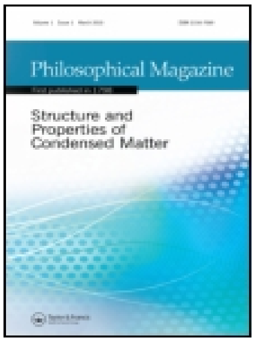

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Low-temperature thermoluminescence study of GaSe:Mn layered single crystals

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ABSTRACT
Mangan doped GaSe single crystals have been studied by thermoluminescence measurements performed with various heating rates between 0.4 and 1.0 K/s in the temperature range of 10–300 K. Thermoluminescence spectra exhibited four distinguishable peaks having maximum temperatures at 47, 102, 139 and 191 K revealing the existence of trapping levels in the crystals. Curve fitting and initial rise methods were applied to observed peaks to determine the activation energies of four trapping levels. Capture cross-sections of each level were also evaluated using the obtained energy values. Moreover, heating rate dependencies of the obtained peaks were investigated. It was shown that increase in the heating rate resulted in the decrease in thermoluminescence intensity and shift of the peak maximum temperatures to higher values. Discrete, single trap behaviour was established for acceptor level related with the peak at 191 K by analysing the sequentially obtained peaks with different stopping temperatures between 15 and 65 K.

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Semiconductors; crystals; defects; luminescence

1. Introduction

The A(III) B(VI)-type semiconducting compound of GaSe which has a layered structure can be considered as a promising candidate for the production of optoelectronic devices in red and blue visible region [1–3]. The intralayer bond type has strong ionic–covalent while van der Waals interactions dominate the interlayer bonds [4]. Researchers have investigated the electrical and optical properties of these type crystals to get knowledge about possible usage area in technology. Approximately, 2.0 eV of band gap energy for GaSe was reported in previous studies [5,6]. GaSe crystals have been studied to expand the application areas in technology. Second-order non-linear optical susceptibility and high birefringence make the undoped crystal suitable for infrared and terahertz generation [7–11]. Previously, photoluminescence (PL) measurements have been performed for undoped GaSe crystals in the temperature range of 10–300 K in the wavelength range from 635 to 750 nm [12]. Two broad bands reaching maximum intensity at 644 and 695 nm were reported.

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Thermoluminescence (TL) investigations at low temperatures (10–300 K) have also been accomplished for undoped GaSe crystals [13]. Results of the analysis exhibited the presence of four trapping levels centred at 140, 180, 240 and 370 meV for as-grown GaSe. Effect of the annealing on the defects in the as-grown crystal has been studied. The determined trap levels have not completely been eradicated by annealing of the crystal with increasing temperature up to 500 °C. Four peaks attributed to the trap levels with activation energies 110, 160, 180 and 330 meV were obtained. However, it was reported that the intensity of the observed peaks after annealing dropped dramatically (~100 times) indicating the concentration of trapping levels to be decreased.

In addition to investigations on undoped GaSe layered crystals, researchers have also given great attention to GaSe crystals doped with different elements. Hsu et al. [14] have investigated the optical and electrical properties of GaSe:Er crystals by means of Hall, deep-level transient spectroscopy (DLTS) and PL measurements. In their study, two acceptor levels were revealed with energies of 65 and 158 meV. Previously, Shigetomi et al. [15] studied GaSe crystals doped with Mn by PL measurements at low temperatures (above 77 K). They obtained the emission spectrum corresponding to the transition from the conduction band to the acceptor level with energy of 1.82 eV. As a result, an acceptor level above the valence band was established centred at 300 meV. Hall-effect, PL and photo-current response of p-type GaSe:Te crystals have also been reported by Shigetomi et al. [16]. They claimed that two observed acceptor levels at 20 and 80 meV originate from the interstitial Te atoms. The same authors expanded their works on GaSe crystals by using different dopants from group V elements [17]. They used PL and Hall-effect technique to investigate the effect of As, Bi and Sb dopants on the crystal properties. Analysis of the obtained data revealed the existence of one donor level at about 80 meV associated with hydrogen-like donor level below the conduction band and one acceptor level at about 600 meV attributed to defects or defect complexes above the valence band. Moreover, Cui et al. [18] focused on indium-doped GaSe crystals to characterize the defects and/or impurity levels. DLTS measurements performed in the temperature range of 100–350 K resulted with four peaks having maxima at 127, 160, 248 and 319 K. They suggested that the trapping levels related to peaks observed at 127 and 248 K occurred due to the defects arising from indium dopant. On the other hand, peaks observed at 160 and 319 K were believed to originate from the natural defects like dislocations or stacking faults in the undoped crystal. p-type GaSe:Cr crystals produced by Bridgman method were also studied to obtain information on its electrical properties [19]. The authors determined an activation energy of 18 meV for acceptor level of GaSe:Cr crystals using a technique based on the temperature dependencies of hole concentration.

The purpose of this manuscript is to report the results of low-temperature thermoluminescence measurements on GaSe:Mn crystals which is an effective experimental method to study the defects and/or impurities in the crystals. They may play an important role for the performance of devices used in technological area. They can not only lower the internal quantum efficiency but also render light generation impossible. Moreover, some scattering centres may occur due to the defects and this leads to diminish carrier mobility which causes not to be developed high-frequency operation. For this reason, revealing the existence of trapping centres due to defects and/or impurities will encourage the researchers and/or producers working in related areas.

2. Experimental details

Mn of 99.999% purity was doped at 1 at. % to the stoichiometric melt of gallium selenide. Raw materials were enclosed in the well-cleaned quartz tube with a tip at the bottom under 10^{-5} Torr. The single crystals were grown by the conventional Bridgman technique. The growth was carried out in a vertical furnace through a thermal gradient of 30 °C/cm between the temperatures 1000 and 650 °C at a rate of 0.5 mm/h. The samples were cleaved from ingots (dark red in colour) with a razor blade perpendicular to the ***c***-axis of the crystal. The dimensions of the bulk sample used in TL measurements were 18 mm × 8 mm × 1 mm. The studied sample has electrically p-type conductivity as determined using the hot-probe method.

Home-made experimental set-up built around a closed cycle helium gas cryostat (Advanced Research Systems, Model CSW-202) was used for TL measurements carried out at low temperatures (10–300 K). The pressure in the cryostat was decreased to approximately 10^{-3} Torr. The sample temperature was governed using a Lakeshore Model 331 temperature controller. A blue light source (~470 nm) and a photomultiplier (PM) tube were attached to the optical access port of the cryostat (quartz window) by a measurement chamber. Illumination with blue light lasted 600 sec at 10 K. An expectation time of 120 sec was waited for excited charge carriers to be brought equilibrium. Then, the sample was heated up to room temperature. Some mirrors and lenses were placed between the sample and PM tube (Hamamatsu R928; spectral response: 185–900 nm) to direct and to focus the luminescence emitted from the sample to the PM tube working in photon counting regime. The output signal of the PM tube was converted into TTL pulses by a fast amplifier/discriminator (Hamamatsu Photon Counting Unit C3866). The counter of data acquisition module (National Instruments, NI-USB 6211) was employed to count the TTL pulses. LabView™ graphical development environment designed for TL measurements was improved for our experimental set-up by writing a program to control whole measurement system/devices. The schematic diagram of the measurement set-up is presented in Figure 1.

3. Results and discussion

Figure 2 shows a typical TL curve for GaSe:Mn crystal in the temperature range 10–225 K with heating rate of $\beta = 1.0$ K/s. At the beginning of the experiments, we have performed the measurements in the10–300 K range. However, since no TL peak is detected above 225 K, we have carried out the further experiments in the 10–225 K range. Three overlapping peaks (A, B and C) and one isolated peak D were observed with maximum temperatures (T_{max}) nearly at 47, 102, 139 and 191 K, respectively.

Curve fitting and initial rise methods are the best-known and useful methods among the analysis techniques valid for experimental TL curves. We applied those of two methods to the observed TL curves and calculated the activation energies of trap levels in the Mn doped crystal associated with the curves.

Chen and McKeever stated that the intensity of thermoluminescence (I_{TL}) emitted from any trap centres in a luminescent material is characterized by the following equations [20],

$$I_{TL} = n_0 \nu \exp\left\{-\frac{E_t}{kT} - \int_{T_0}^{T} \frac{\nu}{\beta} \exp\left(-\frac{E_t}{kT}\right) dT\right\} \tag{1}$$

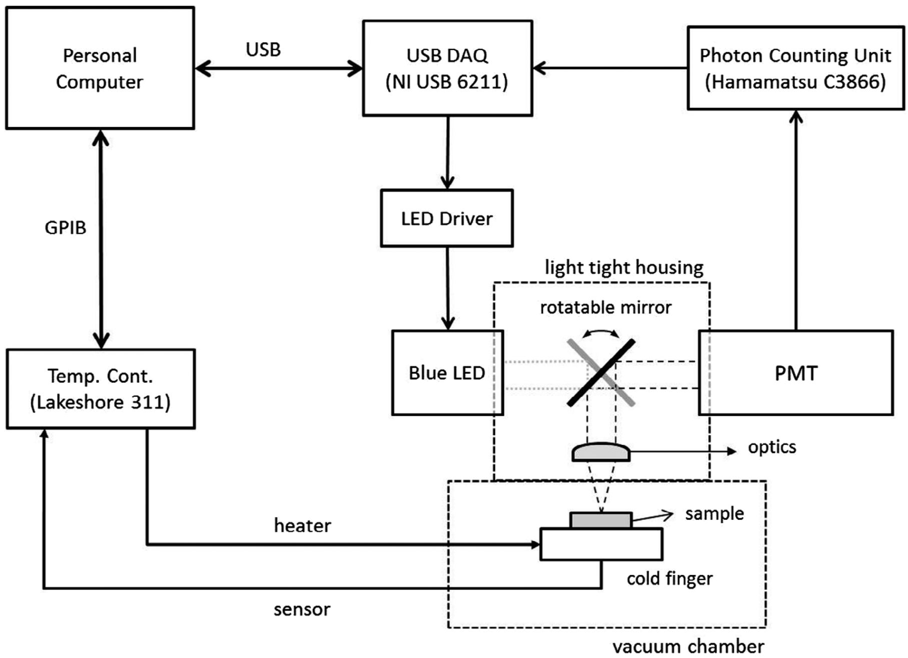

Figure 1. Simplified block diagram of the TL measurement set-up.

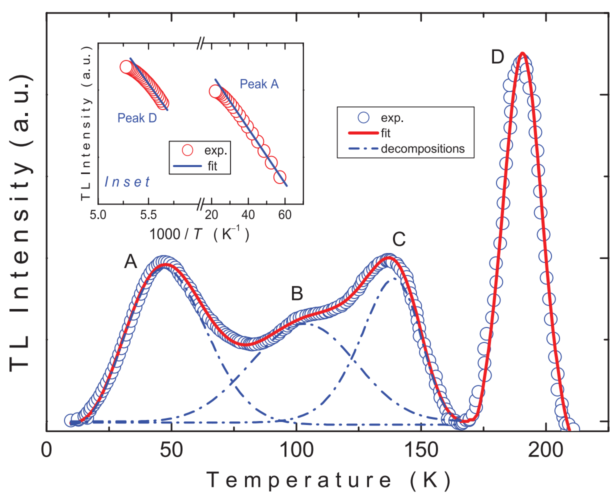

Figure 2. (colour online) Experimental TL glow curve (open circles) of GaSe:Mn crystal with heating rate of 1.0 K/s. Solid curve shows the fit to the experimental data. Dash-dotted lines show the decomposed curves obtained by fitting the experimental TL curve. Inset: TL intensity vs. 1000/*T*. The circles present the experimental data and the solid lines represent the theoretical fit.

$$I_{\mathrm{TL}} = n_0 \nu \exp\left(-\frac{E_{\mathrm{t}}}{kT}\right)\left[1 + (b-1)\frac{\nu}{\beta}\int_{T_0}^{T} \exp(-E_{\mathrm{t}}/kT)\mathrm{d}T\right]^{-\frac{b}{b-1}} \tag{2}$$

Equations (1) and (2) are related to the first and non-first order kinetics, which declare retrapping of charge carriers in the material are negligible and non-negligible, respectively. In the equations, n_0, v, E_t, β, T_0 and b correspond to the initial number of charge carriers occupying trap level, the attempt-to-escape frequency, the activation energy of the trap level, the heating rate, the starting temperature of heating process and the order of the kinetics, respectively. We applied curve-fitting method to successively obtain glow curves with the help of these equations. For the first three peaks (A, B and C), Equation (1) gave the best-fit result with the activation energy values of $E_{tA} = 8 \pm 1$ meV, $E_{tB} = 34 \pm 2$ meV and $E_{tC} = 130 \pm 6$ meV. This is an evidence for the trap levels that characteristics of the first-order kinetics are exhibited. Since the last peak (labelled as D in Figure 2) was distinctively independent of the others, we analysed it separately. Application of Equation (1) was unsuccessful for fitting of the peak D. Therefore, Equation (2) was utilized several times by varying the parameter b between 1 and 2. The best fit was accomplished with the value of $b = 1.2$, which indicates that the mixed order of kinetics dominates the trap level corresponding to the TL peak D [20]. At the end of fitting process, activation energy of trapping centre was calculated as $E_{tD} = 388 \pm 15$ meV. The capture cross-section (S_t) values were also computed using the found energy values in the following equation

$$S_t = \frac{\exp(t_m)t_m^3\beta k}{N_v \upsilon_{th}(2b + t_m)E_t}, \text{ where } t_m = \frac{E_t}{kT_{max}}$$

In the above equation, $N_v = 2(2\pi m_h^* kT/h^2)^{3/2}$ is the effective density of states in the valence band and υ_{th} is thermal velocity of a free hole. S_t values were calculated as 2.6×10^{-26}, 4.4×10^{-26}, 6.8×10^{-23} and 2.2×10^{-17} cm^2 for traps related with the peaks A, B, C and D, respectively, using the effective mass $m_h^* = 0.8m_0$ value for GaSe crystals in the equation responsible for the effective density of states [21].

Initial rise method can be used for TL curves without considering the type of the kinetics order dominating the trapping states during the excitation process of charge carriers in trap levels. This method is independent of kinetics order and basically depends on the initial tail of the TL curves. Thermal excitation of charge carriers to the non-localized states from trap levels give rise to TL emission proportional to $\exp(-E_t/kT)$ [20]. Following this relation, one can plot logarithmic variation of the TL intensity as a function of reciprocal of the respective temperature. Activation energy of trap levels can be computed using the slope of this graph, which yields a straight line with the value ($-E_t/k$). In the present study, we could only analyse the peaks A and D since the peaks B and C overlap with peak A and each other. Obtained activation energies of trap levels were reported as $E_{tA} = 7 \pm 1$ meV and $E_{tD} = 386 \pm 15$ meV (see inset of Figure 2).

At this point, we focused to compare found energy values with previous studies on related crystals reported by several authors [13,22,23]. The acceptor levels with activation energies of 130 and 388 meV are close to those observed in undoped GaSe crystals (140 and 370 meV) [13]. These levels may be associated with native defects or defect complexes [22]. In our experiments on GaSe:Mn crystals, we have not observed the acceptor levels with energy of 180 and 240 meV reported in undoped GaSe crystals by TL measurements [13]. It is generally adopted that the major acceptors in undoped GaSe are gallium vacancies V_{Ga} [23]. Doped with mangan, most of the gallium vacancies are occupied by mangan atoms, and the Mn_{Ga} defects may be non-radative centres. Thus, in the TL spectra of GaSe:Mn

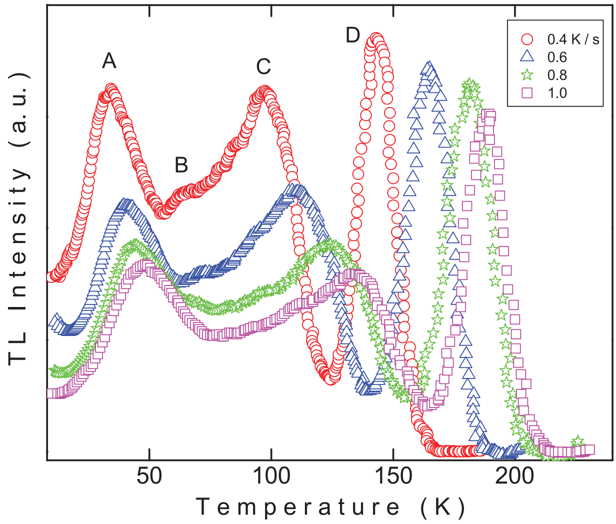

Figure 3. (colour online) Experimental TL curves of GaSe:Mn crystals with different heating rates between 0.4 and 1.0 K/s.

crystals, the absence of bands with activation energies of 180 and 240 meV may probably be associated with the recovery of these defect levels by Mn atoms doping.

The shallow acceptor level of 34 meV observed in our study of GaSe:Mn crystal by low-temperature TL experiments may be attributed to the Mn atoms by analogy with the results of work on GaSe:Cu crystals [22], where shallow acceptor level of 40 meV was assigned to the Cu atoms. As for the extremely shallow level of 8 meV, which was not previously observed in any GaSe crystals doped with various atoms, this level probably may be attributed also to the Mn atoms.

In order to better comprehend the TL mechanism of the indicated trap levels, heating rate dependence of the glow curves was investigated. Figure 3 shows the detected TL glow curves of Mn-doped GaSe crystals obtained by heating the sample with various β values ranging from 0.4 to 1.0 K/s with step of 0.2 K/s. One can easily observe the alteration in the position and shape of the TL curves as a response to variation of heating rate in the graph. Clearly, T_{max} value of each glow curves shifts to higher temperatures with increase in heating rates. Some explanations for this behaviour were reported by Anishia et al. [24] in their TL study on lithium magnesium borate phosphors. As the charge carriers occupying the trap levels are excited with small heating rate of β_1, one can obtain a TL curve with maximum temperature T_{max1}. If the charge carriers are stimulated with heating rate of β_2, which is bigger than β_1, the trapped charge carriers recombine with the opposite charge carriers at the recombination centre more quickly. This means that less charge carriers are released from the trap levels with thermal energy kT_1 by performing the heating process with β_2. Same amount of charge carriers can only be released from the trap levels with an energy kT_2 ($T_2 > T_1$) as the heating rate of β_2 is used for excitation. Therefore, the maximum temperature of a new TL curve (T_{max2}) shifts to higher temperatures. Figure 4 illustrates the heating rate dependence of TL intensity and T_{max} values of TL curves. As expected, increase in heating rate resulted in increase of T_{max} value. In addition, increase in the heating rate

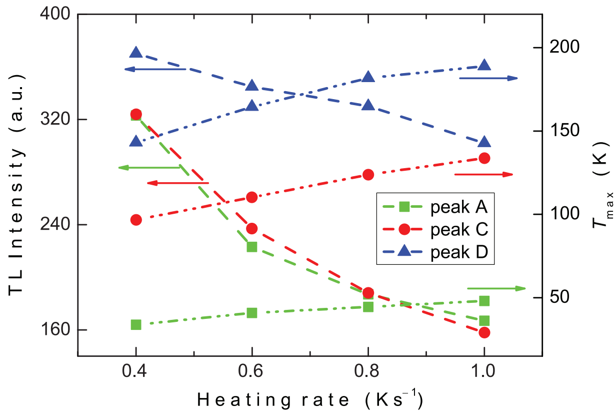

Figure 4. (colour online) Heating rate dependencies of TL intensities and peak maximum temperatures.

led to the decrease in the TL intensity, which can be assigned to thermal quenching whose efficiency gets prominent at higher temperatures [25–27].

Thermoluminescence properties of the trap levels in GaSe:Mn crystals was studied in detail utilizing from a well-known technique based on the thermal cleaning of the shallowest levels of the traps by varying the stopping temperature (T_{stop}) [20,24]. The experimental procedure of this technique was performed as follows: The sample was cooled down to T_{stop} temperature and illumination was carried out at this temperature for 600 sec. Then, the temperature was decreased to starting temperature ($T_0 = 10$ K). Afterwards, the sample was heated up to 300 K with constant rate of 1.0 K/s. Same condition was applied for each pre-heating treatment achieving for different T_{stop} values ranging from 15 to 65 K. In these wise, trapped charge carriers occupying shallowest levels were emptied and only contribution to obtained TL curves was provided from liberation of charge carriers at deeper levels. In the literature, there are many examples explaining the characteristics of traps centre. Some trap states exhibit the properties of continuous distribution of traps especially in highly defective materials while the others consist of single trap level. The applied experimental method is very successful to understand the behaviour of trap centre. In continuous traps which reveal the existence of many overlapping peaks, increase in the T_{stop} values leads to shift of initial tail and T_{max} value of each glow curves to higher temperatures distinguishably, while the high-temperature side of the curves is not affected [20,28–30]. However, in the single trap case, T_{max} of the curves remains unchanged and low- and high-temperature sides of the curves tends to decrease symmetrically as the T_{stop} value is increased [20]. The only alteration is the number of charge carriers releasing from trap level. Figure 5 depicts the TL curves detected with different T_{stop} values between 15 and 65 K. Since the peaks observed at low temperatures (peaks A, B and C) were overlapping, we only characterized the trap level corresponding to peak labelled as D. As can be seen from the figure, T_{max} values of the curves can be considered as nearly same with negligible variation of ~2 K (difference between the maximum temperature of first and last peaks). The TL intensity of the curves decreases in both the temperature sides symmetrically as the stopping temperature is increased. Inset of Figure 5 shows the T_{stop} variation in T_{max} and E_t values of each glow curves. Activation energies were calculated using the initial rise method and all of the values were found very close each other. This is another evidence for trap to be attributed to discrete, single level.

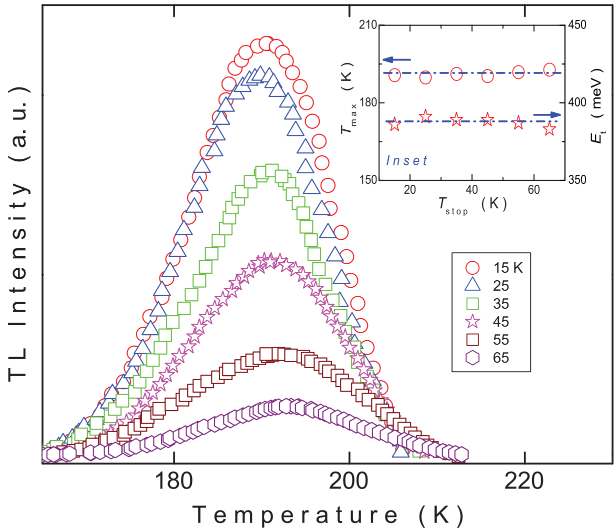

Figure 5. (colour online) Glow curves of GaSe:Mn crystals at different T_{stop} temperatures at heating rate $B = 1.0$ K/s. Inset: The dependence of peak maximum temperatures and activation energies on T_{stop} values. The dash-dotted lines are only guide for readers.

The average activation energy of the single trap level was calculated as 388 ± 15 meV utilizing the energy values of each glow curves. This value is good agreement with the values evaluated by previous analysis technique.

4. Conclusions

TL investigations have been accomplished for GaSe:Mn single crystals with the help of the analysis of the observed glow curves in the temperature range 10–225 K. Activation energies were found as 8 ± 1, 34 ± 2, 130 ± 6 and 388 ± 15 meV for trapping levels corresponding to peaks with maxima at 47, 102, 139 and 191 K, respectively, by utilizing curve-fitting and initial rise methods. Curve-fitting method also established that the trap levels related with peaks A, B and C were dominated by first-order kinetics, while the trap level associated with peak D exhibited the properties of mixed-order kinetics. The capture cross-section values of trap levels were found as 2.6×10^{-26}, 4.4×10^{-26}, 6.8×10^{-23} and 2.2×10^{-17} cm^2, respectively. The acceptor levels with energies of 130 and 388 meV were believed to be related to defects or defect complexes that were also obtained in undoped GaSe. However, the acceptor levels centred at 8 and 34 meV were ascribed to Mn atoms. Moreover, thermal quenching effect was revealed for the trap levels by studying heating rate dependencies ranging from 0.4 to 1.0 K/s. It was demonstrated that the acceptor level related with peak D behave as single trap level.

Disclosure statement

No potential conflict of interest was reported by the authors.

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