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Fine ceramics (advanced ceramics, advanced technical ceramics) — Characteristic of piezoelectric properties under high-load conditions —

Part 1: Resonant-antiresonant method under high-temperature conditions

Céramiques techniques (céramiques avancées, céramiques techniques avancées) — Caractéristique des propriétés piézoélectriques en conditions de charge élevée —

Partie 1: Méthode résonante-antirésonante à des températures élevées

Reference number
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Foreword

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

A list of all parts in the ISO 21819 series can be found on the ISO website.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Characteristic of piezoelectric properties under high-load conditions —

Part 1:
Resonant-antiresonant method under high-temperature conditions

1 Scope

This document specifies a method of measuring piezoelectric properties of piezoelectric fine ceramics and other piezoelectric devices under high-temperature conditions, where the electromechanical coupling coefficient is determined based on measurements of resonance/antiresonance frequencies using impedance analysers.

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.

IEC 60584-1, *Thermocouples — Part 1: Reference tables*

IEC 60584-2, *Thermocouples — Part 2: Tolerances*

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*

EN 50324-3, *Piezoelectric properties of ceramic materials and components — Part 3: Method of measurement — High power*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 50324-1, EN 50324-2 and EN 50324-3 and the following apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
resonant-antiresonant method
method where the ratio of mutual conversion between electrical energy and mechanical energy is measured based on the resonance frequency and antiresonance frequency of piezoelectric fine ceramics and other piezoelectric devices

3.2
resonance frequency
f_r
lower of two frequencies at which admittance or impedance between electrodes of a transducer achieves zero phase in the vicinity of the target vibration mode

3.3
antiresonance frequency
f_a
higher of two frequencies at which admittance or impedance between electrodes of a transducer achieves zero phase in the vicinity of the target vibration mode

4 Symbols

$\Delta k(t)$ Rate of change of electromechanical coupling coefficient (%)

k_t Electromechanical coupling coefficient after holding for t min after the test piece reaches the test temperature ± 2 °C

k_1 Electromechanical coupling coefficient after holding for 1 min after the test piece reaches the test temperature ± 2 °C

5 Principle

Under high-temperature conditions, the polarization within piezoelectric ceramics becomes unstable and there is thus a risk that the piezoelectric properties might deteriorate as time advances. In order to take both the high-temperature environment and elapsed time into consideration, the electromechanical coupling coefficient is measured in a high-temperature environment as a function of time from the time point where the high-temperature conditions are reached.

Note that other methods of measuring piezoelectric properties, such as electric field-induced strain and/or resonance vibration displacement, can also be considered. However, this document makes use of the measurement of the electromechanical coupling coefficient via the resonant-antiresonant method due to the ease of measurement.

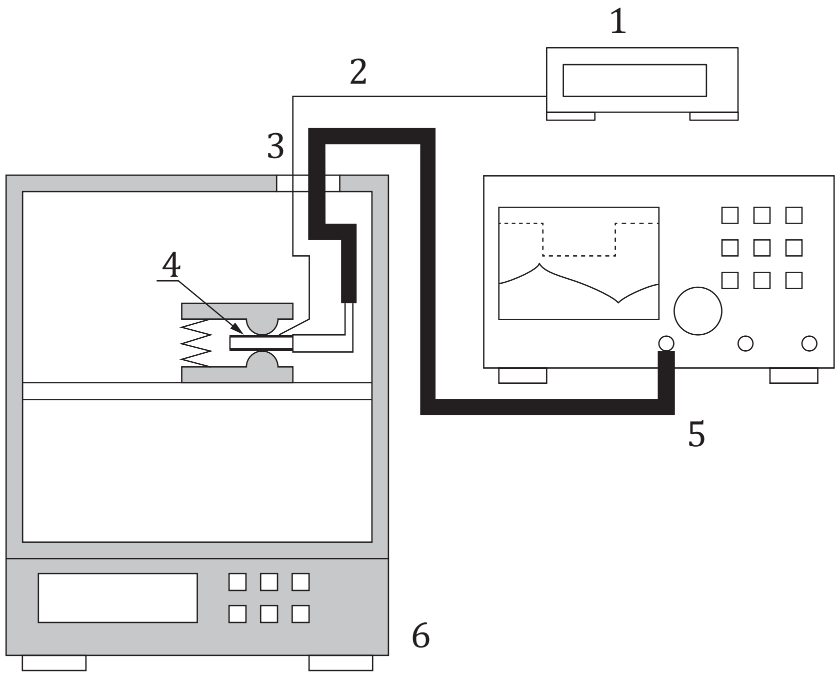
6 Specimens

This measurement method is not limited to the measurement of materials, but can also be applied to all piezoelectrics and other piezoelectric devices that are intended to operate in a high-temperature environment.

7 Measurement equipment

7.1 General

This clause details the apparatus used for measurement. Figure 1 explains the configuration of the measurement apparatus.



Key

1 temperature measurement
2 thermocouple
3 through-hole
4 test piece
5 impedance analyser
6 thermostatic chamber

Figure 1 — Schematic diagram of measurement apparatus configuration

7.2 Impedance analyser

An instrument for obtaining the impedance, which is a ratio of voltage and current, and the admittance, which is the inverse, by applying alternating voltage to piezoelectric fine ceramics and piezoelectric devices while scanning through a range of frequencies.

7.3 Thermostat chamber

A thermostat chamber used for this method shall be fit to be used in the full service temperature range, from ambient temperature to test temperature, with a temperature fluctuation range of ± 5 °C or better, and have a structure that allows recording the internal temperature continuously using thermocouples. It is recommended that the thermostat chamber is equipped with an air blower inside, allowing forced air ventilation/circulation in order to be able to keep the temperature within the chamber uniform.

7.4 Thermocouple

Thermocouples prescribed by IEC 60584-1 and IEC 60584-2.

7.5 Recorder

An instrument capable of recording temperature continuously.

7.6 Test piece holder

No particular shape is specified for a test piece holder, but consideration shall be given such that the holder does not affect the target vibration mode of the device under test.

8 Measurement conditions

The measurement temperature conditions and measurement items are as follows. Note that the test temperature shall be at the Curie point or lower.

a) Measurement start temperature (temperature of test piece at test start): (25 ± 5 °C) (ambient temperature).

b) Test temperature (temperature measured at the test piece or near the test piece): 100 °C or higher.

c) Program for raising temperature (rate of temperature change): within ± 10 °C/min.

d) Test piece temperature (temperature measured by a thermocouple reaches the test temperature ± 2 °C).

e) Measurement end temperature (temperature of test piece at test end): (25 ± 5 °C) (ambient temperature).

f) Measurement items: test piece temperature, electromechanical coupling coefficient, resonance frequency and antiresonance frequency.

It is permissable to measure the admittance values at the resonance and antiresonance frequencies, the equivalent circuit constants at the target vibration mode, as well as the relative permittivity and dielectric loss at 1 kHz as required.

NOTE The electromechanical coupling coefficient is defined according to the object to be measured. For example, Formula (1) is defined by using resonant frequency f_r and antiresonance frequency f_a and this is the electromechanical coupling coefficient k in the noted vibration mode.

$$k = \sqrt{1 - \frac{f_r^{\,2}}{f_a^{\,2}}} \tag{1}$$

where

k is the electromechanical coupling coefficient;

f_r is the resonant frequency;

f_a is the antiresonance frequency.

9 Measurement procedure

The measurement is carried out according to the following procedure.

a) Place a test piece in a thermostatic chamber.

b) Connect the test piece and an impedance analyser, for example using a test piece holder, such that data can continuously be obtained from the test piece placed in the thermostatic chamber.

It is recommended that a short lead wire is used for the test piece holder because measurement values obtained using a long lead wire might be different from those obtained when the test piece and the impedance analyser are connected without using a lead wire.

c) Calibrate the impedance analyser to eliminate any impact from the impedance in the lead wire and other connections on the measurement results.

d) Attach a thermocouple to the test piece or place it near the test piece. At this point, it is recommended that the thermocouple is connected to a recorder in order to record the temperature continuously.

e) Measure the initial value at ambient temperature [measurement item listed in Clause 8 f)].

NOTE This temperature is equivalent to the test piece temperature at the test start, in Clause 8 a).

f) Raise the temperature in the thermostatic chamber to the test temperature as described in Clause 8 d) and measure the temperature 1 min after the test piece temperature reaches the test temperature ± 2 °C.

NOTE The test temperature and the temperature indicated by the thermostatic chamber are not necessarily the same. Moreover, there are cases where the temperature indicated by the thermostatic chamber and the test piece temperature might be different.

g) Keep the test piece temperature at the test temperature ± 2 °C and measure the value at least three more times at sufficient time intervals [measurement items listed in Clause 8 f)].

NOTE Sufficient intervals refer, for example, to 1 min later, 10 min later, 100 min later or 1 000 min later.

h) Lower the temperature in the thermostatic chamber to the ambient temperature and measure the value when the test piece temperature drops down to 25 ± 5 °C (ambient temperature) [measurement item listed in Clause 8 f)]. In addition, record the time required for the test piece temperature to drop down to the ambient temperature at which the measurement is made.

This temperature is equivalent to the test piece temperature at the test end in Clause 8 e). It is better to wait for a sufficient period of time before making the measurement to allow the temperature to settle, rather than measuring immediately after the test piece temperature reaches the ambient temperature.

10 Calculation procedures of measured results

10.1 Calculation of measurement data

Round off resonance frequency and antiresonance frequency to three decimal places.

Round off electromechanical coupling coefficient, each admittance value, equivalent circuit constants and dielectric loss at 1 kHz to two decimal places.

Round off relative permittivity at 1 kHz to the integer number.

10.2 Rate of change of electromechanical coupling coefficient

Obtain the electromechanical coupling coefficient k based on the resonance frequency f_r and antiresonance frequency f_a defined by Formula (1) and then obtain the rate of change in electromechanical coupling coefficient $\Delta k(t)$ using Formula (2), rounding off $\Delta k(t)$ to two decimal places.

$$\Delta k(t) = \frac{k_t - k_1}{k_1} \tag{2}$$

11 Test report

The following items shall be reported for measurement:

a) the number of this document, i.e. ISO 21819-1:2018;

b) measurement date, testing supervisor;

c) measurement data of type, material, shape and thickness of test piece;