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Comparison of fluctuating potentials and donor-acceptor pair transitions in a Cu-poor Cu_2ZnSnS_4 based solar cell

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The structure of the electronic energy levels of a single phase Cu_2ZnSnS_4 film, as confirmed by Raman Scattering and x-ray diffraction, is investigated through a dependence on the excitation power of the photoluminescence (PL). The behavior of the observed asymmetric band, with a peak energy at $\sim$1.22 eV, is compared with two theoretical models: (i) fluctuating potentials and (ii) donor-acceptor pair transitions. It is shown that the radiative recombination channels in the Cu-poor film are strongly influenced by tail states in the bandgap as a consequence of a heavy doping and compensation levels. The contribution of the PL for the evaluation of secondary phases is also highlighted. © *2014 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4899057]

The quaternary compound Cu_2ZnSnS_4 (CZTS) has been presented as a promising absorber layer in the thin film photovoltaics technology.[1–3] The power conversion efficiency of CZTS based solar cells has increased from 0.66% in 1996,[4] to the current record value of 9.2%,[2] which is far behind the records for Cu(In,Ga)Se_2 (CIGS) and polycrystalline silicon.[5,6] The higher efficiency was reported for devices where the CZTS layer is prepared with a Cu-poor and Zn-rich composition.[7] In these conditions, it is well known that the fluctuating potentials critically affect the CZTS structure of the electronic energy levels,[8–13] which is detrimental for the solar cells efficiency.[14,15]

Photoluminescence (PL) is a well suited technique for the investigation of radiative and non-radiative channels in semiconductors and heterostructures.[8,16–19] A few different models have been considered for the assignment of the luminescence in CZTS: free-to-bound transition,[20] donor-acceptor pair (DAP) recombination,[21–23] quasi-DAP (QDAP) recombination,[24] and radiative channels involving fluctuating potentials.[8–13,25,26] Therefore, more studies are needed to fully understand the electronic properties of CZTS. In this work, we present a PL study for a Cu-poor CZTS thin film, grown by sulphurization of a structure of precursor layers deposited by sputtering/evaporation. The observed asymmetric band is well described by the electrostatic fluctuating potentials model as we will show.

The studied solar cell is based on a CZTS thin film deposited on a Mo-coated soda lime glass substrate and prepared on a conventional 2-step approach: (i) sputtering/evaporation of precursor layers and (ii) rapid thermal processing.[3,27] Energy dispersive X-ray spectroscopy operated at an acceleration voltage of 25 kV was performed and metallic atomic ratios of $[Cu]/([Zn]+[Sn]) = 0.81$ and $[Zn]/[Sn] = 0.99$ were obtained, which are close to the ones of champion cells. The cross-section of the solar cell (see Fig. 1) reveals a compact film and window layers conformal over the CZTS layer.

X-ray diffraction (XRD) measurements (configuration $\theta - 2\theta$), using the Cu-K_α line, were made to evaluate the structural properties of the samples (see Fig. 2(a)). Several diffraction peaks are seen, including the ones from the Mo layer (40.3° and 73.4°) and from the window layer (SnO_2 at 26.2°). The main peaks of CZTS ((112), (204), and (312) reflections) are present, but its identification is confirmed by the observation of the unique tetragonal peaks at 16.1°, 18.0°, 29.5°, 33.7°, and 69.1°.[8] With the XRD data, we estimated the lattice parameters to be $a = 5.454$ Å and $c = 10.95$ Å, which allows us to calculate the ratio $c/2a = 1.004$, showing that the cell is tegragonal. It is well known that some peaks, mostly the main ones ((112), (204), and (312) reflections) of the CZTS structure might be superimposed with other phases like cubic-Cu_2SnS_3 (CTS) and cubic-ZnS. Furthermore, we

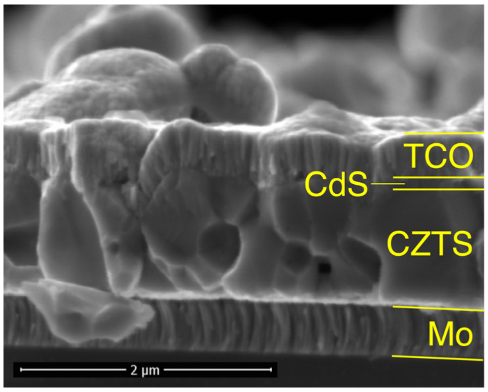

FIG. 1. Scanning electron microscopy image of the cross-section of the CZTS solar cell showing the Mo, CZTS, CdS, and transparent conductive oxide (TCO) layers.

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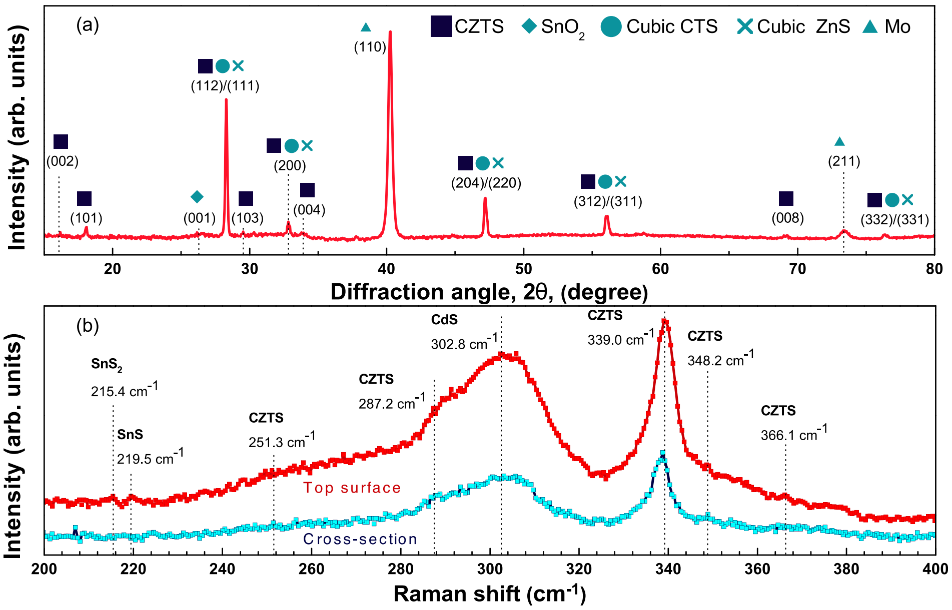

FIG. 2. (a) XRD diffractogram and (b) Raman spectra measured on the top surface, and on a cross-section configuration, of the solar cell.

also performed Raman spectroscopy (RS) using a LabRaman Horiba 800 with the 532 nm laser line, to evaluate the presence of secondary phases. In order to overcome the limited depth penetration of the laser beam, we performed top surface and cross-sectional measurements (see Fig. 2(b)). No significant differences are observed between the top and the cross section measurements. In both cases, we can observe the CZTS vibration modes[8] at 251.3, 287.2, 339.0, 348.2, and 366.1 cm^{-1}. The peak at 302.8 cm^{-1} corresponds to CdS, since the measurements were performed in the full device. No evidence is found for the presence of CTS and ZnS secondary phases in the scrutinized layer. The only small evidence of secondary phases are the two peaks at 215.4 and 219.5 cm^{-1}, which, if were stronger, could be related to SnS$_2$ and SnS phases, respectively. However, due to its very low relative intensities and the fact that also no evidences were seen in XRD, we conclude that the amount of possible secondary phases in our absorber layer is at most residual.

PL was performed using a Bruker IFS 66v Fourier Transform Infrared spectrometer, equipped with a liquid nitrogen cooled Ge detector, and a helium flux cryostat. The excitation source was the 514.5 nm line of an Ar$^+$ laser (spot diameter of ∼1 mm). PL spectra taken at 7 K and with an excitation power values in the range 0.6–100 mW are shown in Fig. 3. The observed emission corresponds to a broad and asymmetric band. For an excitation power of 100 mW, the peak energy and the full width at half maximum (FWHM) are 1.22 eV and 0.16 eV, respectively. On the low energy side of the emission, an approximate exponential decrease is observed, whereas on the high energy side, the intensity decrease is more abrupt. This band shows a deviation of 0.28 eV from the assumed bandgap energy (∼1.5 eV) for CZTS. The characteristics of the emission are close to the ones described in literature by different authors.[8,13,20,26–28]

As the excitation power decreases, a red shift of the emission and a slight increase of the FWHM values occur. This effect takes place with no significant change of the shape of the band. In order to analyze the power dependence, all of the PL spectra were fitted with two Gaussian components. In Fig. 4(a), it shows the dependence of the peak energy (E) of the band on the excitation power (P). In the literature,[29,30] this dependence is often described by the following relation:

$$E = \beta \ln(P/P_0), \qquad (1)$$

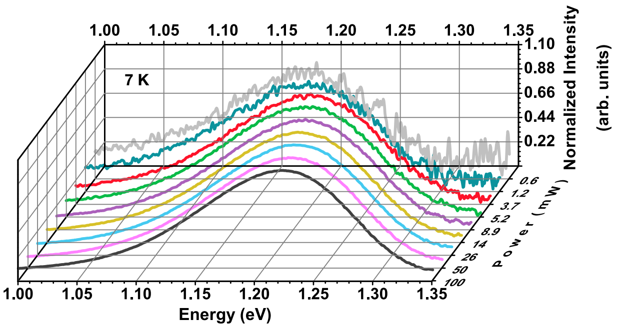

FIG. 3. Photoluminescence spectra measured at 7 K of a CZTS-based solar cell for different values of the excitation power. The values of the PL intensity are normalized to the maximum value in each spectrum.

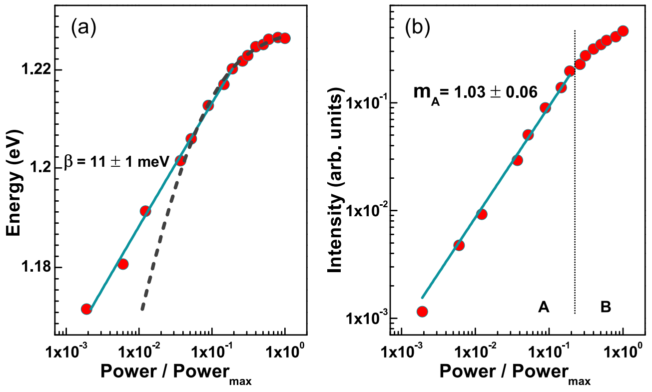

FIG. 4. Dependence on the excitation power of (a) peak energy and (b) PL integrated intensity of the broad and asymmetric band. The excitation power values were normalized to the maximum one (100 mW). The solid lines in (a) and (b) represent the fits of Eqs. (1) and (3), respectively, to the experimental points. The dashed line in (a) corresponds to the fit of Eq. (2) to the experimental points. A and B in (b) identifies the low and high excitation power regimes, respectively.

where β characterizes the energy shift and P_0 is a fitting parameter. However, Eq. (1) can only describe the experimental points that, for the log representation in Fig. 4(a), are in the linear regime observed for the lower excitation power values range. In that regime, a value of $\beta = 11 \pm 1\,\text{meV}$ was obtained. β was not previously estimated for CZTS, and the obtained value is similar to reported ones for CIGS.[30]

One of the models considered for the discussion of the nature of the radiative transitions in CZTS considers DAP transitions. Close pairs give origin to sharp lines, whereas for sufficiently distant pairs, a broad band is observed.[16] For the latter, Zacks and Halperin[31] proposed an analytical expression for the relation of the excitation power with the peak energy of the observed band

$$\mathrm{P} \propto \frac{(\mathrm{E}-\mathrm{E}_\infty)^3}{\mathrm{E_B}+2\mathrm{E}_\infty-2\mathrm{E}}\exp\left(-\frac{2\mathrm{E_B}}{\mathrm{E}-\mathrm{E}_\infty}\right), \tag{2}$$

where $E_\infty = E_g - E_D - E_A$ is the limiting energy of infinity distant pairs, E_g is the bandgap energy value of the semiconductor, E_D and E_A are the ionization energies of the donor and acceptor, respectively, $E_B = e^2/\epsilon R_B$, and R_B is the Bohr radius. As is shown by the gray dashed line in Fig. 4(a), Eq. (2) is unable to describe the experimental data in the lower excitation power regime. Even if we consider the fit shown in Fig. 4(a), for which the value of $E_g = 1.5\,\text{eV}$ was considered, an estimated value of $E_D - E_A = 2.35\,\text{eV}$ was achieved. This value has no physical meaning for CZTS. Additionally, no sharp lines are observed in the high energy side of the broad band, as expected for polycrystalline materials. Thus, the DAP model cannot explain the observed luminescence.

The dependence of the PL intensity (I) on the excitation power (see Fig. 4(b)) can be parameterized by[32]

$$\mathrm{I} \propto \mathrm{P}^{\mathrm{m}}, \tag{3}$$

where m is an adjustable parameter. The data evidence a diminishing of the slope as the excitation power increases. This behavior is commonly observed when more than two orders of magnitude are covered.[32] The deviation to Eq. (3) can be interpreted as a progressive saturation of the energy levels involved in the radiative recombination. The estimated value for the low (A) excitation power regime is $m_A = 1.03 \pm 0.02$. Usually, values of $m > 1$ are interpreted as related with excitonic radiative recombination, whereas values of $m < 1$ are related with radiative transitions involving defects. In our case, the estimated value of m_A is in the transition between both regimes. Thus, it is not straightforward the conclusion regarding the possible localization of the charge carriers.

Previously, the possible presence of four secondary phases was discussed: CTS, ZnS, SnS, and SnS_2. From these phases, CTS and SnS have bandgap values in the range (~0.96–1.07 eV),[33,34] which is lower than the value of CZTS. Thus, the expected related emission could be present in the low energy side of the observed asymmetric band. However, we should note that for the energy range 0.8–1.1 eV, the detectivity of the Ge detector is very high and no additional component is observed in our film in that range (see Fig. 3). Actually, if the observed emission is composed of more than one component, with origin in different phases, it is expected a different dependence on the increase of the excitation power, which will result in a modification of the shape of the whole emission. This effect is not observed for our film. Concerning the other two possible secondary phases, the bandgap is much higher than the one of CZTS and no influence on the measured luminescence is expected. Therefore, PL supports the conclusion from XRD and RS measurements that the possible presence of those phases must be residual.[35]

In the literature, two models used to discuss the emission in CZTS are (i) DAP transitions[21–23] and (ii) band tail states created by electrostatic fluctuating potentials.[8–13,25,26] The appearance of these types of radiative transitions depends mainly on the doping level of the material as well as the degree of compensation.[36] For DAP transitions, the doping level must be low and thus the interaction between defects is not significant. Therefore, the charge carriers are located in discrete levels inside the bandgap. On a second case, for highly doped and compensated semiconductors, a large concentration of defects, where most of them are charged, exists. The interaction of these randomly distributed defects results in electrostatics fluctuating potentials along the film, resulting in the appearance of tails states in the bandgap. The conduction and valence bands follow the same electrostatic fluctuating potentials which results in no bandgap energy variations throughout the film. These fluctuations influence strongly the structure of the electronic energy levels with severe consequences on the radiative transitions observed for the semiconductor.[36] The study of the dependence on excitation power of PL contributes to the distinction between transitions involving DAP and fluctuating potentials.[10] In the case of DAP transitions, it is predicted that, as the excitation power increases, distant pairs become saturated and progressively closer ones will be populated, which will cause a blue shift of the emission according to Eq. (2).[16,31] The estimated values of β (see Eq. (1)) are up to a few meV.[37–39] On the other hand, the fluctuating potentials model assumes the existence of tail states with different depth in the bandgap. The deeper ones are separated by longer distances and are statistically less probable. For a lower excitation power regime, populating these deeper wells is favored. The increase of the excitation power will promote populating more shallow tails states, which has a higher density. This change from deeper to shallower tail states is reflected on a blue shift of the emission. The values of β are higher than the ones estimated for DAP transitions. Also, an increase of β is expected as the doping level and the degree of compensation increases.[29,37] The estimated value of β, 11 meV, is higher than the common values found for DAP transitions reported for other semiconductors.[29,37,40] Also, Eq. (2) is unable to describe the whole set of data (see Fig. 4(a)). Furthermore, the shape of the band is clearly asymmetric, being described by a high FWHM (0.16 eV). Thus, this behavior cannot be ascribed to DAP transitions, but is typical of radiative transitions involving electrostatic fluctuating potentials.

Regarding the dependence of integrated intensity on excitation power, the obtained slope ($m_A = 1.03 \pm 0.02$) is a transition value between the regimes from localized to non-localized charge carriers.[8,32] The existence of electrostatic fluctuating potentials along the film with the resulting formation of tail states in the bandgap will allow some degree of localization of the charge carriers, compatible with the estimated value of m. The observed behavior of the emission supports that the radiative transitions in Cu-poor CZTS are influenced by electrostatic fluctuating potentials.

In summary, we have shown that PL gives strong contributions to both structural identification and understanding of the electronic properties of CZTS. In the first case, the PL results allow us to infer that there are no evidences of secondary phases as supported by XRD and RS. Concerning the radiative channels, we performed a deep investigation of the dependence of the PL on the excitation power and compared our results with two theoretical models: DAP transitions and fluctuating potentials. The DAP model assumes a power dependence of the energy with β values up to few units, whereas for the fluctuating potentials, the same dependence should give β values of the order of 10 meV or even higher. The asymmetric shape of the band, a large blue shift ($\beta = 11 \pm 1\,\text{meV}$), and a m parameter ($m_A = 1.03 \pm 0.02$) close to 1 are compatible with a highly doped and compensated film, for which the radiative transitions are influenced by the occurrence of fluctuating potentials. Clearly, our results are not in accordance with the DAP model.

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Equation (2) in our original manuscript[1] was published incorrectly. Equation (2) should be read as[2]

$$\mathrm{P} \propto \frac{(\mathrm{E}-\mathrm{E}_\infty)^3}{\mathrm{E_B}+\mathrm{E}_\infty-2\mathrm{E}}\exp\left(-\frac{2(\mathrm{E_B}-\mathrm{E}_\infty)}{\mathrm{E}-\mathrm{E}_\infty}\right), \qquad (1)$$

where P is the excitation power, E is the peak energy, $E_\infty = E_g - E_D - E_A$, $E_B = E_\infty + \frac{e^2}{4\pi\epsilon R_B}$, ϵ is the permittivity of the semiconductor, and R_B is the Bohr radius. Since the mistake only happened on the document, the fit of this equation to the experimental points shown in Fig. 4(a) was done with the right form of the equation. Thus, this correction does not affect the conclusions.

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