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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for piezoelectric constant d_{33} of piezoelectric ceramics by direct quasi-static method

Céramiques techniques (céramiques avancées, céramiques techniques avancées) — Méthode d'essai pour déterminer la constante piézoélectrique d_{33} *des céramiques piézoélectriques par méthode quasi-statique directe*

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Foreword

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

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for piezoelectric constant d_{33} of piezoelectric ceramics by direct quasi-static method

1 Scope

This document specifies how to measure the piezoelectric constant d_{33} of piezoelectric ceramics using a direct quasi-static method (d33 meter method, Berlincourt method).

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 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20507 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
piezoelectric constant
d_{33}
part of the piezoelectric fundamental equation (*d*-form):

$$S = s^{E}T + dE \text{ (inverse piezoelectric effect)} \quad (1)$$

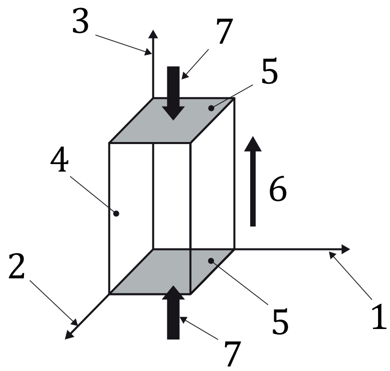
$$D = dT + \varepsilon^{T}E \text{ (direct piezoelectric effect)} \quad (2)$$

where

S	is strain;
T	is stress, in N/m^2;
E	is electric field (strength), in V/m;
D	is electric flux density, in C/m^2;
ε^{T}	is free permittivity (assuming constant T), in F/m;
s^{E}	is elastic compliance (assuming constant E), in m^2/N.

The piezoelectric constant *d* in Formula (1) expresses the inverse piezoelectric effect, while *d* in Formula (2) expresses the direct piezoelectric effect. The unit of *d* in each equation is [m/V] and [C/N], respectively, and they are equivalent. The subscript 33 in the symbol d_{33} indicates the vibration mode; the two digits indicate electric axis (first) and mechanical axis (second), respectively. Figure 1 shows a schematic diagram of poling (polarization) direction and the axes of piezoelectric specimen. Since descriptions of piezoelectric ceramics typically use the polarization axis direction as axis 3, in this case, the polarization axis direction (axis 3) indicates electric flux density D_3 along axis 3 when stress T_3 is added. Now, assuming electric field *E* is constant (for example, 0) in Formula (2) that expresses the direct piezoelectric effect, d_{33} can be found using Formula (3):

$$d_{33} = [\delta D_3 / \delta T_3]_E \quad (3)$$



Key

1 axis 1
2 axis 2
3 axis 3
4 specimen
5 electrodes
6 polarization direction
7 stress, T_3

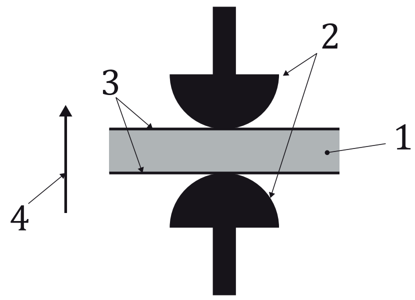
Figure 1 — Schematic diagram of poling (polarization) direction and the axes of piezoelectric specimen

4 Measurement principle

Figure 2 shows a conceptual diagram. Specimen (1), which is a piezoelectric ceramic plate, is coated with electrode (3) on both the upper and lower surfaces, and polarization treatment is applied in the third axis direction (from bottom to top or top to bottom). The specimen is held fixed by contact probes (2), which are loaded with a dead load (specimen gripping force). Denoting the electrode area (i.e. the area of the surface to which stress is applied) by *A* [m^2], the surface charge by *Q* [C], and the applied alternating force by *F* [N], the electric flux density *D* becomes $D = Q/A$ and the stress *T* is expressed as $T = F/A$, which implies that Formula (3) can be rewritten as Formula (4):

$$d_{33} = (Q/A)/(F/A) = Q/F \quad (4)$$

Now, if an alternating force *F* is applied to the specimen from the bottom (or top) of the probe, an alternating charge *Q* is generated on the upper and lower electrodes of the specimen. As a consequence, by applying an alternating force *F* with known amplitude, the d_{33} value can be obtained from Formula (4) by measuring the generated charge *Q*. The generated charge *Q* is calculated by measuring the AC current in a circuit with virtual ground or AC voltage of a standard capacitor with a sufficiently larger capacity than the specimen. In principle, the d_{33} value can then be obtained by using Formula (4), but in practice the measured alternating current signal will be obtained as a root-mean-square value (RMS). This means that the obtained d_{33} value (Q/F value) is not absolute; instead, the Q/F value is only proportional to the true d_{33} value. Therefore, the final d_{33} value of the measured specimen shall be obtained relative to calibration measurements using standard specimens with known d_{33} values in advance.



Key

1 specimen (piezoelectric ceramic plate)

2 probe

3 electrode

4 polarization direction (3 axis)

Figure 2 — Conceptual diagram

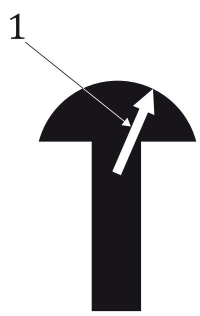
5 Equipment and instruments

5.1 Test equipment

The test equipment is composed of a force unit that holds a specimen in a fixed position and applies alternating force and a measuring unit that measures the generated charge. In addition, it is recommended that a force gauge is attached in the vicinity of the contact probes in order to monitor the dead load when the specimen is held.

5.2 Probes

The test probes shall have spherical surfaces in the areas that apply load to the specimen, as shown in Figure 3. The radius of the spherical surface shall be at least 3 mm; the greater the better. However, flat probes (probes whose contact surface is flat) shall be avoided.



Key

1 radius

Figure 3 — Probe

6 Specimens

6.1 Standard specimens

The piezoelectric constant d_{33} of standard specimens is obtained utilizing the resonance-antiresonance method prescribed by IEC 60483 (hereinafter referred to as the resonance method). The shape of the standard specimens shall conform to 6.2. Ideally, the standard specimens shall be made from the same material as the specimens to be measured, but can be substituted with specimens with different chemical composition if standard specimens of the same composition cannot be obtained.

6.2 Shape and size of specimens

The shape of the specimens shall be such that they allow for the forming of electrodes on the entire surface so that the electric charge Q corresponding to D_3 can be measured, and shall allow application of alternating force F in the polarization axis direction. In addition, if a tabular oscillator shaped like a circular or rectangular plate – a typical ceramic shape – is used, the thickness of the specimens (distance between the two probes) shall be at least 1,5 mm. 2 mm or greater is recommended. Moreover, the ratio between the thickness and diameter of each specimen (thickness/diameter) shall be at least 0,1 or larger.

In the case of a rectangular plate with a thickness t, a width W, and a length L, both t/W and t/L shall be larger than 0,1 at the same time.

7 Test method

7.1 Test environment

The test shall be conducted at temperatures of 15 °C to 30 °C.

If the room temperature is near the phase transition temperature and phase transition is likely to occur due to application of stress, take care as the result may differ significantly from the d_{33} value obtained using the resonance method.

NOTE There are possible effects by temperature stability of a standard specimen.

If specimens are easily affected by humidity, the humidity shall be adjusted in order to minimize its influence.

7.2 Test conditions

The test shall be conducted under the following conditions.

7.2.1 Specimen fixing position

The specimen shall be fixed at the geometric centre of the surface to which the alternating force F is applied.

7.2.2 Specimen gripping force

The specimen gripping force shall be the minimum force within the range 0,5 N to 10 N that keeps the specimen still. If the gripping force cannot be measured, it is advisable to fix the specimen with a force that keeps the specimen immovable, but keep the same gripping force as for the standard specimens under the same conditions.

The local compression stress applied to the specimen depends on the radius of the contact probes used. Therefore, if probes with small radii are used, a smaller gripping force is recommended.

7.2.3 Frequency of alternating force

The measurement frequency shall be between 90 Hz and 300 Hz, preferably around 100 Hz.

Use the same frequency when measuring the standard specimen and the test specimen.

Avoid frequencies that are integer multiples of the power supply frequency to be used for the instrument.

Test frequency should be far below the resonance frequency of the test specimen and also away from the resonance of the system.

7.2.4 Amplitude of alternating force

The amplitude of the alternating force shall be in the range 0,1 N to 0,4 N (RMS).

7.2.5 Measuring time

The measuring time here means the time elapsed after fixing the specimen with the probes until the d_{33} value is obtained. It is recommended that the measuring time is recorded by keeping time series data of the d_{33} measurements and determining the time required for the value to stabilize. If the value does not stabilize within 1 min, set the measuring time to 1 min while noting clearly that the d_{33} value was obtained after 1 min had elapsed (1-min value).

7.2.6 Number of measurements

It is recommended that three to five measurements are obtained for both the front and back surfaces of each specimen. After each measurement, disengage the contact probes from the specimen once before fixing the specimen again.

8 Calculation

Obtain the piezoelectric constant d_{33} using Formula (5), based on the obtained measurement results of positive and negative piezoelectric constant d_{33}.

$$d_{33} = (|d_{33}(+)| + |d_{33}(-)|)/2 \qquad (5)$$

The piezoelectric constant d_{33} of the material shall be determined by computing the average measurement values of the front and back surfaces of the specimens.

9 Report of test results

As test results, report the following items:

a) document number;

b) test date;

c) test facilities, test equipment and probe shape;

d) standard specimens, measurement test materials and their shapes;

e) test conditions (temperature, humidity, fixing position, gripping force, frequency of alternating force, amplitude of alternating force, measuring time);

f) measurement results.