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Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement method of piezoelectric strain at high electric field

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National foreword

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**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Measurement method of piezoelectric
strain at high electric field**

*Céramiques techniques — Méthode de mesurage de la contrainte
piézoélectrique à champ électrique élevé*





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Foreword

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The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement method of piezoelectric strain at high electric field

1 Scope

This International Standard specifies the measurement method of piezoelectric strain at high electric field for high power piezoelectric devices. This International Standard is intended to be used to determine the piezoelectric strain coefficient of the materials by measuring strain vs. electric field:

- applied electric field: 0 to 2 MV/m;
- frequency of electric field: 0,1 to 1 Hz.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60122-1, *Quartz crystal units of assessed quality — Part 1: Generic specification*

IEC 60483, *Guide to dynamic measurements of piezoelectric ceramics with high electromechanical coupling*

EN 50324-1, *Piezoelectric properties of ceramic materials and components — Part 1: Terms and definitions*

EN 50324-2, *Piezoelectric properties of ceramic materials and components — Part 2: Method of measurement — Low power*

3 Terms and definitions, and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60122-1, IEC 60483, EN 50324-1, EN 50324-2 and the following apply.

3.1.1

applied wave form

shape of voltage applied to specimen as a function of time

Note 1 to entry: Triangular waves are used in this International Standard.

3.1.2

signal generator

apparatus which controls the applied wave form

3.1.3

power source

high-voltage amplifier which generates an electric-field (E-field) applied to the specimen

3.1.4

E-field induced strain

strain of the specimen which is induced by applying the E-field

Note 1 to entry: The value is given by the ratio of displacement to specimen thickness.

3.1.5

displacement meter

apparatus which measures the E-field induced strain

3.1.6

mobile contact

mobile electrode which simultaneously holds the specimen and applies voltage to the specimen

3.1.7

maximum E-field

E_{\max}

maximum value of applied E-field

3.1.8

maximum strain

S_{\max}

strain at maximum E-field

3.1.9

residual strain

S_r

strain when E-field is removed

3.1.10

strain vs. E-field curve

continuously plotted curve of strain as a function of E-field

3.2 Symbols

f frequency

d_{\max} = S_{\max}/E_{\max}

E_h electric field at which the strain difference between the up- and down-curves shows a maximum

S_h strain difference between the up- and down-curves at E_h

H_{\max} = S_h/S_{\max}

4 Measuring environment

The measurement should be carried out in an environment free from acoustic noise and vibrations. It is desirable to control the measurement temperature 25 ± 5 °C and relative humidity less than 60 %.

5 Specimens

5.1 General

The test specimen shall be of any ceramics that can be cut into desired shapes such as disks and quadrilateral plates. There is no limit in specimen size and shape, if the specimen is held horizontally with a small tilt.

5.2 Shape and size

Specimens are made by cutting out from ceramics or single crystals. The shape of the specimen should be disk or quadrilateral plate, with smooth surfaces. Thickness distribution less than $\pm 1\%$ is desirable within the plate. The standard dimensions of the specimen are 0,3 mm - 1,0 mm in thickness, 5 mm - 15 mm in size, and 15 - 25 in the ratio of size to thickness. A desirable example of such disc is 0,5 mm in thickness and 10 mm in diameter.

5.3 Electrodes

Electrodes should be deposited on both sides of the specimen plate, leaving the edge region free. This edge region should have an area of less than 10 % of the sample surface. The electrode is desirable with tough adhesion and without deterioration. An example of such electrode metal is Au, Ag and Pt.

5.4 Polarization

The specimen should be treated by a poling procedure. Polarized direction should be shown on the specimen surface. Typically the positive side of the sample is marked with a dot or cross.

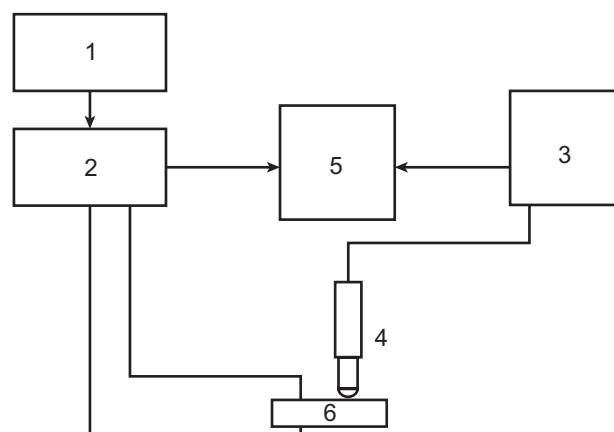
6 Principle

The strain of the specimen is induced by the piezoelectric effect when an electric field is applied between the two electrodes of the specimen. The strain is detected as the thickness variation of the specimen using a displacement meter. The strain vs. E-field curve is drawn, and the piezoelectric constant is calculated from this curve.

7 Measurement equipment

7.1 Equipment

[Figure 1](#) shows an example of a system block diagram for piezoelectric strain measurement.



Key

- 1 signal generator
- 2 power source
- 3 displacement meter
- 4 displacement sensor
- 5 monitor
- 6 specimen

Figure 1 — Schematic diagram of piezoelectric strain measurement

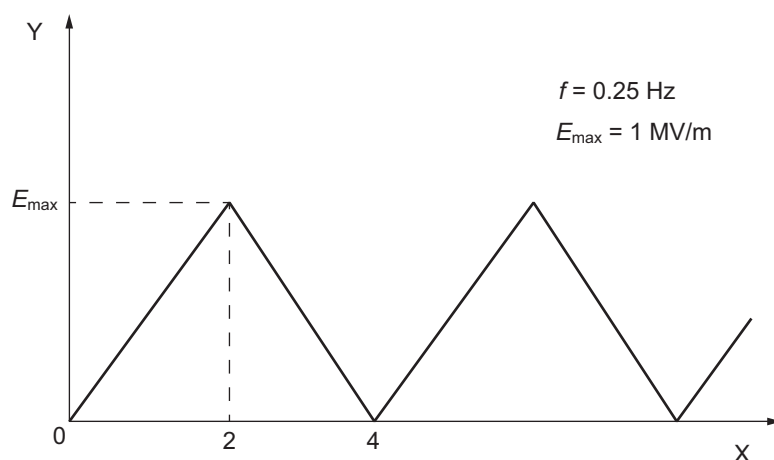
7.2 Components of equipment

7.2.1 Signal generator. A function generator is recommended to generate the triangular waveform with the frequency range of 0,1 Hz to 1 Hz. [Figure 2](#) shows an example of an applied waveform.

7.2.2 Power source. A power source which can generate voltages as high as several kV should be used.

7.2.3 Displacement meter. A measuring accuracy of ± 10 nm is needed for the displacement sensor. Both contact and non-contact sensors can be used. Different kinds of measuring system which have the required measuring accuracy can be used, such as differential inductive displacement gauge, magneto-resistance type linear displacement sensor, laser interferometer, capacitive method, etc.

7.2.4 Monitor. Instruments such as analog-to-digital converters and oscilloscopes are used for monitoring the relation between applied E-field and displacement, and for data acquisition.



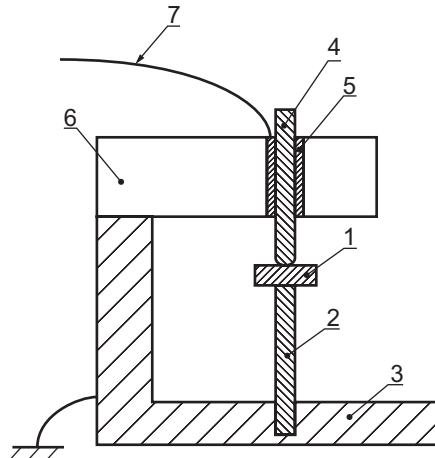
Key

X time (s)

Y E (MV/m)

Figure 2 — Example of applied wave form

7.2.5 Specimen holder. The specimen can be held horizontally or vertically. The specimen is placed between a stationary contact and a moving contact. The tip of the moving contact is recommended to have a sphere shape with a radius less than 2 mm. The tip of the stationary contact should be flat with a diameter less than 30 % of specimen diameter. The mechanical stress to the specimen shall be kept as low as possible in the measurement. The specimen holder has a structure which stably holds the specimen under applied E-field between both electrodes of the specimen. [Figure 3](#) shows an example of specimen holder structure. The bottom of the stationary contact is fixed in a metal base which is connected to the ground. The moving contact is supported by a linear bearing, which is fixed in a polymer block and is connected by a wire to the power source.



Key

- 1 specimen
- 2 stationary contact
- 3 metal stand
- 4 moving contact
- 5 linear bearing
- 6 polymer block
- 7 wire to power source

Figure 3 — Example of specimen holder structure

7.3 Environment of specimen holder

The specimen holder should be placed on an anti-vibration stage. A cover of the specimen holder is recommended to remove acoustic noise and to maintain a constant specimen temperature. It is effective to prevent discharge by immersing the specimen in isolation oil, although the use of isolation oil is not mandatory.

7.4 Calibration of measurement equipment

The measurement system shall be calibrated by using a reference sample (see [Annex A](#) for an example), which is accompanied by measured S_{max} data using the calibrated equipment. Measurement conditions and procedures for the reference sample are specified in [Clause 8](#) and [Clause 9](#), respectively. It is desirable that the measured strain at 1 MV/m is within the permissible range of the reference sample. If the measured value is out of range, it is recommended firstly to confirm movement of the mobile contact. If the mobile contact moves normally, then it is recommended to carry out calibration of signal generator, power source and displacement meter.

8 Measurement conditions

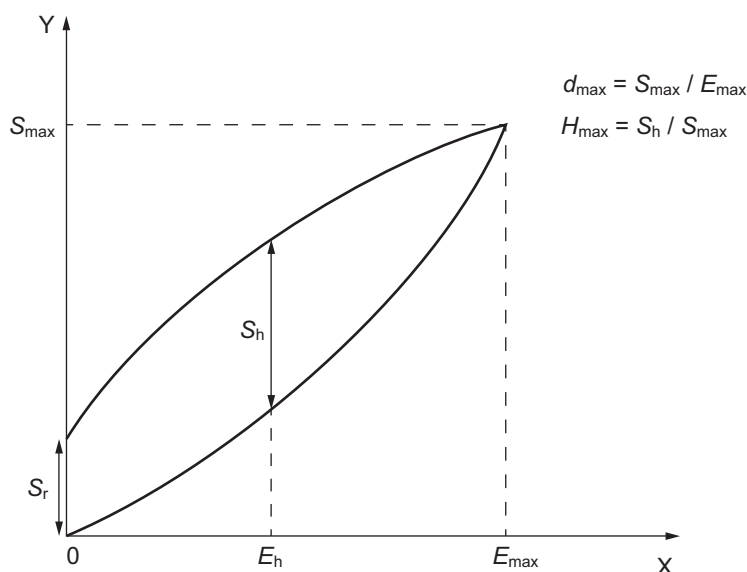
Strain vs. E-field curves are measured with unipolar change of E-field: the direction of the E-field should be parallel to the specimen poling direction. Triangular wave excitation should be used for the measurement. One cycle is defined by a ramp from 0 MV/m to the maximum E-field, the up-curve, followed by a ramp down to 0 MV/m, the down-curve, continuously. The frequency of the E-field is defined as the inverse time of one cycle. The time interval between each cycle can be chosen arbitrarily. The maximum E-field is less than 2 MV/m.

9 Measurement procedures

- a) Measure the specimen thickness repeatedly three times using a micrometre. Calculate average thickness from the measured values.
- b) Place the specimen in the specimen holder.
- c) Measure the strain vs. E-field curves using a series of 5 triangular wave cycles. Record the measurement data of cycle numbers 3, 4 and 5. The number of sampling data points in one curve should be larger than 100.

10 Calculations and results

Analyse the strain vs. E-field curves and calculate the parameters, S_{\max} , E_{\max} (MV/m), d_{\max} (m/MV), E_h (MV/m), S_h , H_{\max} and S_r , as shown in [Figure 4](#). Then record the values of the parameters for each curve with three significant figures.



Key

- X E-field (MV/m)
Y strain

Figure 4 — Analysis method of strain vs. E-field curve

11 Test report

A test report should be written in order to include the experimental results properly in the written form. The experimental conditions shall be indicated with the following information:

- a) the test date, laboratory and name(s) of the operator(s);
- b) the product type of specimen and name if possible; product-type may include material, shape, composition, etc.
- c) the measured thickness of the specimen;
- d) a list of the equipment used for the test, with schematic diagrams and photographs of the measurement system and specimen holder;
- e) a description of the measurement environment of the specimen holder;

- f) the setup conditions (maximum applied voltage, maximum applied E-field, frequency of the E-field, and time interval between tests i.e. waiting time) of each measurement;
- g) the measurement results of cycle number 3, 4 and 5, presented in a table and graph forms (see [Figure 4](#)).

Annex A (informative)

Example of reference sample

Specifications of an example reference sample¹⁾ are as follows:

- a) material: PZT ceramics
- b) type: N82
- c) Specifications:
 - Curie temperature $T_c > 350$ °C;
 - diameter = 17,8 mm;
 - thickness = 1,0 mm;
 - electromechanical coupling factor in radial direction $K_r(20$ °C) = 0,619 2 ± 0,000 5;
 - S_{\max} values at 1 MV/m are shown in [Table A.1](#).

Sample should be stored in a box filled with inert gas.

Table A.1 — S_{\max} values at $E_{\max} = 1$ MV/m

Temperature °C	S_{\max} ($\times 10^{-4}$)	Permissible range ($\times 10^{-4}$)
10	3,04	±0,15
15	3,11	±0,15
20	3,16	±0,16
25	3,27	±0,16
30	3,37	±0,17

1) Reference material manufactured by NEC Tokin Co. Ltd. and distributed by Japan Fine Ceramics Centre, 2-4-1 Mutsuno, Atsuta-ku, Nagoya, 456-8587 Japan. Tel:+81-52-871-3500, Fax:+81-52-871-3599 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

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