

TECHNICAL SPECIFICATION

**Nanomanufacturing – Key control characteristics –
Part 8-4: Metal-oxide interfacial devices – Activation energy of electronic trap
states: Low-frequency-noise spectroscopy**

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**Nanomanufacturing – Key control characteristics –
Part 8-4: Metal-oxide interfacial devices – Activation energy of electronic trap
states: Low-frequency-noise spectroscopy**

INTERNATIONAL
ELECTROTECHNICAL
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**NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –****Part 8-4: Metal-oxide interfacial devices – Activation energy of electronic
trap states: Low-frequency-noise spectroscopy**

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

Draft	Report on voting
113/865/DTS	113/876/RVDTS

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Specification is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

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.

Future subparts of IEC 62607-8 will carry the new general title *Metal-oxide interfacial devices* as cited above. Titles of existing subparts in this series will be updated at the time of the next edition.

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

- reconfirmed,
- withdrawn, or
- revised.

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INTRODUCTION

Nano-enabled metal-oxide interfaces, such as an oxide nanolayer sandwiched by metal electrodes, are the essential components of IoT and AI devices for computing. Nano-enabled functions derived from the nanoscale metal-oxide interface and the oxide nanolayer appear, such as a significant change in electrical resistance. Analogue resistance change is the typical characteristic which possesses the large potential for non-von Neumann information processing. More concretely, the metal-oxide interfacial device is an indispensable element in the product-sum circuit that records the learning process as the analogue resistance change. It is known that the analogue resistance change occurs electronically in oxide interfacial layers regardless of the filamentary conductance. Since the electrical resistance is affected by electrons scattering in a material, it is extremely important to standardize the technique for evaluating electron traps in that material. Low-frequency-noise spectroscopy (LFNS) measurement is the powerful and unique tool to evaluate the activation energy of the electron trap states, which is one of the most essential electronic properties – especially in devices with the nano-scaled conductive path.

This document specifies a measurement protocol to evaluate the electronic trap states by LFNS in nano-enabled metal-oxide interfacial devices.

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 8-4: Metal-oxide interfacial devices – Activation energy of electronic trap states: Low-frequency-noise spectroscopy

1 Scope

This part of IEC 62607 specifies a measurement protocol to determine the key control characteristic

- activation energy of electronic trap states

for metal-oxide interfacial devices by

- low-frequency-noise spectroscopy

The noise spectra peak temperatures are obtained within a designated temperature range. Activation energies are then calculated based on the frequency dependence of the peak temperatures to analyse the energy levels associated with the electronic trap states. The activation energy is determined by the temperature dependence of the capture time at electron traps under the assumption that it is described by an Arrhenius function.

- In metal-oxide interfacial devices, electrical conductance is observed through an oxide nanolayer sandwiched between metal electrodes.
- The size of the conductive path in metal-oxide interfacial devices is dependent on the current value and is usually nanoscale in diameter, taking the form of a filamentary wire. This evaluation method is useful for analysing the electronic trap states in nanowires and other miniaturized devices that have nanolayers.

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 terminology databases for use in standardization at the following addresses:

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

3.1.1 General terms

3.1.1.1

device under test

DUT

sample attached to an apparatus for evaluation of a specific physical property such as electrical resistance or current–voltage behaviour

3.1.2 Terms specific to this document

3.1.2.1

electronic trap state

state which traps a carrier, for example, an electron, in the nanoscale metal-oxide interface and the oxide nanolayer

3.1.2.2

metal-oxide interfacial device

electronic component that consists of metal electrodes and an oxide nanolayer

EXAMPLE Non-volatile and volatile memories, and metal-oxide-semiconductor field-effect transistors (MOSFETs).

Note 1 to entry: The oxide layer is located between the metal electrodes, and the electrical conductance is observed through this layer.

Note 2 to entry: Metal-oxide interfacial devices play an important role in various electronic applications and are commonly used in various fields such as electronics, electrical engineering, and energy storage.

3.1.2.3

activation energy

measure of the minimum energy required to initiate a thermally activated electronic, physical, or chemical process

Note 1 to entry: The activation energy is an important parameter that is often described by the Arrhenius equation, which models the temperature dependence of chemical reaction rates and other thermally activated processes.

3.1.2.4

low-frequency-noise spectroscopy

technique used to measure and analyse low-frequency fluctuations in electrical signals in electronic devices to gain information about the underlying physical mechanisms causing the noise

Note 1 to entry: The fluctuations are analysed in the frequency domain to determine the spectral distribution of the noise and to obtain information about the underlying physical mechanisms causing the noise.

Note 2 to entry: Low-frequency-noise spectroscopy is commonly used to study the properties of materials and devices at the nanoscale, including the behaviour of electrons, the distribution of energy levels, and the dynamics of charge transport. The mechanism of noise is evaluated in terms of the number of charge carriers fluctuating due to the capture and emission processes of carriers at electron traps with certain activation energies.

Note 3 to entry: The information obtained from low-frequency-noise spectroscopy can be used to improve the performance and reliability of electronic devices, as well as to gain insights into the fundamental physics of materials and systems.

3.2 Abbreviated terms

AI artificial intelligence

IoT Internet of Things

LFNS low-frequency-noise spectroscopy

4 General

4.1 Measurement principle

To measure the activation energy for electronic trap states in metal-oxide interfacial devices, low-frequency-noise spectroscopy can be used to determine the energy required for an electron to escape from a trap state.

To perform this measurement, a metal-oxide interfacial device is typically subjected to a small bias voltage and the low frequency noise signals are measured over a range of temperatures between T_1 and T_2 . The noise signals are then analysed to determine the activation energy of the trap states, with which electrons fluctuate due to the capture and emission processes.

The activation energy is determined by fitting the temperature dependence of the noise signals to an Arrhenius function, which relates the capture and the emission times of electrons at trap states to the activation energy. This fitting process allows for the determination of the activation energy and provides information about the energy distribution of trap states within the device.

4.2 Sample preparation of the DUT

Report the stacking structure of DUT, such as the material and the thickness of the metal-oxide interfacial device. In addition, it is important to report the fabrication process information, such as the method of deposition, the shape of the DUT, and the electronic contact.

In order to avoid resistance measurement artifacts, the electric resistance of the substrate (R_{sub}) should be higher than that of the DUT (R_{dut}). The parallel current path in the substrate becomes smaller with the ratio of $R_{\text{dut}}/R_{\text{sub}}$. This value should be smaller than the error bar of the data. The electronic contact resistance, for example between the contact pad and the probe, should be minimized.

4.3 Experimental setup and apparatus

The measurement setup to perform the measurement procedure for low-frequency-noise spectroscopy (LFNS) according to this document is shown in Figure 1.

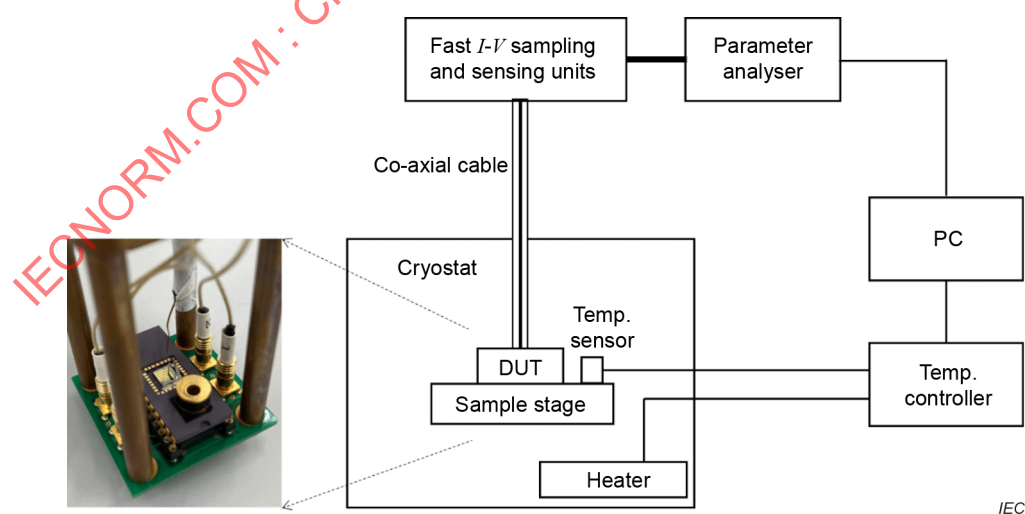


Figure 1 – An example of sample placement and experimental system

The measurement system consists of the following elements.

- Temperature control system (cryostat, temperature sensor, heater, temperature controller): The system controls the temperature range from cryogenic to room temperature. The lower limit of the range is better to be as lower as possible, for example, 4 K. In general, DUT shall be cooled to a lower temperature to measure lower activation energies.
- Fast I - V sampling and sensing unit: It should be possible to change the sampling frequency from approximately 10 Hz to 10^6 Hz. The electrical contacts connected to DUT are required to allow signal acquisition in the frequency range. Co-axial cables and probes are required to be compatible with high frequencies. Typically, a waveform generator that has a fast current–voltage (I - V) measurement function is used as the sampling unit. In some cases, a low-noise amplifier combined with a spectrum analyser can be added to the unit.
- Parameter analyser: A parameter analyser is required to be able to measure the resistance value of DUT, which is the object of noise measurement, with sufficient accuracy for the noise level, for example, 100 times larger than the signal-to-noise ratio. I - V measurements should be conducted in the form of a Kelvin connection; however, the connection method is not necessarily required if the contact resistance between DUT and the cable is small enough to be neglected.

5 Measurement procedure

- 1) Pre-measurement treatment: The sample mounting rod is inserted into the cooling chamber. After the operation, the air is evacuated by a scroll pump and helium is filled repeatedly. Typically, the chamber pressure is reduced to the range from 30 Pa to 40 Pa for the vacuum pumping. In the following helium filling, the pressure is raised almost equal to atmospheric; it is important to adjust the pressure slightly higher than the atmospheric pressure.
- 2) I - V characteristics evaluation: The sample is cooled from room temperature to the target temperature, T_1 . Before LFNS measurements, I - V characteristics evaluations are performed at the temperature T_1 . It is important to pay attention to the resistance value of the sample during the operations of 1) and 2) so that the desirable range of the value is maintained. The applied voltage is adjusted as needed to keep the resistive state adequate.
- 3) LFNS measurement: High-speed current sampling is performed at an applied voltage for the temperature range of T_1 to the target temperature, T_2 . Magnitude of the voltage is controlled to maintain proper resistive state. The lowest temperature, T_1 , and target temperature, T_2 , are determined according to the specifications of the temperature control system. In general, lower temperatures allow evaluation of smaller activation energies. Also, the rate of temperature change should be sufficiently slower than the time required for data processing at each measurement point.
- 4) Post I - V characteristics evaluation: I - V characteristics evaluations are performed at the target temperature, T_2 , to make sure there are no problems with electrical contact.

Table 1 shows the LFNS measurement sequence and parameters.

Table 1 – LFNS measurement sequence and parameters

	Step	Parameter 1	Parameter 2	Parameter 3	Parameter 4
1	Pre-measurement treatment	Remarks			
2	I - V characteristics evaluation	Temperature T_1	Applied voltage		
3	LFNS measurement	Temperature range [T_1 to T_2]	Voltage	Number of points	Sampling frequencies
4	Post I - V characteristics evaluation	Temperature T_2	Applied voltage		

6 Data analysis and interpretation of results

6.1 General

The activation energy of the electronic trap states is evaluated by the following analysis of the relationship between frequency (f) and peak temperature.

6.2 Peak determination

The noise spectrum is calculated by Fourier transforming the sampling data ($I-t$) of the current (I) value on the time (t) axis. The peak temperature is determined in the spectrum.

6.3 Interpretation of results

The noise analysis is performed based on previous studies [1], [2], [3], [4]¹. The capture time (τ) for an electron trap can be expressed as:

$$\tau = \exp(E_A/k_B T) / (n_e v_e \alpha_T) \quad (1)$$

where

E_A is the activation energy for the trap state;
 k_B is the Boltzmann constant;
 T is the temperature (K);
 n_e , v_e , and α_T are the electron density, electron thermal velocity, and capture cross-section, respectively, assuming that the emission time from the electron trap is equal to τ .

By focusing on the given frequencies (f) and peak temperatures, the activation energy E_A can be evaluated using the Formula (2) [1]:

$$\ln(T^{0.5}/f) = (E_A/k_B T) + C \quad (2)$$

where

C is a constant.

The effective density of the band states is assumed to be less temperature-dependent [1].

In the case that the density of state is considered to possess temperature dependence, Formula (3) can be applied [2], [3]:

$$\ln(T^2/f) = (E_A/k_B T) + C \quad (3)$$

where

C is a constant.

¹ Numbers in square brackets refer to the Bibliography.

According to Formula (2) or Formula (3), E_A can be determined from an Arrhenius plot of $\ln(T^{0.5}/f)$ versus $1/T$ or $\ln(T^2/f)$ versus $1/T$. The plot is performed using the peak temperatures extracted from fitting of the noise spectra. The value of the coefficient of determination, R^2 , which is obtained by linear regression in the process of E_A calculation, can be recorded. The value shows how close the data points are to the fitted line, namely the degree of certainty of the regression, thus implying how certain the obtained value of E_A is. An R^2 value closer to 1 indicates a better fitting. Typical examples of the analysis of measurement results are shown in Annex A, Annex B and Annex C.

7 Reporting data

The following data at a minimum shall be reported:

- sample description;
- T_1 : start temperature;
- T_2 : end temperature;
- temperature time history;
- frequency setting parameters;
- I - V characteristics at T_1 ;
- I - t characteristics;
- R^2 value obtained through Arrhenius fitting;
- activation energy.

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Annex A (informative)

Case study: Low-frequency-noise spectroscopy measurement of metal-oxide interfacial device

A.1 General

LFNS was evaluated in the sample with a structure of TaO_x nanolayer (60 nm) sandwiched by TiN electrodes. The stacking structure is shown in Figure A.1.

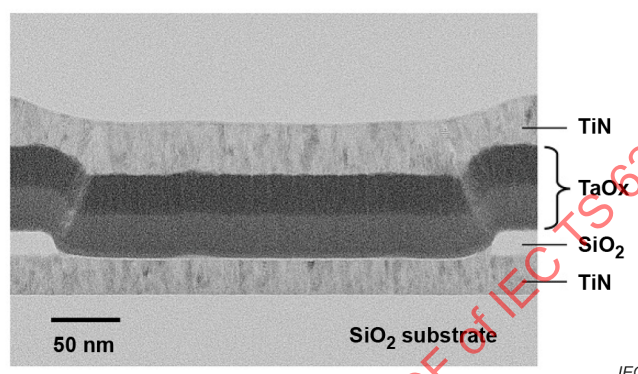


Figure A.1 – Transmission electron microscopy image of TiN/ TaO_x /TiN

A.2 LFNS measurement

– Step 1: Pre-measurement treatment

The sample mounting rod was inserted into the cooling chamber. After the operation, the air was evacuated by a scroll pump and helium was filled repeatedly. The chamber pressure was reduced to approximately 30 Pa to 40 Pa for the vacuum pumping, and through the following helium filling, adjusted slightly above atmospheric (approximately 0,01 MPa higher than the atmospheric pressure).

– Step 2: I - V characteristics evaluation

The sample was cooled from the room temperature to 3 K, which was the target temperature T_1 . Before LFNS measurements, I - V characteristics evaluations were performed at the temperature T_1 with an applied voltage of 0,1 V.

NOTE None of additional resistive control was conducted for this sample during Steps 1 and 2, because the sample was maintained in a high resistive state adequately.

– Step 3: LFNS measurement

High-speed current sampling was performed at the set voltage of 0,1 V for the temperature range from 3 K to 300 K (T_1 to T_2) with the following conditions:

- fixed number of points: 5×10^4 points;
- sampling frequencies: 10 Hz to 10^6 Hz.

– Step 4: Post I - V characteristics evaluation

I - V characteristics evaluations were performed at 300 K, the target temperature T_2 , to make sure there were no problems with electrical contact.

Table A.1 shows the LFNS measurement sequence and parameters.

Table A.1 – LFNS measurement sequence and parameters

	Step	Parameter 1	Parameter 2	Parameter 3	Parameter 4
1	Pre-measurement treatment	-			
2	I - V characteristics evaluation	Temperature 3 K	Applied voltage $\pm 0,1$ V		
3	LFNS measurement	Temperature range 3 K to 300 K	Voltage 0,1 V	Number of points 5×10^4	Sampling frequencies 10 Hz to 10^6 Hz
4	Post I - V characteristics evaluation	Temperature 300 K	Applied voltage $\pm 0,1$ V		

A.3 Data analysis

The obtained data are processed prior to analysis step. The set of sampling data (I - t) of the current (I) value on the time (t) axis (expressed in amperes [A]) is Fourier transformed and then converted into a spectrum (unit [A^2/Hz]). The frequency-dependent data of the spectral intensity at each temperature are converted into the temperature-dependent spectral intensity in each frequency. Figure A.2 shows the Fourier-transformed spectra derived from the sampling I - t data that were measured in A.2.

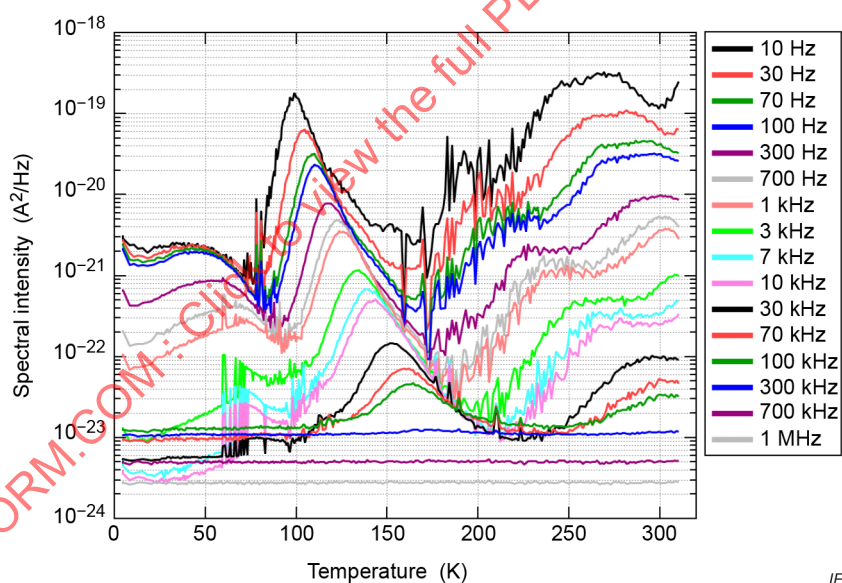


Figure A.2 – Changes in noise spectra as a function of temperature at the given frequencies

Curve fitting is executed on each spectrum at the preset frequency and the spectral intensity is calculated. Lorentzian peak is used as the curve fit function.

$$y = y_0 + \frac{A}{(x - x_0)^2 + B}$$

The peak temperature on each spectrum of the transformed data was determined by the curve fitting as shown in Figure A.3.

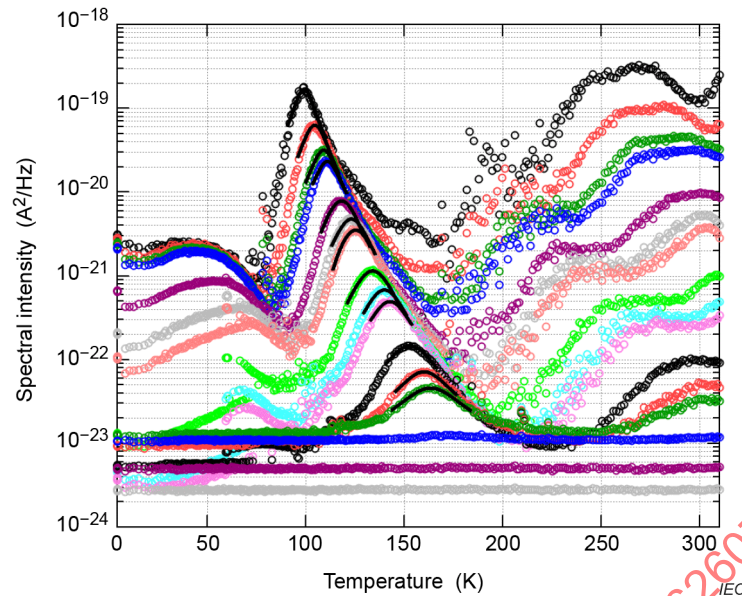


Figure A.3 – Changes in noise spectra as a function of temperature at the given frequencies with the fitting curves to determine the peak temperatures

Arrhenius plot of $\ln(T^{0.5}/f)$ versus $1/T$ is performed using the peak temperatures extracted from the fitting, according to Formula (2), to determine the activation energy. By linear regression of the plot with the best coefficient of determination (R^2 value), the slope of the fitted line is obtained.

Using the slope, the activation energy was calculated as 0,194 eV by the following formula:

$$E_A = b \times k_B$$

where

E_A is the activation energy for the trap state;

b is the slope of the Arrhenius plot;

k_B is the Boltzmann constant in the unit of eV/K;

The peak temperature of each noise spectrum and the Arrhenius plot are as shown in Table A.2 and Figure A.4, respectively.

Table A.2 – Peak temperature at each frequency

Frequency	Peak temperature	$\ln(T^{0.5}/f)$	$1/T$
10 Hz	99,2	-0,004 18	0,010 1
30 Hz	105	-1,08	0,009 56
70 Hz	109	-1,9	0,009 16
100 Hz	111	-2,25	0,009 01
300 Hz	118	-3,32	0,008 46
700 Hz	123	-4,14	0,008 12
1 kHz	125	-4,49	0,007 98
3 kHz	134	-5,56	0,007 46
7 kHz	140	-6,38	0,007 14
10 kHz	143	-6,73	0,007 01

Frequency	Peak temperature	$\ln(T^{0,5}/f)$	$1/T$
30 kHz	153	-7,79	0,006 54
70 kHz	160	-8,62	0,006 25
100 kHz	163	-8,96	0,006 12

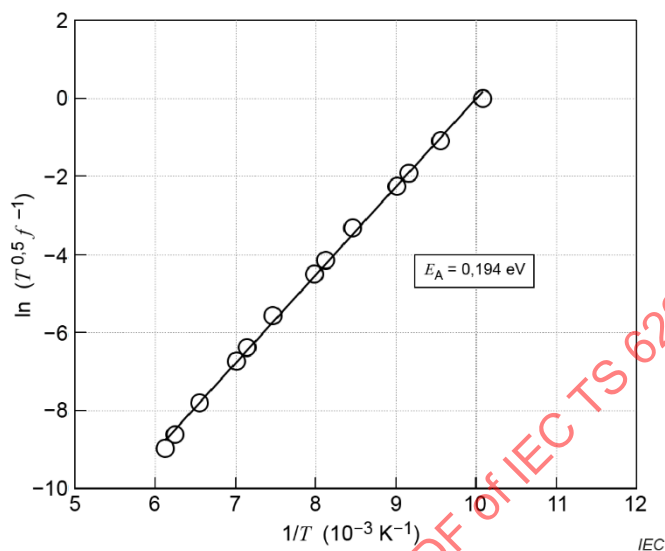


Figure A.4 – Arrhenius plot using the peak temperatures

To compare the R^2 value, the Arrhenius plot of $\ln(T^2/f)$ versus $1/T$ is also performed according to Formula (3). The peak temperature of each noise spectrum and the Arrhenius plot are as shown in Table A.3 and Figure A.5, respectively. The comparison result is shown in Table A.4.

Table A.3 – Peak temperature at each frequency, $\ln(T^2/f)$

Frequency	Peak temperature	$\ln(T^2/f)$	$1/T$
10 Hz	99,2	6,89	0,010 1
30 Hz	105	5,9	0,009 56
70 Hz	109	5,14	0,009 16
100 Hz	111	4,81	0,009 01
300 Hz	118	3,84	0,008 46
700 Hz	123	3,08	0,008 12
1 kHz	125	2,75	0,007 98
3 kHz	134	1,79	0,007 46
7 kHz	140	1,03	0,007 14
10 kHz	143	0,711	0,007 01
30 kHz	153	-0,251	0,006 54
70 kHz	160	-1	0,006 25
100 kHz	163	-1,32	0,006 12

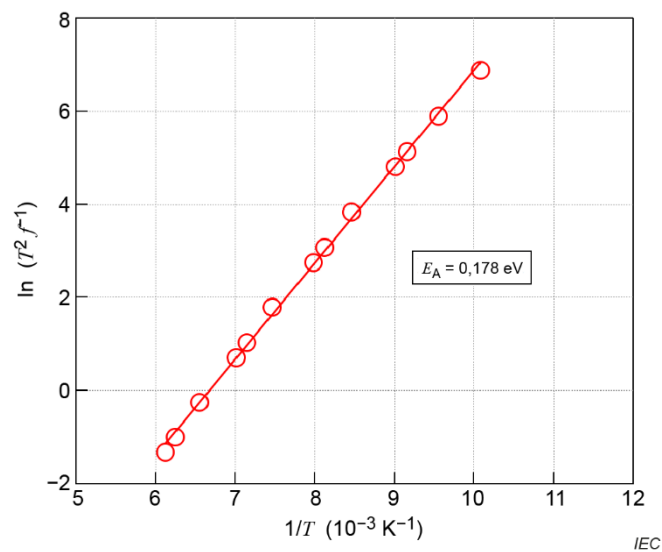


Figure A.5 – Arrhenius plot using the peak temperatures, $\ln(T^2/f)$

Table A.4 – R^2 values and activation energies

Parameter	$\ln(T^{0,5}/f)$	$\ln(T^2/f)$
R^2	0,998	0,998
activation energy, E_A (eV)	0,194	0,178

Annex B (informative)

Case study: Low-frequency-noise spectroscopy measurement of metal-oxide interfacial device

B.1 General

LFNS was evaluated in the sample with a structure of TaO_x nanolayer (60 nm) sandwiched by TiN electrodes. The same sample as used in the case study of Annex A was measured again to evaluate the activation energy of the electron trap in the lower resistive state. The resistance value was adjusted electronically by applying voltage to be approximately 300 times lower than that of Annex A study.

NOTE The method of controlling resistive states is similarly applied to, for example, IoT and AI devices [5].

B.2 LFNS measurement

– Step 1: Pre-measurement treatment

The sample mounting rod was inserted into the cooling chamber. After the operation, the air was evacuated by a scroll pump and helium was filled repeatedly. The chamber pressure was reduced to approximately 30 Pa to 40 Pa for the vacuum pumping, and through the following helium filling, adjusted very slightly above atmospheric (approximately 0,01 MPa higher than the atmospheric pressure).

– Step 2: I - V characteristics evaluation

The sample was cooled from the room temperature to 3 K, which was the target temperature T_1 . Before LFNS measurements, I - V characteristics evaluations were performed at the temperature T_1 with an applied voltage of 1,5 V. Prior to the I - V evaluation, resistance value of the sample was adjusted to be lower than that of the Annex A study.

– Step 3: LFNS measurement

High-speed current sampling was performed at the set voltage of 0,1 V for the temperature range from 3 K to 300 K (T_1 to T_2) with the following conditions:

- fixed number of points: 5×10^4 points;
- sampling frequencies: 10 Hz to 10^6 Hz.

– Step 4: Post I - V characteristics evaluation

I - V characteristics evaluations were performed at 300 K, the target temperature T_2 , to make sure there were no problems with electrical contact.

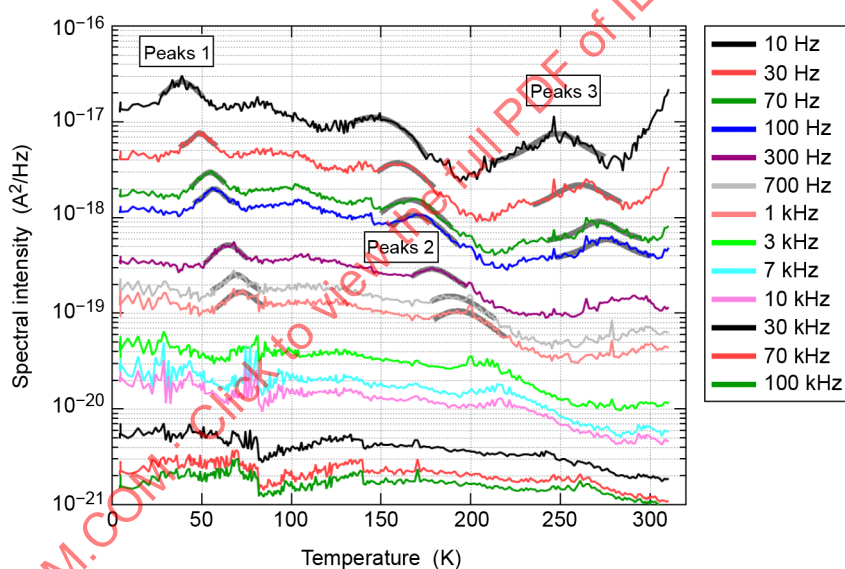
Table B.1 shows the LFNS measurement sequence and parameters.

Table B.1 – LFNS measurement sequence and parameters

	Step	Parameter 1	Parameter 2	Parameter 3	Parameter 4
1	Pre-measurement treatment	-			
2	I - V characteristics evaluation	Temperature 3 K	Applied voltage $\pm 1,5$ V		
3	LFNS measurement	Temperature range 3 K to 300 K	Voltage 0,1 V	Number of points 5×10^4	Sampling frequencies 10 Hz to 10^6 Hz
4	Post I - V characteristics evaluation	Temperature 300 K	Applied voltage 0,1 V		

B.3 Data analysis

The noise spectrum was calculated by Fourier transforming of the sampling data (I - t). The peak temperature was determined by the curve fitting as shown in Figure B.1.



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Figure B.1 – Changes in noise spectra as a function of temperature at the given frequencies with the fitting curves to determine the peak temperatures

Arrhenius plot of $\ln(T^{0.5}f)$ versus $1/T$ was performed using the extracted peak temperatures, according to Formula (2), for the three series of peaks (Peaks 1 to Peaks 3). The plots and the derived activation energy of each series are as shown in Figure B.2.

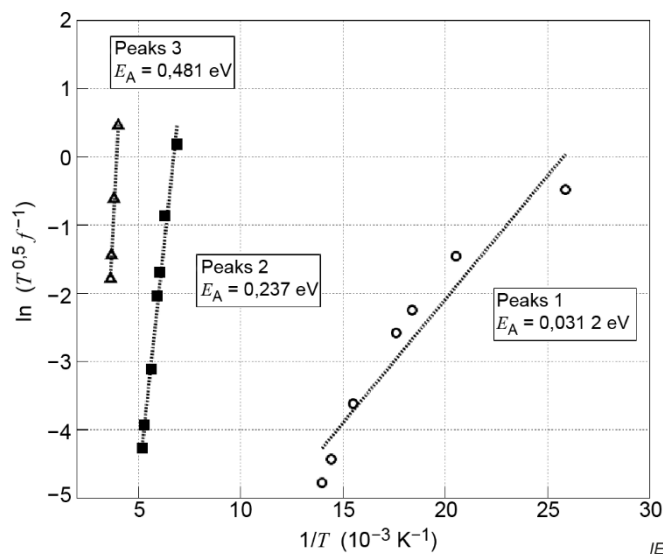


Figure B.2 – Arrhenius plot using the peak temperatures

To compare the R^2 values, the Arrhenius plot of $\ln(T^2/f)$ versus $1/T$ was also performed for the three series of peaks, according to Formula (3). The plots and derived activation energy of each series are as shown in Figure B.3. The comparison result is shown in Table B.2.

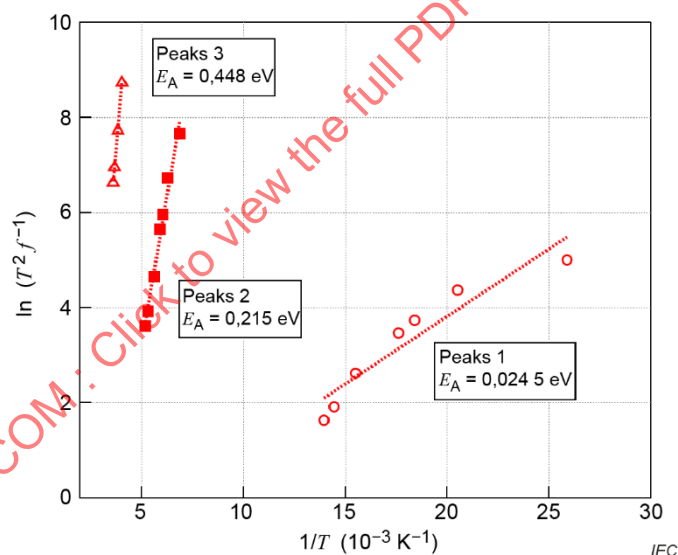


Figure B.3 – Arrhenius plot using the peak temperatures, $\ln(T^2/f)$

Table B.2 – R^2 values and activation energies

Parameter	Arrhenius plot	Peaks 1	Peaks 2	Peaks 3
R^2	$\ln(T^{0.5}/f)$	0,922	0,987	0,999
	$\ln(T^2/f)$	0,895	0,985	0,999
activation energy, E_A (eV)	$\ln(T^{0.5}/f)$	0,031 2	0,237	0,481
	$\ln(T^2/f)$	0,024 5	0,215	0,448