Identification of Excitons and Biexcitons in Sb$_2$Se$_3$ under High Photoluminescence Excitation Density

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The near-band-edge emission of Sb$_2$Se$_3$ microcrystals is studied in detail under high photoluminescence excitation density using a pulsed UV laser ($\lambda = 266$ nm, pulse width 0.6 ns). Based on the peak energy positions and the excitation power density and temperature dependencies ($T = 3$–$110$ K) of the photoluminescence spectra, the emission is interpreted as a recombination of two pairs (A and B) of free excitons and biexcitons appearing because of valence band splitting. The magnitude of the valence band splitting due to the crystal field ($\Delta_{9} = 20–22$ meV) is estimated in the Brillouin zone center.

1. Introduction

Development of photovoltaic (PV) materials consisting of earth abundant elements is an ultimate criterion to meet the increasing demands for electricity production. The current thin-film PV technologies are mostly dominated by two favorite compounds, i.e., CdTe and Cu(In,Ga)Se$_2$ (CIGS). However, the high price of In and Ga as well as the toxicity of Cd may limit their widespread use. Alternative quaternary Cu$_2$ZnSn(S,Se)$_4$ (CZTSSe) type compounds, comprising abundant and low-cost elements like Cu, Zn, Sn, S, and Se, are also potential candidates for next-generation PV technologies. However, kesterites have turned out to be very challenging on their way toward highly efficient thin film PV due to strong recombination of photogenerated charge carriers via various routes leading to short minority carrier lifetime (i.e., magnitude lower than in CIGS, CdTe, etc.) and diffusion length and resulting in large open-circuit voltage deficit of kesterite solar cells.

It was shown that due to the very high absorption coefficient the photons with wavelengths $\lambda > 800$ nm are completely absorbed within the first 400 nm of Sb$_2$Se$_3$ film, enabling much thinner absorber layers compared to the usual thin film solar cells.

Despite extensive research there are still problems related to various point defects or electronic band structure of this compound. Capacitance spectroscopy studies have shown the presence of various deep donor and acceptor defects. Defect states in polycrystalline Sb$_2$Se$_3$ have been studied also using temperature and laser intensity-dependent photoluminescence (PL). The low-temperature ($T = 10$ K) PL spectrum was consisted of three bands at 0.94, 1.10, and 1.24 eV. The PL bands at 1.24 and 0.94 eV were found to originate from the distant and close donor–acceptor pair recombination, respectively, and the third PL band at 1.10 eV was proposed to be related to the grain boundaries. Recent theoretical calculations showed that the defect chemistry of quasi-1D Sb$_2$Se$_3$ is rather complicated compared to conventional semiconductors. In particular, it has been discussed that the identical defects located on nonequivalent atomic sites might have different properties. All kinds of defect studies are therefore extremely important as usually defects act as powerful recombination channels and reduce the performance of the solar cell.
Besides the complicated defect structure, the electronic band structure of Sb$_2$Se$_3$ is also not fully understood. There are contradictory reports regarding the nature of the band gap (direct/indirect), as well as a significant range of values for its energy. There now exists a broad consensus that the band (direct/indirect), as well as a significant range of values for the energy gap of Sb$_2$Se$_3$ at room temperature (RT) is generally considered to be indirect with a direct gap lying at $\approx$0.1 eV to higher energy.$^{[13]}$ Moreover, the temperature dependence of indirect and direct band gaps is different and therefore the energy difference between them is found to be only about 40 meV at $T = 0$ K.$^{[13]}$ At the same time, a slightly smaller difference of about 25 meV at $T = 0$ K was also reported.$^{[18]}$ In most of the studies, absorption measurements and Tauc’s plot are used to determine the bandgap energy, but it is extremely difficult to distinguish between so closely situated indirect and direct band gaps with this method. Moreover, the absorption spectrum is often disturbed by the Urbach tail.$^{[11,19]}$ Photoelectance spectroscopy (PR) is considered to be more reliable method, but unfortunately this technique probes exclusively direct optical transitions that are present at singularities of the joint optical density of states. According to PR measurements made on polycrystalline samples, the bandgap energy at $T = 0$ K was around 1.32 eV.$^{[16,18]}$ There is less information about band structure of single-crystalline Sb$_2$Se$_3$ samples. Recent optical measurements made on quasi-1D Sb$_2$Se$_3$ single crystals revealed that the absorption edge at low temperatures can shift about 10 meV depending on polarization conditions and even more at RT.$^{[20]}$ This anisotropy affects also the shape of absorption spectrum and could cause additional ambiguity to experimental results. Additionally, the valence band splitting in the center of the Brillouin zone by a crystal field and the spin–orbit interaction add extra features to the optical absorption and reflectance measurements. Unfortunately, PR measurements were not able to detect these valence band splittings, because the broadening parameter obtained from PR spectra was too high. Very recently, the presence of four different excitonic transitions was reported by using absorption and reflectance measurements at $T = 11$ K and at $T = 300$ K.$^{[20]}$ These transitions are due to the crystal field ($\Delta_c$) and spin–orbit ($\Delta_m$) interactions leading to valence band splitting in the center of the Brillouin zone center and are called as A, B, C, and D excitons. The low-temperature ground state energy positions of these excitons were measured at 1.299, 1.312, 1.347, and 1.538 eV for A, B, C, and D excitons, respectively. Due to crystal symmetry rules, A exciton is allowed in $E\parallel c$ polarization while the B exciton in $E\perp c$ polarization. It was additionally shown in Sb$_2$Se$_3$ single crystals $\Delta_c = 13$ meV and $\Delta_m = 35$ meV.$^{[20]}$ All these aforementioned results show that different methods are also needed to improve the understanding of the electronic band structure of Sb$_2$Se$_3$ crystals in more detail.

Excitons are most often studied by low-temperature PL spectroscopy, which, however, requires very high-quality crystals, where other recombination channels are suppressed. Alternatively, it is possible to dramatically increase the probability of free exciton emission by the generation of very high concentration of electron–hole pairs.$^{[21]}$ This can be done by using very high laser power. However, when continuous wave (cw) laser is used, there is a high risk of overheating the crystal before reaching the required power. Therefore, pulsed lasers with very short laser pulses must be used.$^{[22]}$ It was estimated that a pulsed laser can produce 15 times more excitons and about 150 times more biexcitons than cw excitation under the same experimental conditions.$^{[23]}$ There are also requirements for the laser wavelength. It should be as short as possible in order to increase the density of generated free excitons. In Sb$_2$Se$_3$, the absorption coefficient at $\lambda < 400$ nm is reaching $10^6$ cm$^{-1}$ and the electron–hole pairs are generated in a thin surface layer with a thickness of only a few tens of nanometers.$^{[13]}$ In the present paper, we report the first detection of excitonic emission in Sb$_2$Se$_3$ microcrystals by using pulsed UV laser and analyze the properties of detected A and B free excitons and biexcitons.

### 2. Excitons and Biexcitons

The free exciton binding energy in 3D crystals is usually calculated using a simple hydrogen model$^{[24]}

$$E_b = \frac{\mu R_y}{m_e^* m_h^*},$$

(1)

where $\mu = (m_e^* m_h^*) / (m_e^* + m_h^*)$ is the reduced mass of exciton, $m_e$ is the electron mass, and $R_y$ is the Rydberg constant. Sb$_2$Se$_3$ is a compound with quite high static permittivity, i.e., $\varepsilon_r \geq 24$ and therefore the free exciton binding energy is expected to be very small. As already mentioned, due to the crystal field splitting of valence band we expect at least the presence of A and B excitons in this material.$^{[26]}$ Effective mass of electrons in Sb$_2$Se$_3$ is reported to be $m_e = 0.67 m_0$ and the effective mass of holes in the upper valence bands A and B is $m_h = 3.32 m_0$ and $m_h^* = 3.83 m_0$, respectively.$^{[20]}$ However, different values of electron and hole effective masses were also reported: $m_e = 0.365 m_0$ and $m_h = 0.42 m_0,$$^{[27]}$ $m_e^* = 0.316 m_0$ and $m_h^* = 0.31 m_0.^{[28]}$ These values give the free exciton binding energy in the range of $E_{BA} = 4–9$ meV and the exciton radius is about 7 nm. The separation between A and B excitons in Sb$_2$Se$_3$ at low temperature is expected to be $\Delta_e = 13$ meV.$^{[26]}$ It is also known that Sb$_2$Se$_3$ is a semiconductor with a layered structure in which the Sb and Se atoms are connected with three other atoms of the opposite type, which in turn are connected within the crystal through weak secondary bonds. It was shown that the free exciton binding energy in layered crystals can be higher than in ordinary 3D crystals.$^{[29]}$

First studies of biexcitons were made using CuCl crystals, see ref. [30] and references therein. It was discovered that the properties of biexciton emission are very different from free exciton emission. First, the integral intensity of biexciton emission $\Phi_{XX}$ shows a superlinear growth with the increase in excitation intensity $I$ while the integral intensity of free exciton emission $\Phi_X$ is growing linearly. The experimental data can usually be fitted by the simple power law of the form $\Phi \propto I^m$, where $m = 1$ and $m = 2$ for free excitons and biexcitons, respectively.$^{[11,13]}$ Therefore, at higher laser, power biexciton emission
is typically dominating. Another difference of free exciton and bie exciton properties is related to the temperature dependence of the peak position. Free exciton emission usually follows the temperature dependence of the bandgap energy while the bie exciton peak position is showing more rapid redshift. The bie exciton emission happens by the radiative annihilation of the electron and hole pairs, which is described by the following equation:

\[ E_{XX} = E_X + E_h \]

where \( E_X \) is the energy of the exciton and \( E_h \) is the energy of the hole. The binding energy of the bie exciton is given by:

\[ K_{XX} = K_X \]

\[ (2E_X - E_{XX}^b) \cdot \frac{\hbar^2 K_{XX}^2}{2M_{XX}} = h\nu \]

where \( E_X \) is the energy of the free exciton, \( E_{XX}^b \) is the energy of the bie exciton, \( \hbar \) is the reduced Planck constant, \( h\nu \) is the energy of the emitted photon, \( M_X \) is the mass of the exciton, and \( M_{XX} \) is the effective mass of the bie exciton.

The Sb\(_2\)Se\(_3\) sample for PL measurements consisted of several dozen randomly oriented microcrystals. Most of these microcrystals had a needle-like shape with flat well-formed facets, see Figure 1a and Figure S1a in the Supporting Information. Therefore, all additional measurements were done using these microcrystals.

3. Results and Discussion

At first, we measured PL of three different Sb\(_2\)Se\(_3\) samples: microcrystals grown at Se source temperatures of 450 and 380 °C and a bigger single crystal, see Figure S2 in the Supporting Information. All samples show very similar PL spectra, but the most intense emission and the lowest low energy tail we got from sample grown at Se source temperature of 450 °C, see Figure S2a in the Supporting Information. Therefore, all additional measurements were done using these microcrystals.

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Figure 1b presents a deconvoluted Raman spectra of Sb\(_2\)Se\(_3\) microcrystals fitted using Lorentzian function to resolve the peaks. Spectra were measured at RT using two different lasers. It can be seen that by using red laser (\( \lambda = 633 \) nm) we were able to reveal also a peak at 81 cm\(^{-1}\). All observed peaks belong to Sb\(_2\)Se\(_3\) and were found and theoretically predicted also in refs. [16,35–37]. The X-ray diffraction (XRD) pattern of the studied Sb\(_2\)Se\(_3\) microcrystals is presented in Figure 1c. According to the XRD analysis, the material crystallizes in orthorhombic structure of Sb\(_2\)Se\(_3\) with space group Pbnm #62. All detected XRD peaks belong to Sb\(_2\)Se\(_3\). According to the peak positions, estimated lattice parameters are: \( a = 1.1784 \) nm,
$b = 0.3980 \text{ nm}$, and $c = 1.1650 \text{ nm}$. These values are in good agreement with the values found in literature and in crystallographic database.\cite{16,38} Figure 2a,b illustrates the PL spectra of Sb$_{2}$Se$_{3}$ microcrystals at different temperatures, measured with laser peak power density of 660 kW cm$^{-2}$ ($\lambda = 266 \text{ nm}$). At $T = 3 \text{ K}$, only two slightly asymmetric biexciton peaks can be detected labeled as XX$_B$ and XX$_A$, but with increasing the temperature the XX$_B$ peak vanishes and a new peak X$_B$ appears at higher photon energy. All these peaks show a redshift with increasing temperature.

For spectral fitting of PL peaks, we used an asymmetric hyperbolic secant function

$$I = \frac{I_0}{\exp[(E-E_0)/W_L] + \exp[-(E-E_0)/W_H]} \quad (5)$$

where $I$ is the PL intensity, $I_0$ is a parameter related to the maximum intensity of the PL peak, $E_0$ is a parameter which is related to the peak position of the PL peak, $E$ is a photon energy, $W_L$ and $W_H$ are parameters related to the peak width from low and high energy side of the peak, respectively. This fitting function was successfully used in slightly disordered 2D materials, where exciton and trion peaks were weakly asymmetric.\cite{39,40} For all peaks $W_L > W_H$. Theoretical calculations also have shown that the shape of biexciton peak is asymmetric, but the actual shape is still not completely settled.\cite{34} All PL spectra were showing also a wide background at lower photon energies and this background was fitted using Gaussian shape. Results of these spectral fittings are given in Figure 2d.

The full-width at half-maximum (FWHM) of the peaks at $T = 3 \text{ K}$ is in the range of 10–30 meV. This rather small width is typical for PL peaks having excitonic origin. Additional peak X$_A$ becomes more apparent, when the laser power was reduced, see Figure 2d and Figure 3c. The model of detected free exciton and biexciton peaks is presented in Figure 2c and peak positions are given in Table 1.
In Figure 3a, the peak positions of the free exciton $X_B$ and biexcitons $XX_B$ and $XX_A$ depending on the sample temperature are shown. The peak position of $X_A$ free exciton is given at $T = 3$ K as a red dot. The temperature dependence of the $X_B$ peak position was fitted using Varshni expression [41]

$$E_{\text{max}} = E_0 - \alpha T^2 / (\beta + T)$$

where $E_0$ is the zero Kelvin peak position. The Varshni parameters $E_0$, $\alpha$, and $\beta$ determined for $X_B$ are, respectively, $1.333$ eV, $0.77$ meV K$^{-1}$, and $354$ K. The same expression was used also for $E_g$, where obtained parameters were $1.317$ eV, $0.90$ meV K$^{-1}$, and $358$ K. The temperature dependence of $E_g$ is given as a green line in Figure 3a. The slight difference of parameters $\alpha$ and $\beta$ for the $X_B$ peak position and $E_g$ is mostly related to small difference between temperature dependence of $E_g$ and $E_E$, because the free exciton peak usually follows the temperature dependence of bandgap energy. At the same time, the temperature dependence of $XX_B$ peak position shows more noticeable redshift and this is typical for biexciton peak position. Unfortunately, the intensity of the $XX_A$ peak dropped rapidly with temperature and we could not determine the behavior of this peak at higher temperatures, where this peak is expected to show similar to $XX_B$ temperature dependence. According to these obtained values of peak positions, the value of the crystal field splitting is $\Delta_{\text{cr}} = 20-22$ meV.

This is slightly higher than the value obtained from absorption measurements in ref. [20].

Next, the analysis of the temperature quenching of the PL peaks was carried out, see Figure 3b. An Arrhenius plot of the resulting integral intensity was fitted using a model of a single recombination channel proposed in ref. [42]

$$\Phi(T) = \Phi_0 \left[ 1 + \alpha_1 T^{1/2} + \alpha_2 T^{3/2} \exp\left( -E_a / kT \right) \right]$$

where $\Phi_0$ is the integral intensity of the PL peaks at 0 K, $\alpha_1$ and $\alpha_2$ are process rate parameters, and $E_a$ is the activation energy. It turned out that all peaks show quite similar thermal quenching activation energy $E_a = 20.0 \pm 0.8$ meV, but parameters $\alpha_1$ and $\alpha_2$ were different for different peaks. At low excitation density, the quenching process of biexciton emission is mainly due to the thermal dissociation of biexciton into free excitons while the temperature quenching of excitonic lines is due to the thermally activated depopulation of the free excitonic level and/or to the activation of nonradiative recombination centers. The similar thermal activation energy for free

| Peak label | Peak position at $T = 3$ K [eV] | Assignments | Binding energy [meV] |
|-----------|-----------------------------|--------------|---------------------|
| $X_B$     | 1.333 (estimated)           | Free exciton (B) | $-$                |
| $XX_B$    | 1.322                       | Biexciton (B)  | 11                  |
| $X_A$     | 1.311                       | Free exciton (A) | 6                  |
| $XX_A$    | 1.302                       | Biexciton (A)  | 9                  |

Table 1. Detailed features and transition assignments of the near-band-edge PL peaks in Sb$_2$Se$_3$ microcrystals. Calculated binding energies are given in the last column.
exciton and biexciton peaks exceeding about twice the binding energies is probably an indication of nonradiative processes at high excitation density. At higher temperatures, the generation rate of biexcitons and free excitons is quite high and despite increasing thermal dissociation the concentration of both exciton features is notable. However, the role of the exciton–phonon and exciton–exciton scattering is increasing with temperature and thus the overall intensity of PL peaks starts to decrease. High thermal activation energy of exciton emission exceeding the exciton or biexciton binding energy was also found in MAPbBr3 perovskite quantum dots and in CuInSe2. It was concluded that the usual dissociation of free excitons or biexcitons into the continuum states and trapping by the nonradiative centers cannot be the only process leading to the thermal quenching of PL. However, additional studies are needed to specify the mechanism of thermal quenching of these PL peaks.

Additional proof of free exciton and biexciton emission can be found from laser power dependence of our PL peaks. By decreasing the laser power, we did not notice any shift of peak position of all PL peaks and the XA PL peak is more exposed at lower laser power, see Figure 3c. The integral intensity dependence of PL peaks on laser power is presented in Figure 3d. As predicted by theory, both biexciton peaks (XXA and XXB) show superlinear growth with m = 2 while the intensity of free exciton peak (X) increases linearly with m = 1.

According to peak position of observed free exciton and biexciton peaks at T = 3 K, we calculated also the binding energies, see Table 1. The binding energy for the Xa free exciton was not determined, because we do not have definite value for E\textsuperscript{b}. However, we believe that the binding energy is very close to the value of Xb binding energy. Unfortunately, we were not able to detect any sign of indirect transitions predicted by other groups. Apparently, other methods are needed to uncover indirect transitions in Sb\textsubscript{2}Se\textsubscript{3}.

### 4. Conclusions

In conclusion, we present a detailed study of free exciton and biexciton emissions in Sb\textsubscript{2}Se\textsubscript{3} microcrystals. Due to the crystal field splitting of the valence band (\(A_{\gamma} = 20–22\) meV), we observed two different free exciton PL peaks and two different biexciton peaks at T = 3 K: Xa, XB, XXA, and XXB centered at 1.311, 1.333, 1.302, and 1.322 eV, respectively. The binding energy of biexcitons was 9 and 11 meV for A and B biexcitons, respectively. Both biexciton peaks (XXa and XXb) show a superlinear growth while the intensity of free exciton peak (X) increases linearly on laser power. The temperature dependence of XXB peak position shows noticeable redshift typical for biexciton peak position. This shift is faster than the normal temperature shift caused by the temperature dependence of the bandgap energy. The fast shift with temperature is attributed to the contribution of the bieexcitonic kinetic energy. We also found that all PL peaks show quite similar thermal quenching activation energy \(E_q = 20.0 \pm 0.8\) meV and this is probably related to the activation of common nonradiative recombination channel.

### 5. Experimental Section

The Sb\textsubscript{2}Se\textsubscript{3} microcrystalline powder was synthesized in a two-temperature zone furnace in a sealed evacuated quartz ampoule from Sb metal powder (5N, ~200 mesh powder, Alfa Aesar) and elemental Se pellets (4N purity). 0.1 g Sb metal powder was poured into one side of a quartz ampoule by using polymer funnel and as a chalcogen source 0.118 g Se pellets were introduced in the other side. The volume of used ampoule was ~25 mL with inside diameter of 1.0 cm and length of 32.5 cm. The precursors inside the ampoule were degassed under dynamic (continuous) vacuum-pumping process for 1 h at RT and the ampoule was sealed at a vacuum level of 1.3 × 10\textsuperscript{−2} mbar (0.01 torr). Sealing was done by using O\textsubscript{2} + propane flame. After that, sealed ampoule was placed into a pre-heated two-zone tube furnace, where the temperature of both zones was controlled and regulated precisely. In such an arrangement, the vapor pressure of the Se source was determined and controlled by the lowest temperature in the system. The temperature zone in which the Sb powder was located was kept at 500 °C and the Se source temperature was kept at 450 °C, which corresponds to the partial pressure of Se of 16.4 mbar (~12.3 torr). Additionally, a second growth was also made with a Se source temperature of 380 °C corresponding to the Se pressure of 2.67 mbar (~2 torr). The formation process of Sb\textsubscript{2}Se\textsubscript{3} took place at these temperature regimes for 96 h. Finally, the ampoule was taken out from the furnace with the Se source side ahead and cooled down to RT with a cooling rate of 120 °C min\textsuperscript{−1}. The obtained Sb\textsubscript{2}Se\textsubscript{3} microcrystalline powder contained well-formed monocristalline needles with flat and shiny facets and additionally some polycrystalline fragments of Sb\textsubscript{2}Se\textsubscript{3}. At disposal, a bigger single crystal made by liquid-phase epitaxy (LPE) was seen, see Figure 5b,c in the Supporting Information.

XRD patterns were recorded using Smartlab (Rigaku) diffractometer equipped with rotating 9 kW Cu anode X-ray tube. Powder sample was measured in Bragg-Brentano geometry in 10°–70° 2θ angle range with 0.02° step. Reference XRD pattern was simulated from Sb\textsubscript{2}Se\textsubscript{3} CIF file obtained from crystallographic open database. Lattice parameters were calculated manually to match experimental XRD peak positions based on orthorhombic crystal system.

The shape and surface morphology of the synthesized microcrystals were studied with a high-resolution scanning electron microscope (SEM), Zeiss MERLIN.

For the PL excitation, a diode pumped Nd:YAG solid state 266 nm laser was used with the pulse width 0.6 ns and repetition rate 19 kHz. The maximum peak power was 0.75 μJ and the maximum average power was 7.15 mW. The maximum peak power density was estimated to be 660 kW cm\textsuperscript{−2}. The laser beam was not focused to avoid damaging the sample and the pump power was reduced by a gradient UV (fused silica) filter.

PL was dispersed by using the 500 mm focal length monochromator (Andor SR-500i; grating 600 g mm\textsuperscript{−1} blazing at 1000 nm) and focused into thermoelectrically cooled InGaAs photodetector (IGA-030-TE2-H; Electro-Optical Systems Inc.). The conventional lock-in detection system (SR830; Stanford Research Systems) was used to extract the emission signal. The PL measurements were carried out in the temperature range of 3–110 K by mounting the samples (several dozen randomly oriented microcrystals) on the cold finger of a closed-cycle helium cryostat (Janis SHI-4; Lake Shore Cryotronics, Inc.).

Raman measurements were carried out using a Horiba LabRAM HR800 μ-Raman system equipped with a cooled multichannel CCD detection system in the backscattering configuration with a spectral resolution better than 1 cm\textsuperscript{−1}. A He-Ne laser (wavelength \(\lambda = 633\) nm) and Nd:YAG laser (wavelength \(\lambda = 532\) nm) were used for excitation. The laser spot size was about 5 μm in diameter.

Supporting information for additional SEM images of synthesized Sb\textsubscript{2}Se\textsubscript{3} representative PL spectra of different samples, and deep PL is available from the Wiley Online Library or from the author.
Supporting Information
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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
The data that support the findings of this study are available from the corresponding author upon reasonable request.

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