COMPARISON BETWEEN METHODS FOR THE MEASUREMENT OF THE $D_{33}$ CONSTANT OF PIEZOELECTRIC MATERIALS

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The piezoelectric strain constant is one of the key parameters that determines the conversion efficiency between electrical and acoustical energy for piezoelectric materials widely used in acoustic transducers and sensors. For most transducers working in longitudinal mode, the longitudinal piezoelectric strain constant, $d_{33}$, is of particular significance, especially for sensor and transducer arrays. The natural question arises on how to measure such a constant. Typical approaches include the quasi-static method, the dynamic resonant method and the laser interferometry method. The reported measurement systems were developed at the National Institute of Metrology China and the methods were applied on the same samples for comparison purposes. The quasi-static method is simple to operate and more suitable for most PZT materials. With regards to the resonant method, there are special requirements with regards to the sample size, the necessity for an LCR analyzer as well as the quality of the fixture, all of which play a significant role in the measurement itself. The method relying on laser interferometry, uses the reverse piezoelectric effect to obtain the displacement when an AC voltage is applied on the samples. Comparison results showed that for the P5 material, the value of the $d_{33}$ constant obtained by the resonant method is approximately 7% lower than that of the quasi-static method, while the value of the constant based on the laser interferometry method is always higher than that of the quasi-static method. When the thickness-to-diameter ratio of the piezoelectric ceramic is set to 0.5, the values of $d_{33}$ obtained by the quasi-static and the laser interferometry method are very close indeed. Potential reasons that contribute towards the difference between the methods, as well as the traceability of the quasi-static method, are discussed.
Keywords: $d_{33}$ constant, piezoelectric materials, quasi-static method, dynamic resonant method, laser interferometry method.

1. Introduction

Piezoelectric materials are widely used in acoustic transducers and sensors. When designing piezoelectric actuators and sensors, it is important to consider the parameters of the material. The piezoelectric strain constant is one of the key parameters that determines the conversion efficiency between electrical and mechanical energy. For most transducers working in longitudinal mode, the longitudinal piezoelectric strain constant $d_{33}$ is of particular significance, especially for sensor and transducer arrays. Standard practices use multiple measurement methods in order to provide the value of the coefficient under consideration. At present, the three most popular approaches are the
quasi-static method, the dynamic resonant method (frequency method) and the laser interferometry method [1].

The natural question relates to the particular method that is most accurate to measure the piezoelectric strain constant. Previous research has compared the differences between these methods. The most comprehensive work by Fialka [2] investigated all three approaches for the measurement of piezoelectric coefficients and came to the conclusion that all could be regarded as equivalent with respect to the measurement accuracy. However, there are still several key questions that need to be answered. One of them, is that the measurements are performed at different frequencies for different methods, while the piezoelectric strain constant is, obviously, a function of frequency. For example, the dynamic resonant method obtains the constant at the resonant frequency of the samples, which is significantly different from the frequency used for the quasi-static method. To compare the three methods for piezoelectric strain constant measurements, the three systems were developed at the National Institute of Metrology China. Each of these methods were studied and a critical comparison between them was carried out.

2. Measurement systems and test samples

Because of the relative ease of operation, the quasi-static method is widely used in the PZT industry. The laser interferometry method on the other hand is mostly used in laboratory environments, because of the necessity for vibration isolation. With the dynamic resonant method, most of the parameters, in addition to the piezoelectric strain constant, can be measured.

2.1 Quasi-static system

The quasi-static method is based on the principle of the direct piezoelectric effect. When a low frequency force is applied on a polarized piezoelectric ceramic, the stressed surface will generate an alternating electric charge. By combining the measured charge and the applied force, the piezoelectric constant can be calculated. For such measurements, the recommended frequency range of the force is in the range 20 Hz to 200 Hz and the applied force from 0.1 N to 0.4 N. Figure 1 shows the schematic diagram of the measurement setup. Different to the commercial quasi-static measurement apparatus such as type ZJ-3, the dynamic force transducer (type Sinocera CL-YD-303) was selected instead of a reference piezoelectric material. An Agilent signal generator (type 33220A) was used to drive the shaker (type JZK-2) in order to generate the required force in the range 0.1 N to 0.4 N. A tension gauge was used to monitor the preload which was used to hold the sample in place during measurements. The outputs of the force sensor and the PZT piece under test were connected to a charge amplifier (type B&K 2690). Subsequently, the signals were captured by an oscilloscope (type T&K 4034) and the value of $d_{33}$ could thus be calculated from the charge-to-force ratio.

![Figure 1: Block diagram and picture of the quasi-static system](image-url)
2.2 Dynamic resonant system

In a given mechanical or electrical system, certain vibrations can naturally occur after external excitation conditions. The free vibration frequency of the system depends on its physical size and, of course, the smaller the size, the higher the free vibration frequency. With regards to resonant characteristics, large amplitude oscillations can be generated by a small force within a specific frequency range. For piezoelectric materials, the resonant effect can be induced by applying an alternating voltage on polarized ends of the material instead of mechanical stress. Each sample has a variety of vibration modes and selection of different directions can induce different resonant effects. To ensure pure longitudinal resonance, test samples need to meet certain requirements, with one example being that the length of the circular column should be 2.5 times larger than its diameter for a sample of such particular shape.

The equivalent circuit of piezoelectric materials is usually composed of the parallel connection of two parts; one is the static capacitance $C_0$, the other is the series connection of inductor $L$, capacitance $C$, and resistance $R$ which represent its dynamic characteristics. In the process of the measurement, the alternating current in sweeping-frequency mode is used to excite the piezoelectric material under test, the impedance characteristics of which are analyzed to obtain its series and parallel resonant frequencies ($f_s$, $f_p$ respectively) as well as other characteristic parameters under consideration. The sinusoidal excited signal with the same amplitude in the sweeping-frequency process is used in the measurement. When the frequency of the excitation signal is equal to the natural frequency of piezoelectric crystal in vibration mode, the real part of its admittance takes a maximum value and the imaginary part is close to zero. With the frequency sweeping, the admittance circle, which represents the real and imaginary part of the piezoelectric crystal’s admittance, is drawn on a coordinate system. Subsequently, series and parallel resonant frequencies $f_s$ and $f_p$, the resonant frequency $f_r$, anti-resonant frequency $f_a$, maximum impedance frequency $f_m$, minimum impedance frequency $f_m$, can also be obtained through the admittance circle [3]. The main equations related to parameters calculation are shown as follows.

The free dielectric constant $\varepsilon_{33}^{T}$ is given by:

$$\varepsilon_{33}^{T} = \frac{C_T l_t}{A}$$  \hspace{1cm} (1)

where $C_T$ is the free capacitance, $l_t$ is the sample thickness and $A$ is the sample area.

The longitudinal electromechanical coupling constant $k_{33}$ can be calculated from:

$$k_{33} = \frac{\pi}{2} \frac{f_s}{f_p} \cot\left(\frac{\pi}{2} \frac{f_s}{f_p}\right)$$  \hspace{1cm} (2)

The short-circuit elastic compliance constant $s_{33}^E$ is given by:

$$s_{33}^E = \frac{1}{4(1-k_{33}^2)l_t^2 f_p^2 \rho}$$  \hspace{1cm} (3)

where $\rho$ is the sample density. The longitudinal piezoelectric strain constant is given by:

$$d_{33} = k_{33} \sqrt{\varepsilon_{33}^T s_{33}^E}$$  \hspace{1cm} (4)

An LCR meter (type E4980A) was used to measure the impedance characteristics of the piezoelectric ceramic material. The data acquisition and processing between computer and impedance analyzer was performed using LabVIEW. The details of automatic measurement system have previously been reported in the literature [4]. The system mainly consisted of an impedance analyzer VISA interface and mode setting, input of physical parameters of piezoelectric material, frequency step setting, impedance and admittance graphic display, and output of measurement parameters, as shown in Fig. 2. The six characteristic frequency points, electro-mechanical coupling coefficient, dielectric constant, free capacitance, elastic compliance constant and piezoelectric constant of piezoelectric ceramic material.
zoelectric ceramics can be measured. The program could perform $d_{33}$ and $d_{31}$ measurements of piezoelastic ceramic columns which meet the necessary size requirements.

![Dynamic Resonance Method Measuring System](image1)

**Figure 2**: Program interface of the dynamic resonant system

### 2.3 Laser interferometry system

The laser interferometry method is based on the principle of the reverse piezoelectric effect. After the application of a polarization voltage, the piezoelectric ceramics will generate micro-range displacements in the thickness direction where the external electric field is applied to. The piezoelectric equation is given by:

$$S = sE + dE$$  \hspace{1cm} (5)

where $S$ is the strain, $E$ is the applied electric field, $sE$ is the elastic compliance constant and $T$ is the stress. When $T$ is set to zero, the longitudinal piezoelectric strain constant $d_{33}$ is written as:

$$d_{33} = S_3 / E_3$$  \hspace{1cm} (6)

where $S_3$ is the displacement at the longitudinal direction and $E_3$ is the electric field.

The sample under test was fixed on a platform (probe table) by gas adsorption, as shown in Fig. 3.

![System of the laser interferometer method](image2)

**Figure 3**: System of the laser interferometer method

Two probes provided the AC voltage signal on the sample and the telescope was used to observe the contact details. A commercial laser interferometer (Polytec OFV552) was used to measure the displacement of the upper surface of the sample.

### 2.4 Test samples

Soft piezoelectric ceramic cylinders, PZT5, all provided from the same manufacturer were selected as the test samples. For the dynamic resonant method, the diameter of the samples was 5 mm and the height was 15 mm, with 5 samples in total tested. As there are no specific dimension require-
ments for the quasi-static method and laser interferometry methods, PZT5 samples with diameter of 10 mm and thickness of 2 mm, 5 mm and 8 mm respectively were used in order to compare the measurement results between the two methods.

3. Influence factors of each method

Every method has its own influence factors. To compare the test results from different methods, it is obviously necessary to know such factors and control them. The $d_{33}$ constant is a function of temperature for most piezoelectric materials, so all the tests were carried out in a laboratory room with the temperature controlled to $20^\circ C \pm 0.2^\circ C$.

3.1 Quasi-static method

Regarding this particular method, the preload, excitation force and frequency, shape of the holding heads and loading time, can potentially affect the measurement results. Experimental results showed that while the preload had different effects on hard and soft ceramics materials, the frequency and the amplitude of the exciting force had a significant impact on the results [1,5].

With the preload increasing, the $d_{33}$ measurement of hard materials was influenced much less compared to that of the soft material. When the preload increased to 10 N, the measurement results showed significantly low levels of fluctuations. Therefore from an empirical perspective, for both the soft and hard piezoelectric ceramics the preload should be no less than 10 N.

For the test samples discussed in section 2.4, the parameters of the quasi-static method were frequency of excitation force 110 Hz, amplitude of the force 0.25 N, with the preload at 10 N. Hemispherical holding heads were used to apply the force.

3.2 Dynamic resonant method

The $d_{33}$ value obtained by the dynamic resonant method, was obtained by means of simultaneously solving piezoelectric equation, wave equation and dynamic equation according to the Mason equivalent equation. The influencing factors include sweeping-frequency step, fixture, clamp impedance and environmental interference conditions.

The key issue in the dynamic resonant method is how to obtain the ideal admittance circle, so the accuracy of the measured impedance at each frequency is as high as possible. The measurement accuracy of frequencies has a corresponding relationship with the sweeping-frequency step of excitation signal and the step depends on the rate change of the impedance angle of the piezoelectric materials. By taking into account the optimization between the measurement accuracy and the measurement time, the sweeping-frequency step of the samples discussed in section 2.4 was set to 50 Hz. The fixture also played an important role in the measurement process and, for this reason, a tweezer contact test fixture 16334A was chosen. The two polarizing electrode surfaces of test sample were clapped by the fixture and then were directly connected to the impedance analyser.

3.3 Laser interferometry method

The commercial laser interferometer was used to measure the displacement when an AC voltage was applied on the test sample. The influencing factors include the fixture, the excitation field strength and frequency.

During the measurement of piezoelectric materials, the polarization field varies between 0.6-1.5 kV/mm. To measure the piezoelectric materials longitudinal strain constant, the applied AC voltage should be significantly less than the polarization strength. Otherwise, a reversal of the domain in the material might be caused, which as a result produces a non-linear behaviour in the charge-electric strength; with this in mind, it was maintained in the range 0.05 kV/mm to 0.1 kV/mm at 1 kHz during the test. The method of how to secure and fix the samples under test is equally very important,
as additional stress can be introduced in an unsuitable fixed mode. As shown in Fig. 3, the test sample was set on the platform by gas absorption.

4. Comparison result and discussion

4.1 Comparison and discussion

The measurement results are shown in Table 1. For each sample and method, a total of 3 repeat measurements were performed. It may be seen that even for samples from the same manufacturer, the results show a small deviation in the \( d_{33} \) value even with the same measurement method. It should be noted that the measurement frequency of quasi-static method was 110 Hz while the resonant frequency was approximately 90 kHz. The laser interferometer measurements were performed at 1 kHz.

From the values presented in Table 1, it may be seen that the measurement of the \( d_{33} \) constant from dynamic resonant method has the lowest value. According to the curve of the piezoelectric constant for the PZT5 material as a function of frequency (supplied by Bourlincurt), the difference of \( d_{33} \) between 90 kHz and 110 Hz is approximately 7% while the low frequency part it appears to be higher. For example, the test result of sample 1 by the quasi-static method was 414 pC/N, and 7% less of it was 385 pC/N. The value is very close to that measured by the dynamic resonant method. With regards to the laser interferometry method, the test results were slightly higher in value compared to the quasi-static method for cylindrical samples.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Quasi-static method</th>
<th>Dynamic resonant method</th>
<th>Laser interferometry method</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>414±16</td>
<td>380±8</td>
<td>420±9</td>
</tr>
<tr>
<td>2</td>
<td>410±15</td>
<td>367±9</td>
<td>422±8</td>
</tr>
<tr>
<td>3</td>
<td>396±15</td>
<td>364±7</td>
<td>415±9</td>
</tr>
<tr>
<td>4</td>
<td>424±17</td>
<td>376±8</td>
<td>435±8</td>
</tr>
<tr>
<td>5</td>
<td>410±16</td>
<td>374±8</td>
<td>421±9</td>
</tr>
</tbody>
</table>

It should be noted that for the majority of the samples, the thickness of the sample was always less than the diameter. In order to compare the test results for such samples, the piezoelectric ceramic materials with a diameter of 10 mm and thickness of 2, 5 and 8 mm were selected respectively. The results measured by the laser interferometry and the quasi-static method were shown in Fig. 4. The measured longitudinal strain value of \( d_{33} \) by the laser interferometry method was 12.7% larger than the quasi-static method. When the ratio of thickness to diameter of piezoelectric ceramics is 0.5, the measurement results of the two methods were very similar indeed, and actually very close to the true \( d_{33} \) value of the piezoelectric ceramics PZT5 [6].

Both the quasi-static and laser interferometry methods are not ideal measurement procedures for longitudinal piezoelectric constants. The differences are mainly caused by the different boundary conditions in different methods. When the laser interferometer was used to measure the displacement of the sample with AC electric field applied, the material itself is stretched and compressed alternately in the direction of the thickness. In the quasi-static method, a preload is required, such as 10 N, in order to hold the sample between the measurement heads and therefore the sample is always in compression. The stress caused by the preload could potentially reduce the output of the charge induced by the AC force. In fact, the ideal measurement needs to be in a zero-field measurement, both for the laser interferometry and quasi-static method. For example, the test results from the quasi-static method need to be extrapolated to zero preload conditions.
The piezoelectric film is one of the extreme status of piezoceramic chips. Lefki and Dormans [7] studied the longitudinal piezoelectric constant measurement of piezoelectric film by direct and reverse piezoelectric effect. When the displacement is measured, the piezoelectric constant is determined by:

\[
d_{33}^{(1)} = \frac{S_3}{E_3} = d_{33} - 2d_{31} \frac{s_{13}^F}{(s_{11}^E + s_{12}^E)}
\]  

while when the quasi-static method is used, the constant measured by direct piezoelectric effect is given by:

\[
d_{33}^{(2)} = d_{33} - 2d_{31} \frac{(s_{13}^E + \sigma/Y)}{(s_{11}^E + s_{12}^E)}
\]

where \(d_{31}\) is the transverse piezoelectric constant, \(\sigma\) is Poisson's ratio, \(Y\) is Young's modulus and \(s_{\alpha\beta}^E\) is the elastic compliance constant \((\alpha, \beta=1,2,3)\).

From Eq. (7) and (8), it may be seen that the transverse piezoelectric constant \(d_{31}\) reduces the measurement result of the real \(d_{33}\) in both direct and reverse piezoelectric effect methods. Without any corrections, the measured result is less than the real value, while for the quasi-static method, the value is even smaller and the difference depends on the ratio of \(\sigma\) to \(Y\). The difference for piezoelectric films should be more significant than for bulks due to the influence of bending, but the bulks should, in principle, show similar differences.

4.2 Traceability of quasi-static \(d_{33}\) meters

Quasi-static \(d_{33}\) meters are widely used in the piezoelectric industry; inside such meters, a comparison PZT bulk is used to carry out the measurement. The question arises on how to calibrate it and it is therefore natural to consider that another reference PZT cylinder can be used to calibrate the apparatus. If the dynamic resonant method is used to calibrate the reference bulk, the corresponding result is the \(d_{33}\) value at its resonant frequency. The correction from the resonant frequency to the frequency at which the \(d_{33}\) meter works, could also potentially lead to further errors.

Within a certain deviation range, the laser interferometry method could be used as a primary calibration technique to obtain the longitudinal strain constants of piezoelectric bulks and to calibrate quasi-static instruments. The difference and possible reasons were discussed in section 4.1.

The dynamic force sensor can also be used as a calibration means to obtain the \(d_{33}\) value of reference bulks, as discussed in section 2.1. For precise calibration of dynamic force sensors, the \(d'_{33}\) of reference bulks could be measured by the charge and force at a specific frequency of interest.
5. CONCLUSION

For the measurement of the longitudinal piezoelectric constant, typical approaches include the quasi-static method, the dynamic resonant method and the laser interferometry method. Comparison results for the P5 material showed that the value of the $d_{33}$ constant obtained by the dynamic resonant method is approximately 7% lower than the quasi-static method, while the value based on the laser interferometry method is systematically higher than the quasi-static method. When the thickness-to-diameter ratio of the piezoelectric ceramic is set to 0.5, the values of $d_{33}$ obtained by the quasi-static and laser interferometry methods are very close indeed. Both of the quasi-static method and the laser interferometry method are not ideal measurement approaches for longitudinal piezoelectric constants. The differences were mainly caused by the different boundary conditions between the investigated methods.

Reference bulks can also be used to calibrate $d_{33}$ meters in industry. When the laser interferometry method is used to calibrate such reference bulks, the difference between the methods should be considered. If the dynamic resonant method is selected, a suitable correction of $d_{33}$ value at different frequencies should be applied. The dynamic force sensor with precise calibration could also be used to calibrate the reference bulk at the same frequency as the $d_{33}$ meter.

ACKNOWLEDGEMENTS

The work was supported by the Natural Science Foundation of China (No.51205378 and 51575502) and Special Fund for Scientific Research in the Public Interest (201310010).

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