite Cu2ZnSnS4 are occupied by Zn while the 4d sites in stannite Cu2CdSnS4 are occupied by Cu. There are three possible ways in which perfectly ordered kesterite Cu2ZnSnS4 can transform into perfectly ordered stannite Cu2CdSnS4 (Fig. 2).

i. Kesterite to stannite transformation at a particular Cd content. In this model, before the kesterite to stannite transformation, Cd replaces Zn in the 2d site in the kesterite structure, and after the transformation, Cd replaces Zn in 2a sites in the stannite structure. This model is shown in Fig. 2a, with an example of the transformation occurring at Cd/[Cd + Zn] = 0.40.

ii. Continuous transformation from kesterite to stannite with Cd partially occupying the 2a and 4d sites. In this model, the
occupancy of the sites depends on the degree of transformation from kesterite to stannite, which depends on the Cd content. We assume a linear relationship between the degree of transformation and the Cd content. Hence, in this model, with increasing Cd content, Cu from the 2a site replaces Zn/Cd in the 4d sites [note that in the partially substituted Cu$_2$Zn$_{1-x}$Cd$_x$SnS$_4$, we use Wyckoff notations for stannite Cu$_2$CdSnS$_4$]. The 2a sites left behind by Cu are occupied by Zn and Cd. This continues until all Zn are replaced with Cd, when all Cu from 2a sites go to 4d sites, and all 2a sites are occupied by Cd, that is, till we get the stannite Cu$_2$CdSnS$_4$ structure shown in Fig. 1.

iii. Continuous transformation from kesterite to stannite, with Cd occupying only the 2a sites. In this model, Cu from the 2a sites replaces Zn in the 4d sites. The 2a sites left behind by Cu are occupied by Cd, so that Cd and Zn are never present on the same Wyckoff sites.

In the sections below, these three models are referred to as model 1, model 2, and model 3, respectively. Having enumerated the possible models, we simulate the XRD patterns for these models at Cd/[Cd + Zn] = 0.0, 0.2, 0.4, 0.6, 0.8, and 1.0, and compare the simulated XRD patterns to the experimentally measured patterns for the same compositions.

3.1.2. Simulated and measured XRD patterns. We use the XRD data in the 2$\theta$ range 15–24 degrees to study the structure of Cu$_2$Zn$_{1-x}$Cd$_x$SnS$_4$ (Fig. 3; XRD patterns in the 2$\theta$ range 10–60 degrees shown in Fig. S1†). This narrow range is chosen because the 002, 101, and 110 peaks in this range are relatively insensitive to the change in c/a ratio of the tetragonal structure, while other strong XRD peaks are more sensitive to the c/a ratio than to the space group symmetry. Hence these peaks can reliably be used to differentiate between stannite and kesterite. The simulated XRD patterns for the three models show distinct trends.

For model 1 (Fig. 3a), before the kesterite to stannite transformation, the intensity of the 101 peak is significantly higher than that for peaks 002 and 110; after the transformation, the 101 peak can barely be observed. In Fig. 3a, although it is assumed that the transformation occurs for Cd/[Cd + Zn] > 0.4, the trend in the peak intensities observed above will remain the same irrespective of the Cd/[Cd + Zn] ratio. On the other hand, it can clearly be observed in the experimental XRD patterns...
that there is no drastic change in the peak intensities as expected in model 1. The change in the peak intensities in the experimental XRD patterns are gradual and span over the entire Cd/[Cd + Zn] range. Hence, we conclude that the kesterite to stannite transformation does not occur at any particular Cd/[Cd + Zn] ratio, but rather, is a continuous transformation, either according to model 2 or model 3.

For model 2 (Fig. 3b), the intensity of the 101 peak decreases gradually with increasing Cd/[Cd + Zn] ratio. This is consistent with the trend in the intensity of the 101 peak in the experimental XRD pattern (Fig. 3d). However, the relative intensities of the three peaks are not consistent with those observed in the experimental patterns. For example, in the simulated patterns, at Cd/[Cd + Zn] = 0.40, where the best photovoltaic performance is obtained, the relative intensities of the three peaks are almost the same as those observed for Cd/[Cd + Zn] = 0. This is not observed in the experimental patterns, where the intensities of the three peaks at Cd/[Cd + Zn] = 0.4 are similar. The differences in the relative intensities of the peaks between the simulated and observed XRD patterns are even more significant for higher Cd/[Cd + Zn] ratios. At Cd/[Cd + Zn] = 0.60, the simulated patterns show a higher peak intensity for 101 peak as compared to 002 and 110 peaks, while the experimental patterns show an opposite trend – higher peak intensities for 002 and 110 compared to 101. Hence, based on comparisons of the relative intensities of the peaks, we conclude that the kesterite to stannite transformation does not follow model 2.

Finally, an excellent agreement is observed in peak positions, peak intensities, and relative peak intensities between the simulated and experimental XRD patterns. Hence, we conclude that model 3 best describes the structural changes that occur as Zn is substituted with Cd. The continuous structural change from kesterite to stannite in this model is also supported by the evolution of peak positions in Raman spectra, that show a gradual change in the position of the main A1/A1 peak (Fig. S2†). In model 3 (Fig. 2c), the Cd that is ‘substituting Zn’ does not replace Zn at the 2d sites. Rather, Cu from 2a sites replaces Zn at 2d sites, and Cd occupies the 2a site left behind by Cu. Hence, the argument used to justify the substitution of Zn with Cd – that is, the difference in the ionic size – needs to be reinterpreted. In the following sections, we study the structural differences that could explain the optoelectronic effects of Cd substitution.

3.3. Cu–Zn and Cu–Cd disorder

3.3.1. Computational results. We used a model based on cluster expansion (fitted on DFT data) and Monte-Carlo (MC) simulations to evaluate how cation disorder depends on temperature in both Cu2ZnSnS4 and Cu2CdSnS4. The computed heat capacity of Cu2ZnSnS4 shows a sharp peak at 530 K.
(Fig. S3†), indicative of a second-order phase transition in agreement with previous computations and Raman spectroscopy. In contrast, such a second-order phase transition is not present for Cu₂CdSnS₄. Indeed, while Cu₂ZnSnS₄ undergoes phase transition from the ordered kesterite structure of the space group I \( \overline{4} \) at low temperatures (Fig. 1) to the disordered structure of the space group \( I \overline{4} 2m \) at high temperatures, for Cu₂CdSnS₄ the structural changes are absent since the ordered stannite structure already exhibits the space group \( I \overline{4} 2m \), thereby suppressing the order-disorder phase transition.

Fig. 4a shows the temperature dependence of the long-range order parameter \( S \), defined as \( S = [P_{\text{Cu}}(\text{Cu}) - f(\text{Cu})]/[1 - f(\text{Cu})] \), where \( P_{\text{Cu}}(\text{Cu}) \) is the occupancy of a Cu atom in the native Cu sites for the ordered structure, and \( f(\text{Cu}) \) is the fraction of Cu atoms, which is equal to 2/3 as the Sn atoms are found immobile below 1200 K according to our MC simulations. The order parameter \( S \) ranges from 1 for an ordered structure to 0 for a fully disordered structure. For Cu₂ZnSnS₄, \( S \) decreases abruptly towards 0 above the critical temperature of \( \sim 530 \) K, as the Cu occupancy at the cationic sites approaches the random limit (Fig. 4b). Compared to Cu₂ZnSnS₄, the lack of a second-order phase transition leads to a less drastic decrease of \( S \) for Cu₂CdSnS₄. As a result, Cu₂CdSnS₄ is significantly more ordered than Cu₂ZnSnS₄ for typical synthesis temperatures of 770–900 K (highlighted in grey in Fig. 4a–c).

### 3.3.2. Differential scanning calorimetry

To support the computational finding that Cu₂ZnSnS₄ undergoes an order-disorder phase transition and Cu₂CdSnS₄ does not, we performed differential scanning calorimetry measurements in conjunction with thermogravimetric analysis (DSC/TGA). In the case of Cu₂ZnSnS₄, we observe a peak in the heat flow at \( \sim 520 \) K (Fig. 4d), in remarkable agreement with the prediction of a sharp peak in the heat capacity for Cu₂ZnSnS₄ at \( \sim 530 \) K (Fig. S3†). Further, TGA curve for Cu₂ZnSnS₄ around this temperature shows no mass loss/gain, suggesting that the peak in the heat flow is due to the enthalpy of the order-disorder phase transformation. In case of Cu₂CdSnS₄ a peak is observed at similar temperatures close to 520 K, although a clear weight change was also observed at this point, suggesting that this peak in the heat flow is not related to a phase transformation (Fig. 4e). Hence, we infer that the second-order phase transformation is suppressed in Cu₂CdSnS₄, in accordance with the theoretical predictions.

### 3.3.3. Implications of the order-disorder phase transition on Cu-Zn and Cu-Cd disorder

Regardless of the technique used for deposition of Cu, Zn, Sn, and S/Se on Mo-glass substrates, the absorbers for kesterite are annealed at a high temperature \( \sim 770–900 \) K in an S/Se atmosphere. At these temperatures, there is a significant disorder present in Cu₂ZnSn(S,Se)₄. As the sample is cooled to room temperature, an order-disorder transformation takes place, as shown in Fig. 4b. If the transformation is not continuous – that is, the transformation occurs below a certain critical temperature – as in the case of Cu₂ZnSnS₄, then there needs to be a nucleation and growth process for the new phase. However, if the transformation is continuous – that is, the transformation occurs continuously as temperature is decreased – as in the case of Cu₂CdSnS₄, the absorbers for kesterite are more likely to be single crystalline.
Cu2CdSnS4, then the need for the potentially kinetically slower nucleation and growth step is circumvented.

In the case of continuous transformation, the nucleation and growth steps are replaced by a continuous cation redistribution step, in which it is easier for kinetic reasons to equilibrate the system. Moreover, the ordering for Cu2CdSnS4 begins at high temperatures (that is, close to the sulfurization temperature of about 850 K) as soon as the sample is slowly cooled after sulfurization, while the ordering in Cu2ZnSnS4 only begins at low temperatures (that is, below the order-disorder transformation temperature of 530 K). Hence, the thermal energy available for the ordering process in Cu2CdSnS4 is larger than that available for Cu2ZnSnS4. Hence, under the same cooling rates, Cu2CdSnS4 would be more ordered than Cu2ZnSnS4.

We qualitatively explore the effect of structural disorder on optoelectronic properties of Cu2ZnSnS4 and Cu2CdSnS4 by comparing the photoluminescence characteristics. It has recently been suggested that Cu2Zn + ZnCu disorder does not directly affect the optoelectronic performance and can indirectly affect the more deleterious defects such as the 2CuZn + SnZn, which can degrade the photovoltaic performance. We hypothesize that the suppression of CuCd due to the reduced CuCd + CdCu disorder in Cu2CdSnS4 as compared to Cu2Zn + ZnCu in Cu2ZnSnS4 could lead to enhanced suppression of CuCd containing defect clusters such as 2CuCd + SnCd compared to 2CuZn + SnZn. Since these defects are expected to affect the optoelectronic properties, we propose that the enhanced photoluminescence yield for Cu2CdSnS4 as compared to Cu2ZnSnS4 is partly due to the reduced 2CuZn/Cd + SnZn/Cd defects, which in turn are suppressed due to the reduced CuCd + CdCu compared to CuZn + ZnCu. However, it must be noted that Cd substitution itself directly affects the formation energy of 2CuZn/Cd + SnZn/Cd, and suppresses these defects by increasing the formation energy of 2CuCd + SnCd in Cu2CdSnS4 compared to 2CuZn + SnZn in Cu2ZnSnS4. Hence, the increased photoluminescence yield of Cu2CdSnS4 compared to Cu2ZnSnS4 could be a result of both the direct (Cd increasing the formation energy of 2CuCd + SnCd) and indirect (Cd reducing the CuCd disorder, thereby suppressing other CuCd containing deleterious defects such as 2CuCd + SnCd) mechanisms.

We note that the distinct behaviour of cation disorder in Cu2CdSnS4 in comparison to Cu2ZnSnS4 is not expected from earlier DFT calculations, which showed that the CuCd + CdCu antisite pair has the same formation energy, and hence the same thermodynamic equilibrium concentration, as the CuZn + ZnCu pair in Cu2ZnSnS4. Our MC simulations indicate that such studies based on diluted point-defect models may have difficulty in extrapolating to the correct description of cation disorder, in particular when phase transitions become relevant. In typical defect calculations, the isolated point defects were placed in an ordered system corresponding to the lowest-energy structure, thereby discarding collective interactions among defects and phase change. In the present case, the higher cation ordering of Cu2CdSnS4 is indeed driven by the absence of phase transition.

The study of order-disorder transformation using first-principles calculations and differential scanning calorimetry presented here can be used as a general strategy to predict the relative degree of disorder in complex multi-element compounds, and the XRD simulations combined with experimental data can be used to study the detailed structural characteristics without resorting to resource-intensive techniques such as neutron diffraction. Hence, the methods used in this report can be applied to screen emerging materials for applications where defects and disorder limit the desired optimal performance.

4. Conclusions

We studied structural effects of the substitution of Zn with Cd in Cu2ZnSnS4 to reveal a two-way cation redistribution as Cd is incorporated into the Cu2ZnSnS4 structure. We found that the intuitively expected substitution of Zn positions with Cd does not occur, but rather, Cd occupies the 2a sites – which are occupied by Cu in Cu2ZnSnS4 – and Cu from these sites replace Zn at 2d sites. Hence, the explanation hitherto used for the structural reasons that lead to improved performance of Cu2(Zn,Cd)SnS4 as compared to Cu2ZnSnS4 – that is, the increase in the formation energy of deleterious point defects due to the substitution of Zn with a larger Cd cation – need to be reinterpreted. Using MC simulations, we showed that Cu2CdSnS4 has more structural order compared to Cu2ZnSnS4, and that this can lead to improved optoelectronic properties even if the thermodynamic formation energy calculated using diluted point-defect models for disorder-inducing defects for Cu2CdSnS4 and Cu2ZnSnS4 is the same. We further proposed how this difference in structure affects the photoluminescence characteristics. Hence, we showed how the structural properties are correlated to the optoelectronic properties in this system, and we believe that the methods employed here can be used study other complex semiconductors.

Conflicts of interest

There are no conflicts to declare.

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