IEC TS 62607-8-2

Edition 1.0 2021-01

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics –
Part 8-2: Nano-enabled metal-oxide interfacial devices – Test method for the polarization properties by thermally stimulated depolarization current

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 8-2: Nano-enabled metal-oxide interfacial devices – Test method for the polarization properties by thermally stimulated depolarization 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 62607-8-2, which is a Technical Specification, has been prepared by IEC technical committee 113: Nanotechnology standardization for electrical and electronic products and systems.

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

Draft TS	Report on voting
113/539/DTS	113/562/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 document 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 document will remain unchanged until the stability date indicated on the IEC website under "http://webstore.iec.ch" in the data related to the specific document. At this date, the document will be

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- withdrawn,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

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INTRODUCTION

Thermally stimulated depolarization current (TSDC) measurement has been a widely used method for acquiring information about electric polarization phenomena of various materials such as dielectrics, ferroelectrics, semiconductors, ceramics, plastics, and other organic materials for the past several decades. Recently, TSDC measurement became recognized as a powerful tool to evaluate polarization and depolarization, relaxation time, charge-storage and activation energy in advanced electronic materials including nano-enabled materials and devices. Accordingly, a standardized protocol for TSDC measurement will be useful to add validity to the experimental data for the purposes of productization of nano-enabled materials and devices. The reference sample for the reproducible TSDC measurement is also important.

This document offers a measurement method to be developed for determining polarization properties of metal-oxide interfacial devices using TSDC.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 8-2: Nano-enabled metal-oxide interfacial devices – Test method for the polarization properties by thermally stimulated depolarization 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. The latter is frequently called thermally stimulated depolarization current (TSDC). This part of IEC 62607 focuses on the latter method, and specifies the measurement procedures to be developed for determining polarization properties of metal-oxide interfacial devices.

This document includes:

– outlines of the experimental procedures used to measure TSDC,
– 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
sample on which to evaluate a specific physical property such as electrical resistance or *I-V* behaviour

[SOURCE: IEC 62607-2-1]

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

3.1.3
thermally stimulated depolarization current
TSDC
current flowing through an external circuit connecting to DUT, originated from the release of stored dielectric polarization due to raising temperature

3.2 Abbreviated terms

DUT device under test

TSC thermally stimulated current

TSDC thermally stimulated depolarization current

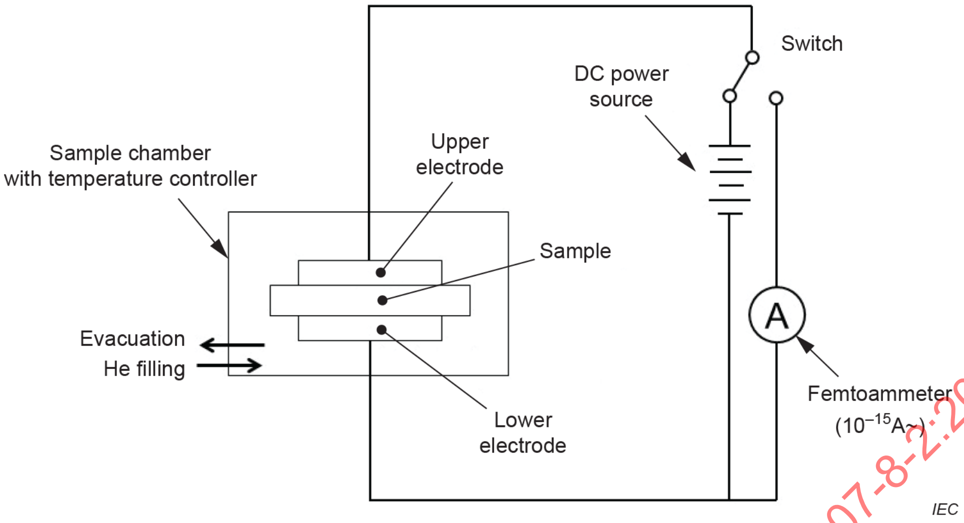
$SrTiO_3$ strontium titanium trioxide, strontium titanate

4 Measurement of TSDC

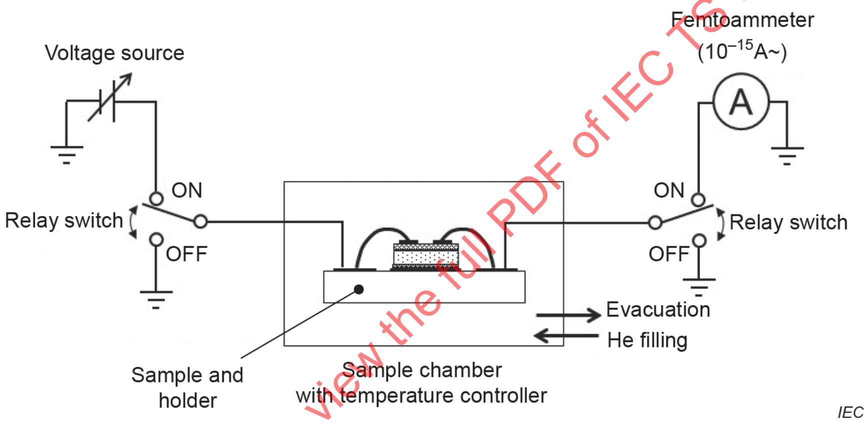
4.1 General

4.1.1 Measurement system

An accurate and reproducible test protocol of TSDC is standardized. Examples of an accurate and reproducible standardized test protocol of TSDC are shown in Figure 1. In the diagrams, each sample is attached to the sample holder with (a) upper and lower electrodes or (b) contact probes. The photos of these sample holders are shown in Figure 2 a) and b), respectively.



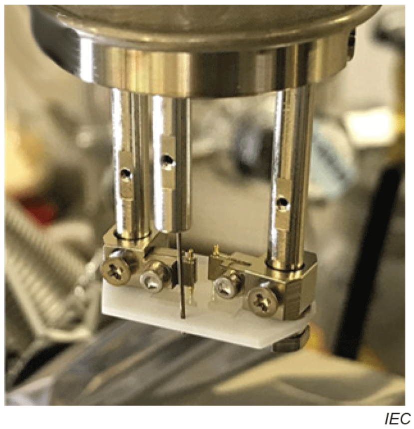
a) Sample is attached to the sample holder with upper and lower electrodes



b) Sample is attached to the sample holder with contact probes

Figure 1 – Examples of the experimental schematic diagram of TSDC

a) upper and lower electrodes

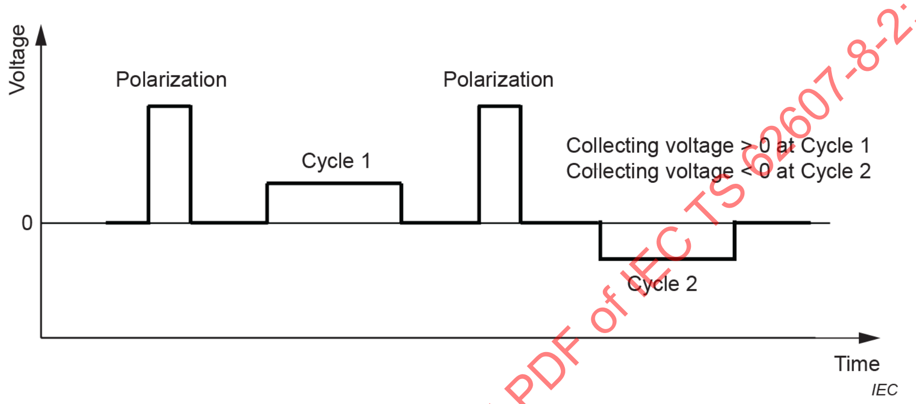


b) contact probes

Figure 2 – Photos of sample holders

4.1.2 TSDC measurement sequence

Examples of temperature control sequence for TSDC measurements are shown in Figure 3 a) In the case that the polarization is produced by the voltage, the sign of the current depends on that of the polarization voltage. On the other hand, the sign of the collecting voltage is considered not to affect the polarity of the polarization current. Therefore, the current of the depolarization process is obtained by subtracting the detrapped current from the observed current as shown in Figure 3 b). When the collecting voltage is applied in Cycles 2 and 4, the background current of the system can be obtained. If difference is evidenced between the currents of Cycles 2 and 4, it is recommended to check the status of the TSDC measurement setup to determine the origin of the background current. Furthermore, because the polarity of the pyroelectric current depends on that of the temperature slope, the difference between currents at Cycles 3 and 4 is assumed to be the pyroelectric current; in case that the pyroelectric current is evidenced, the contribution should be subtracted from the measured current [1].

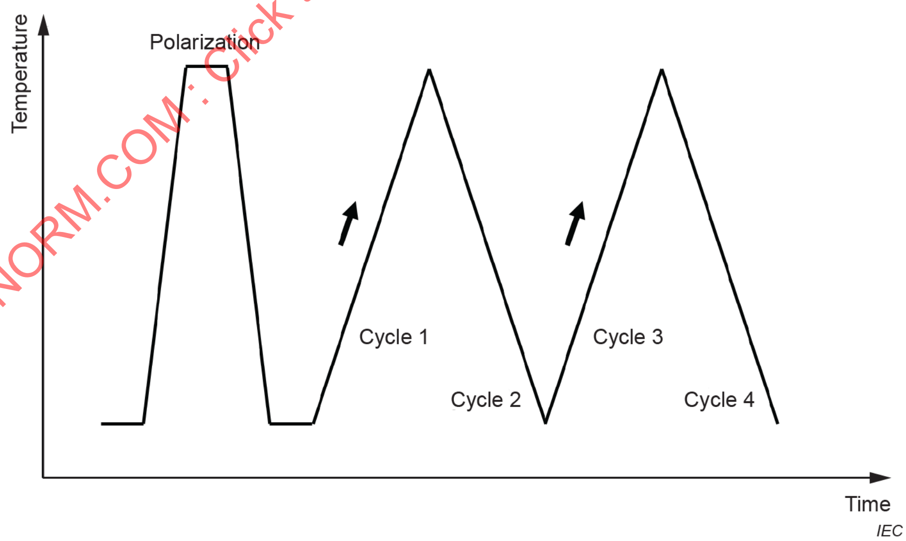


Detrapping process: $i_{\text{cycle 1}} - i_{\text{cycle 2}} = 2 \times i_{\text{detrap}}$

Depolarization process: $i_{\text{cycle 1}} - i_{\text{cycle 2}} = 0$

The depolarization current does not depend on the sign of the collecting voltage.

a)



b)

Figure 3 – Visualization of TSDC measurement sequence

4.1.3 Expression of temperature dependency of TSDC value

The temperature dependency of TSDC value shall be expressed by plotting TSDC (A) on the ordinate against temperature on the abscissa [2].

4.2 Sample preparation

TSDC 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. Furthermore, parameters of the polarization process – such as a polarization temperature, polarization (or applied) voltage and/or a plasma exposure – should be described in detail.

4.3 Experimental procedures

- Step 1: Pre-measurement treatment.
- Step 2: Polarization.
- Step 3: Cooling – Cool the sample to the starting temperature.
- Step 4: Measurement – Measure TSDC while heating up to the targeted end temperature.
- Step 5: Hold time – Keep the sample at the target temperature to stabilize the sample condition.
- Step 6: Cooling – Cool the sample again to the starting temperature.
- Step 7: Measurement – Repeat Step 4.
- Step 8: Post-measurement treatment – Set back to the room temperature before taking the sample out from the sample chamber.

NOTE 1 For Step 1, as an example, baking or annealing in a controlled environment can be done to vaporize absorbed water on the sample surface.

NOTE 2 For Step 2, temperature/voltage/time parameters applied in the process are reported. In the case that DUT is polarized before TSDC measurement, e.g. by corona charging, the conditions are reported in the sample preparation section.

NOTE 3 If necessary to determine whether the generated current is derived from detrapping of charges or a depolarization process, the polarity of the current and voltage is changed in Step 4. The current generated by the depolarization process does not depend on the sign of the collecting voltage.

Table 1 – TSDC measurement sequence steps and parameters

Step		Parameter 1	Parameter 2	Parameter 3	Parameter 4
1	Conditioning (pre-measurement treatment)	Temperature	Voltage	Time	
2	Polarization	Polarization temperature	Polarization voltage	Hold time	
3	Cooling	Target temperature	Cooling rate	Voltage	Hold time
4	Measurement	End temperature	Heating rate	Collecting voltage	
5	Hold time	Hold time			
6	Cooling	Target temperature	Cooling rate	Hold time	
7	Measurement	End temperature	Heating rate	Collecting voltage	
8	Post-measurement treatment	Target temperature	Heating rate	Hold time	

5 Reporting data

The following data at a minimum shall be reported:

- measurement parameters shown in Table 1;
- sample description;
- TSDC measurement configuration;
- TSDC signal as a function of temperature.

6 Data analysis / interpretation of results

6.1 General

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

6.2 Determination of the polarization charge

The polarization charge is evaluated by the integration of the TSDC current versus the temperature.

6.3 Peak method

The activation energy of the polarization state is estimated by the temperature dependence of the TSDC peak position.

Annex A
(informative)

Case study – TSDC measurement of $SrTiO_3$

A.1 General

Basic TSDC measurement sequences are shown in Table A.1. The measurement sequence described below is summarized in steps.

- Step 1: Pre-measurement treatment

  A suitable pre-measurement treatment varies on the material being measured, and can change depending on the situation or environment. To obtain reliable TSDC spectra and reproducible results, the polarization process (shown in sample preparation) and the pre-measurement treatment conditions should be identical in a series of measurements.

  In the case of $SrTiO_3$ measurement, the bare surface of the single-crystal $SrTiO_3$ substrate was used for the TSDC measurements. Two Au contact probes were touched on the surface. After installation of the sample, baking was done to vaporize moisture on the sample surface.

- Step 2: Polarization

  The sample is polarized by applying a bias voltage at high temperature. In this $SrTiO_3$ measurement, the sample was set in the sample chamber and heated up to the target temperature (373 K), followed by holding for 10 min under a polarization voltage (50 V) before starting Step 3.

- Step 3: Cooling

  Set target temperature (Parameter 1) and cooling rate (Parameter 2), then cool the sample to the target temperature.

- Step 4: Measurement (first run)

  TSDC measurement is done at the heating-up cycle. Firstly, turn the voltage-source switch and the ammeter relays on. Then, heat 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 an induction response.

- Step 5: Hold time

  When heating up to the targeted end temperature, hold it for 10 min to stabilize the sample condition.

- Step 6: Cooling

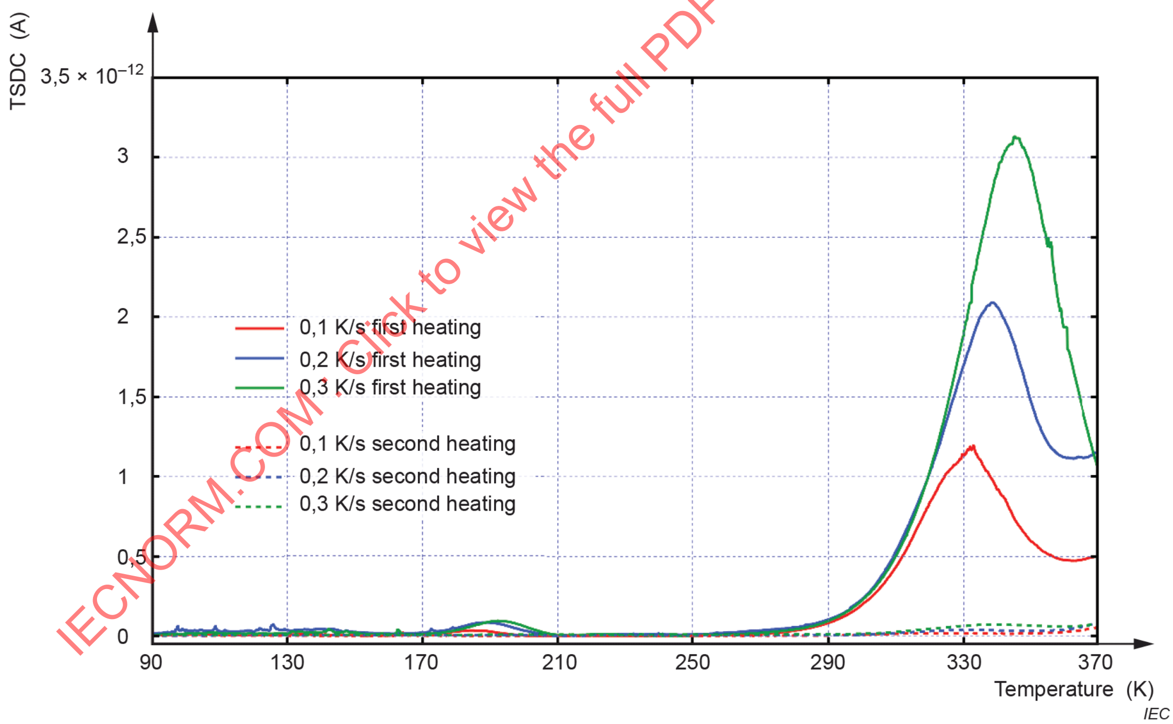
  Cool the sample again to the targeted starting temperature.

- Step 7: Measurement (second run)

  Repeat Step 4 to ensure that the depolarization wholly occurred in the first run. If DUT is fully depolarized/detrapped and no pyroelectric effect is confirmed, the background current can be estimated in the second run, as shown in Figure A.1.

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

	Step	Parameter 1	Parameter 2	Parameter 3	Parameter 4
1	Conditioning (pre-measurement treatment)	Temperature -	Voltage -	Time -	
2	Polarization	Polarization temperature 373 K	Heating rate 0,1 K/s to 0,3 K/s	Polarization voltage 50 V	Hold time 10 min
3	Cooling	Target temperature 90 K	Cooling rate 20 K/min	Voltage 50 V	Hold time 10 min
4	Measurement (first run)	End temperature 373 K	Heating rate 0,1 K/s to 0,3 K/s		
5	Hold time	Hold time 10 min			
6	Cooling	Target temperature 90 K	Cooling rate 20 K/min	Hold time 10 min	
7	Measurement (second run)	End temperature 373 K	Heating rate 0,1 K/s to 0,3 K/s		
8	Post-measurement treatment	Target temperature -	Heating rate -	Hold time -	



TSDCs of the first and the second heating periods in the measurement sequence are compared by multiple heating rates. The peak currents above 300 K in the second run, which are considered to be the background current, become much weaker than in the first run. The observed peak positions approximately agree with the results in the previous report [3].

Figure A.1 – TSDC data comparison by heating rate

A.2 Estimating activation energy of polarization state by peak method

When the temperature of TSDC peak is determined, the activation energy of the polarization state can be estimated by the following equations [4]:

$$\tau(T) = \tau_0 \exp\left(\frac{E_a}{k_B T}\right) \tag{A.1}$$

where τ is the relaxation time which governs the relaxation process, τ_0 is a preexponential factor, E_a is the activation energy and k_B is Boltzmann's constant. τ_0 is expressed by

$$\tau_0 = \frac{k_B T_m^{\ 2}}{\beta E_a} \exp\left(-\frac{E_a}{k_B T_m}\right) \tag{A.2}$$

where β is the heating rate and T_m is the temperature at the current peak. Equation (A.2) can be rearranged as

$$\ln\left(\frac{T_m^{\ 2}}{\beta}\right) = \frac{E_a}{k_B T_m} - \ln\left(\frac{k_B}{\tau_0 E_a}\right) \tag{A.3}$$

Therefore, E_a can be determined from the Arrhenius plot of $\ln\left(T_m^{\ 2}/\beta\right)$ versus $1/T_m$.

- Step 1: Deciding peak positions

  The second derivative of TSDC curve is used to elucidate the peak temperature, as shown in Figure A.2. Derived derivatives of TSDCs with multiple heating rates can be plotted with the original TSDCs and reading the peaks of the derivatives helps to find specific peak positions of the TSDC curves. Thus, the peak temperature T_m can be decided for each TSDC.

- 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.3.

- Step 3: Calculation

  Multiplying the slope by Boltzmann's constant and dividing by the electron charge constant, the activation energy (eV) is obtained, as shown in Table A.2.

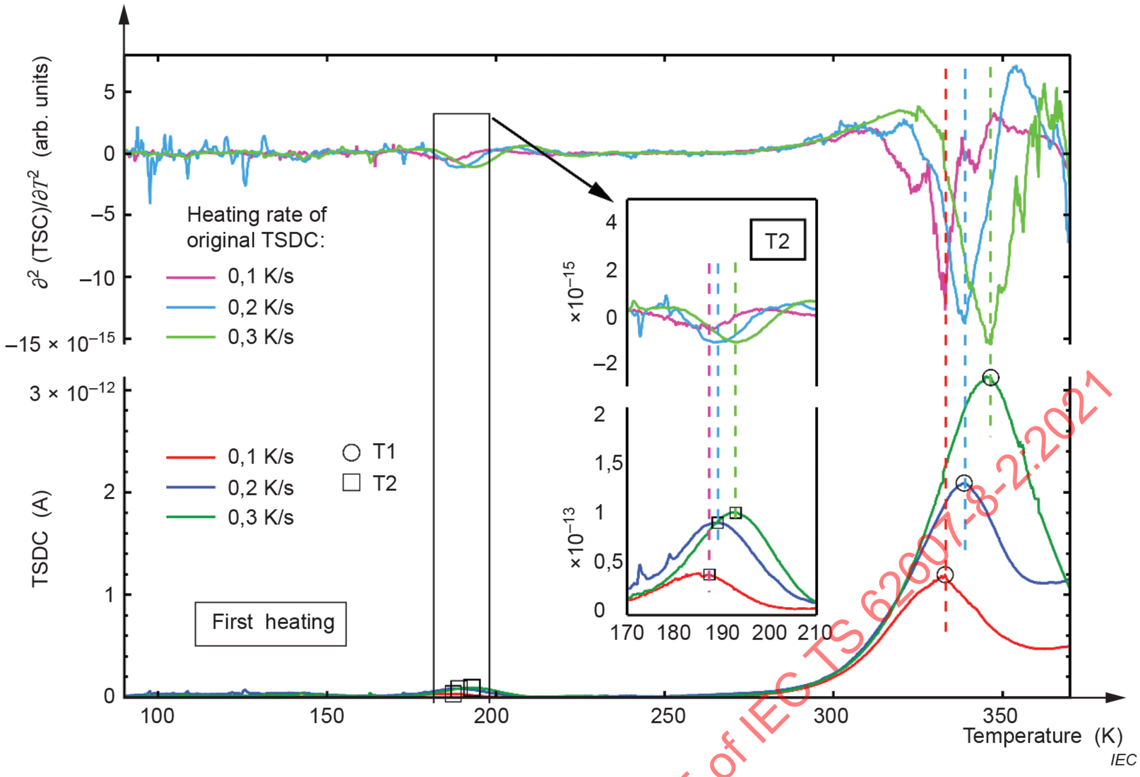


Figure A.2 – Determination of TSDC peak positions using the second derivative curves

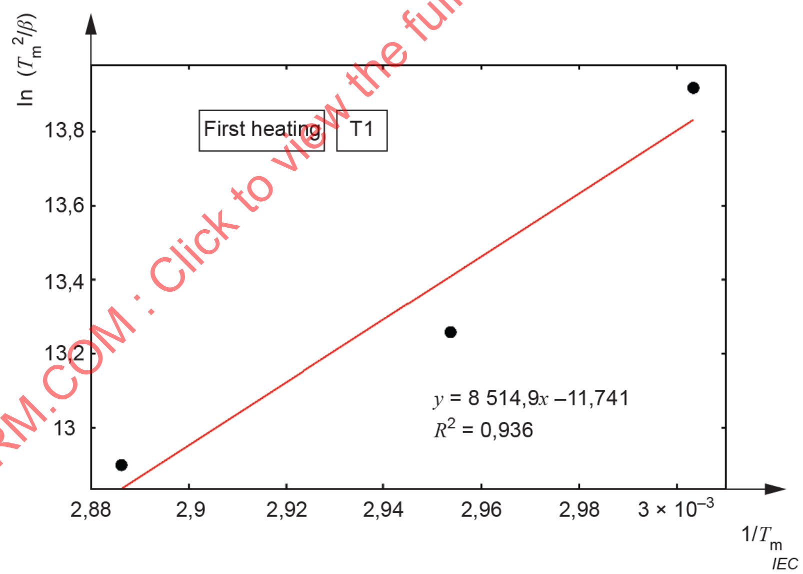


Figure A.3 – Arrhenius plot of ln(T_m^2/β) versus 1/T_m

Table A.2 – Activation energies in the first heating for y = ln(T_m^2/β)

Peak	T2	T1
R^2 **value**	0,864	0,936
Activation energy (eV)	0,54	0,73