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Edition 1.0 2020-04

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 8-1: Nano-enabled metal-oxide interfacial devices – Test method for defect states by thermally stimulated current

IEC TS 62607-8-1:2020-04(en)

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 8-1: Nano-enabled metal-oxide interfacial devices – Test method for defect states by thermally stimulated current

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Technical Specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC TS 62607-8-1, which is a Technical Specification, has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems.

The text of this Technical Specification is based on the following documents:

DTS	Report on voting
113/493/DTS	113/510/RVDTS

Full information on the voting for the approval of this Technical Specification can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 62607 series, published under the general title *Nanomanufacturing – Key control characteristics*, can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

Thermally stimulated current (TSC) measurement has been a simple and widely used method to get information about charge trapping and electric polarization phenomena of various materials such as dielectrics, ferroelectrics, semiconductors, ceramics, plastics, and other organic materials for the past several decades. Recently, TSC measurement has been recognized as a versatile tool to evaluate defect states and structures in advanced electronic materials including nano-enabled materials and devices. The defect states in devices such as metal-oxide interfacial devices, C-60 FETs, organic LEDs and emerging photovoltaic cells act as charge carrier traps influencing their performance and reliability. As such, a standardized protocol for TSC measurement will be useful to add validity of the experimental data for the purposes of productization of nano-enabled materials and devices. The reference sample for the reproducible TSC measurement is also required.

This document offers a measurement method to be developed for determining defect states of nano-enabled metal-oxide interfacial devices, which is suitable for evaluating the electronic state even though the resistance of the device changes widely.

NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –

Part 8-1: Nano-enabled metal-oxide interfacial devices –
Test method for defect states by thermally stimulated current

1 Scope

There are two types of thermally stimulated current (TSC) measurement methods, classified by the origin of the current. One is generated by the detrapping of charges. The other one is generated by depolarization. This part of IEC 62607 focuses on the former method, and specifies the measurement method to be developed for determining defect states of nano-enabled metal-oxide interfacial devices.

This document includes:

- outlines of the experimental procedures used to measure TSC,
- methods of interpretation of results and discussion of data analysis, and
- case studies.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 80004-1, *Nanotechnologies – Vocabulary – Part 1: Core terms*

3 Terms, definitions, and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1.1
device under test
DUT
representative sample device used in testing

[SOURCE: IEC 62876-2-1:2018, 3.1.2, modified – In the definition, the word "sample" has been added.]

3.1.2
thermally stimulated current
TSC
current flowing through an external circuit connecting to DUT, originated from the electricity trapped at low temperature and released due to raising temperature

3.2 Abbreviated terms

DUT	device under test
sccm	standard cubic centimetres per minute
TSC	thermally stimulated current
GaAs	gallium arsenide
Ta_2O_5	ditantalum pentaoxide, tantalum oxide

4 Measurement of TSC

4.1 General

The typical test set-up for measuring TSC is shown in Figure 1.

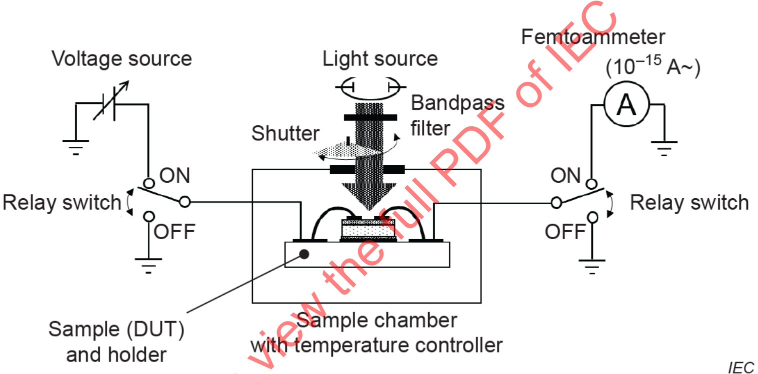

Figure 1 – Structure of TSC measurement device

Temperature dependence of TSC value shall be expressed by plotting TSC (A) on the ordinate against temperature on the abscissa.

4.2 Sample preparation

TSC signal is very sensitive to physical and chemical conditions at the sample/electrode interface. The surface preparation before the electrode formation process should be mentioned in the standardization protocol, as shown in Figure 1 and Table 1.

4.3 Experimental procedures

- Step 1: Conditioning (Pre-treatment conditions) – Restore the sample to a state where all trapped charges are released.
- Step 2: Cooling – Cool the sample to the trapping temperature.
- Step 3: Holding time – Keep the sample at the trapping temperature to stabilize the sample condition.
- Step 4: Trapping – Done by optical injection or voltage injection, or a combination of both.
- Step 5: Measurement – Measure TSC while heating up to the targeted end temperature.
- Step 6: Ending (Post-measurement treatment) – Set back to the room temperature before taking the sample out from the sample chamber.

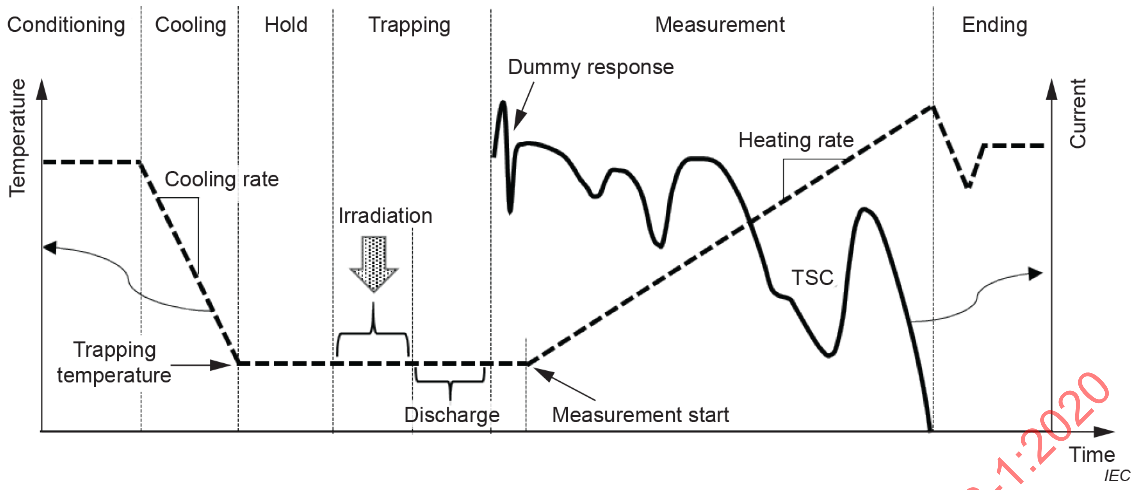

Figure 2 – Visualization of TSC measurement sequence

Table 1 – TSC measurement sequence steps and parameters

Step		**Parameter 1**	**Parameter 2**	**Parameter 3**	**Parameter 4**
1	Conditioning (Pre-measurement treatment)	Conditioning temperature	Holding time	Releasing voltage	
2	Cooling	Trapping temperature	Cooling rate		
3	Hold	Holding time			
4	Trapping (photoexcitation)	Wavelength (bandpass filter)	Light irradiation time	Holding time	Discharge time
	Trapping (Voltage/Current injection)	Applied voltage	Current	Holding time	Discharge time
5	Measurement	End temperature	Heating rate	Collecting voltage	
6	Ending (Post-measurement treatment)	Target temperature	Heating rate	Holding time	

5 Reporting data

- Measurement parameters shown in Table 1.
- Sample description.
- TSC measurement configuration.
- TSC signal as a function of temperature.

6 Data analysis / interpretation of results

6.1 General

In order to evaluate defect states by using TSC spectra, the temperature dependence is analysed. The possible methods are shown in 6.2, 6.3 and 6.4. The typical example of TSC measurement and the analyses are shown in Annex A. The detailed descriptions of the methods are shown in Annex B.

6.2 Peak method [1][1]

A well-known method to approximate trap energy level, through extracting the slope from an Arrhenius plot.

6.3 T_{start}–T_{stop} method [2] [3]

This method is best effectively used when trap temperature peaks are convoluted and need to be separated (e.g. useful for TSC measurements of organic semiconductors). The method should be applied in the case that the energy distribution is narrow; for example, the single or the deconvoluted TSC peak originated from a single trap level.

NOTE The method is also called fractional TSC measurement or thermal sampling.

6.4 Initial rise method [4]

When considering a small area of the rising slope of a peak in a TSC curve, the approximate trap energy level can be determined from the slope. The method should be applied in the case that the energy distribution is narrow; for example, the single or the deconvoluted TSC peak originated from a single trap level.

NOTE The method is a useful way to apply the TSC method for the quality control, because the data analysis can be performed only by one heating-up step.

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[1] Numbers in square brackets refer to the Bibliography.

Annex A
(informative)

Case study

A.1 TSC measurement of Au/GaAs (reference sample)

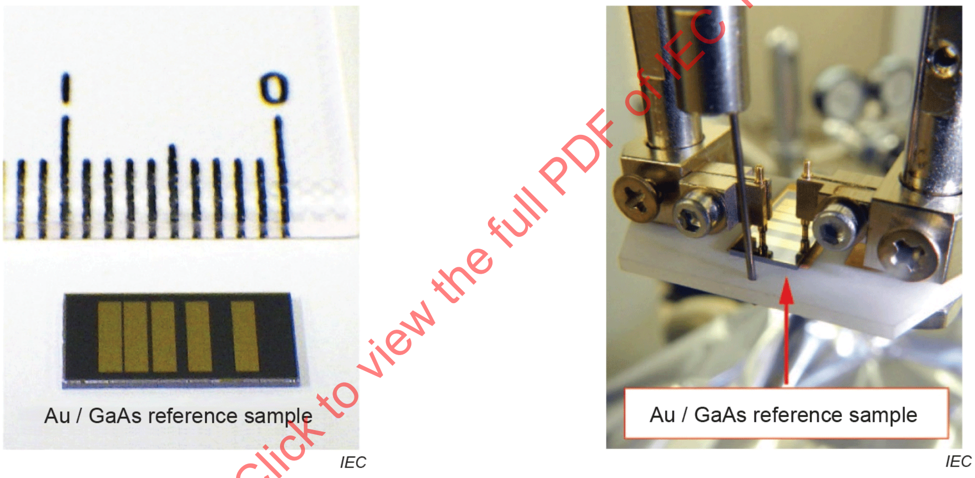
A.1.1 General

Basic TSC measurement sequences are shown in Figure A.1 to Figure A.4 and Table A.1. The measurement sequence described below is summarized in steps. To obtain the reliable TSC spectra, a sample interface treatment is highly important as TSC signal is very sensitive to the interface. As a case study, three types of samples in different interface treatment are prepared and compared:

a) no interface treatment;

b) with the passivation treatment by ammonium sulfide ($(NH_4)_2S_x$);

c) with the cleaning treatment by tetra methyl ammonium hydroxide.



a) **b)**

Figure A.1 – Photos of (a) the Au electrode configuration on GaAs reference sample, and (b) sample setting

- Step 1: Pre-treatment conditions

  Suitable pre-treatment conditions vary on the material being measured, and can change depending on the situation or environment. However, to obtain reliable TSC spectra and reproducible results, restoring a sample to a state where all trapped charges are released is the most important factor to consider before beginning any measurement. Example conditioning methods are as below.

  – Bake or perform annealing in a controlled environment to vaporize absorbed water on the sample surface.

  – Acclimate a sample in a sample chamber for a long time. The temperature in the sample chamber must be well-controlled.

  When performing the sample conditioning in a TSC measurement device, replace the chamber air with He gas before acclimating samples as the atmospheric moisture might affect TSC measurement results. An example of a conditioning protocol is heating up to 368 K and holding for 5 h while applying 0,1 V to release all trapped charge.

- Step 2: Cooling

  Set trapping temperature (Parameter 1) and cooling rate (Parameter 2), then cool the sample to the trapping temperature. Cooling rate can be set at will, but the fastest speed, 20 K/min, is recommended. In this case, a manual handling of liquid nitrogen refilling is also needed to control the cooling rate.

- Step 3: Hold

  When coming down to the trapping temperature, hold it for 20 min to 40 min to stabilize the sample condition.

- Step 4: Trapping

  Trapping is done at low temperatures by voltage injection or optical injection, or a combination of both so that the carriers can remain frozen in the trap levels. When performing carrier injection after the first holding time, turn the voltage-source switch and the ammeter relays on (Figure A.2), then observe the current (Parameter 2) while applying an injection voltage (Parameter 1).

  If the carrier is generated by photoexcitation, select an appropriate bandpass filter (Parameter 1, Figure A.2) for wavelength adjustment, then irradiate light for a set time period (Parameter 2) to the sample. Light irradiation time should always be less than or equal to holding time. Even when performing only light irradiation, it is possible to observe the current output during optical injection by leaving the ammeter relay active.

  After the trapping phase, turn both the voltage source and ammeter relay off, and release the excessive trapped charge which is not fixed at low temperature.

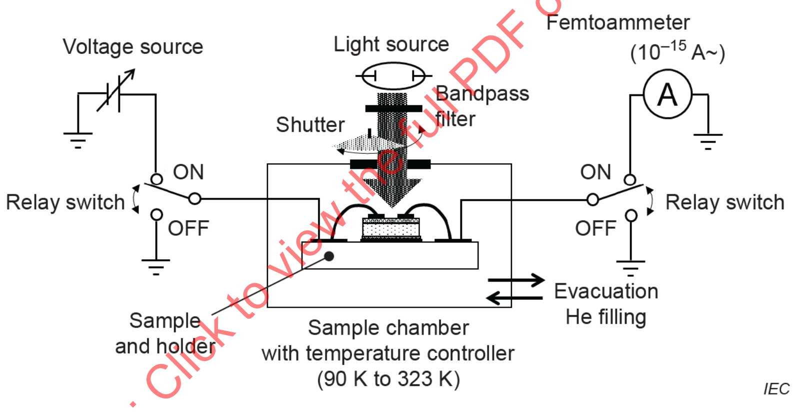


Figure A.2 – Structure of TSC measurement device

- Step 5: Measurement

  TSC measurement is done at the heating-up phase. Firstly, turn the voltage-source switch and the ammeter relays on. Then, apply the collecting voltage (Parameter 3) while temperature is rising up to the end temperature (Parameter 1). Take a dummy response step in the first minute since a quick current response occurred right after turning on the ammeter relay due to the induction response.

  In general, the polarity of the injection voltage and collecting voltage should be opposite. However, in case of TSC measurement, this can be flexible depending on a sample condition and/or a measurement protocol. Before using a new sample, it is important to find an optimum measurement protocol including the polarity of the injection/collecting voltage.

- Step 6: Post-measurement treatment

  After completion of the measurement, the temperature of the sample chamber easily drops to below freezing temperature as the liquid nitrogen still remains in the cooling container. Hence, before taking the sample out of the sample chamber, the chamber should be once heated up to the target temperature (Parameter 1) and cooled down to the room temperature by setting an appropriate holding time (Parameter 3).

  Recommended setting in this measurement case (cooled down to 90 K): Heating up to 353 K at 5 K/min, holding time of 100 min.

Table A.1 – TSC measurement sequence steps and parameters / case study

Step		Parameter 1	Parameter 2	Parameter 3	Parameter 4
1	Conditioning (Pre-measurement treatment)	Conditioning temperature 368 K	Holding time 5 h	Releasing voltage 0,1 V	
2	Cooling	Trapping temperature 90 K	Cooling rate 20 K/min		
3	Hold	Holding time 30 min			
4	Trapping (photoexcitation)	Wavelength (bandpass filter) 630 nm	Light irradiation time 300 s	Holding time 5 min	Discharge time 5 min
	Trapping (Voltage/Current injection)	Applied voltage 0 V	Current 0 A	Holding time 0 min	Discharge time 0 min
5	Measurement	End temperature 323 K	Heating rate 3 K/min to 20 K/min	Collecting voltage 0,2 V	
6	Ending (Post-measurement treatment)	Target temperature 353 K	Heating rate 5 K/min	Holding time 100 min	

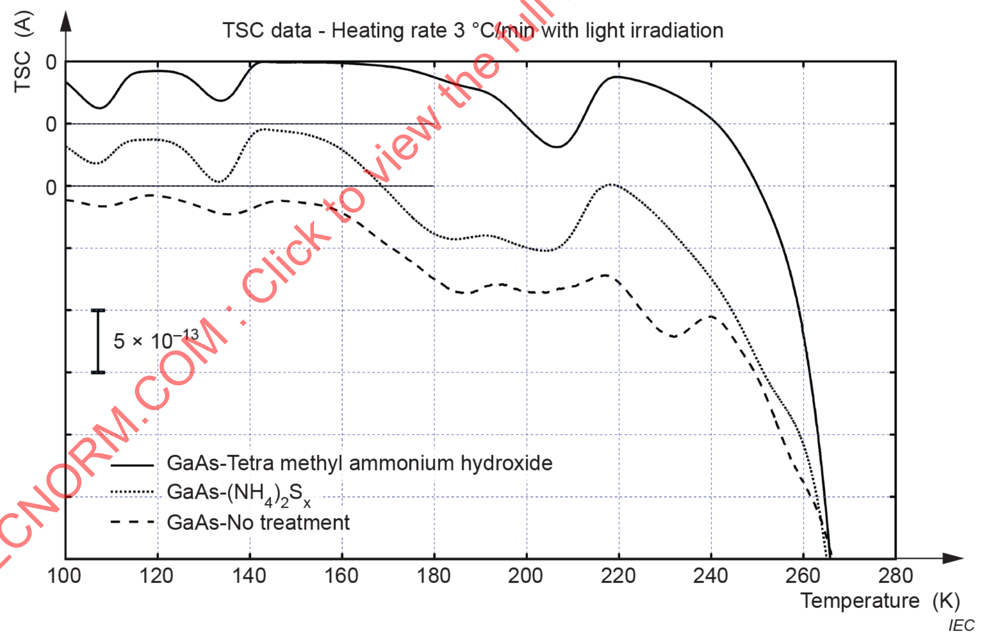


Figure A.3 – TSC data comparison by samples

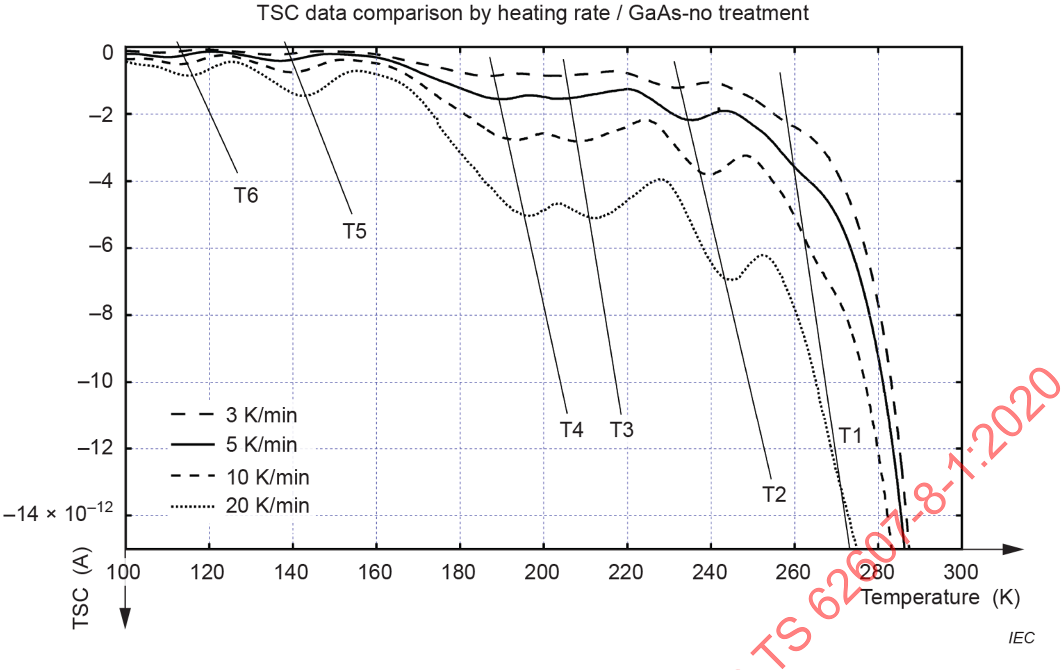


Figure A.4 – TSC data comparison by heating rate

According to the previous study [5], T2 is assigned as an electron trap, while T3, T4, T5, T6 are hole traps. T3, T4, T5, T6 can be identified as $\mathrm{As_{Ga}}^{++}$, $\mathrm{Ga_{As}V_{Ga}}$ (or $\mathrm{V_{As}}$), $\mathrm{V_{Ga}}$ and $\mathrm{Ga_{As}}^{--}$, respectively. As shown in Figure A.3, the surface preparation passivates the electron traps (T2). Since T4 decreases without changing T5, the surface preparation by tetra methyl ammonium hydroxides is considered to decrease $\mathrm{V_{As}}$. These changes occur only at the surface of GaAs (and/or the interface between Au and GaAs). Obviously TSC measurement is a powerful method to get information about defect states of nanomaterials.

A.1.2 Estimating activation energy of defect states by peak method

When the temperature of TSC peak is determined, the activation energy of the peak trap can be estimated by the following equations [5]:

$$E_\mathrm{a} = k_\mathrm{B} T_\mathrm{m} \ln \frac{N \sigma v k T_\mathrm{m}^{2}}{\beta E_\mathrm{a}} \quad (1)$$

where E_a is the energy level of a given trap, T_m, the temperature at the current peak, N the effective density of states, σ the capture cross-section, v the thermal velocity of carriers, k_B Boltzmann's constant, and β the heating rate during the thermal scan. The parameters N, v, and σ are temperature dependent and related to carrier type. Two methods can be used for the determination of the energy level of a trap. Since Equation (1) can be rearranged as

$$\ln\left(\frac{T_\mathrm{m}^{2}}{\beta}\right) = \frac{E_\mathrm{a}}{k_\mathrm{B} T_\mathrm{m}} - \ln \frac{N \sigma v k}{E_\mathrm{a}} \quad (2)$$

E_a can be determined from the Arrhenius plot of $\ln\left(T_\mathrm{m}^{2}/\beta\right)$ vs. $1/T_\mathrm{m}$. Alternatively, with the temperature dependence of N and v considered, E_a can be calculated approximately using

$$E_a = k_B T_m \ln \frac{{T_m}^4}{\beta} . \quad (3)$$

- Step 1: Deciding peak positions

  The second derivative of TSC curve is used to elucidate the peak temperature as shown in Figure A.5. Derived derivatives of TSCs with multiple heating rates can be plotted with the original TSCs, and reading peaks of the derivatives helps to find specific peak positions of the TSC curves. Thus, peak temperature T_m can be decided for each TSC.

- Step 2: ${T_m}^2$ plot

  The relation between $\ln\left({T_m}^2 / \beta\right)$ and $1/T_m$ is plotted for each observed peak. By linear regression of the plot with the best coefficient of determination (R^2 value), the slope is obtained as shown in Figure A.6.

- Step 3: Calculation

  Multiplying the slope by Boltzmann's constant and dividing by the electron charge constant, the activation energy (eV) is obtained.

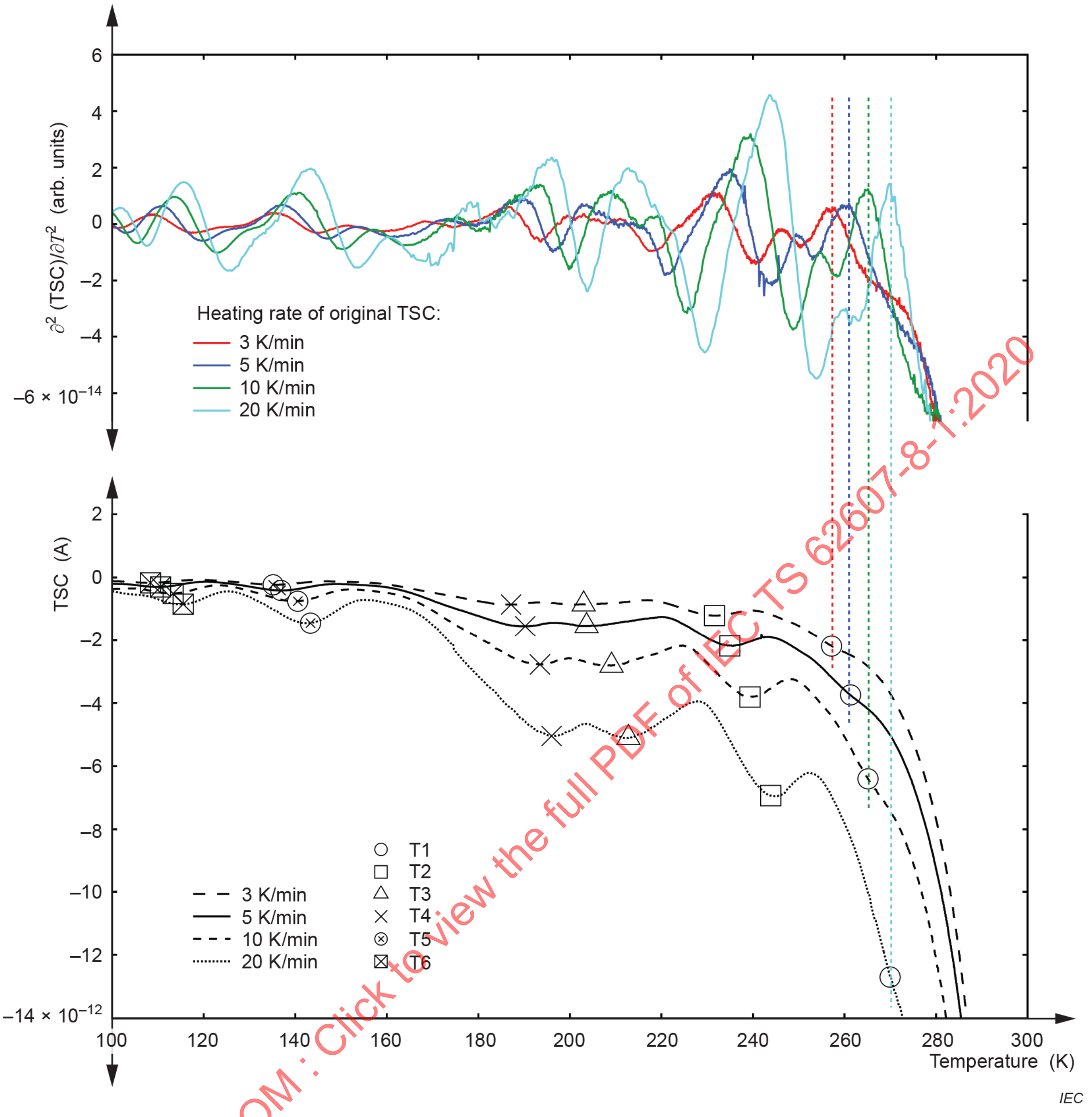
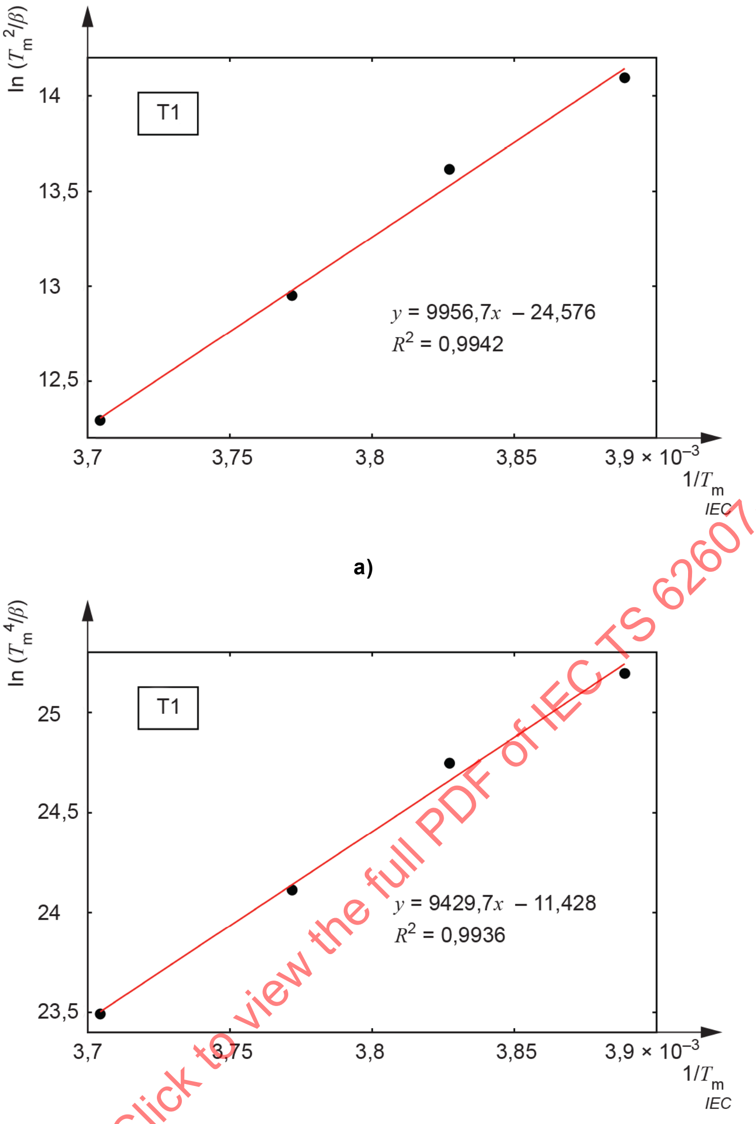


Figure A.5 – Determination of TSC peak positions using the second derivative curves



a)

b)

Figure A.6 – Arrhenius plots of (a) ln(T_m^2/β) vs. 1/T_m and (b) ln(T_m^4/β) vs. 1/T_m

Table A.2 – Activation energies of T1 to T6 for y = ln (T_m^2/β)

Peak	T6	T5	T4	T3[a]	T2	T1
R^2 value	0,992	0,994	0,985	1,000	1,000	0,994
Activation energy (eV)	0,26	0,35	0,64	0,69	0,71	0,86

[a] An outlier was removed from the T3 dataset.

Table A.3 – Activation energies of T1 to T6 for y = ln (T_m^4/β)

Peak	T6	T5	T4	T3[a]	T2	T1
R^2 value	0,991	0,993	0,983	1,000	1,000	0,994
Activation energy (eV)	0,25	0,33	0,61	0,65	0,67	0,81

[a] An outlier was removed from the T3 dataset.

The activation energies and the values of the coefficient of determination, R^2, of the peaks T1 to T6 are shown in Table A.2 and Table A.3. The R^2 values of T_m^2 plot and T_m^4 plot are about the same but those of T_m^2 plot are slightly better than the case of the T_m^4 plot. It suggests that the temperature dependence, which is included as the parameters consist of T_m^4 as shown in Equation (3), has no significant influence on these observed peaks.

A.2 TSC measurement of Ir/Ta$_2$O$_5$

A.2.1 General

TSC measurements of Ir/Ta$_2$O$_5$ samples are shown in Figure A.7 to Figure A.11 and Table A.4. Metal oxides frequently show broad TSC spectra, therefore the performance of TSC measurement device with the reference samples should be examined prior to the measurements.

Table A.4 – TSC measurement sequence steps and parameters / case study (2)

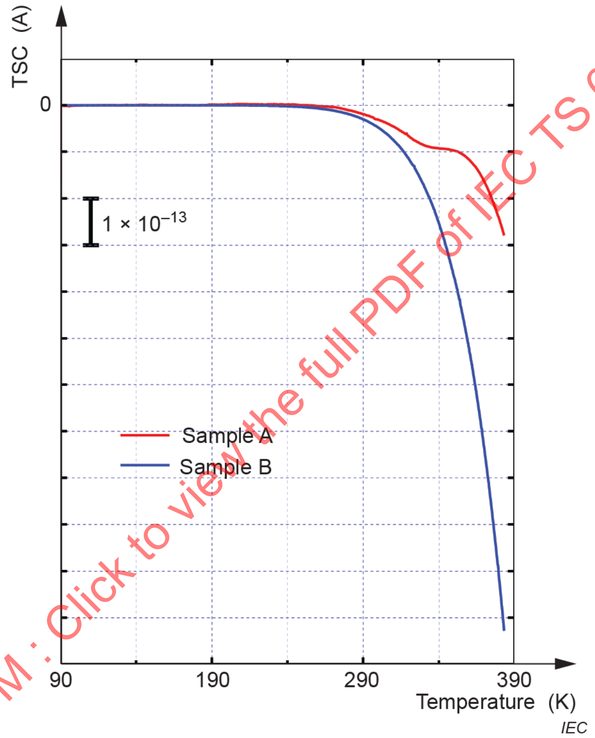
Step		Parameter 1	Parameter 2	Parameter 3	Parameter 4
1	Conditioning (Pre-measurement treatment)	Conditioning temperature 368 K	Holding time 2 h to 3 h	Releasing voltage -	
2	Cooling	Trapping temperature 90 K	Cooling rate 20 K/min		
3	Hold	Holding time 30 min			
4	Trapping (photoexcitation)	Wavelength (bandpass filter) White light	Light irradiation time 300 s	Holding time 5 min	Discharge time 5 min
	Trapping (Voltage/Current injection)	Applied voltage 0 V	Current 0 A	Holding time 0 min	Discharge time 0 min
5	Measurement	End temperature 383 K	Heating rate 3 K/min to 20 K/min	Collecting voltage 0,01 V to 3 V	
6	Ending (Post-measurement treatment)	Target temperature -	Heating rate -	Holding time -	

The metal-oxide samples with the interface of Ir/Ta$_2$O$_5$ were deposited on a thermally oxidized Si substrate by a sputtering method. Three types of samples were prepared by changing gas flow during the Ta$_2$O$_5$ deposition. These conditions are summarized in Table A.5. The thicknesses of Ir and Ta$_2$O$_5$ layers are 60 nm and 100 nm, respectively. The electrode configuration was the same as that shown in the case study of Clause A.1; Au/GaAs.

Table A.5 – Conditions of Ta_2O_5 sputtering deposition

Sample	Ar (sccm)	O_2 (sccm)	N_2 (sccm)
A	47,5	2,5	–
B	50	–	–
C	19	1	10

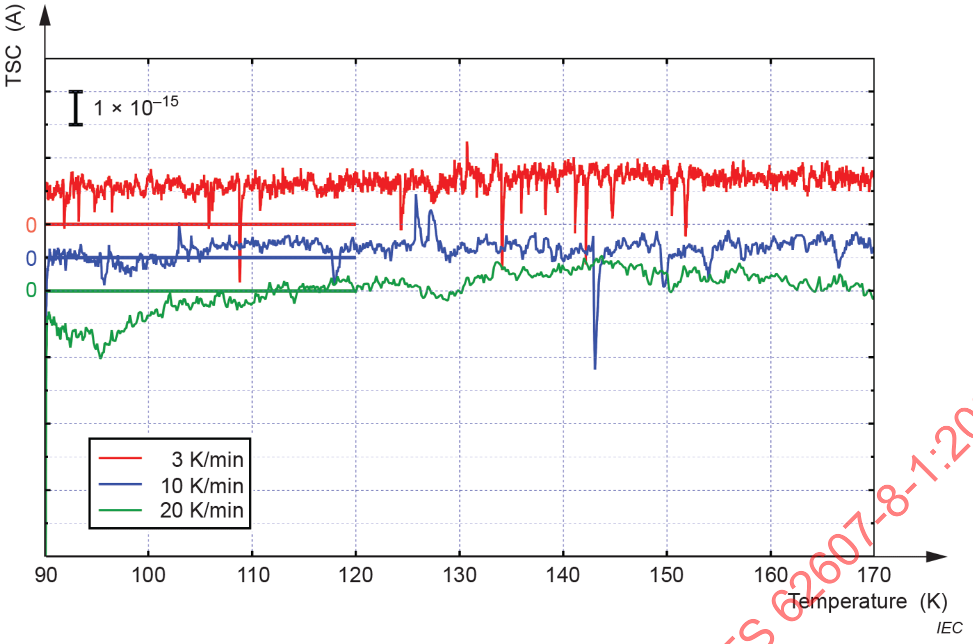
NOTE Ta_2O_5 were deposited on a thermally oxidized Si substrate by radio frequency (RF) magnetron sputtering (RF: 300 W, rotation: 10 rev/min). Gas flow rates of Ar, O_2 and N_2 were controlled to produce three types of samples: Sample A, Sample B and Sample C.



Sample A: Ta_2O_5 (Ar: 47,5 sccm, O_2: 2,5 sccm) (red line) and Sample B: Ta_2O_5 (Ar: 50 sccm, Ar 100 %) (blue line). Heating rate and collecting voltage of both samples were 20 K/min and 3 V, respectively.

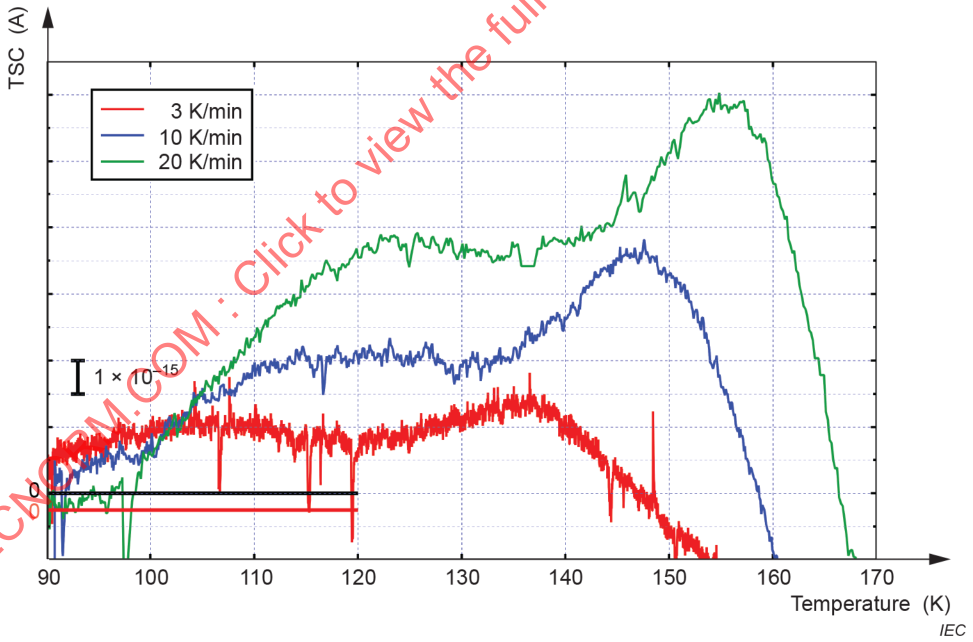
NOTE The plotted TSC of Sample B are 1:50 000 scale [0,000 02 times] of the original data.

Figure A.7 – TSC data comparison by samples



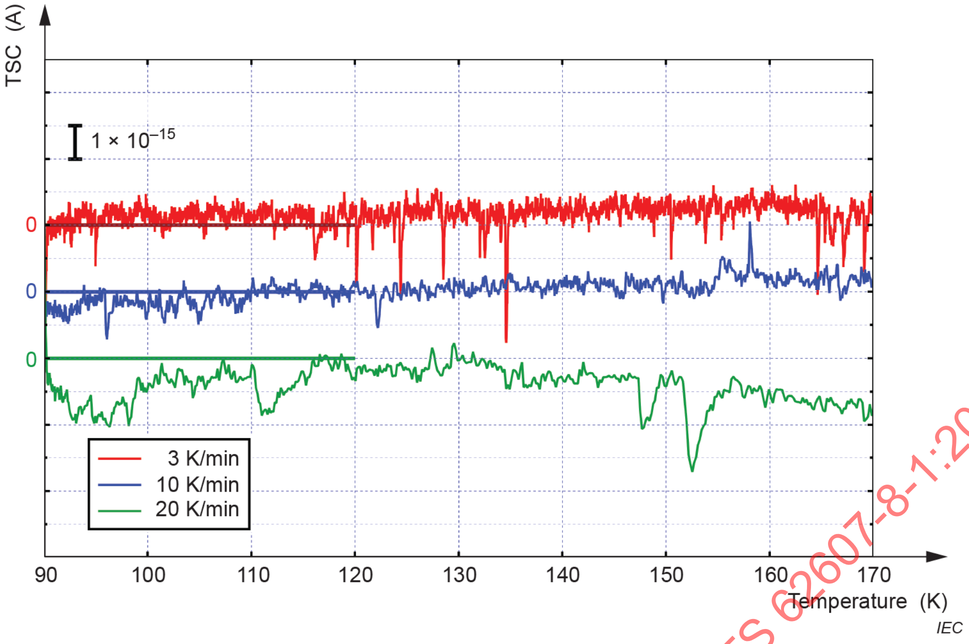
Collecting voltage was 3 V.

Figure A.8 – TSC data comparison of Sample A by heating rate



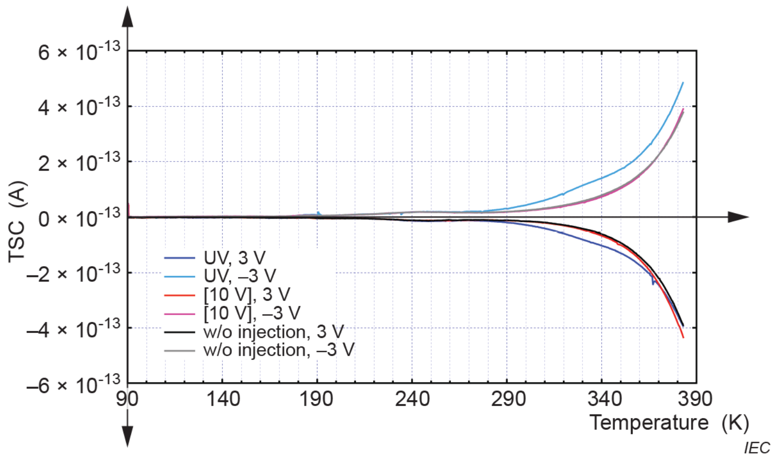
Collecting voltage was 0,01 V.

Figure A.9 – TSC data comparison of Sample B by heating rate

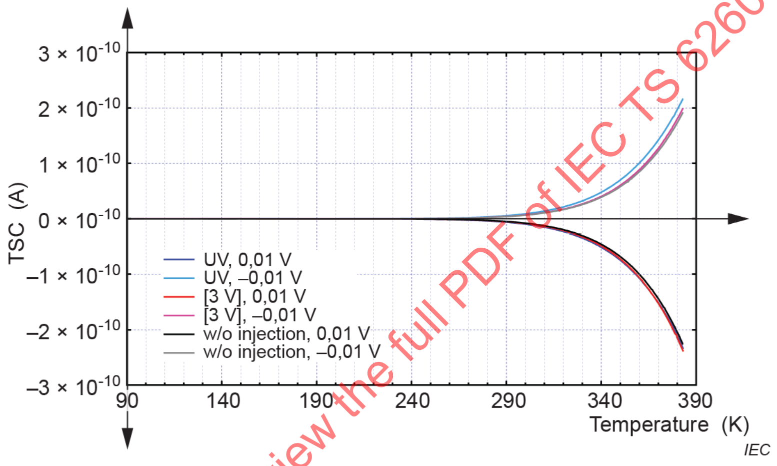


Sample C: Ta_2O_5 (Ar: 19 sccm, O_2: 1 sccm, N_2: 10 sccm). Collecting voltage was 3 V.

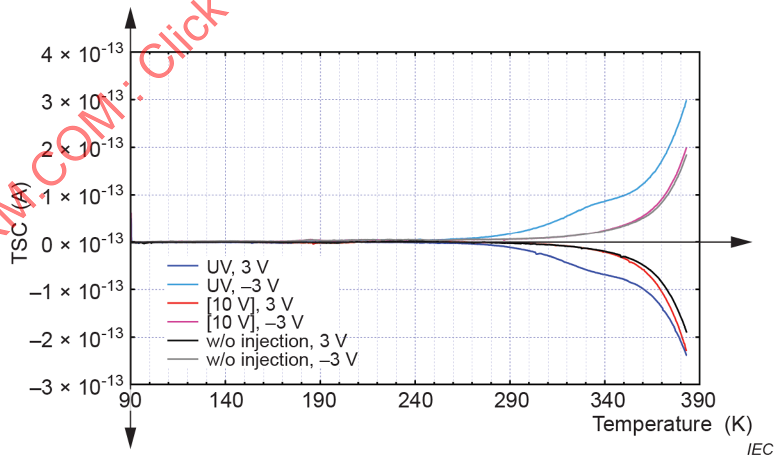
Figure A.10 – TSC data comparison of Sample C by heating rate



Sample A (Ar + O_2). Collecting voltage ±3 V



Sample B (Ar 100 %). Collecting voltage ±0,01 V



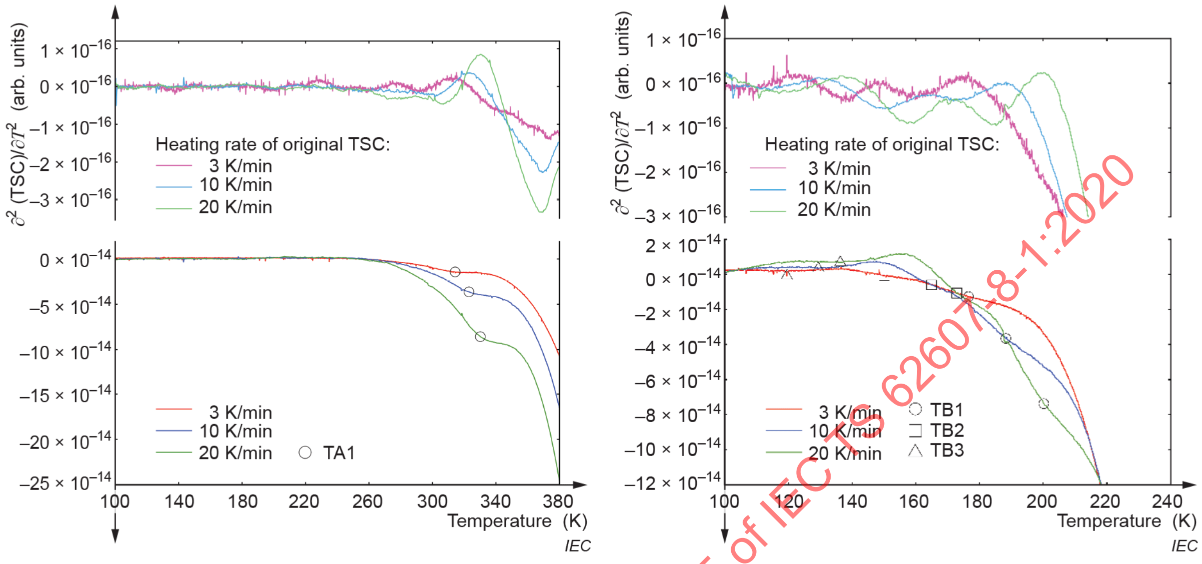
Sample C (Ar + O_2 + N_2). Collecting voltage ±3 V

Each legend shows a combination of carrier injection method and applied collecting voltage during TSC measurement. Dark blue line: Photoexcitation (UV) and positive collecting voltage. Light blue line: Photoexcitation (UV) and negative collecting voltage. Red line: Voltage injection and positive collecting voltage. Pink line: Voltage injection and negative collecting voltage. Black line: No carrier injection and positive collecting voltage. Grey line: No carrier injection and negative collecting voltage.

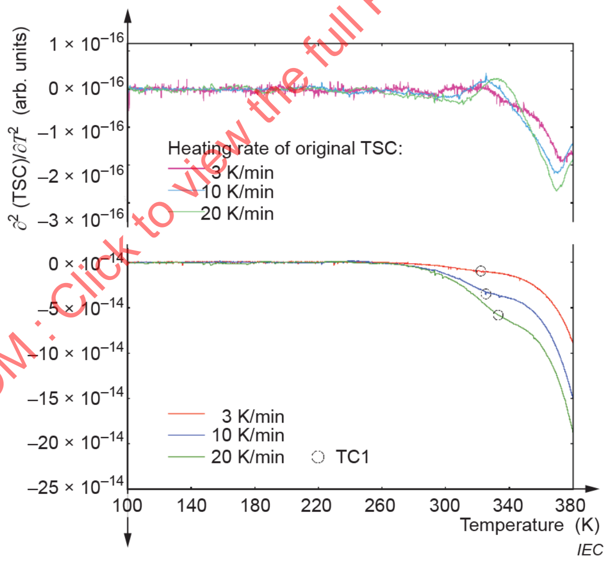
Figure A.11 – TSC data comparison by carrier injection method (Samples A, B and C)

A.2.2 Estimating activation energy of defect states by Peak method

The activation energies of the TSC peaks of Ir/Ta_2O_5 samples were determined in the same manner shown in A.1.2. The peak positions of Samples A, B and C were identified using the second derivative curves (Figure A.12), then $T_m{}^2$ and $T_m{}^4$ plots were performed (Figure A.13).

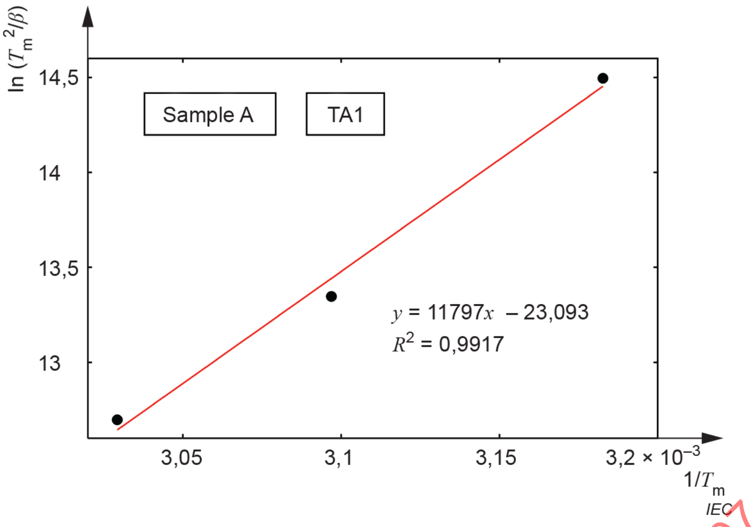


Sample A **Sample B**

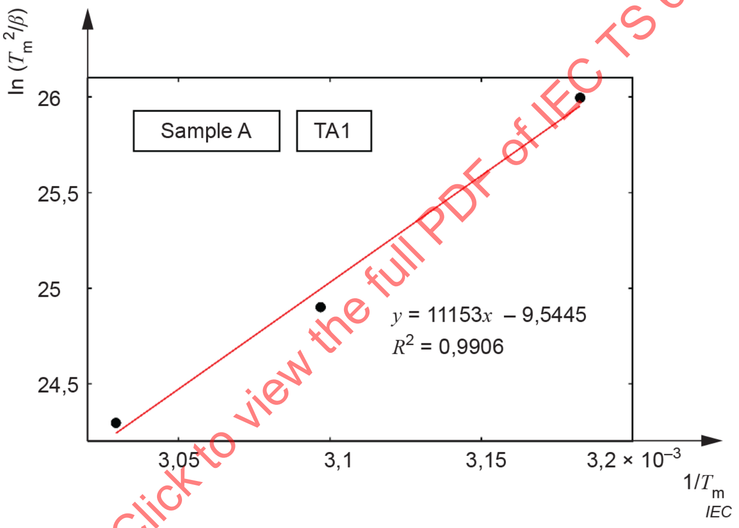


Sample C

Figure A.12 – Samples A, B and C: Determination of TSC peak positions using the second derivative curves



a) **ln($T_m{}^2/\beta$) vs. 1/T_m**



b) **ln($T_m{}^4/\beta$) vs. 1/T_m**

Figure A.13 – Arrhenius plots for TA1, Sample A

Table A.6 – Activation energies of Samples A, B and C

Sample	**A**	**B**			**C**
Peak	**TA1**	**TB3**	**TB2**	**TB1**	**TC1**
Equation: y = ln($T_m{}^2/\beta$)					
R^2 value	0,992	1,000	0,996	0,980	0,855
Activation energy (eV)	1,02	0,14	0,16	0,21	1,43
Equation: y = ln($T_m{}^4/\beta$)					
R^2 value	0,991	0,999	0,995	0,972	0,845
Activation energy (eV)	0,96	0,12	0,13	0,18	1,37

NOTE The energy levels of the peak of Sample A (TA1) and that of Sample C (TC1), either located above 300 K, were determined to be 1,02 eV and 1,43 eV, respectively. Sample B, with peaks observed below 200 K as TB1 to TB3, was estimated to have the energy levels of 0,21, 0,16 and 0,14 eV, respectively.

Activation energies of Samples A, B and C are summarized in Table A.6. The TSC peak above 300 K is consistent with the previously reported defect D [6], which is related to the deep donor observed in the ultrathin (physical thickness < 10 nm) Ta_2O_5 film. On the other hand, TSC peaks below 200 K are not assigned well. Since these peaks in a lower temperature range appeared only in Sample B in which the Ta_2O_5 layer is deposited with no oxygen gas supply, the detrap processes are reasonably considered to be originated from oxygen vacancies with relatively shallow activation energies.