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Time resolved photoluminescence on Cu(In, Ga)Se₂ absorbers: Distinguishing degradation and trap states

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Recent reports have suggested that the long decay times in time resolved photoluminescence (TRPL), often measured in Cu(In, Ga)Se₂ absorbers, may be a result of detrapping from subbandgap defects. In this work, we show via temperature dependent measurements, that long lifetimes >50 ns can be observed that reflect the true minority carrier lifetime not related to deep trapping. Temperature dependent time resolved photoluminescence and steady state photoluminescence imaging measurements are used to analyze the effect of annealing in air and in a nitrogen atmosphere between 300 K and 350 K. We show that heating the Cu(In, Ga)Se₂ absorber in air can irreversibly decrease the TRPL decay time, likely due to a deterioration of the absorber surface. Annealing in an oxygen-free environment yields a temperature dependence of the TRPL decay times in accordance with Schockley Read Hall recombination kinetics and weakly varying capture cross sections according to T^{0.6}. *Published by AIP Publishing*. [http://dx.doi.org/10.1063/1.4977707]

Improving thin film solar cells relies on the accurate knowledge of the physical properties of the absorber and functional layers. Among others, the minority carrier lifetime is a very important parameter since it is governed by various radiative and nonradiative loss channels of the photoexcited carriers, which determine the open-circuit voltage of finished solar cells. Time resolved photoluminescence (TRPL) is a well-known technique to measure the carrier lifetimes, and the theoretical description for high quality III-V semiconductors is well established. In contrast, the description of TRPL data measured on absorbers used in thin film solar cells is still not fully understood. The material quality is inferior to that of III–V semiconductors and non-idealities arising from defects, interface recombination, internal electric fields, compositional gradients, and grain boundaries make a precise determination of the true minority carrier lifetime difficult.

Despite the fact that the measured TRPL transients can often not be described by a single exponential decay for reasons already mentioned; it has been shown by numerous studies in different material systems, such as CdTe^{2,3} and CIGSe^{4,5} that there is a correlation between the minority carrier lifetime and the open-circuit voltage of the devices. Moreover, it has been shown that, depending on the excitation wavelength and the interface quality, TRPL measurements are prone to measure too low lifetimes since interface recombination influences the decay curve. Finally it is also quite well established that, in order to get representative data, the absorbers have to be measured directly after growth and that the amount of surface oxidation has to be kept as low as possible. This will be of central importance throughout this manuscript, and we will show that degradation has to be prevented before other effects, such as minority carrier

Recently, Maiberg and coworkers published a detailed analysis of the TRPL technique using a combination of modelling, analytical calculations, and experiment.^{8–10} A large number of situations were analysed to explain the origin of non mono-exponential decay curves in TRPL. In particular, the influence of minority carrier trapping has been discussed.⁹ Considering the Shockley Read Hall (SRH) recombination for a p-type semiconductor with a donor-like trap state close to the conduction band 1,11,12 trapping becomes important when the trap capture times of electrons are smaller than the effective recombination time and the re-emission time to the conduction bands is large. In this case, the electron is trapped for a long time followed by re-emission to the conduction band where either another trapping process or recombination takes place. The overall effect is that the recombination is retarded and a long decay time is observed in the TRPL measurement despite the fact that the real minority recombination time is significantly smaller. From a practical point of view, this situation can be very misleading since TRPL is often used for process optimization of solar cells, and minority carrier trapping violates the correlation of lifetime and open-circuit voltage. The influence of trapping in the TRPL measurements can be discerned by temperature-dependent measurements. 9,10 Although Maiberg et al. measured a decay time of 300 ns at room temperature, this decay time decreased to 80 ns after heating to 330 K, and complementary simulations suggested the SRH lifetime to be below 10 ns for their sample. It is an important question whether detrapping is a general cause of the observed long lifetimes in CIGSe or whether the results reported by Maiberg et al. are to be correlated with the specific growth parameters used for the CIGSe samples that are studied.

trapping (which will be discussed in the next paragraph), can be analyzed.

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In order to investigate this issue, we have carried out similar temperature-dependent TRPL measurements on CIGSe absorber layers, however, with varied environmental measurement conditions. Identical samples were measured in air and in a nitrogen atmosphere. Moreover, we compare the TRPL results with photoluminescence imaging data. By this, we are able to show that trapping is not responsible for long lifetimes in our CIGSe samples.

The experiments have been carried out on CIGSe samples prepared by multi stage coevaporation. Details of the experimental growth conditions can be found in Reference 13 and in the supplementary information (IV, QE, CV, and Quasi-Fermi level splitting determination are also in the supplementary information). After growth, the sample was broken into small pieces, which were stored in a glove box to prevent severe oxidation. Each experiment was then performed on a new piece of absorber, freshly removed from the glove box. This allowed us to investigate a large number of different measurement conditions on the same sample from the same growth run, which is a prerequisite for a systematic study. We will see that even with careful handling/ storage, an initial degradation of the fresh absorber is difficult to be avoided. The TRPL measurements are performed in a custom setup which uses a pulsed 660 nm laser source and time-correlated single photon counting with a InGaAs photomultiplier. The time resolution of the system is approximately 400 ps. The excitation spot size in all experiments described below is approximately 40 micrometers in diameter. In this study, the photon density per pulse has been set between $\approx 2.6 \times 10^{11}$ and 5.3×10^{11} photons/cm². This translates to a maximum generated carrier density in the CIGSe sample of $\approx 4-8 \times 10^{15} \text{ cm}^{-3}$ if an absorption coefficient of $1.6 \times 10^4 \text{ cm}^{-1}$ (@ 660 nm) is assumed. For the temperature dependent TRPL measurements, the sample was placed on a small ceramic hotplate in air. The sample temperature is measured using a small Pt100 temperature sensor placed on top of a small dummy absorber layer. After each heating step, the sample temperature is kept constant for five minutes followed by the TRPL measurement. In addition, measurements were performed in a cryostat filled with N₂ gas. PL imaging was carried out in a custom system described elsewhere, ¹⁴ where the same ceramic hotplate was used for the temperature dependent measurements. The system has been calibrated to absolute photon numbers in order to be able to compare the lifetime measurements with the radiative recombination rate. More details including the quasi-Fermi level splitting measurements of the absorber can be found in the supplementary information.

Figure 1 depicts a CIGSe sample measured under different environmental conditions. In all cases, the sample has first been measured at room temperature followed by gradual heating up to approximately 350 K. Afterwards, the cooldown has been performed in the same temperature steps in order to study the reversibility of the observed processes. We analyze the background corrected data by fitting a biexponential model to the TRPL transients as defined in Equation (1) where $A_{1,2}$ are the amplitudes, and $\tau_{1,2}$ the lifetime values.

$$I(t) = A_1 \times \exp(-t/\tau_1) + A_2 \times \exp(-t/\tau_2).$$
 (1)

In the present case, $\tau_{1,2}$ exhibit the same temperature dependence (see supplementary information) and consequently we decided to use a weighted average value τ for the lifetime as defined in Equation (2)

$$\tau = \frac{A_1 \tau_1 + A_2 \tau_2}{A_1 + A_2}.\tag{2}$$

The TRPL spectra of the absorber measured in air are depicted in Figure 1(a) together with the deduced lifetimes presented in Figure 1(c). The stars in Figure 1(c) indicate the measurements during heating up whereas the dots depict the

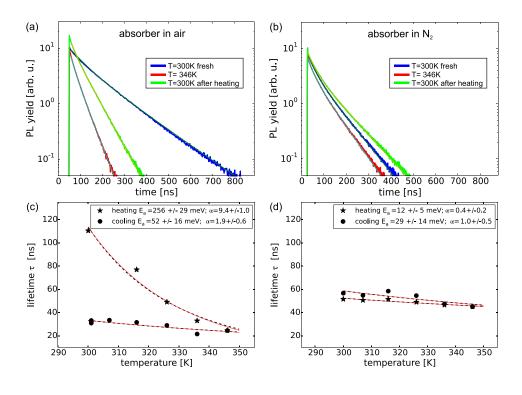


FIG. 1. Temperature dependence of the CIGSe lifetime measured in (a) ambient conditions and (b) in a nitrogen atmosphere. The three extreme cases of the TRPL transients (room temperature before and after the temperature sweep and the transient at the highest investigated temperature of $T = 346 \,\mathrm{K}$) are depicted. (c) and (d) Extracted lifetimes according to Equations (1) and (2) for the complete temperature sweep. The values in (c) correspond to the sample measured in air whereas the values in (d) correspond to the measurements in N2. The fits in (c) and (d) have been with Equation (3) assuming either a capture cross section $\sigma(T) \propto T^{\alpha}$ or σ according to Eq. (4).

lifetimes during cool down. It can be clearly seen that the decay time is severely affected by the heating in air since we see a clear decrease in τ that does not recover after reaching room temperature again. This indicates that the absorber changes with heating in air thus leading to a permanent reduction of the lifetime.

In contrast, the TRPL results of the same absorber measured in N_2 [Figures 1(b) and 1(d)] are very different. We do not see a significant decrease of the lifetime, and the temperature dependence of τ is very weak and for the most part reversible. The values before and after the heat treatment change by less than 10%. In this case, we even observe a small increase of almost 10% of the lifetime after the heating cycle. Consequently, we can rule out the severe degradation effects here.

The data also reveal that the bare absorber measured in air exhibits a higher initial lifetime (110 ns) compared to the measurements performed in nitrogen (\approx 50 ns). This can be explained by the fact that not all measurements could be performed simultaneously. The measurements on the bare absorber in air were executed first. Although, for subsequent measurements, the samples were kept in a glovebox some degradation in the initial lifetime was always observed for these later performed characterizations. This is a well known phenomena in CIGSe. As soon as the absorber is in air, there is some degradation (see for example, Reference 15).

From our data, we can conclude that temperature dependent measurements in air will be strongly influenced by degradation. However, the question remains whether the observed temperature dependence measured in N_2 can still arise from a de-trapping process or whether the observed dependence is compatible with Schockley Read Hall recombination kinetics. In the SRH model, the lifetime can be expressed as

$$\tau(T) = \frac{1}{v_{th}(T)N_d\sigma_d(T)},\tag{3}$$

where the thermal velocity is given by v_{th} , N_d is the density of deep traps, and σ_d the capture cross section. The thermal velocity is proportional to \sqrt{T} whereas the temperature dependence of the capture cross section is somewhat unclear. A temperature dependence of the lifetime of $T^{-0.5}$ has been measured in InAs/GaSb superlattices ¹⁶ which translate into a temperature independent σ_d whereas a temperature dependence of $\sigma_d \propto T^{-0.8}$ has been found for $\text{Cu}_2\text{ZnSnS}_4$ at low temperatures. ¹⁷ Maiberg et~al. assumed a T^1 dependence for σ_d in Reference 9 and a temperature dependence of $T^{4.5}$ in Reference 10 for CIGSe, the justification of which is not clear to us. In previous works, ¹⁸ a thermally activated capture cross section, as denoted in Equation (4) has been used to describe the multi phonon emission (MPE) of deep defects in GaAs and InP.

$$\sigma_d = \sigma_\infty \exp\left(\frac{-E_a}{k_B T}\right). \tag{4}$$

A more complex model has been discussed by Rosenwaks¹⁹ taking into account, MPE in conjunction with tunneling processes at low temperatures, which simplifies to an exponential dependence similar to Equation (4) at higher temperatures ($E_a \ge k_B T$). We will therefore analyse our measurements either

assuming $\sigma_d \propto T^\alpha$ where α is the fit parameter or by assuming an exponential dependence according to Equation (4) where the activation energy E_a is the fit parameter. In Figures 1(c) and 1(d), the average lifetime values are fitted with an exponential dependence (dashed line) as in Equation (4) and with a T^α behaviour (red solid line). It becomes immediately clear that from our experimental data, we are unable to distinguish between the two different temperature dependencies. However, for the measurement in nitrogen, the deduced exponent α is between 0.4 and 1.0 which translates into an activation energy below 30 meV. If we neglect the small differences between heating up and cooling down and perform a weighted average, we end up with an activation energy of 16 meV or an exponent $\alpha=0.6$.

These results are in contrast to the measurements in air where the activation energy is much larger ($E_a = 256 \, \text{meV}$). However, as discussed above, this is a direct consequence of degradation and should not be considered as a property of the CIGSe absorber. We would like to stress that the observed temperature dependence of σ_d in N_2 is consistent with the SRH recombination, and stands in contrast to the assumption of $\sigma_d \propto T^{4.5}$ as in Reference 10. In our case, the deduced activation energies of σ_d are, in average, smaller than the thermal energy which rules out detrapping as the dominant process determining the decay curves.

In order to gain further insights into the mechanism of degradation in air, we performed the temperature dependent photoluminescence measurements in conjunction with TRPL. This is relevant for solar cell devices since they operate at AM1.5 equivalent continuous illumination and not under pulsed illumination as in the case of TRPL.

The radiative recombination rate R is given by

$$R(t) = B \times \left[(p_0 + \Delta p(t)) \cdot (n_0 + \Delta n(t)) - n_i^2 \right]. \tag{5}$$

The hole (electron) concentration in equilibrium is denoted as $p_0(n_0)$ whereas the optically generated carriers are denoted as $\Delta p(t)$ and $\Delta n(t)$ for holes and electrons, B is the radiative recombination coefficient, and n_i is the intrinsic carrier concentration. For low injection conditions in p-type semiconductor (which is the correct description for AM1.5 illumination) Equation (5) can be approximated by

$$R(t) = Bp_0 \Delta n(t). \tag{6}$$

We assume an exponential decay of the minority carriers given as $\Delta n(t) = \Delta n_0 \cdot \exp(\frac{-t}{\tau})$ where the total lifetime τ is given by the sum of the inverse radiative and non-radiative lifetimes¹

$$\frac{1}{\tau} = \frac{1}{\tau_R} + \frac{1}{\tau_{nR}}. (7)$$

Using the expression for $\Delta n(t)$ and further integrating leads to the final expression for the (internal) PL yield

$$Y = Bp_0 \Delta n_0 \tau. \tag{8}$$

From Equation (8) it becomes clear that, if the injection stays constant, variations in the PL yield are either linked to changes in lifetime τ or in the equilibrium hole concentration.

In order to compare TRPL and PL in a meaningful way, the lifetime was measured on a fresh part of the sample in air in order to get the initial state of the absorber. We measure an average lifetime of approximately 49 ns. The sample is subsequently transferred to the PL imaging system (in ambient air) which is equipped with a heating stage. The temperature is varied between room temperature and 360 K and spectrally and laterally resolved absolute photon numbers are measured. Afterwards, the sample is cooled down using the same temperature steps. After the PL imaging measurements, the sample is immediately transferred to the TRPL setup where the lifetime is measured a second time in air.

The results of this measurement are presented in Figure 2. The PL yield as a function of the sample temperature is shown in Figure 2(a), and the inset shows a photoluminescence image of the investigated absorber measured at RT. From the image, it becomes immediately clear that the PL yield exhibits some lateral variations which we attribute to non-uniformities in the material quality. However, the changes in intensity are uniform despite these inhomogeneities (see supplementary material). The averaged PL yield is approximately 2×10^{18} photons/(m² s), which corresponds with an external PL efficiency of approximately 3.3×10^{-4} and a quasi-Fermi level splitting of 655 meV (see supplementary material). This is in accordance with the assumption that the dominant recombination process in our absorber is non-radiative. If we assume a radiative recombination coefficient B of $6 \times 10^{-11} \text{ cm}^{-3} \text{ s}^{-1}$ (Ref. 20) and a net doping of 5×10^{15} cm⁻³ (measured on one of the solar cell by CV profiling shown in the supplementary information) the radiative lifetime is 3.3 μ s. Using the formulas presented in Reference 21 and the measured external photoluminescence efficiency,

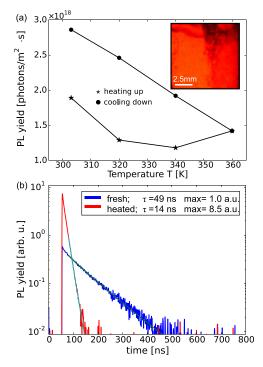


FIG. 2. (a) The average PL yield measured at different sample temperatures. The inset shows a PL image of the sample prior to heating. (b) TRPL transients for the absorber before and after the heat treatment. The inset gives the average lifetime values as deduced from Equation (1). The max. values correspond to counts right after the pulse.

we deduce a non-radiative lifetime of 51 ns. This is in good agreement with the measured lifetime value of 49 ns considering that the B value is not accurately known. The detailed calculation can be found in the supplementary material.

From the integrated PL images presented in Figure 2(a), we do observe an increase in the PL yield after heating up and completed cooling down which indicates either a larger bulk lifetime or significant changes in doping (see Eq. (8)). The TRPL measurement on the same sample, however, shows a strong decrease in the measured lifetime in air as depicted in Figure 2(b). We measure a decrease of τ by a factor of 3.5 in TRPL (Figure 2(b)), whereas the PL yield increases by a factor of 1.5(Figure 2(a)). In order for the two measurements to be consistent (c.f. Eq. (8)) p_0 needs to increase by approximately a factor of 5. From Equation (6) follows that for t = 0, the measured yield in TRPL is equal to $R(t=0) = B \cdot p_0 \cdot \Delta n_0$. This means that changes in the transient right after the pulse can also be linked to variations in p_0 . In TRPL, we measure roughly a factor of 8 increase in the count rates right after the pulse (Figure 2(b)). This is roughly consistent with the PL imaging data which gave a factor 5 as discussed in the preceding paragraph. The difference can be explained by the fact that the analyzed areas are different. The TRPL is measured on an area of 40 µm diameter whereas the PL imaging has been measured on 1 cm². Indeed we can get an estimate of the total yield in TRPL if we integrate all counts. The ratios between the two measurements are 2.3. In order for the data to be consistent with Eq. (8), p₀ has to increase by a factor of 8 in order to account for a difference in lifetime by a factor of 3.5. The factor of 8 is exactly what we measure in TRPL right after the pulse as indicated in the inset of Figure 2(b).

The fundamental reason for the change of p_0 is not clear at present. At this point, we can only speculate, and more work is necessary in order to fully understand the consequences of these measurements. The most plausible explanation is to attribute the measured changes in PL and TRPL to an enrichment of Na at the surface due to the air anneal. Na is known to increase the p-type carrier concentration which is consistent with the data. It has been shown that the Na segregation at the front surface is triggered by oxygen (during TCO formation²²). Oxygen itself might also play a role in the observed process. Early work on CuInSe₂ suggests that oxygen increases the p-type conductivity via removal of compensating donors.²³

The most important outcome of this part of the work is that intensity changes in TRPL need to be taken into account since they reflect changes in doping which will change the photoluminescence measurements. These changes already occur at slightly elevated temperatures in air. The excellent agreement of TRPL and PL imaging (measured close to AM1.5 intensity) corroborates the conclusion that we do measure lifetimes and not detrapping times. Moreover, the experiments corroborate that the degradation effect is independent of the initial state of the absorber (110 nm in Figure 1 vs. 49 ns in Figure 2).

In summary, we have shown that the CIGSe absorbers investigated in this study do have long minority carrier lifetimes which are not influenced by trapping/detrapping processes. We highly suggest that temperature dependent measurements (300–350 K) are carried out in nitrogen (or

with an additional passivation layer) in order to reduce the degradation effects. From our measurements, we find a capture cross section which is weakly temperature dependent with an exponent of $\alpha=0.6$. Finally, we have shown that the reduction of the lifetime is accompanied with an increase in doping.

See supplementary material for solar cell results, including IV, QE, and CV are presented. Evaluation of the lifetime values (not statistically averaged) are shown.

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