Revealing Defect Centers in $PbWO_4$ Single Crystals Using Thermally Stimulated Current Measurements

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Abstract

The trap centers have significant impact on the electronic properties of lead tungstate ($PbWO_4$), suggesting their crucial role in optoelectronic applications. In the present study, we investigated and revealed the presence of shallow trap centers in $PbWO_4$ crystals through the utilization of the thermally stimulated current (TSC) method. TSC experiments were performed in the 10-280 K range by applying constant heating rate. The TSC spectrum showed the presence of a total of four peaks, two of which were overlapped. As a result of analyzing the TSC spectrum using the curve fit method, the activation energies of revealed centers were found as 0.03, 0.11, 0.16 and 0.35 eV. The trapping centers were associated with hole centers according to comparison of TSC peak intensities recorded by illuminating the opposite polarity contacts. Our findings not only contribute to the fundamental understanding of the charge transport mechanisms in $PbWO_4$ crystals but also hold great promise for enhancing their optoelectronic device performance. The identification and characterization of these shallow trap centers provide valuable insights for optimizing the design and fabrication of future optoelectronic devices based on $PbWO_4$.

Keywords: $PbWO_4$, $PbMoO_4$, defects, optoelectronic applications, thermally stimulated current, thermoluminescence

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1. Introduction

Lead tungstate ($PbWO_4$) is a widely recognized inorganic compound prized for its distinctive structural and optical characteristics, making it valuable in various applications. $PbWO_4$ is classified within the tetragonal crystal system, featuring a crystal structure comprising Pb^{2+} cations and WO_4^{2-} anions.[1,2] The arrangement of WO_4^{2-} anions occurs in a tetrahedral manner, while the spaces between the tetrahedra are occupied by Pb^{2+} cations. The strong bonding between the WO_4^{2-} anions and Pb^{2+} cations contributes to the compound's robust and unyielding crystal lattice. Previous studies have reported the compound's lattice parameters as $a = b = 5.42$ Å and $c = 12.04$ Å.[3] $PbWO_4$ demonstrates notable characteristics such as a high refractive index and a wide bandgap of 3.20 eV, rendering it valuable for applications in scintillators, X-ray detectors, and optoelectronic devices.[4-7] The material's high refractive index facilitates efficient light collection and detection, while its wide bandgap contributes to low dark noise and superior energy resolution. Due to its high density, $PbWO_4$ proves to be an exceptional choice for radiation detection and imaging applications.[6] Its density allows for effective absorption and attenuation of ionizing radiation, making it particularly suitable for deployment in nuclear medicine and radiological applications.[8] Extensive studies have focused on optimizing $PbWO_4$ for diverse applications, while ongoing research endeavors aim to exploit its properties for novel applications across various fields. $PbWO_4$ serves as a suitable host material for rare earth elements, enabling the development of visible light emitting phosphors for applications such as light emitting diodes. Notably, $PbWO_4$ doped with Sm^{3+} emits orange-red light, while $PbWO_4$ doped with Yb^{3+}:Ho^{3+}:Tm^{3+} displays multi-color luminescence.[9,10]

The revealing of defect centers in semiconductor materials is of great importance for technological applications. Defects have the ability to affect the electrical, optical and structural properties of semiconductors. Identifying defect centers allows researchers to develop strategies to improve the performance of the devices they design using these semiconductor materials. It is necessary to reveal defect centers in order to determine important points for technological applications such as performance optimization, design, engineering, reliability, durability, process optimization of materials.[11,12] Lead and oxygen vacancies, lead Frenkel defects, color centers arising from the presence of impurities and structural defects, radiation-induced defects are some of the types of defects presented in $PbWO_4$.[13-15] One of the methods used to determine defect centers in semiconductor and insulating materials is thermally stimulated current (TSC).[16-18] TSC measurements are a powerful and well-known experimental method for studying defects in materials, revealing parameters of trap centers. In this method, the trapping

of the charge carriers in the trap centers in any low-temperature material is provided as a result of irradiation of the material with a suitable source. Then the radiation is terminated and the material is heated in the dark and the trapped charge carriers are provided to the excitation with the help of heat energy to the delocalized bands. These excited charge carriers contribute to the measured current which can be analyzed to reveal information about the defect centers/trap levels. Our aim in this study is to determine the shallow trap centers of $PbWO_4$ single crystals, which have never been examined before, by TSC method performed in the temperature range of 10-280 K. As a result of the analysis of the observed TSC spectrum, the activation energies of the shallow trap centers were determined. The results obtained in this paper will make a significant contribution to the studies on optoelectronic applications of $PbWO_4$ compound.

2. Experimental details

$PbWO_4$ single crystals were produced using the commonly employed Czochralski method. Starting materials, namely high-purity lead oxide (PbO) and tungsten oxide (WO_3), were meticulously weighed to achieve the desired $PbWO_4$ composition. These materials were combined in an appropriate stoichiometric ratio and placed within a platinum crucible. Subsequently, the crucible, containing the materials, was subjected to high temperatures exceeding 1000 °C in an oxygen-rich environment within a furnace. Continuous stirring of the melt ensured thorough homogenization of the components. A rod with a seed crystal of $PbWO_4$ attached to it was immersed into the melt. The seed crystal was gradually extracted from the melt at a controlled rate of 2 mm/h, while the melt maintained a constant temperature. As the seed crystal ascended, the formation of a $PbWO_4$ single crystal occurred through the crystallization process originating from the melt. Throughout the growth procedure, precise control of rotation and cooling rates was exercised, maintaining values of 20 rpm and 30 °C/h, respectively. Following growth, the $PbWO_4$ single crystal underwent polishing to achieve a reflective surface. To determine its structural properties, X-ray diffraction analysis was performed employing a Rigaku Miniflex diffractometer utilizing a Cu-Kα (λ = 1.54Å) radiation source.

To enable electrical contact, a sandwich geometry configuration was adopted, utilizing opposite surfaces of the sample (see inset of Figure 1). The front surface was connected with a conductive copper wire using silver paste, while the back surface was secured to a sample holder that was grounded. Low-temperature TSC experiments were conducted within a closed-cycle helium gas cryostat (Advanced Research Systems, Model CSW 202), which maintained the sample temperature between 10 and 280 K. A light-emitting diode was affixed to the

cryostat's quartz window. During the TSC measurements, the sample temperature was lowered to T_0 = 10 K, and the front surface was exposed to light with energy exceeding the material's band gap for 600 seconds at T_0. A bias voltage of V_1 = 1 V was applied during illumination, utilizing a Keithley 228 A voltage/current source. Following the cessation of the light source and a brief period in darkness, the sample temperature was raised to 280 K at a constant heating rate of 1.0 K/s, employing a Lake-shore temperature controller and maintaining a bias voltage of V_2 = 100 V. The resulting current during the heating process was measured using a Keithley 6485 picoammeter. All measuring devices were controlled through a project prepared with the Labview graphical development program and TSC measurements were completed.

3. Results and discussions

In our recent papers, we have presented the linear, nonlinear, spectroscopic ellipsometry and temperature-dependent band gap energy properties of the $PbWO_4$ crystal studied in the present paper to obtain information about trap centers. As a result of the analysis of temperature-dependent transmission measurements, it was seen that when the temperature was reduced from 300 to 10 K, the band gap energy increased from 3.20 to 3.35 eV.[4] A photograph of our crystal was also presented in the same reference, and it was seen that the crystal exhibiting high quality optical characteristics was brittle and slightly yellowish. Transmittance and reflectance experiments were performed to get linear and nonlinear optical characteristics of the $PbWO_4$ crystal.[19] Bandgap energy, static refractive index, static dielectric constant, single oscillator and dispersion energies, Urbach energy, nonlinear refractive index, first-order and third-order nonlinear susceptibilities of the compound were reported in the study. Spectral dependencies of various optical parameter like components of complex dielectric function, dielectric relaxation time, dissipation factor, surface and volume loss functions of the $PbWO_4$ crystal were reported utilizing ellipsometry measurements.[20]

Some of the grown crystals were pulverized for XRD measurements and the measurements were performed on the powder form of the material. Since the orientations will be limited in the measurements taken on the crystal, this causes very few (usually one or two) peaks to appear in the XRD pattern. In the case of powder, since different powder particles exhibit different orientations, more diffraction peaks are visible in the XRD pattern. The XRD pattern of the powder form of the grown $PbWO_4$ crystal, depicted in Figure 1, exhibited seven distinct diffraction peaks, located approximately at 28.90º, 46.95º, 49.20º, 53.75º, 57.80º, 58.95º, and 75.60º. These peaks corresponded to the (400), (204), (220), (116), (312), (224),

and (404) planes, respectively, signifying their association with the tetragonal crystalline structure, as specified by the JCPDS Card No: 08-0476.

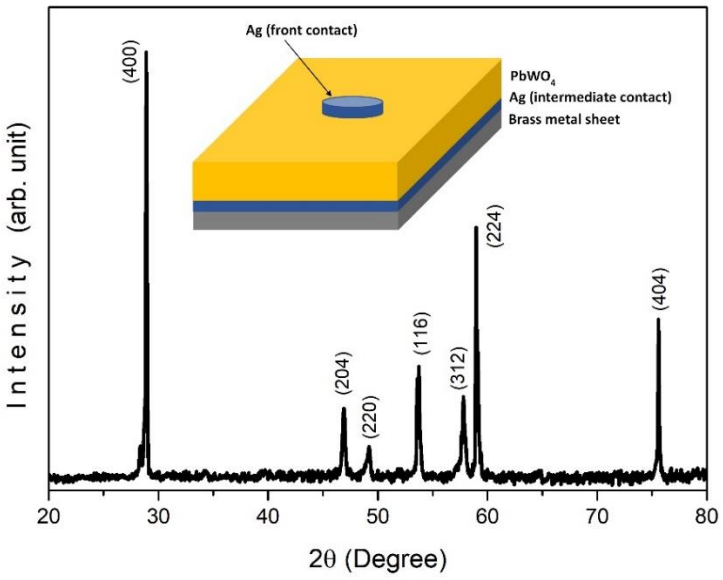

Figure 1. XRD pattern of the $PbWO_4$ powder.

TSC measurements were conducted on $PbWO_4$ crystal over a temperature range of 10-280 K, employing a constant heating rate of β = 1.0 K/s. In these measurements, three peaks emerged with maximum temperatures of approximately 86, 154 and 237 K, as depicted in Figure 2a. In addition to these peak points, it can be seen that a peak of around 175 K overlaps with a peak of around 150 K. Positive and negative voltage polarities were applied separately to the illuminated surface of the material to obtain information about the type of trap center (hole or electron trap) and TSC curves were recorded at each configuration. Figure 2b gives an example of a schematic representation in which the illumination is applied to the positive contact. Figure 2a illustrates the TSC curves of $PbWO_4$ crystal obtained under forward and reverse bias conditions for the observed trapping center. In this experimental approach, the contact on the illuminated surface of the crystal was linked to either the positive or negative terminals of the applied voltage, and TSC experiments were conducted for both scenarios. Illuminating the crystal surface generated both types of charge carriers; however, only one type was propelled throughout the entire field zone, while the opposite type was swiftly collected depending on the bias voltage.[21] Only the illuminated terminal type carrier remained trapped throughout the excitation. Figure 2 revealed that when the polarity of the illuminated contact was positive, the TSC curve exhibited the highest intensity, indicating the distribution and

subsequent trapping of holes in the sample. Consequently, the observed peaks can be characterized as a hole trap.

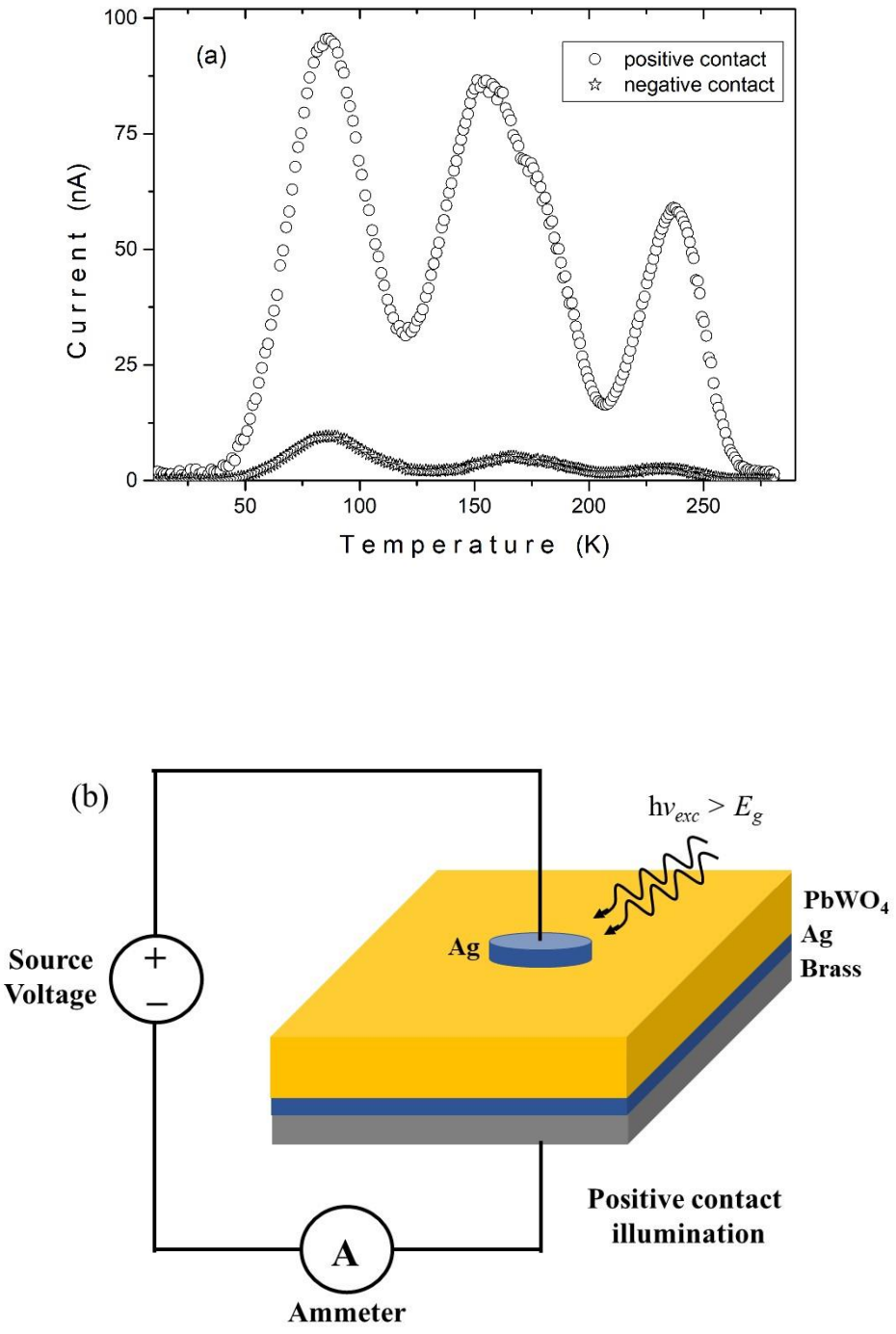

Figure 2. (a) TSC glow curves obtained for illumination of positive and negative contacts. (b) Schematic representation of the illumination of the positive contact.

Multiple techniques exist for determining the activation energies of trapping centers, and in this study, both the curve fitting and initial rise methods were employed. The curve fit method is theoretically based on the fitting of the TSC curve, whose temperature-dependent function is defined, with appropriate software programs. The temperature-dependent current (I_{TSC}) functions of any TSC peak are given as follows[22]

$$I_{\mathrm{TSC}} = Cexp\left\{-\frac{E_{\mathrm{t}}}{kT} - \int_{T_0}^{T}\frac{\nu}{\beta}\exp\left(-\frac{E_{\mathrm{t}}}{kT}\right)dT\right\} \quad \text{(first-order kinetics)} \quad (1)$$

$$I_{\mathrm{TSC}} = Cexp\left(-\frac{E_{\mathrm{t}}}{kT}\right)\left[1+(b-1)\frac{n_0\nu}{\beta N}\int_{T_0}^{T}\exp\left(-E_{\mathrm{t}}/kT\right)dT\right]^{-\frac{b}{b-1}} \quad \text{(non-first order kinetics)} \quad (2)$$

where C is a constant, n_0 is initial trapped concentration, N is total concentration of the trap centers, ν is attempt-to-escape frequency, and b is order of kinetics parameter which gets value $1< b \leq 2$ for non-first order of kinetics. First and non-first order kinetics correspond to the cases of slow and fast retrapping, respectively. When slow retrapping occurs, there is a higher likelihood for the electrons that are excited to the valence band from trap centers to contribute to the current rather than undergoing retrapping. The dominant processes in situations involving slow or negligible retrapping involve the release of holes from the trapping centers and their contribution to the current when an electric field was applied to the sample. In the case of fast retrapping, the holes are more likely to be trapped again. Specifically, when the curve fitting method was utilized on the TSC curve concerning four trapping centers, a satisfactory alignment between the theoretical considerations and experimental data was achieved (see Figure 3). The fitting process proved successful, particularly in cases involving slow retrapping. This outcome suggests that retrapping is negligible for the identified traps in the $PbWO_4$ crystal. In Figure 3, the solid line represents the experimental data, and it exhibits a well-fitted four peaks characterized by activation energies of E_{tA} = 0.03 eV, E_{tB} = 0.11 eV, E_{tC} = 0.16 eV and E_{tD} = 0.35 eV. The peak maximum temperatures of the deconvoluted curves were found from the fitting process as T_A = 86.3 K, T_B = 149.4 K, T_C = 178.1 K and T_D = 237.5 K.

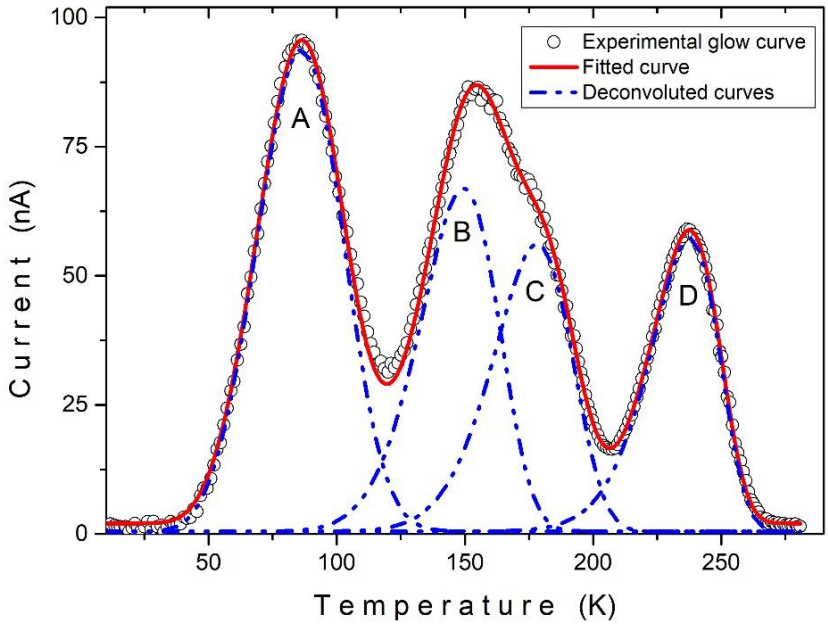

Figure 3. Experimental TSC spectrum of $PbWO_4$ crystal. Open circles are experimental data. Solid and dash-dotted curves show the total fit to the experimental data and deconvoluted curves, respectively.

In thermally stimulated current (TSC) experiments, sometimes multiple TSC peaks can overlap, making it difficult to accurately distinguish and analyze individual peaks. To address this issue, a thermal cleaning process is often used to separate the overlapping peaks.[22,23] The thermal cleaning process is applied by heating the material to a certain cleaning temperature (called as $T_{cl.}$), then lowering it back to its initial low temperature, and finally taking measurements over the entire temperature range without any additional lighting. When we apply it in this way, it is ensured that the trap centers where the escape from the trap centers begins/continues/ends at the temperature of T_{cl} may be emptied and others are not affected. As a result of such an application, overlapping peaks are separated. As a result of the fitting of the curve, which appears to be a single peak around 150 K in Figure 3, it was understood that this peak consisted of two single peaks. To prove this and increase the reliability of the fit process, the thermal cleaning method was used. Looking at the individual peaks in Figure 3, it is seen that the excitation of the charge carriers from the trap center related to peak B starts from ~100 K while excitation of the charge carriers from the trap center related to peak C starts from ~125 K. Several different cleaning temperatures were applied to select a temperature at which peak B was emptied as much as possible and peak C was not affected as much as possible. TSC

curves obtained as a result of different cleaning temperatures were examined and it was seen that the most suitable cleaning temperature was 120 K. The TSC curve obtained by using a cleaning temperature of $T_{cl.}$ = 120 K is shown in Figure 4. This curve revealed the presence of two peaks around 175 and 237 K. It can be easily said that these two peaks belong to the C and D trap centers revealed from the fit in Figure 3. The TSC curve obtained as a result of thermal cleaning was analyzed using the curved fit method, and the activation energies and peak maximum temperatures of the observed peaks were determined as E_{tC} = 0.17 eV, E_{tD} = 0.36 eV, T_C = 179.1 K and T_D = 238.1 K.

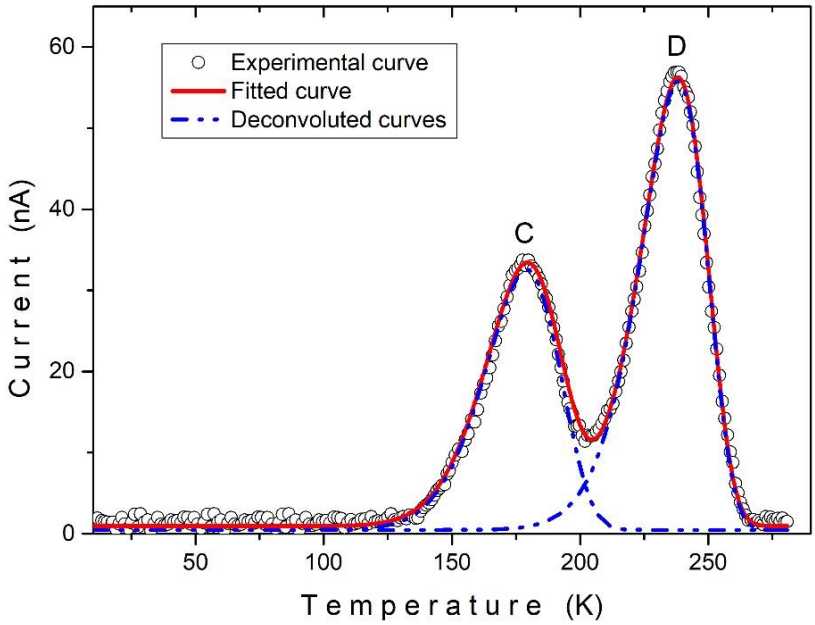

Figure 4. Experimental TSC spectrum (open circles) of $PbWO_4$ crystal after applying thermal cleaning process.

The primary approach for assessing the activation energies of trap levels is the initial rise method.[24,25] This method revolves around the observation that the integrals in the equations of both first and non-first order reactions (Eqs. 1 and 2) become significantly small when charge carriers begin to transition from trap levels. Consequently, the exponential terms within these integrals closely approximate unity within this initial temperature range. As a result, the TSC (thermally stimulated current) intensity can be expressed as follows

$$I_{\mathrm{TSC}} = Cexp(-E_t/kT). \quad (3)$$

According to this equation, the ln(I_{TSC}) vs. 1/T plot, which exhibits a linear behavior for the first part where the TSC peak starts to rise, can be used to find the activation energy. In this

method, when the relevant graph for the overlapping peaks is plotted, only the activation energy of the first peak is obtained. Therefore, we used the initial rise method to find the activation energies of peak A from the TSC curve (Figure 3) we obtained without thermal cleaning and peak C from the TSC curve (Figure 4) we obtained after thermal cleaning. Figure 5 shows the drawings of ln(I_{TSC}) *vs. 1/*T obtained using the TSC curves in the respective figures. As a result of the linear fitting of the plots, the activation energies of peak A and C were found to be E_{tA} = 0.03 eV and E_{tC} = 0.16 eV. The compatibility of these values with the values obtained as a result of the curve fit method can be considered as evidence of the reliability of the analysis results.

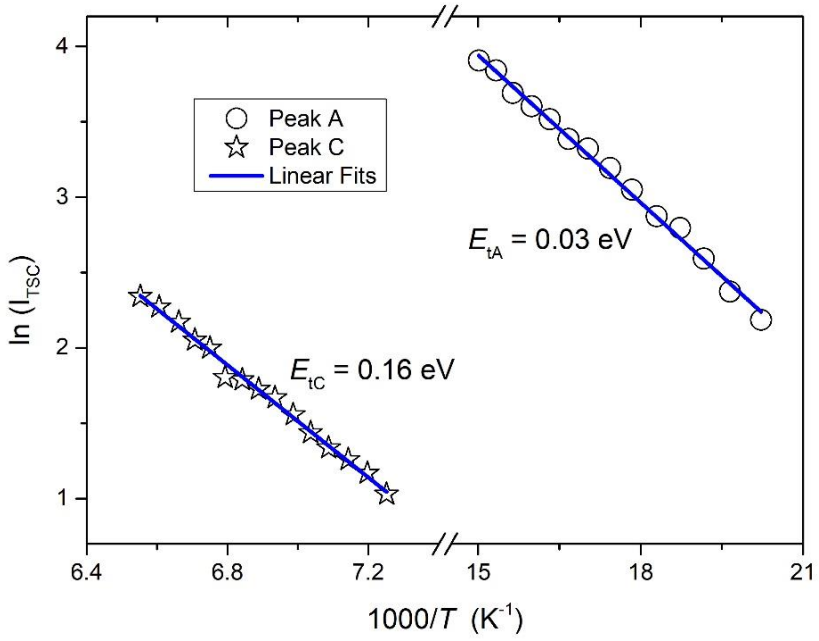

Figure 5. Initial rise method application plots according to Eq. (3) for peaks A and C.

At this point, it is useful to make a general assessment of the trap centers revealed in the $PbWO_4$ crystal. The main intrinsic defects that exist in the $PbWO_4$ compound are the oxygen and lead vacancies. In the study on thermally stimulated luminescence (TSL) measurements of the $PbWO_4$ crystal, it was noted that the peaks that appeared in the range of 100-300 K on the TSL curve belonged to hole trap centers, especially those associated with WO_3-type oxygen vacancies.[13] It was also reported that the locations of the WO_4 groups in the lead-deficient crystal regions could be linked to the hole centers that contribute to the thermally stimulated green emission. In another paper on TSL studies of $PbWO_4$ single crystals, two peaks around 178 and 170 K were observed when the sample was irradiated at 85 K with 4.7 eV irradiation

source.[14] When the irradiation was applied at 140 K, two peaks around 178 and 192 K were observed. Authors proposed that the peak around 190 K with activation energy of 0.43 eV is responsible for the thermal destruction of the hole O^-(I) type centers. Furthermore, the paper also linked the activation energy of 0.14 eV to the formation of O^-(I) type centers, which are created when excitons near La^{3+} or Y^{3+} ions associated with V_{Pb} disintegrate.

4. Conclusion

In order to determine the shallow trap centers of $PbWO_4$ single crystals, the TSC method was applied in the temperature range of 10-280 K. The TSC spectrum showed peaks indicating the presence of four trap centers. As a result of analyzing the observed TSC peak using the curve fit method, the peak maximum temperatures and activation energies of revealed centers were found as follows: E_{tA} = 0.03 eV, E_{tB} = 0.11 eV, E_{tC} = 0.16 eV, E_{tD} = 0.35 eV, and T_A = 86.3 K, T_B = 149.4 K, T_C = 178.1 K, T_D = 237.5 K. Using the thermal cleaning method, the overlapping B and C peaks were separated from each other, ensuring the reliability of the curve fit analysis results. When the TSC method was applied, the positive and negative contacts were illuminated separately. When the positive contact was illuminated, the observed peak was found to have a higher intensity/current. This showed that the revealed trap centers were hole-type. By unraveling the shallow trap centers in the $PbWO_4$ crystal, we open up avenues for optimizing the design and fabrication of optoelectronic devices, leading to enhanced functionalities and superior performance in various applications such as solid-state lighting, photovoltaics, and optical sensors.

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