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Semiconductor devices – Micro-electromechanical devices – Part 30: Measurement methods of electro-mechanical conversion characteristics of MEMS piezoelectric thin film

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SEMICONDUCTOR DEVICES – MICRO-ELECTROMECHANICAL DEVICES –

Part 30: Measurement methods of electro-mechanical conversion characteristics of MEMS piezoelectric thin film

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The text of this International Standard is based on the following documents:

FDIS	Report on voting
47F/286/FDIS	47F/289/RVD

Full information on the voting for the approval of this International Standard 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 62047 series, published under the general title *Semiconductor devices – Micro-electromechanical devices*, can be found on the IEC website.

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SEMICONDUCTOR DEVICES – MICRO-ELECTROMECHANICAL DEVICES –

Part 30: Measurement methods of electro-mechanical conversion characteristics of MEMS piezoelectric thin film

1 Scope

This part of IEC 62047 specifies measuring methods of electro-mechanical conversion characteristics of piezoelectric thin film used for micro sensors and micro actuators, and its reporting schema to determine the characteristic parameters for consumer, industry or any other applications of piezoelectric devices. This document applies to piezoelectric thin films fabricated by MEMS process.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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
unimorph beam
beam composed of piezoelectric thin film on substrate

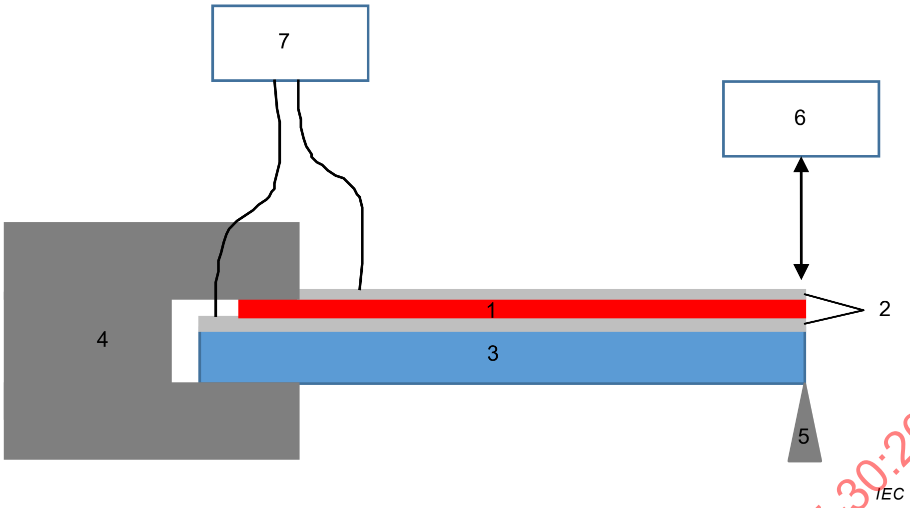
3.2
direct transverse piezoelectric coefficient
transverse piezoelectric coefficient of the piezoelectric thin film calculated from generated charge or voltage caused by strain or stress

3.3
converse transverse piezoelectric coefficient
transverse piezoelectric coefficient of the piezoelectric thin film calculated from strain or stress caused by electric field or voltage

4 Test bed of MEMS piezoelectric thin film

4.1 General

These measuring methods of the transverse piezoelectric properties apply to the unimorph beam. Symbols and designations of test bed are shown in Table 1.



Key

1 thin film under testing

2 electrodes

The Electrodes are contacted with the top side and bottom side surface of the thin film under testing.

3 substrate

4 clamp

5 linear actuator (not in use for converse piezoelectric measurement)

6 displacement meter

7 electric measurement instrument (i.e. voltmeter, charge meter, ammeter, oscilloscope, or lock-in amp) in direct piezoelectric measurement, power source (function generator and amplifier) in converse piezoelectric measurement

Figure 1 – Test bed of direct and converse transverse piezoelectric coefficient of MEMS piezoelectric thin film

Table 1 – Symbols and designations of test bed

Kind of properties	Symbol	Unit	Designation
Dimension of cantilever specimen	l	m	length of cantilever
	w	m	width of cantilever
	h_s	m	thickness of base cantilever
	h_p	m	thickness of piezoelectric thin film
Electro-mechanical conversion properties	$e_{31,f}$	C/m^2	effective transverse piezoelectric coefficient
	$e^d{}_{31,f}$	C/m^2	effective transverse piezoelectric coefficient (direct effect)
	$e^c{}_{31,f}$	N/Vm	effective transverse piezoelectric coefficient (converse effect)
	$e^c{}_{31,f}(V_{in,0})$	N/Vm	extrapolated effective transverse piezoelectric coefficient at 0 V (converse effect)
	$e^c{}_{31,f}(V_{in,min})$	N/Vm	minimum effective transverse piezoelectric coefficient (converse effect at the lowest V_{in})
	$e^c{}_{31,f}(V_{in,max})$	N/Vm	maximum effective transverse piezoelectric coefficient (converse effect at the highest V_{in})
	d_{31}	m/V	transverse piezoelectric coefficient (d-form)
Electrical properties	C	F	capacitance of piezoelectric thin film
	V_{out}	V	output voltage
	V_{in}	V	input peak-to-peak voltage
	$tan\ \delta$		dielectric loss
Mechanical properties	$s_{11}{}^E$, $s_{12}{}^E$	m^2/N	elastic compliances of piezoelectric thin film
	D_{in}	m	input tip displacement
	D_{out}	m	output tip displacement
	E_s	N/m^2	Young's modulus of base cantilever
	ν_s		Poisson's ratio of base cantilever
	E_p	N/m^2	Young's modulus of piezoelectric thin film
	y_c	m	position of neutral plane of the unimorph cantilever from the bottom

4.2 Functional blocks and components

4.2.1 General

Figure 1 provides fundamental configurations consisted of functional blocks or components for test bed of direct and converse transverse piezoelectric coefficient of MEMS piezoelectric thin film. Details of the functional blocks or components named as keys are provided in 4.2.2 to 4.2.6.

4.2.2 Clamp

The clamp holds one end of the unimorph beam to make a cantilever.

NOTE The clamping condition is confirmed by mechanical Q factor at the resonance of the cantilever.

4.2.3 Linear actuator

The linear actuator provides displacement to the tip of the cantilever with triangular wave. It is used for direct piezoelectric measurements.

4.2.4 Displacement meter

The displacement meter measures the tip displacement of the cantilever.

4.2.5 Electric measurement instrument

In measurements of direct piezoelectric effect, the thin film under testing generates electric output between electrodes. An electric measurement instrument (i.e. voltmeter, charge meter, ammeter, oscilloscope, or lock-in amplifier) measures the generated voltage, charge or current synchronizing with the displacement by the linear actuator.

4.2.6 Power source

In measurements of converse piezoelectric effect, the power source applies an electric input signal between the top and bottom electrodes. The input sinusoidal signal from the function generator is amplified by the power amplifier.

5 Thin film under testing

5.1 General

The top surface of the MEMS piezoelectric thin film is coated with a top electrode to measure output voltage in direct transverse coefficient measurement, or to provide input voltage in converse piezoelectric coefficient measurement.

The thickness of the base material of the unimorph beam shall be much larger than that of the thin film under testing, typically at least 100 times, in order to approximate the neutral plane of the unimorph beam to be the half of the substrate thickness. The theoretical equation of the neutral plane is described in Clause A.5 (this approximation is used in the Stoney's formula).

Generally, the thickness of the electrodes should be much smaller than that of the thin film under testing.

In the case of ferroelectric thin films, a poling treatment is indispensable to align the polar direction and maximize their piezoelectric characteristics.

5.2 Measurement principle

In general, the effective transverse piezoelectric coefficient of the piezoelectric thin film is defined as follows (see [1]–[3][1]):

$$e_{31,\mathrm{f}} = \frac{d_{31}}{s_{11}^{\mathrm{E}} + s_{12}^{\mathrm{E}}} \tag{1}$$

The effective transverse piezoelectric coefficient of direct piezoelectric effect ($e^{\mathrm{d}}_{31,\mathrm{f}}$) is calculated as follows:

$$e^{\mathrm{d}}_{31,\mathrm{f}} = \frac{4lC}{3wh_{\mathrm{s}}(1-\nu_{\mathrm{s}})} \cdot \frac{V_{out}}{D_{in}} \tag{2}$$

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

The effective transverse piezoelectric coefficient of converse piezoelectric effect ($e^{c}_{31,f}$) is calculated as follows:

$$e^{c}_{31,f} = \frac{E_s h_s^2}{3l^2(1-\nu_s)} \cdot \frac{D_{out}}{V_{in}} \tag{3}$$

Converse piezoelectric effect strongly depends on the input voltage due to extrinsic piezoelectric contribution. Therefore, the three transverse piezoelectric coefficients $e^{c}_{31,f}(V_{in,min})$, $e^{c}_{31,f}(V_{in,max})$ and $e^{c}_{31,f}(V_{in,0})$ shall be defined from the results of the converse piezoelectric measurements.

$e^{c}_{31,f}(V_{in,min})$ is the lowest value of $e^{c}_{31,f}$, while $e^{c}_{31,f}(V_{in,max})$ is the highest value of $e^{c}_{31,f}$ in the measurements. Input voltage V_{in} of $e^{c}_{31,f}(V_{in,min})$ should be lower than five times the input voltage V_{in} of $e^{c}_{31,f}(V_{in,max})$.

$e^{c}_{31,f}(V_{in,0})$ is the value $e^{c}_{31,f}$ at input voltage V_{in} = 0. It can be obtained from the extrapolation curve of the relationship between $e^{c}_{31,f}$ and V_{in}. The equation of the extrapolation curve can be determined by the examiner, but it should be indicated in the test report.

NOTE 1 Resonance frequency and Q factor are measured by frequency response of the displacement of the unimorph cantilever under the application of sinusoidal voltage between the top and bottom electrodes.

NOTE 2 Relative dielectric constant and dielectric loss are measured by an LCR meter or impedance analyser, typically at the frequency of 1 kHz.

5.3 Measuring procedures of direct transverse piezoelectric coefficient

The measuring procedures consist in the following steps:

a) measure the ambient temperature and relative humidity;
b) clamp one end of the unimorph beam with the thin film under testing to make a cantilever;
c) set the linear actuator to the tip of the cantilever and vibrate the tip of the cantilever with triangular wave;
d) measure the displacement of the tip of the cantilever;
e) measure the output voltage of the thin film under testing.

5.4 Measuring procedures of converse transverse piezoelectric coefficient

The measuring procedures consist in the following steps:

a) measure the ambient temperature and relative humidity;
b) clamp one end of the unimorph beam with the thin film under testing to make a cantilever;
c) apply unipolar sinusoidal voltage to the thin film under testing to vibrate the cantilever; application voltage is in the same direction as for the poling treatment if poling treatment is done to the specimen;
d) measure the input voltage to the thin film under testing;
e) measure the displacement of the tip of the cantilever;
f) input voltage sweeps more than three times, and the final sweeping data are used for the evaluation of $e^{c}_{31,f}$.

6 Test report

The test report shall include at least the following information.

a) Mandatory
   1) overall dimension of the unimorph beam;
      – length;
      – width;
      – thickness.
   2) length of the cantilever;
   3) specification of the thin film under testing;
      – composition;
      – thickness.
   4) deposition process of the thin film;
   5) substrate material and its properties;
      – material;
      – thickness;
      – crystal orientation in case of single crystal;
      – Young's modulus;
      – Poisson's ratio.
   6) electric properties of the thin film under testing;
      – capacitance;
      – dielectric loss.
   7) test environment;
      – temperature;
      – relative humidity.
   8) test conditions of direct transverse piezoelectric coefficient;
      – input tip displacement;
      – waveform applied to the linear actuator;
      – frequency applied to the linear actuator.
   9) test conditions of converse transverse piezoelectric coefficient;
      – input voltage;
      – waveform applied to the thin film under testing;
      – frequency applied to the thin film under testing.
   10) poling treatment conditions;
      – voltage;
      – poling direction;
      – waveform;
      – frequency;
      – poling time;
      – time from the poling to the measurement.

11) test items;
   - $e^{d}_{31,f}$;
   - $e^{c}_{31,f}$(min);
   - $e^{c}_{31,f}$(max).

b) Optional
   1) poling treatment conditions (opposite poling direction to the mandatory);
      - voltage;
      - poling direction;
      - waveform;
      - frequency;
      - poling time;
      - time from the poling to the measurement.
   2) test conditions of converse transverse piezoelectric coefficient;
      - input voltage;
      - waveform applied to the thin film under testing;
      - frequency applied to the thin film under testing.
   3) test items (opposite poling direction to the mandatory);
      - ed31,f;
      - ec31,f(min);
      - ec31,f(max).
   4) extrapolated converse transverse piezoelectric coefficient at 0 V;
      - ec31,f(0);
      - equation of extrapolation curve.
   5) resonance of the cantilever;
      - resonance frequency;
      - Q factor.

Annex A
(informative)

Example of measuring method of MEMS piezoelectric thin film

A.1 General

Annex A describes an example of a measuring method of MEMS piezoelectric thin film. Clauses A.2 to A.5 summarize the sample preparation procedures, poling treatment conditions, material properties for the calculation of the transverse piezoelectric coefficient, measuring procedures and measurement results.

A.2 Sample preparation procedures

The following steps show the sample (see Figure 1) preparation procedures.

a) The sample is a piezoelectric PZT thin film deposited by RF-magnetron sputtering on the unimorph beam of 625 µm thickness of (100) single crystal silicon substrate.
b) A platinum bottom electrode with titanium adhesive layer is preliminarily prepared on the silicon substrate.
c) A platinum top electrode is prepared on the surface of the PZT thin film.
d) Thicknesses of the PZT thin film, the platinum bottom electrode with titanium layer, and the platinum electrode are 3 µm, 0,2 µm and 0,05 µm respectively.
e) The sample is cut out to the beam shape of 20 mm length and 2 mm width by dicing saw along with <110> crystal orientation.
f) One end of the unimorph beam is clamped by the clamp.
g) Gold wires are bonded to the electrodes with silver paste.

A.3 Measuring procedures

A.3.1 Measuring procedures of direct transverse piezoelectric coefficient

The following steps show an example of measuring procedures of the direct transverse piezoelectric coefficient.

a) The sample is placed on the test bed shown in Figure 1, and the tip of the linear actuator is contacted to the tip of the cantilever from the back side.
b) Gold wires bonded to the electrodes are connected to the voltmeter (oscilloscope, FFT, or lock-in amplifier).
c) Before the measurement, a poling treatment is carried out if necessary. Poling treatment conditions are shown in Table A.1.
d) Table A.2 shows the dimension of the cantilever and necessary values to calculate the transverse piezoelectric coefficient.
e) Young's modulus and Poisson's ratio of the cantilever are used from the reference value of silicon of <110> crystal orientation.
f) The input tip displacement is measured by the Doppler laser vibrometer.
g) The input displacement varies between 1 µm and 23,5 µm at 5 Hz of vibration frequency.
h) Table A.3 shows the output voltage and the calculated transverse piezoelectric coefficient $e^{d}_{31,f}$ corresponding to the tip displacement of the cantilever.
i) Figure A.1 plots the input tip displacement of the cantilever and the calculated transverse piezoelectric coefficient $e^{d}_{31,f}$.

j) From the results of the measurements, the direct transverse piezoelectric coefficient $e^{d}_{31,f}$ can be determined as -6,4 C/m^2.

Table A.1 – Poling treatment conditions

Input signal	Conditions
Peak to peak voltage	20 V
Waveform	Negative unipolar sinusoidal wave
Frequency	1 kHz

Table A.2 – Material properties for calculation of direct transverse piezoelectric coefficient

Kind of properties	Material properties
Piezoelectric thin film material	PZT (Zr/Ti = 52/48)
Material of base cantilever	Silicon of (100) crystal orientation
Crystal orientation direction along the length	<110>
Poisson's ratio of base cantilever	0,064
Length of cantilever	17,45 mm
Width of cantilever	2,00 mm
Thickness of base cantilever	625 µm
Thickness of thin film	3,00 µm
Capacitance of thin film	0,056 27 µF
Dielectric loss	0,020 7

Table A.3 – Output voltage and calculated transverse piezoelectric coefficient

Tip displacement [µm]	Output voltage [V]	Direct transverse piezoelectric coefficient $e^{d}_{31,f}$ [C/m^2]
1,16	6,5	6,46
4,71	26,3	6,41
7,9	44,1	6,40
11,94	66,7	6,41
15,88	88,6	6,40
19,54	109,2	6,41
23,55	131,0	6,37

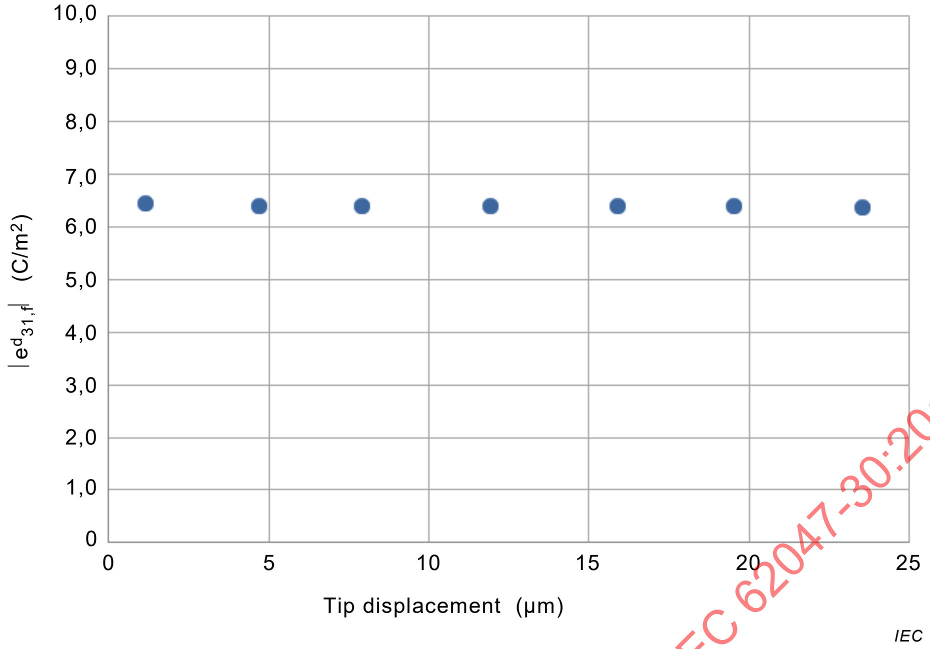


Figure A.1 – Tip displacement and calculated direct transverse piezoelectric coefficient

A.3.2 Measuring procedures of converse transverse piezoelectric coefficient

The following steps show an example of measuring procedures of converse transverse piezoelectric coefficient.

a) The sample is placed on the test bed shown in Figure 1.

b) The gold wires bonded to the electrodes are connected from the power amplifier.

c) Table A.4 shows the dimension of the cantilever and necessary values to calculate the converse transverse piezoelectric coefficient.

d) Young's modulus and Poisson's ratio of the cantilever are used from the reference value of silicon of <110> crystal orientation.

e) Before the measurement, a poling treatment is carried out if necessary.

f) Negative unipolar sinusoidal voltage is applied from the power amplifier to the top electrode.

g) The input voltage is varied between 5 V and 30 V.

h) The input voltage frequency is 400 Hz, and resonant frequency of the cantilever is 2,2 kHz.

i) The output tip displacement is measured by the Doppler vibrometer.

j) The tip displacement is measured by increasing the input voltage from 5 V to 30 V, by decreasing the input voltage from 30 V to 5 V, and again by increasing the input voltage from 5 V to 30 V. The measurement of input voltage and tip displacement is made three times in total.

k) Table A.5 shows the input voltage and the calculated transverse piezoelectric coefficient $e^{\mathrm{c}}{}_{31,\mathrm{f}}$ corresponding to the tip displacement of the cantilever.

l) Figure A.2 plots the relationship between the input peak-to-peak voltage to the piezoelectric thin film and the calculated transverse piezoelectric coefficient $e^{\mathrm{c}}{}_{31,\mathrm{f}}$.

m) From the extrapolated curve in Figure A.2, $e^{\mathrm{c}}{}_{31,\mathrm{f}}(V_{\mathrm{in},0})$ and $e^{\mathrm{c}}{}_{31,\mathrm{f}}(V_{\mathrm{in,max}})$ can be determined as 10,0 N/Vm and 15,0 N/Vm, respectively.

Table A.4 – Material properties for calculation of converse transverse piezoelectric coefficient

Kind of properties	Material properties
Piezoelectric thin film material	PZT (Zr/Ti=52/48)
Material of base cantilever	Silicon of (100) crystal orientation
Crystal orientation along the length	<110>
Young's modulus of base cantilever	$1{,}69 \times 10^{11}$ Pa
Poisson's ratio of base cantilever	0,064
Length of cantilever	17,45 mm
Width of cantilever	2,00 mm
Thickness of base cantilever	625 µm
Thickness of thin film	3,00 µm

Table A.5 – Tip displacement of cantilever and calculated transverse piezoelectric coefficient

	1st time [increasing voltage]		2nd time [decreasing voltage]		3rd time [increasing voltage]	
Input voltage [V]	**Tip displacement** [µm]	**Transverse piezoelectric** coefficient [N/Vm]	**Tip displacement** [µm]	**Transverse piezoelectric** coefficient [N/Vm]	**Tip displacement** [µm]	**Transverse piezoelectric** coefficient [N/Vm]
5	0,74	11,5	0,76	11,8	0,76	11,8
10	1,59	12,4	1,65	12,8	1,62	12,6
15	2,58	13,4	2,64	13,6	2,59	13,5
20	3,66	14,2	3,68	14,3	3,67	14,2
25	4,74	14,8	4,75	14,9	4,72	14,8
30	5,78	15,0	5,78	15,0	5,76	15,0

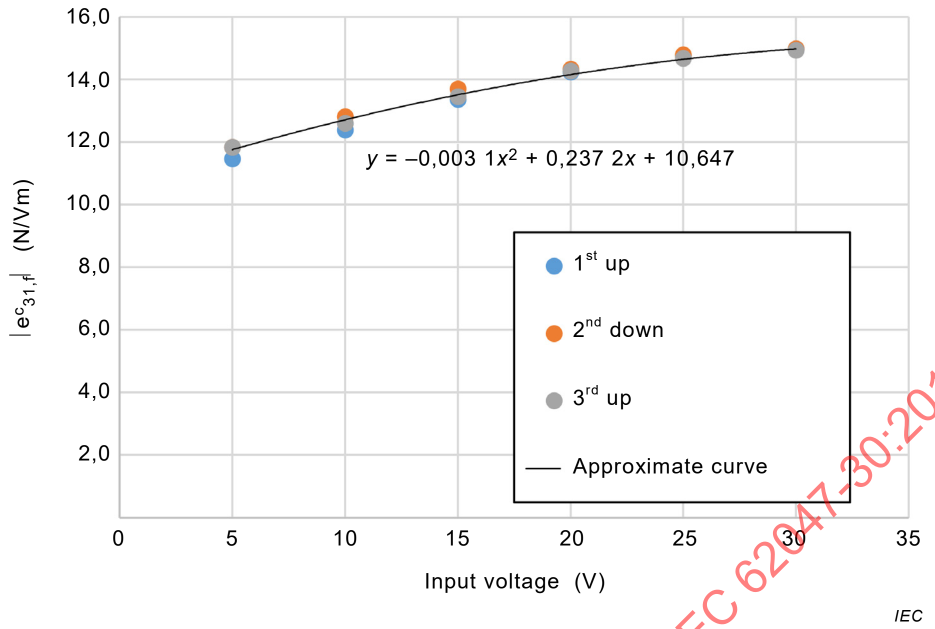


Figure A.2 – Input voltage and calculated converse transverse piezoelectric coefficient