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Investigation of traps distribution in GaS single crystals by thermally stimulated current measurements

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ABSTRACT

Thermally stimulated current (TSC) investigations of *p*-GaS (gallium sulfide) single crystals grown by Bridgman method were achieved by virtue of consecutive experiments carried out at various heating rates in between 0.4 and 1.0 K/s in the temperature range of 10–280 K. One single TSC peak around 148 K and overlapped, incomplete peaks in the end limit temperature of the experiments were observed in the spectrum recorded at constant heating rate of 1.0 K/s. Individual peak was analyzed utilizing curve fitting method. Existence of one trapping level centered at 0.11 eV was revealed by the analyses. Heating rate dependency of obtained TSC curve was also studied and it was shown that TSC intensity decreased besides increase of peak maximum temperature with heating rate. Characteristics feature of trapping mechanism was investigated in detail by employing different stopping temperature between 50 and 110 K. Analyses on T_m–T_{stop} dependency resulted in a presence of quasi-continuously distributed traps with activation energies ranging from 0.11 to 0.55 eV. The revealed trap was thought to be arising from intrinsic defect possibly created by V_{Ga} or antisite S_{Ga}.

1. Introduction

GaS mono chalcogenide is a member of III–VI semiconductor compounds which have attracted great attention of researchers due to their fascinating properties suitable for various device applications in optoelectronics [1–6]. GaS crystals have layered structure consisting of two planes of Ga atoms sandwiched between two planes of S atoms in the sequential form of S–Ga–Ga–S. Bonding within the each layer is covalent type while bonding between layers is weak Van der Waals [7]. Due to this weak interaction between layers, GaS bulk crystals are easily separated into its monolayers which provide convenience to obtain two dimensional very thin sheets. This causes material properties to be enhanced. For these convincing reasons, GaS has been mostly preferred material by researchers to investigate its structural, electrical and optical properties. Three types of crystal structure (β, ε and γ) were attributed to GaS according to stacking of quadruple layers on each other. Bulk GaS has β-phase structure with symmetry group of $D_{6h}^4(P6_3/mmc)$ [8]. It exhibits the properties of hexagonal structure. Lattice constants were reported as $a = 0.3587$ nm and $c = 1.5492$ nm [9]. The resistivity of the GaS was previously reported as 1.25×10^{10} Ω cm along the c-axis [10]. It is known as a wide band gap material. Transmission measurements revealed the presence of direct and indirect optical band-to-band transitions with energies of 2.53 and 2.63 eV, respectively, at room temperature [11]. Tunable band gap characteristics as a result of change of sulfide composition make this crystal promising for optoelectronic and photovoltaic applications [12,13]. It was shown in Ref. [4] that GaS monolayer is a perfect candidate for field-effect-transistor applications. Chitara and Ya'akobovitz gave point to usage of layered GaS in nano-electrochemical resonator showing mega-hertz resonance frequencies [14]. GaS may also be utilized for passivation of GaAs surfaces [15]. GaS_x compositions were shown to be used in lithium ion batteries as an anode [16].

Taking into account the possible use of GaS in a wide range of technological applications, characterization of this material in different aspects gains importance to have advanced quality devices. Defects and/or impurities are the main phenomena affecting performance of fabricated devices. According to literature works, GaS is highly defective material. Recently, photoconductivity studies on undoped *n*-GaS revealed the presence of trap states with energies of 0.1 and 0.4 eV [17]. Three trap levels were observed in GaS by photoluminescence study

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carried out below room temperature [18]. Activation energies of revealed traps were determined as 0.013, 0.017 and 0.151 eV. Thermoluminescence studies on GaS achieved below and above room temperature indicated the existence of three [19] and six [20] trapping levels with energies of 0.052, 0.200, 0.304 eV and 0.98, 1.09, 1.15, 1.76, 1.87, 1.90 eV, respectively. Thermally stimulated current (TSC) measurements on GaS crystals exhibiting *p*-type conductivity were performed in the temperature range of 10–300 K [21]. Six trapping centers with activation energies of 0.05, 0.06, 0.12, 0.63, 0.71, and 0.75 eV were revealed as a result of applied analyses methods. However, the observed TSC curves were not studied in detail in this reference. The aim of the present paper is to expand defect characteristic studies on GaS in the 10–280 K by performing heating rate-dependent, illumination time-dependent and stopping temperature-dependent TSC experiments for the first time. The observed peak was investigated by a well-known experiment technique called as $T_m - T_{stop}$ and distribution of trapping centers were revealed from the analyses. Illumination time dependence of observed TSC curve was investigated in order to determine order of kinetics of associated trap level. Heating rate dependency of TSC spectra was also studied in detail.

2. Experimental

Polycrystals of GaS were synthesized utilizing stoichiometrically added high-purity elements (at least 99.999%). Single crystal form of GaS was obtained by performing the Bridgman growth method. The structural characterization of the grown sample was previously studied by X-ray diffraction [20] and energy dispersive spectroscopy [22] techniques. X-ray diffraction pattern presented sharp and intensive peaks which are associated with hexagonal crystalline structure. Energy dispersive spectroscopy method revealed the atomic compositions of the constituent elements which were well-consistent with chemical formula of GaS. The resulted ingot was easily cleaved to get studied sample. TSC experiments were performed on the GaS bulk samples (see inset of Fig. 1) with dimensions of $10 \times 10 \times 2$ mm^3. Sandwich geometry was achieved for electrical contacts applied to both surfaces of the sample. The schematic representation of the sandwich geometry is shown in the inset of Fig. 1. One of the sample surface was attached to sample holder using silver paste and this side was grounded. A conductive tiny copper wire was attached to the front surface of the sample by a small drib of silver paste. I–V characteristics of the sample prepared in sandwich geometry was investigated at room temperature before placing the crystal into the cryostat and it was seen that the used sample exhibits ohmic behavior as represented in Fig. 1. The electrical conductivity was checked by performing hot-probe technique and *p*-type conductivity was obtained. Since the grown samples were not intentionally doped, the *p*-type conductivity may be due to unintentional doping and/or commonly observed intrinsic defects in GaS. TSC measurements were performed employing a home-made set up. A closed-cycle helium gas cryostat (Advanced Research Systems, Model CSW 202) was used in order to keep environment temperature between 10 and 280 K. The front side of the sample was illuminated by a light emitting diode (generating light at a maximum peak of 2.6 eV) attached to quartz window of cryostat when the temperature was decreased to 10 K. Throughout the illumination, a bias voltage $V_1 = 1$ V was applied using Keithley 228 A voltage/current source. After an expectation time of 120 s was waited, the temperature was increased up to 280 K with a constant heating rate by Lake-shore temperature controller having sensitivity of 10 mK. In the TSC measurements, voltage is applied to the sample to move charge carriers excited from trapping centers to the contact points. Theoretically, the value of the applied voltage is not important for defect characteristics. However, when low voltage is applied, charge carriers would be retrapped and/or make recombination before reaching to the contact. If high voltage is applied, it would create large background current and therefore TSC peaks may not be observed. Therefore, a sufficiently high voltage value which will not create significant background must be chosen. Different voltage values were applied and most suitable voltage was determined as 100 V and during heating process, a bias voltage of $V_2 = 100$ V was applied. The TSC was measured by a Keithley 6485 picoammeter. The measurements were repeated under same conditions for various heating rates between 0.4 and 1.0 K/s. The TSC spectra for all heating rates were recorded using a software (LabView graphical development program).

3. Results and discussions

Fig. 2 depicts TSC curves of GaS single crystals obtained at constant heating rate of 1.0 K/s in the temperature region of 100–280 K for the cases of without illumination, with illumination for forward and reverse bias voltages. Since there was not observed peak(s) below 100 K, this region was not included in the figure. As seen from the figure, TSC curve (triangles) does not present any peak and/or increase in the background

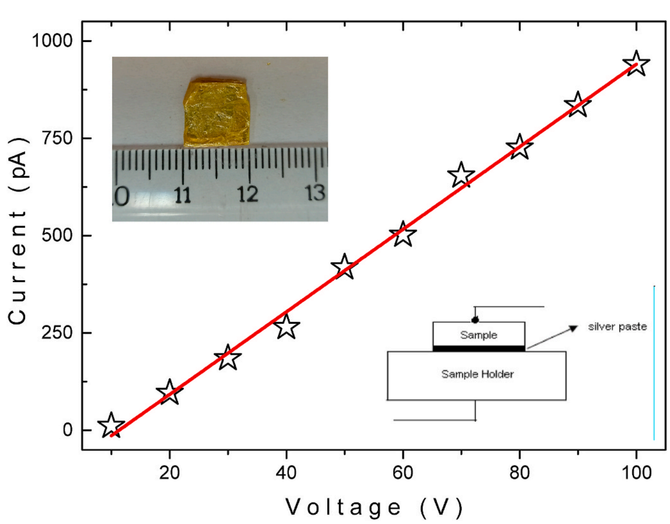

Fig. 1. Room temperature I–V characteristics of GaS bulk crystals prepared by sandwich geometry structure. Stars are the experimental data; solid line indicates the linear fit. Insets show the studied sample and schematic representation of the sandwich geometry.

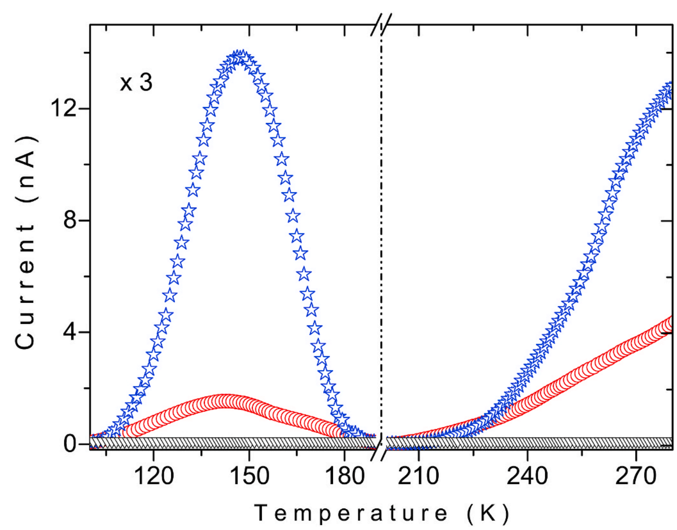

Fig. 2. Experimental TSC curves of GaS single crystals detected with a constant heating rate of 1.0 K/s under forward and reverse bias voltages that illumination was carried out at positive (stars) and negative (circles) contacts, respectively. Triangles represent the curve recorded without illuminating the sample. The observed peak magnitudes were multiplied by 3 for better seen.

current through the applied temperature range. This observation points out that the observed peaks when the sample was illuminated are associated with trapping centers. The TSC glow curves were obtained by connecting the positive (stars) and negative (circles) terminals of the voltage supply to the front surface of the sample and performing measurements for each case under same experimental conditions. In the course of the front surface is exposed to light source, not only electrons but also holes are generated in the vicinity of contact. Regarding to applied bias voltage, one type of the charge carriers is collected quickly whereas the other is swept through the whole field zone. So, the latter one is trapped remarkably much than the other one. As understood from Fig. 2, TSC curve presenting higher intensity (I_{TSC}) was observed if the positive terminal of the voltage supply was applied to illuminated surface of the sample. Otherwise, there was no strongly appearing TSC curve. Therefore, it was thought that the holes were distributed in the crystal and then trapped. This indicated that the observed TSC peak is associated with hole trap in GaS single crystal.

Concentration of filled traps gives significant knowledge about order of kinetics of the trapping levels during thermal release of trapped charge carriers. For first order of kinetics (slow retrapping), thermally released charge carriers from trap level recombine with opposite charge carriers without retrapping. Therefore, variation of concentration of filled traps does not influence the related peak shape and position. However, for non-first order of kinetics, possibility of retrapping of released charge carriers into trap center is much higher than recombination. In this case, variation of concentration of filled traps affects the peak shape and position [23]. Fig. 3 shows TSC curves obtained at a constant heating rate of 1.0 K/s by illuminating the sample at 10 K for different times increasing up to 100 s. As seen from Fig. 3, shape and peak maximum position of TSC peaks are not remarkably changed with illumination time. This means that first order of kinetics dominates the process. Intensities of TSC curves obtained under illumination times of 50 and 100 s are nearly same (see inset). So, the revealed trap level gets full as the sample is illuminated for 100 s. Taking under consideration this fact, all experiments were performed by illuminating the sample for 100 s.

Activation energy (E_t) of revealed trapping level was determined using curve fitting method applied taking under consideration the following theoretical equation giving TSC peak intensity as a function of temperature for first order of kinetic [23].

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

In this equation, C is a constant depending on shape, conductivity properties of the crystal and experimental conditions. T_0 is starting temperature, ν is attempt-to-escape frequency and β is heating rate. Fig. 4 presents experimental TSC peak having peak maximum temperature (T_m) of 148 K obtained at a constant heating rate of 1.0 K/s and its curve fit. Analyses by Eq. (1) applied to experimental data resulted with a successful fit and activation energy of the related trap level was found as 0.11 eV. Previously, some authors studied GaS single crystals to investigate its possible defects. Photoelectrical properties of *n*-type GaS crystals were investigated in Ref. [17,24], and two and one electron trap levels were obtained with energies 0.1 and 0.4 eV, and ~0.13 eV, respectively. *p*-type GaS single crystals were studied in terms of TSC measurements and analyses revealed the presence of six trapping levels at 0.05, 0.06, 0.12, 0.63, 0.71, and 0.75 eV [21]. The authors attributed shallowest hole traps at 0.05, 0.06, 0.12 eV to gallium vacancies (V_{Ga}) while deeper hole traps (0.63, 0.71, and 0.75 eV) were thought to be due to dislocations and/or stacking faults. In the TSC curve reported in Ref. [21], two peaks were observed below 100 K with very small intensities (≈0.06 nA). In the present study, TSC curve did not present peaks in the <100 K region. This point may be due to large background current (≈0.08 nA) observed in our experiments. Moreover, TSC experiments are very sensitive to electrical contacts. The another reason of this point may be due to applied and/or made electrical contacts. In addition, a computational method based on density functional theory was studied to determine intrinsic defects in GaS monolayer in Ref. [25]. According to authors, V_{Ga} and antisite S_{Ga} are the most probable intrinsic defects in *p*-GaS compound. Taking these reports into account, the revealed trap level in the band gap of GaS single crystals with activation energy of 0.11 eV found by curve fitting method was thought as native defect arising due to V_{Ga} or S_{Ga}.

Heating rate is an essential parameter affecting stimulation process of trapped charge carriers. Thus, investigating effect of heating rate on the observed TSC curve gives outstanding information about trapping mechanism. Figs. 5 and 6 indicate TSC spectra recorded for various heating rates between 0.4 and 1.0 K/s in the temperature ranges between 80 and 190 K, and between 190 and 280 K, respectively. As seen from Fig. 5, peak maximum temperature increases, the TSC intensity decreases and TSC peak gets wider with elevating heating rates to keep

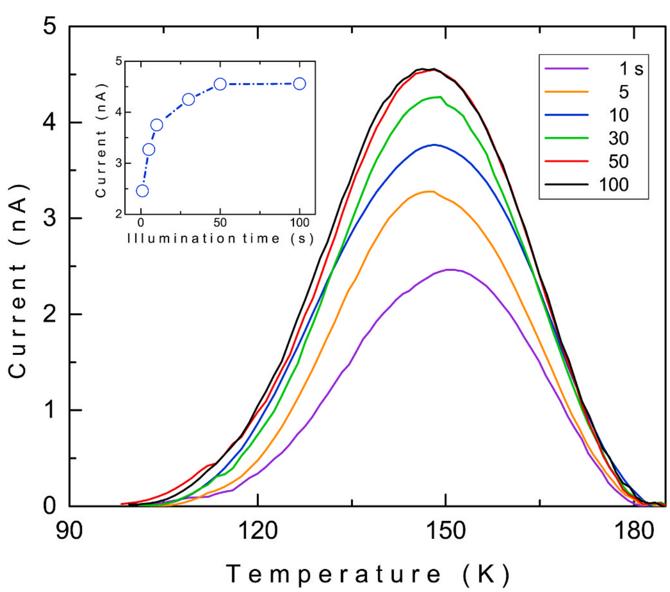

Fig. 3. TSC peaks of GaS crystals obtained at different illumination times in between 1 and 100 s with a constant heating rate of 1.0 K/s. Inset shows illumination time dependency of TSC intensity.

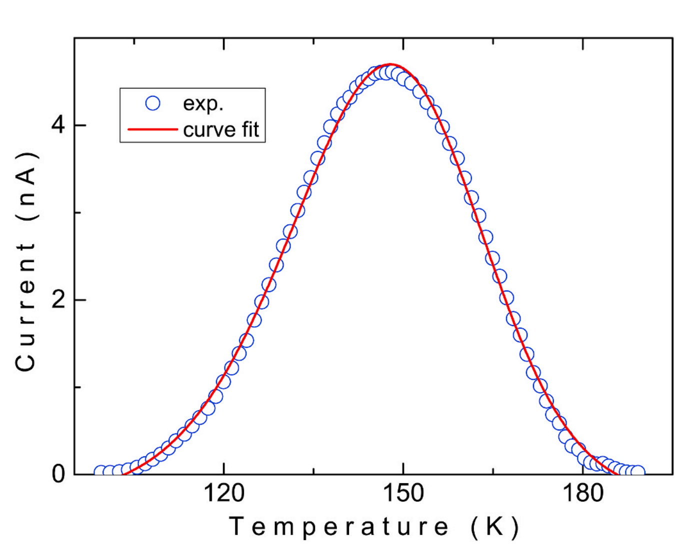

Fig. 4. Experimental TSC peak (circles) of GaS crystals observed in between 100 and 190 K at a constant heating rate of 1.0 K/s and its theoretical curve fit (solid curve).

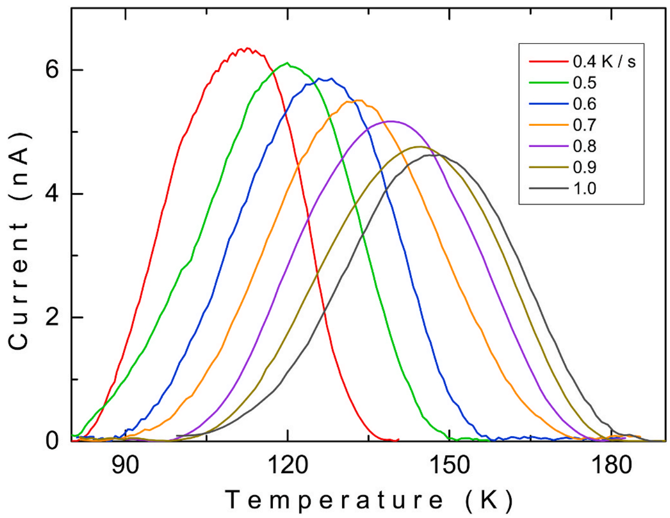

Fig. 5. Experimental TSC curves of GaS crystals recorded for various heating rates of 0.4–1.0 K/s in the temperature range of 80–190 K.

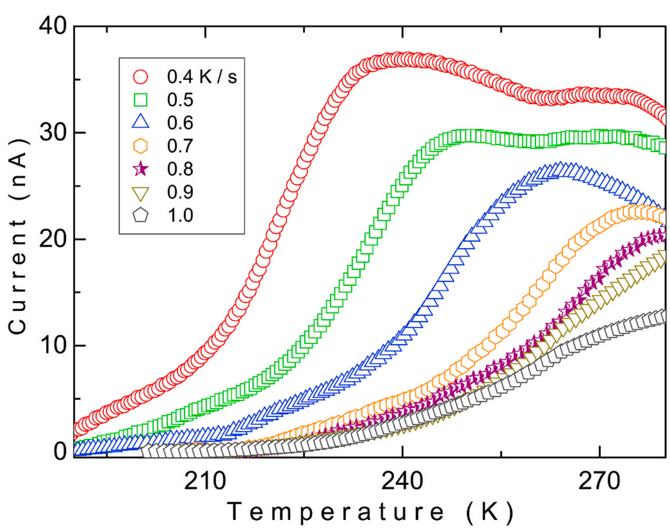

Fig. 6. Experimental TSC curves of GaS crystals recorded for various heating rates of 0.4–1.0 K/s in the temperature range of 190–280 K.

amount of released charges. The same behavior can be seen in Fig. 6. As heating rate of 0.4 K/s is employed, the TSC curve start to ascend around 190 K and lasts above 280 K. Since our experimental set-up allows us studying in the limited temperature region up to 280 K, we cannot detect the whole TSC spectrum which is enough to observe complete curve possibly terminating at temperature above 280 K. Further analyses on TSC curves appeared in temperature region of 190–280 K could not be achieved due to this reason and presence of overlapped few peaks. However, when kept track of ascending part of TSC curves at different heating rates of 0.4–1.0 K/s, it can be easily seen that TSC intensity decreases and T_m value shifts towards higher temperatures with heating rate. Such a shift of T_m may be explained as the following: at low heating rate β_1, trapped charge carriers occupy trap levels for a long time at temperature T_1 and an amount of those are stimulated into delocalized state at this temperature. Employing higher heating rate β_2 causes charge carriers to spend much less time at temperature T_1. In order to stimulate same amount of charge carriers with β_2, higher temperature of T_2 is needed. As a result, appearing TSC curve for higher heating rates is observed to be shifted to higher temperature side [26]. Alteration of the TSC intensity, T_m and full-widths-half-maximum (FWHM) values of TSC peak observed in the temperature region of 80–190 K are seen in Fig. 7. The TSC decreased from 6.4 down to 4.6 nA, the T_m increased from 113 to 148 K and FWHM values increased from 29 to 38 K with increasing heating rate.

Characteristics features of trapping level related to TSC peak having maximum at 148 K was also investigated by studying the distribution of traps through an experimental procedure based on different stopping temperature (T_{stop}). This exploration has importance to be supportive study revealing if the observed TSC peak is associated with a single, discrete trap level or distributed trapping centers. If the trap level is a single trap, T_m of consecutive TSC curves obtained at increased T_{stop} exhibits no change while the intensity decreases. If trapping level is a formation of continuously distributing traps, the T_m is expected to shift besides decreasing intensity of the TSC curve as higher T_{stop} is employed [23]. In order to elucidate which case is valid for revealed trapping level in GaS crystals, experimental process was achieved as explained in the following: the temperature was kept at $T_0 = 10$ K and the sample was illuminated for 100 s at applied bias voltage of 1 V. After an expectation time of 120 s was waited, the sample was heated up to T_{stop} value at bias voltage of 100 V. Then, the temperature was decreased to 10 K and TSC spectrum was detected in between 10 and 200 K at constant heating rate of 1.0 K/s at this voltage value. Consecutive TSC curves were obtained for different T_{stop} values between 50 and 110 K. Fig. 8 illustrates main TSC curve observed at 10 K and the TSC curves obtained at mentioned T_{stop} values at a constant heating rate of 1.0 K/s. It should be noted that this experimental procedure could not be applied to TSC curve appearing at temperature region above 200 K due to same reasons explained previously. As understood from the figure, employing $T_{stop} =$ 50 K led to shift of ascending part of TSC peak and slight decrease in the intensity as compared to main TSC peak obtained at 10 K. This trend robustly continued with increased T_{stop} values up to 110 K in which the intensity decreased almost eight times. Shift of T_m with increasing T_{stop} can be seen in the inset 1 of Fig. 8. This behavior of TSC curves was an indication that shallowest trapping levels were emptied at each sequential T_{stop} values and contribution to TSC curves was provided by charge carriers situated in deeper levels. Taking into T_m behavior due to increasing T_{stop} into account, the revealed trap level was ascribed to presence of quasi-continuously distributed energy levels in GaS crystals. Similar behavior was also observed in Ref. [27–30]. Activation energies of distributed levels were calculated applying curve fitting method to each successive TSC curves. Analyses resulted in increasing activation energy from 0.11 to 0.55 eV (see inset 2 of Fig. 8).

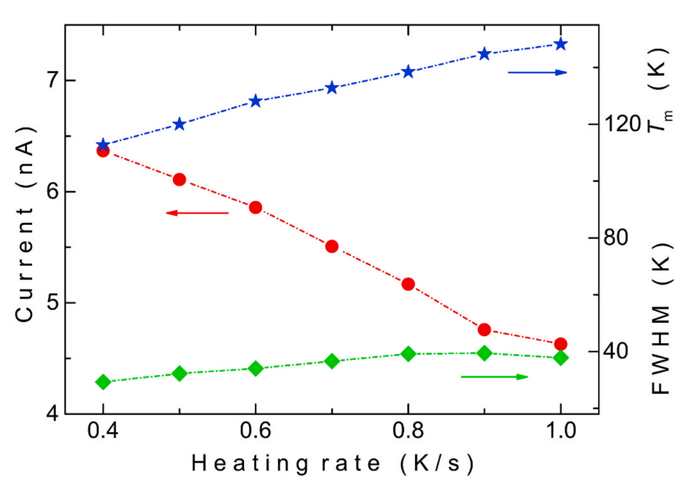

Fig. 7. Heating rate dependency of I_{TSC}, T_m and FWHM of TSC curve obtained in the temperature region between 80 and 190 K.

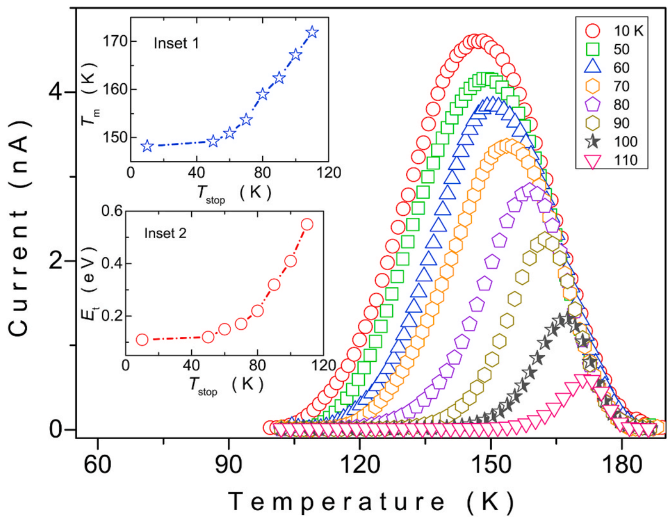

Fig. 8. TSC curves of GaS crystals obtained at different T_{stop} values ranging from 50 to 110 K at constant heating rate of 1.0 K/s. Inset 1 shows $T_m - T_{stop}$ dependency. Inset 2 shows activation energies found for each consecutive T_{stop} values.

4. Conclusion

TSC experiments established for GaS crystals showing *p*-type conductivity were performed in the temperature region between 10 and 280 K with various heating rates. TSC spectrum at 1.0 K/s exhibited an individual peak possessing maximum intensity at 148 K. Curve fitting method was applied to compute activation energy and analysis resulted in a presence of a trap level centered at 0.11 eV. In order to elucidate trapping mechanism, responses of TSC curve to variable heating rates and to different stopping temperatures were explored in detail. As a result of various heating rate measurements, TSC curve exhibited the well-known behavior that T_m and I_{TSC} increased and decreased with increasing heating rate, respectively. In virtue of thermally cleaning procedure with different T_{stop} between 50 and 100 K, quasi-continuously distributed energy levels within the revealed trap were found out with increasing activation energies from 0.11 to 0.55 eV. Obtained shallowest trap level was considered to originate from native defects in the form of V_{Ga} or antisite S_{Ga}.

Author statement

Serdar Delice: Investigation, Formal analysis, Conceptualization, Writing-Original Draft, Mehmet Isik: Investigation, Formal analysis, Conceptualization, Writing-Review and Editing. Nizami M. Gasanly: Supervision, Writing-Review and Editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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