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Calibration of space charge measuring equipment based on the pulsed electro-acoustic (PEA) measurement principle

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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CALIBRATION OF SPACE CHARGE MEASURING EQUIPMENT BASED ON THE PULSED ELECTRO-ACOUSTIC (PEA) MEASUREMENT PRINCIPLE

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IEC 62758, which is a technical specification, has been prepared by technical committee 112: Evaluation and qualification of electrical insulating materials and systems.

The text of this technical specification is based on the following documents:

Enquiry draft	Report on voting
112/206/DTS	112/219/RVC

Full information on the voting for the approval of this technical specification can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC web site under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

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INTRODUCTION

The pulsed electro-acoustic (PEA) method has been used to measure space charge distribution in dielectric materials by many researchers, and it has been accepted, in general, as a useful method to understand the electrical properties of dielectric materials. However, since PEA measurement equipments have been developed/used independently by different researchers over the world, there has not yet been any standard way to evaluate whether a system works properly. The IEC has therefore established a project team to create a standard procedure to evaluate PEA measurement equipment. This technical specification is the result.

CALIBRATION OF SPACE CHARGE MEASURING EQUIPMENT BASED ON THE PULSED ELECTRO-ACOUSTIC (PEA) MEASUREMENT PRINCIPLE

1 Scope

IEC 62758, which is a technical specification, presents a standard method to estimate the performance of a pulsed electro-acoustic (PEA) measurement system. For this purpose, a systematic procedure is recommended for the calibration of the measurement system. Using the procedure, users can estimate whether the system works properly or not.

2 Normative references

None.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
space charge
accumulated charge in materials

Note 1 to entry: This technical specification deals with the space charge in bulk and on surfaces of dielectric materials.

3.2
pulsed electro-acoustic method
PEA
technique for measuring space charge density distribution in solid dielectric materials

Note 1 to entry: In this technique, the pressure wave that is generated from the charge layer in a material specimen by applied pulse voltage to the specimen is observed using piezo-electric transducer attached behind an electrode contacted to the specimen. Details of measurement theory are described in Clause A.1.

3.3
piezo-electric transducer
sensor to detect the intensity of the pressure wave

Note 1 to entry: By applying the pressure wave, the charge is proportionally induced on the surface of the transducer. By connecting an adequate external circuit, the induced charge is converted to voltage signal. In the PEA measurement, the film or plate shaped piezo-electric transducer is usually used. The pressure wave intensity is measured as a voltage signal across the transducer when the wave propagates through the transducer. Details of the measurement procedure are described in A.1.3.

3.4
calibration
set of operations that establish, under specified conditions, the relationship between values of quantities indicated by measuring instrument or measuring system, or values represented by a material measure of a reference material, and the corresponding values obtained by a theoretical model

[SOURCE: IEC 60050-394:2007, definition 394-40-43, modified – the words "obtained by a theoretical model" replace "realized by standards".]

Note 1 to entry: This is the standard way to estimate the performance of a PEA measurement system. In the PEA measurement, the pressure wave generated from the charge layer in the material is measured as a voltage signal. To obtain the charge density distribution, it is necessary to calibrate the measured voltage signal to the charge density distribution. Therefore, in this technical specification, the calibration means the procedure to calculate the charge density distribution from the measured voltage signal.

3.5
deconvolution
procedure to recover the voltage signal from the distorted one

Note 1 to entry: The measured voltage signal is usually distorted by the reflection of the pressure wave at the interfaces between materials constituting the measurement system, the characteristic of the voltage signal detecting circuit and the induced noise with applied pulse voltage. To recover the voltage measured signal, a so-called de-convolution technique is usually used. The details of the deconvolution procedure are described in Clause A.2.

4 Basic theory for measurement

4.1 Permittivity and induced charge density

When a d.c. voltage V_{dc} (V) is applied to a film or sheet shaped dielectric material with thickness of d [m] through the attached electrodes, positive and negative charges with densities of σ_0 and $-\sigma_0$ (C/m^2) are induced at the interfaces between the material and the electrodes. The constant average electric field E_{dc} (V/m) and the charge density are ideally described by the following equations:

$$E_{dc} = \frac{V_{dc}}{d} \tag{1}$$

$$\sigma_0 = \varepsilon E_{dc} \tag{2}$$

Where ε is the permittivity of the dielectric material described with the unit of (F/m). It is also described using the permittivity in vacuum ε_0 = 8,854 x 10^{12} (F/m) as follows:

$$\varepsilon = \varepsilon_0 \varepsilon_r \tag{3}$$

where the non-dimensional coefficient ε_r is called the relative permittivity.

4.2 Charge in dielectrics and Poisson’s law

Here, the axis z is defined in the direction of thickness of a film or a sheet shaped dielectric material. When the charge is accumulated in the material with a volume density of $\rho(z)$ (C/m^3), electric field distribution $E(z)$, under static conditions, is described using the following Poisson’s equation:

$$E(z) = \frac{1}{\varepsilon_0 \varepsilon_r} \int \rho(z) dz \tag{4}$$

The electric potential distribution in the material V(z) is described as

$$V(z) = -\int E(z) dz \tag{5}$$

4.3 Coulombic force of charge in electric field

When charge q (C) is put in the electric field E (V/m), the following Coulombic force F (N) acts on the charge:

$$F = qE \tag{6}$$

When the charge q is homogeneously distributed as a perpendicular layer to z axis, the charge density of the layer σ (C/m^2]) is calculated by using the area of the material S (m^2) as $\sigma = q/S$. Therefore, the pressure wave p (Pa = N/m^2]) generated from the charge layer when the electric field E is applied to the material is

$$p = \sigma E \tag{7}$$

When the above electric field is generated by the pulse voltage with very short duration, the pulse pressure wave generates from each charge layer and it propagates in the material.

4.4 Reflection and transmission of pressure wave

When a pressure wave propagates through the interfaces between different materials, it is divided into transmitted and reflected waves. The ratio of this division is determined by so called acoustic impedance Z (Pa s/m = N s/m^3). The acoustic impedance Z is obtained by the following equation:

$$Z = mu \tag{8}$$

where m (kg/m^3) and u (m/s) are density and acoustic velocity in the material.

When the pressure wave propagates from material 1 to material 2, the transmission and reflection ratios K_t and K_r are described using the acoustic impedances of the materials Z_1 and Z_2 as

$$K_t = \frac{2Z_2}{Z_1 + Z_2} \tag{9}$$

$$K_r = \frac{Z_2 - Z_1}{Z_1 + Z_2} \tag{10}$$

When the pressure wave is generated at the interface between material 1 and 2, the ratio of propagation towards material 2, say K_{g2} is described as

$$K_{g2} = \frac{Z_2}{Z_1 + Z_2} \tag{11}$$

4.5 Maxwell stress

When a voltage V is applied across electrodes attached to a sheet or a film dielectric material with thickness of d and permittivity of ε, the following Maxwell stress F_0 (N) is generated at the interfaces between the material and electrodes:

$$F_0 = \frac{1}{2}\varepsilon\left(\frac{V_{\mathrm{dc}}}{d}\right)^2 = \frac{1}{2}E \times \sigma \tag{12}$$

4.6 Response of linear system

When a delta function $\delta(t)$ (impulse) as a function of time t (s) is input into a linear system, the output of it $h(t)$ is called "transfer function". The relationship between $h(t)$ and $\delta(t)$ is described using the following convolution equation:

$$h(t) = \int_{-\infty}^{+\infty} \delta(\tau) h(t-\tau) d\tau \tag{13}$$

When a certain function voltage $v_{\mathrm{in}}(t)$ inputs the linear system, the output voltage $v_{\mathrm{out}}(t)$ is obtained using $h(t)$ as

$$v_{\mathrm{out}}(t) = \int_{-\infty}^{+\infty} v_{\mathrm{in}}(\tau) h(t-\tau) d\tau \tag{14}$$

In the frequency domain, the above relationship is converted into the following equation:

$$V_{\mathrm{out}}(f) = H(f)\ V_{\mathrm{in}}(f) \tag{15}$$

where $V_{\mathrm{out}}(f)$, $H(f)$ and $V_{\mathrm{in}}(f)$ are functions of frequency f (Hz) converted from $v_{\mathrm{out}}(t)$, $h(t)$ and $v_{\mathrm{in}}(t)$, respectively.

5 Procedure to calibrate the space charge measurement

5.1 Principle of calibration

5.1.1 General

A basic principle of calibration for obtaining charge density distribution from the PEA signal is described below. Generally in calibration for measurement, we need a signal from a measuring object which value is known absolutely. In the case of the PEA measurement for a flat sheet sample, the induced surface charges by applied d.c. voltage at the interfaces between the sample and electrodes are theoretically obtained when the permittivity of the sample is known. Therefore, the following calibration process is based on the ideal measurement of the surface charges under d.c. voltage application.

Consider a virgin (not having space charges in its bulk) dielectric (flat) sheet sample, placed between a set of electrodes. The sample thickness and relative permittivity are d and ε_{r}, respectively. When a small d.c. voltage V_{dc} is applied to the sample, positive and negative surface charges $+\sigma_0$ and $-\sigma_0$ are induced at interfaces between the sample and electrodes, anode and cathode, respectively. Here, the voltage V_{dc} is assumed to be relatively low so that it is not enough to generate any space charge in the bulk of sample. Since these surface charges are located at quite thin layers, they can be treated as impulse (delta) functions on a positional axis z along the thickness of the sample as shown in Figure 1(a). The value of surface charge density σ_0 can be calculated by the following equation:

$$\sigma_0 = \varepsilon_0 \varepsilon_{\mathrm{r}} E_{\mathrm{dc}} = \varepsilon_0 \varepsilon_{\mathrm{r}} V_{\mathrm{dc}}/d \tag{16}$$

where E_{dc} and ε_0 are applied average electric field and the permittivity in vacuum, respectively. Under the electric field E_{dc}, when a pulsive voltage $V_p(t)$ is superimposed on V_{dc}, pulsive pressure waves $p_0(t)$ and $p_d(t)$ are generated from the surface charges (see Annex A). In the PEA method, the pressure wave $p(t)$ generated from the charge distribution $\rho(z)$ is observed using a piezo-electric sensor which transforms the pressure to voltage signal $V_s(t)$ (see A.1.3). Therefore, the calibration procedure enables to transform the obtained $V_s(t)$ to the charge density distribution $\rho(z)$. Since the surface charge density σ_0 can be theoretically calculated using Equation (1), the signal voltage of $V_s(t)$ can be easily calibrated by observing σ_0. On the other hand, the position z can be calculated by the following relationship:

$$z = u_{sa}t \tag{17}$$

where u_{sa} is acoustic velocity in the sample.

However, in general, it is hard to obtain an accurate value of relative permittivity of a sample. Therefore, the actual calibration should be carried out using some parameters that are easily measured. As shown in Figure 1(b), the electric field distribution $E(z)$ in the sample can be obtained by integral calculation of charge density distribution $\rho(z)$. It can be seen that the electric field distribution $E(z)$ in the sample for the calibration measurement shown in Figure 1(b) has a simple rectangular shape with the value of flat portion, $E_{dc} = V_{dc}/d$. The thickness of the sample d and the applied d.c. voltage V_{dc} are easy to measure. Therefore, calibration using the electric field distribution $E(z)$ is proposed in this specification.

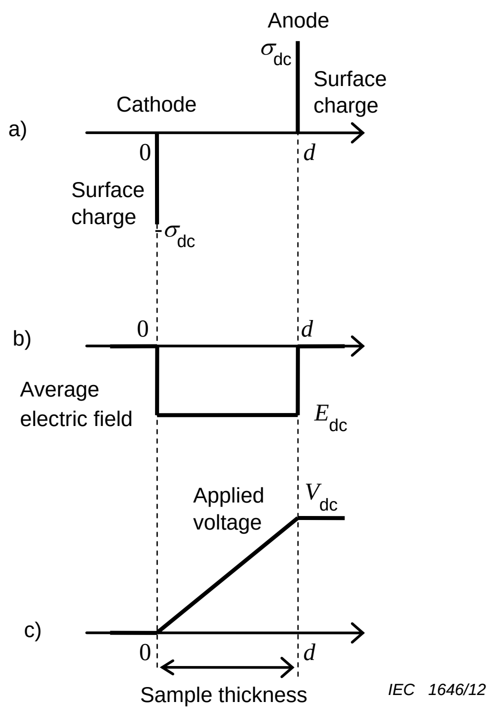


Figure 1(a) – Charge density distribution **Figure 1(b) – Electric field distribution** **Figure 1(c) – Electric potential distribution**

Figure 1 – Theoretical distributions for calibration measurement

5.1.2 Typical result of calibration measurement

Figure 2 shows a typical result of calibration measurement. In this measurement, a PMMA (poly (methyl-methacrylate)) sheet specimen with a thickness of d = 500 μm is used. Figure 2(a) shows charge density distribution obtained by applying a d.c. voltage of V_{dc} = 2 kV to the sample. If the measurement is ideally carried out for the sample without any space charge in its bulk, the charge density distribution should be a pair of delta functions as shown in Figure 1(a). However, they are observed as a pair of peaks with a certain width that is determined by both of the pulse widths t_{vp} of the applied pulse voltage and acoustic wave traveling time t_p passing through the piezo-sensor (see A.1.4). The half-value width, d_r of the first peak in this measurement result is defined as a positional resolution of this measurement. An integral calculation of this peak must be equal to the surface charge density σ_0 shown in Figure 1(a).

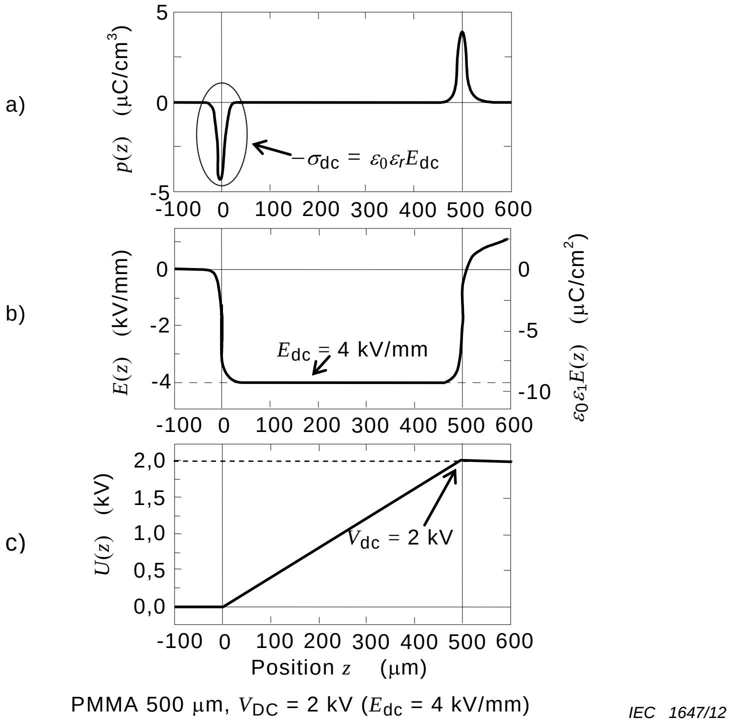


Figure 2(a) – Charge density distribution **Figure 2(b) – Electric field distribution** **Figure 2(c) – Electric potential distribution**

Figure 2 – Typical result of calibration measurement

5.2 Sample preparation

5.2.1 Sample for calibration measurement

A commercially available PMMA sheet with a thickness range of 0,5 mm to 1 mm may be used for the calibration measurement. The calibration measurement shall be carried out under the applied electric field which gives a linear relationship between calculated average electric field and applied d.c. voltage as shown in Figure 10. It is recommended that the electric field strength is within a range of 5 kV/mm to 30 kV/mm, providing the application time is short enough (typically 5 min) not to accumulate space charge. Before the calibration measurement, the thickness of the sample shall be accurately measured using a micrometer. If there are foreign objects or dust on the surface of the sample, the interface adherence between sample and electrodes may be lost. Since the interface adherence is important to make the signal pressure wave smoothly propagate, the sample surfaces shall be cleaned up with soft cloth to remove foreign objects and dusts. A sample with evaporated electrodes may also be used for the calibration measurements.

It should be mentioned here that the PMMA sample could acquire space charges even below 30 kV/mm, above about 303 K and maintain the space charges for a long time. Therefore, samples used for calibration shall either be not subjected to high temperatures in their history or it must be ensured that the voltage levels are not so high as to cause accumulation of space charges when subjected to high temperatures.

5.2.2 Sample placement

Prior to placing the sample between electrodes, in order to help propagation of pressure waves signal at interfaces between electrodes and the sample, both surfaces of the sample should be wetted with commercially available silicone oil. A semi-conductive layer should be placed between the sample and the metal upper electrode to improve the acoustic impedance matching (see A.1.2). Commercially available semi-conductive sheets can be used for this purpose. Adequate force shall be applied to the sample to keep tight contact with the electrodes using a jig mounted to the measurement system.

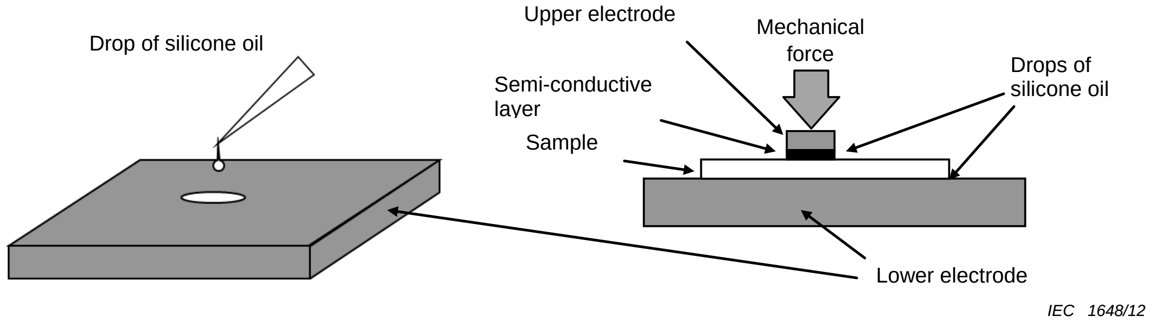


Figure 3 – Drop of silicone oil and sample placement

5.3 Data acquisition

5.3.1 Pulse voltage test

In the PEA method, the signal is obtained by applying a pulse voltage to the sample repeatedly. However, when only the pulse voltage is applied to the sample without any d.c. voltage stress, a very small signal is observed (see A.1.1). Such a signal should be small enough to be neglected in the calibration measurement. Therefore, before the calibration measurement, the signal obtained by only the pulse voltage application shall be observed. The signal shall be obtained with an adequate number of averaging (see 5.3.2). Figure 4 shows a typical measurement result obtained by applying the pulse voltage of 500 V with duration of 14 ns to the PMMA sample with a thickness of 0,5 mm. It may be seen that there is no remarkable signal in Figure 4, and such a kind of result is advisable.

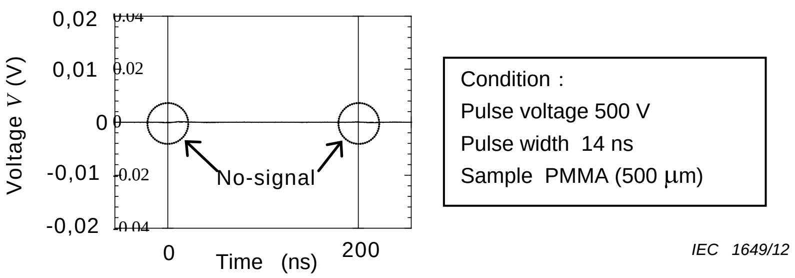


Figure 4 – Pulse voltage application test

5.3.2 Averaging

Generally, in the PEA method, the signal is obtained using the averaging technique that is carried out on the data (obtained by repeatedly applying the pulse voltage to the sample). The suitable minimum number of times for averaging depends on measurement conditions and characteristics of equipment. A signal obtained with a deficient number of times of averaging would be inaccurate for calibration. Figure 5 shows typical waveforms with various averaging number. The waveforms are obtained by applying d.c. voltage of 4 kV and pulse voltage of

500 V with duration of 14 ns to the PMMA sample with a thickness of 1 mm. As shown in Figure 5(a), the waveform without averaging seems to be hidden in white noise. With the number of times of averaging increasing, the noise level decreases as may be seen in Figures 5(b) and 5(c), and consequently the S/N (signal to noise) ratio of the waveform becomes higher. In this example, the wave form shown in Figure 5(c) is preferable for calibration.

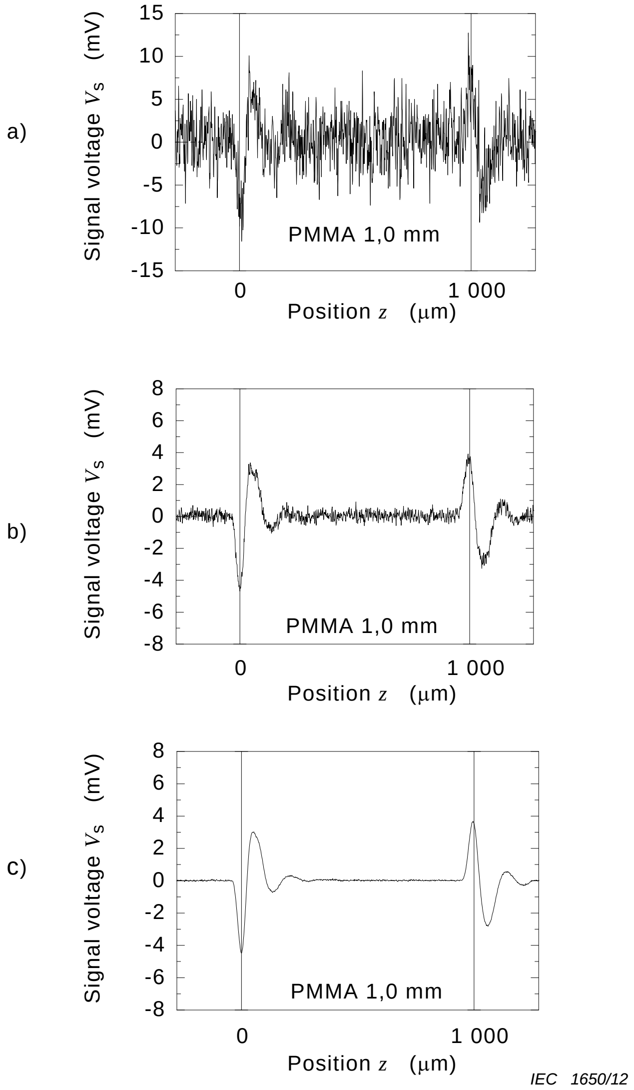
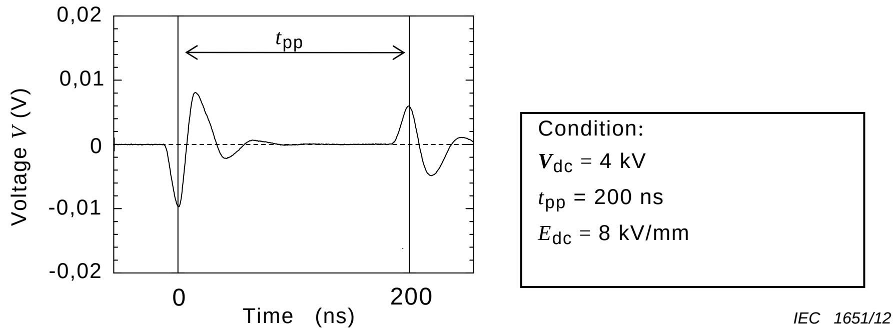


Figure 5(a) – Signal without averaging **Figure 5(b) – Averaged signal (100 times)** **Figure 5(c) – Averaged signal (10 000 times)**

Figure 5 – Dependence of averaging number

5.3.3 Data acquisition for calibration

Immediately after d.c. voltage application to the PMMA sample, the PEA signal wave form is measured by repeatedly applying the pulse voltage with an adequate averaging number. To confirm linearity of the results, measurements should be carried out under at least 3 levels of d.c. electric field E_{dc} below 30 kV/mm. In the obtained waveforms, time duration between peaks t_{pp} must be measured and the acoustic velocity $u_{sa} = d/t_{pp}$, shall be calculated.



Key

t_{pp}	time between first and second peaks
$d/t_{pp} = u_{sa}$	acoustic velocity in sample
$V_{dc}/d = E_{dc}$	d.c. electric field

Figure 6 – Measurement of waveform for calibration

5.3.4 Signal obtained under short circuit condition

After obtaining the signal under d.c. stress, as described above, the d.c. stress is removed, and data is obtained under a short-circuit condition. If the applied d.c. voltage is so excessive that it might cause space charge accumulation, the accumulated space charge should be observed under a short-circuit condition immediately after removal of the d.c. voltage. Therefore, it is necessary to observe the signal waveform under a short-circuit condition immediately after obtaining the data for calibration. Figure 7 shows a procedure and typical results concerning this confirmation. Figure 7(a) shows a waveform obtained under a short-circuit condition before applying d.c. voltage for calibration. This result is the same as the result for “pulse voltage test” mentioned in 4.3.1. Figure 7(b) is the data obtained under d.c. voltage for calibration. Then the data under short-circuit condition shall be taken as shown in Figure 7(c) to compare with the one under short circuit condition before the d.c. voltage application. When the waveform (a) obtained before the d.c. voltage application is the same as the waveform (c) obtained afterwards, it can be said that the d.c. voltage magnitude is proper for the calibration.

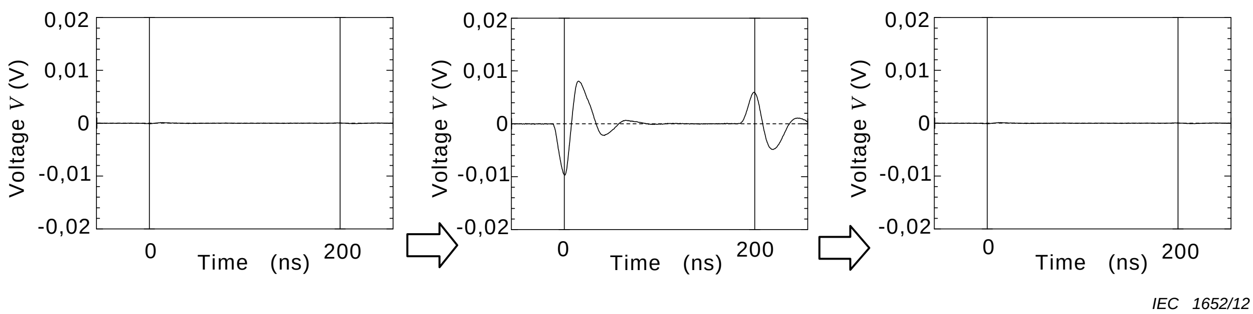


Figure 7(a) – Measurement only with pulse voltage application (under short circuit condition)

Figure 7(b) – Measurement for calibration (under d.c. voltage application)

Figure 7(c) – After measurement (under short-circuit condition)

Figure 7 – Confirmation of absence of space charge accumulation during d.c. voltage application for calibration

5.4 Data processing and calibration

5.4.1 Deconvolution

Obtained data for calibration ordinarily (or generally) includes some distortion because of the acoustic reflection and/or due to characteristic nature of the detection circuit. Therefore, a deconvolution technique is usually applied to the obtained data (see Clause A.2). When the deconvolution technique is carried out, the high frequency noise is increased. Therefore, a low pass filter is also applied in addition to the deconvolution technique. By applying adequate deconvolution and filtering procedure, the waveform with two peaks is obtained as shown in Figure 8(a).

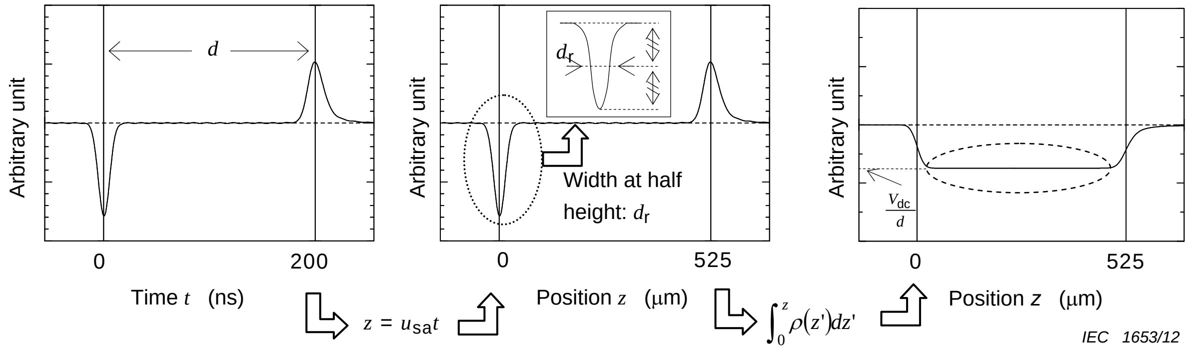


Figure 8(a) – Deconvoluted waveform

Figure 8(b) – Waveform for positional charge density distribution

Figure 8(c) – Waveform for electric field distribution

Figure 8 – Deconvolution and calibration

5.4.2 Calibration for horizontal axis and calculation of waveform for electric field distribution

Using Equation (17), the horizontal axis in "time" shown in Figure 8(a) is converted to "position" as shown in Figure 8(b). In this case, the width at half height of the 1st peak "d_r" shall be measured. The ratio k of d_r to the sample thickness d ($k = d_r/d$ x 100 /%)) is defined as spatial resolution of this measurement. The spatial resolution between 2 % to 10 % is preferable (see A.1.4). To calibrate the vertical axis in charge density distribution, a waveform for the electric field distribution is calculated. By integrating positional charge distribution shown in Figure 8(b), the waveform for electric field distribution is obtained as shown in Figure 8(c).

5.4.3 Calibration for electric field and charge density distributions

As shown in Figure 9(a), the waveform for electric field distribution shall have a flat shape. As shown in Figure 9(b), the vertical axis of the waveform for electric field is decided as the value of flat part is equal to the electric field E_{dc} (= V_{dc}/d). Then the charge density distribution is calculated by differentiation using a nominal value of relative permittivity ε_r, as shown in Figure 9(c). Since the value of the vertical axis in charge density distribution depends on ε_r, it is necessary to specify the numerical value used for the calibration.

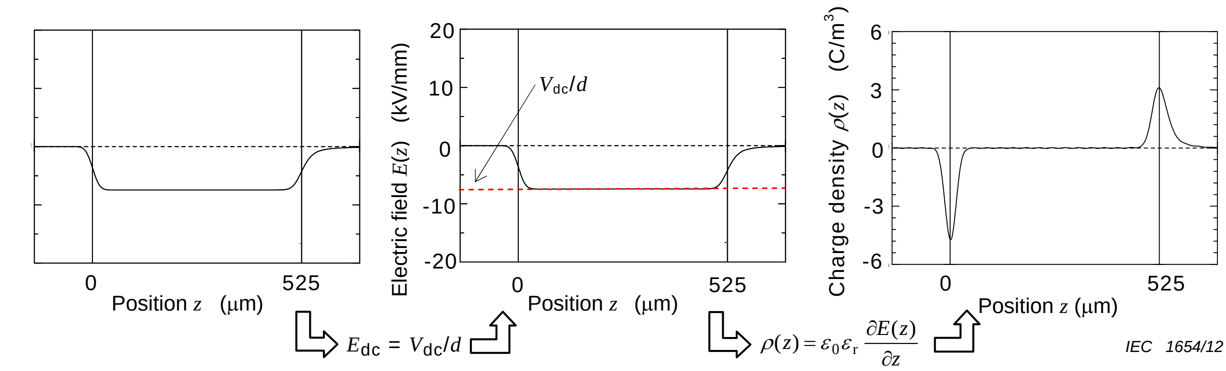


Figure 9(a) – Waveform for electric field distribution

Figure 9(b) – Calibration of electric field distribution using measured average electric field

Figure 9(c) – Calculated charge distribution from calibrated electric field distribution

Figure 9 – Calibration for electric field and charge density distributions

5.4.4 Confirmation of linearity of measurement

When a calibration, as described in 4.4.3, is successfully achieved, the other measurements should be carried out under different electric fields E_{dc} using the same procedure mentioned above in order to confirm linearity of such a calibration. Observations of signals under a short-circuit condition before and after the measurement with d.c. stress are also required to check whether the adequate d.c. stress is applied to the sample. When the data processing and electric field calibration are carried out, the same parameters obtained by the first calibration process have to be used. An additional two or three results should be obtained. When the values of electric fields being proportional to the applied voltages are obtained in all cases, the calibration procedure is considered valid.

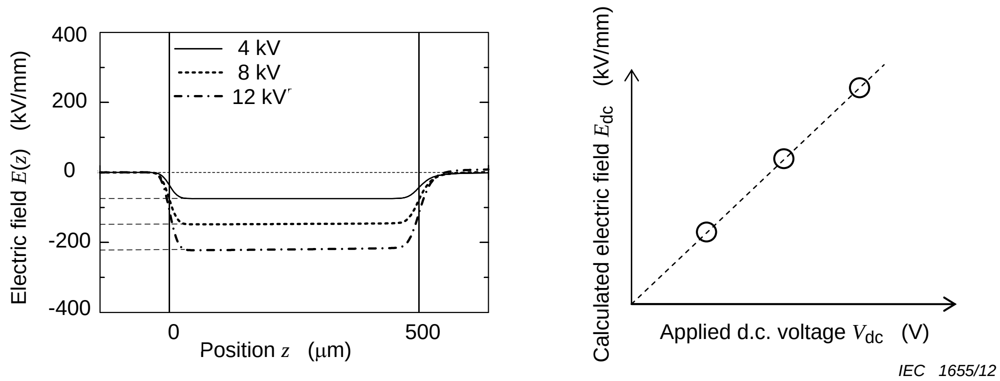


Figure 10 – Confirmation of linearity measurement

5.4.5 Typical test results by expert members of project team

Figures 11 to 16 show typical calibration test results obtained by various research groups of expert members in this project. Table 1 shows sample thickness, permittivity and resolution of measurements. Judging from the results, the calibrated space charge distributions are different because of the usage of different system with different resolutions or permittivities. For example, in the case of measurement results obtained using high resolution system, the first peaks are larger than those obtained using lower resolution system. However, the obtained electric field distributions are mostly the same. It means that the space charge distributions shall be shown with the description of the measurement resolution and the permittivity. Anyway, the electric fields in all results seem to be proportional to the applied average electric fields. Therefore, the calibrations must be fairly carried out in all results.

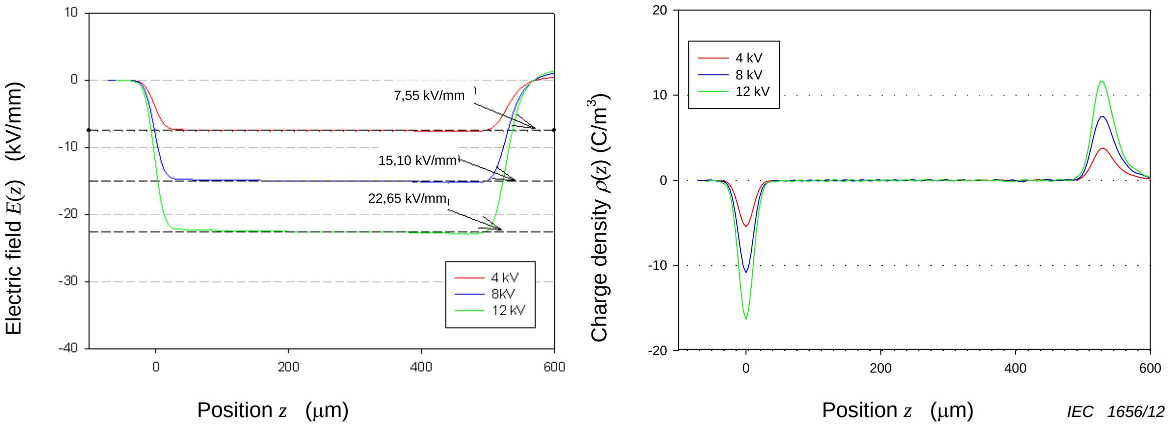


Figure 11(a) – Electric field distribution **Figure 11(b) – Charge density distribution**

Figure 11 – Results of calibration test by research Group A

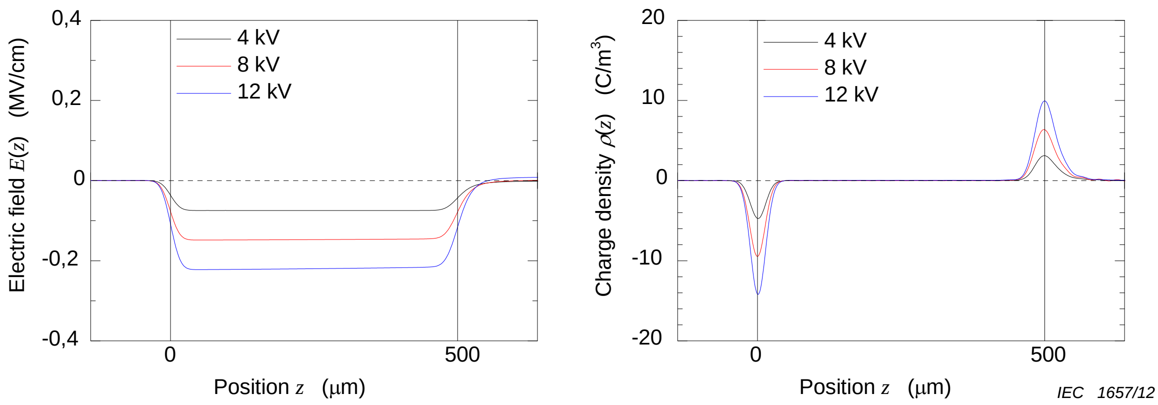


Figure 12(a) – Electric field distribution **Figure 12(b) – Charge density distribution**

Figure 12 – Results of calibration test by research Group B

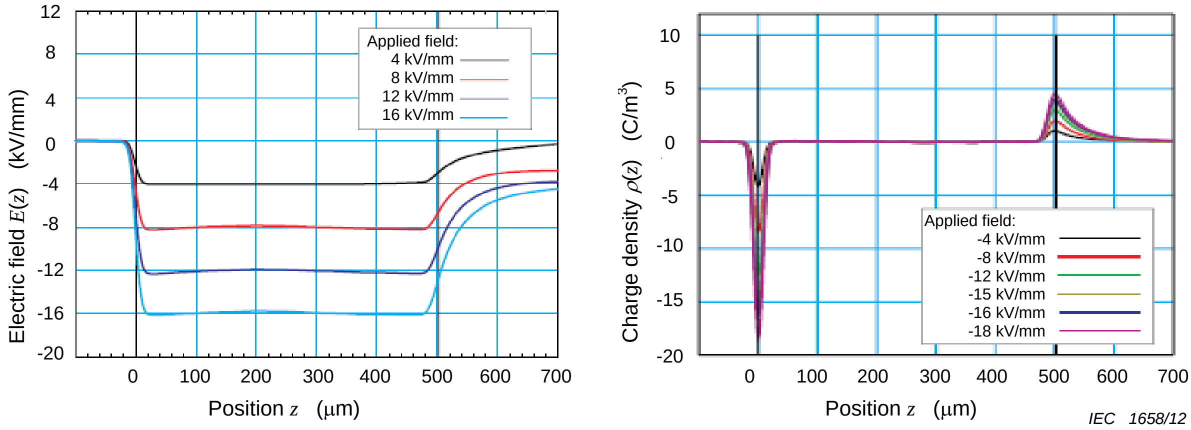


Figure 13(a) – Electric field distribution **Figure 13(b) – Charge density distribution**

Figure 13 – Results of calibration test by research Group C

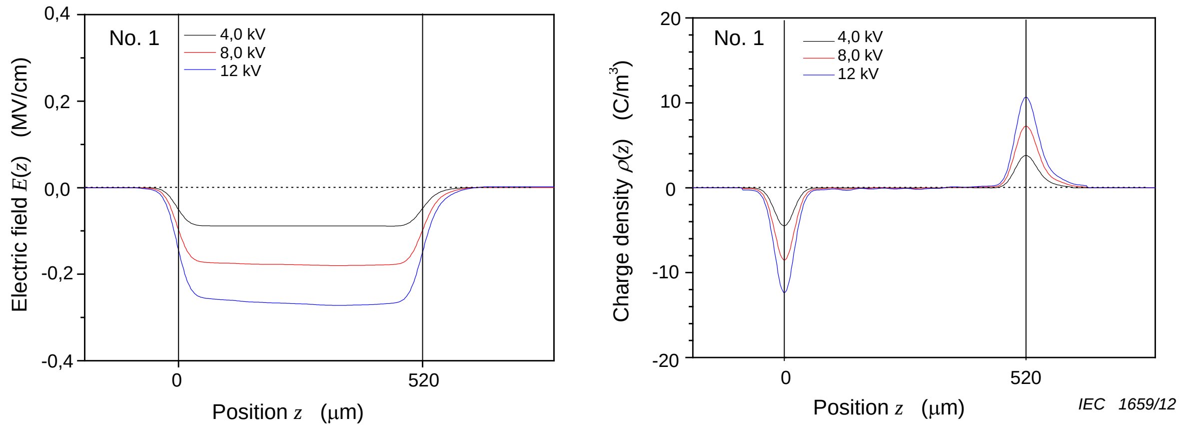


Figure 14(a) – Electric field distribution **Figure 14(b) – Charge density distribution**

Figure 14 – Results of calibration test by research Group D

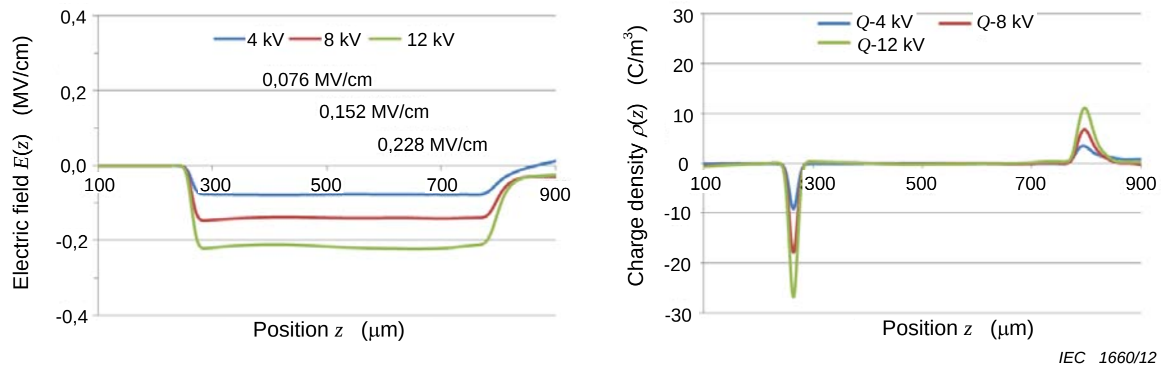


Figure 15(a) – Electric field distribution **Figure 15(b) – Charge density distribution**

Figure 15 – Results of calibration test by research Group E

Table 1 – Measurement resolution

Research group	Sample thickness µm	Relative permittivity	Resolution µm	Resolution %
A	530,	2,6	26	4.9
B	525	2,6	28	5,3
C	502	2,866	18,5	3,7
D	515	2,6	50	9,7
E	525	2,6	18,5	3,5

Annex A
(informative)

Theory of PEA method

A.1 Principle of PEA measurement method

When a pulse voltage is applied to a sample including space charge in its bulk, a pressure wave is generated from each charge. In the PEA method, the pressure wave is detected using the piezo-electric transducer, because the propagating pressure wave has information about the charge from which the pressure wave is generated.

A.1.1 Generation of pressure wave

A.1.1.1 Sample without space charge in its bulk

Schematic diagram of static force when a d.c. voltage is applied to a sample without space charge in the bulk is shown in Figure A.1(a). As shown in this figure, a d.c. voltage V_{dc} applied to a plate-shaped solid sample through metallic electrodes, induces positive and negative charges at interfaces between the sample and electrodes, namely, anode and cathode, respectively. In this condition, a static force F_0, called "Maxwell stress", acting on the induced charges, is described as

$$F_0 = \frac{1}{2}\varepsilon\left(\frac{V_{dc}}{d}\right)^2 \qquad (A.1)$$

where d and ε are the sample thickness and permittivity, respectively. This force always acts as a compression for the sample. The force doesn't generate any pressure wave under static conditions. By applying a pulse voltage $v_p(t)$ superimposing on V_{dc} to the sample in the above condition, the Maxwell stress F on the induced charges generates the pressure wave. The Maxwell stress F is described as

$$F = \frac{1}{2}\varepsilon\left(\frac{V_{dc}+v_p(t)}{d}\right)^2 = \frac{1}{2}\varepsilon\left(\frac{V_{dc}}{d}\right)^2 + \varepsilon\frac{V_{dc}}{d}\frac{v_p(t)}{d} + \frac{1}{2}\varepsilon\left(\frac{v_p(t)}{d}\right)^2 \qquad (A.2)$$

The force described in the first term of Equation (A.2) above, on the right hand side (RHS), does not generate any pressure wave as mentioned above. The forces described in the second and third terms (RHS) generate pulse pressure waves. The force described in the third term of the equation is negligible when the pulse voltage $v_p(t)$ is moderately small. Therefore, the pressure wave generated by the force described in the second term of the equation that is proportional to d.c. stress V_{dc} and pulse voltage $v_p(t)$ is the object to be measured. Since the expansion and compression pressure waves are generated at anode and cathode electrodes, respectively as shown in Figure A.2(b), the polarities of the charges induced on the electrodes are distinguished by measuring the polarity of pressure waves.

A.1.1.2 Sample with space charge in the bulk

A schematic diagram of force when a pulse voltage is applied to the sample including space charge $\rho(z)$ in the bulk at position z is shown in Figure A.1(c). When a pulse voltage is applied to this sample, a pressure wave is generated. Figure A.1(d) shows a schematic diagram of pressure wave generation in the sample including a space charge under d.c. stress V_{dc} superimposed by a pulse voltage $v_p(t)$. In this case, it is assumed that the charge layers with charge density of $\sigma(0)$ and $\sigma(d)$ are induced at anode (position z = 0) and cathode (positon z = d) interfaces, respectively. Furthermore, a space charge $\rho(z)$ within a small width of Δz is also existing in the bulk of the sample. When a pulse voltage superposing the d.c. voltage is applied to the sample, pressure waves are generated from the charges $\sigma(0)$, $\rho(z)$ and $\sigma(d)$ at the same time. Here the pressure waves generated from the charge layers $\sigma(0)$, $\rho(z)$ and $\sigma(d)$ are described as $p_0(t)$, $p_p(t)$ and $p_d(t)$, respectively. The pressure wave is totally described as

$$p(t) = p_0(t) + p_p(t) + p_d(t) = \sigma(0) \times e_p(t) + \rho(z) \times \Delta z \times e_p(t) + \sigma(d) \times e_p(t) \tag{A.3}$$

where $e_p(t)$ (=$v_p(t)/d$) is a pulse electric field that is generated in the sample by the application of pulse voltage $v_p(t)$.

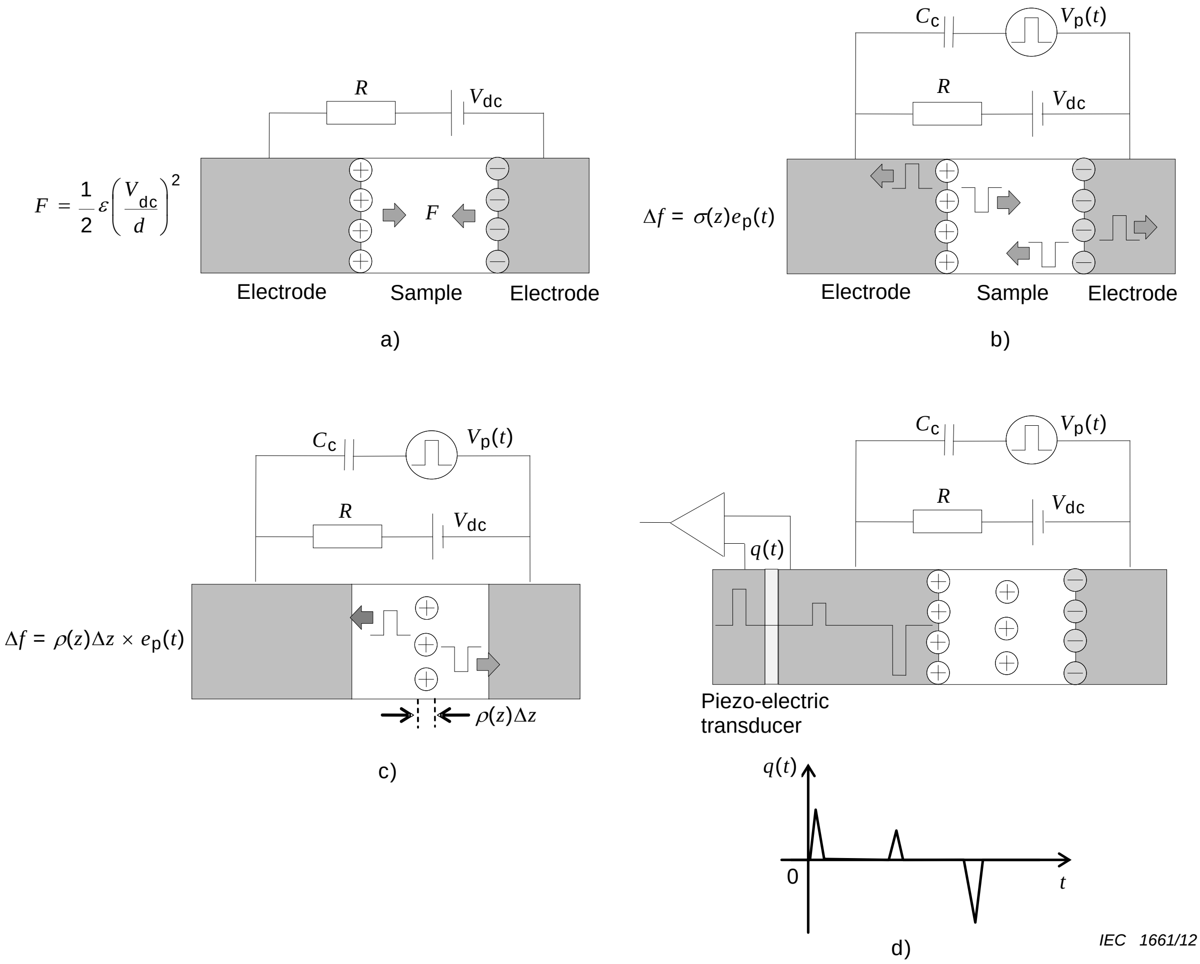


Figure A.1(a) – Static Maxwell stress

Figure A.1(b) – Generation and propagation of pressure wave from induced charges on electrode

Figure A.1(c) – Generation and propagation of pressure wave from space charge in bulk of sample

Figure A.1(d) – Measurement of pressure wave

Figure A.1 – Principle of acoustic wave generation in PEA method

A.1.1.3 Propagation of pressure wave

Figure A.2 shows a schematic diagram of the pressure wave propagation in the PEA measurement system. In the configuration of the PEA system shown in Figure A.2, pressure waves propagating towards a piezo-transducer, which is attached to the grounded Al electrode, are detectable. When a pressure wave is generated at an interface between different materials, the intensity of the pressure waves propagating through the materials depends on the characteristic acoustic impedances of the materials. For example, in the case of a pressure wave $p_o(t)$ generated at the interface between the sample and grounded Al electrode, the pressure wave $p_1(t)$ that is propagating towards the grounded Al electrode is described as

$$p_1(t) = \frac{Z_{\text{Al}}}{Z_{\text{Al}} + Z_{\text{sa}}} p_0(t) \tag{A.4}$$

where Z_{Al} and Z_{sa} are characteristic acoustic impedances of aluminum and sample material, respectively. Here, the characteristic acoustic impedance is defined as the ratio of pressure p to particle velocity in the material v ($Z = p/v$), and it is known that it is obtained by a product of the material density m and acoustic velocity in the material u ($Z = m \times u$). In the case of the pressure wave $p_{\text{p}}(t)$ generated from space charge in the bulk of sample, it is found that a half of it is a pressure wave $p_2(t)$ propagating towards the grounded electrode. When the propagating pressure wave passes through the interface between different materials, some part of it is reflected at the interface. Therefore, it is necessary to consider the transmittance of it. For example, when $p_2(t)$ passes through the interface between the sample and Al electrode, the transmitting pressure wave $p_2'(t)$ is described as

$$p_2'(t) = \frac{2Z_{\text{Al}}}{Z_{\text{Al}} + Z_{\text{sa}}} p_2(t) \tag{A.5}$$

The $p_2(t)$ arrives at the piezo-transducer with a delay of travelling time through the sample compared with arrival of $p_1(t)$. Considering the delay, transmittance and propagation ratios, the observed pressure wave $p(t)$ by piezo-transducer is described using Equation (A.5) as

$$p(t) = \frac{Z_{\text{Al}}}{Z_{\text{Al}} + Z_{\text{sa}}} \sigma(0) e_{\text{p}}(t) + \frac{1}{2} \frac{2Z_{\text{Al}}}{Z_{\text{Al}} + Z_{\text{sa}}} \rho(z) e_{\text{p}}\left(t - \frac{z}{u_{\text{sa}}}\right) \Delta z + \frac{Z_{\text{sa}}}{Z_{\text{ba}} + Z_{\text{sa}}} \frac{2Z_{\text{Al}}}{Z_{\text{Al}} + Z_{\text{sa}}} \sigma(d) e_{\text{p}}\left(t - \frac{d}{u_{\text{sa}}}\right) \tag{A.6}$$

where Z_{ba} is the characteristic acoustic impedance of backing material inserted between the sample and high voltage electrode as shown in Figure A.2. By choosing the backing material, which has the characteristic acoustic impedance close to that of sample material, the coefficients of above three terms come close to each other ($Z_{\text{sa}} = Z_{\text{Al}}$), therefore

$$p(t) = \frac{Z_{\text{Al}}}{Z_{\text{Al}} + Z_{\text{sa}}} \left[\sigma(0) e_{\text{p}}(t) + u_{\text{sa}} \int_{-\infty}^{+\infty} \rho(z) e_{\text{p}}(t - \tau) d\tau + \sigma(d) e_{\text{p}}\left(t - \frac{d}{u_{\text{sa}}}\right) \right] \tag{A.7}$$

where τ is a time variable and defined as $z = u_{\text{sa}} \tau$. In Equation (A.6), the second term of the pressure wave is generated from a single layer of the charge distribution. On the other hand, Equation (A.7) is expressed as the pressure wave generated from total charge distribution using integral form.

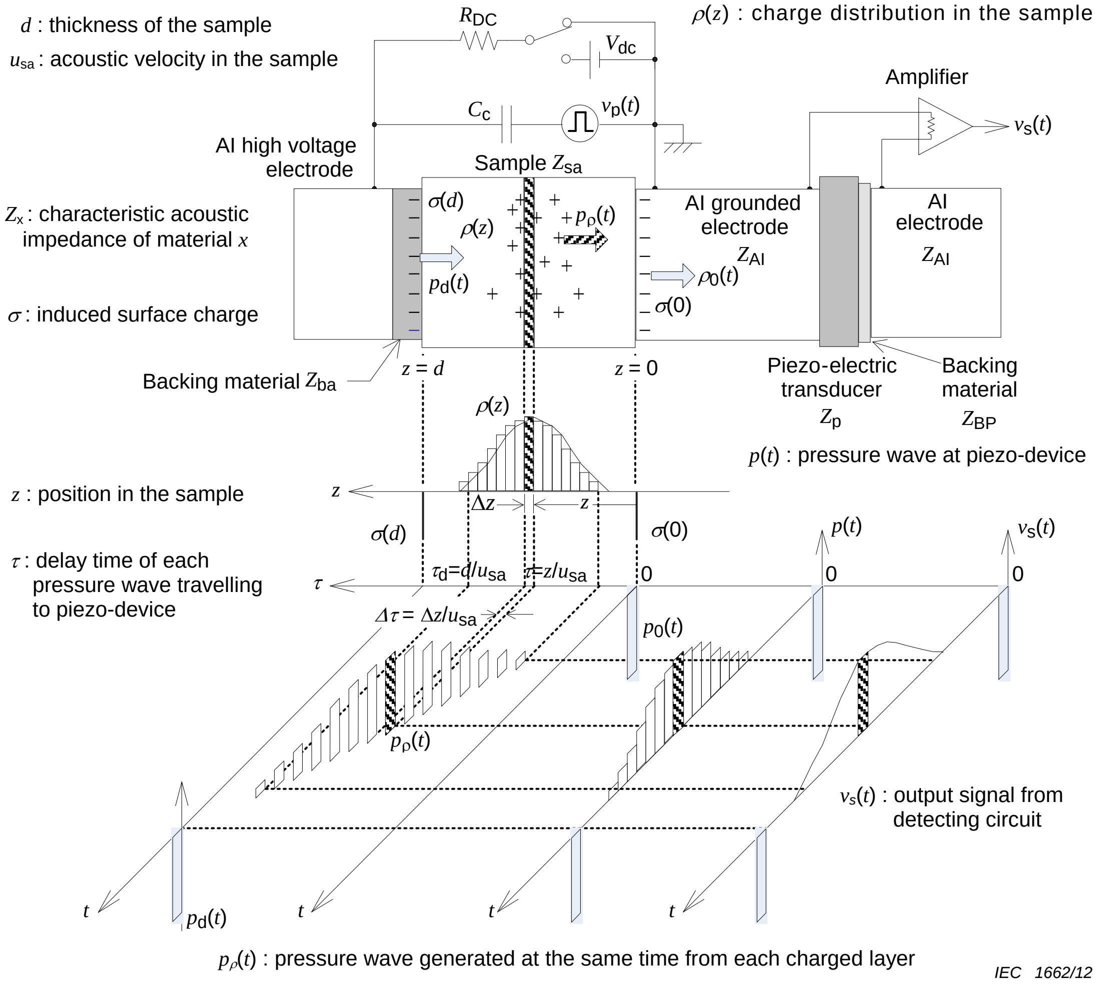


Figure A.2 – Pressure wave propagation in PEA measurement system

A.1.2 Transform from pressure wave to electric signal

Figure A.3 shows a response of piezo-transducer. When a steady pressure wave p is applied to a thick piezo-transducer, an amount of charge q is induced on the surfaces of the transducer. The charge q is described using a piezo-electric constant h_{p} as

$$q = p \times h_{\mathrm{p}} \tag{A.8}$$

When the time-variable pressure wave is travelling through the piezo-transducer with a velocity u_{p}, the piezo-constant h_{p} is described as $h_{\mathrm{p}}(t)$ using variable t. Since the h_{p} is a constant during the pressure wave propagation through the transducer, the function $h_{\mathrm{p}}(t)$ is a constant with duration of Δt_{p} (= b/u_{p}). When a pressure wave with very short duration $\Delta\tau$ is propagating through the transducer with thickness, b, the amount of charge induced is described as a function of time as shown in Equation (A.9):

$$\Delta q(t) = \frac{\Delta z}{b} h_{\mathrm{p}}(t) \times p\left(t - \frac{z}{u_{\mathrm{p}}}\right) \tag{A.9}$$

where t, z and Δz are time, position and width of the pressure wave, respectively. The Δz is expressed as $\Delta z = u_{\mathrm{p}} \times \Delta t$. Integrating for the total pressure wave, the above equation is rearranged while considering the transmittance at the interface between Al electrode and the piezo-transducer, as

$$q(t) = \frac{2Z_{\mathrm{p}}}{Z_{\mathrm{Al}} + Z_{\mathrm{p}}} \frac{u_{\mathrm{p}}}{b} \int_{-\infty}^{+\infty} h_{\mathrm{p}}(t) p(t-\tau) d\tau \tag{A.10}$$

where Z_{p} is a characteristic acoustic impedance of the piezo-transducer.

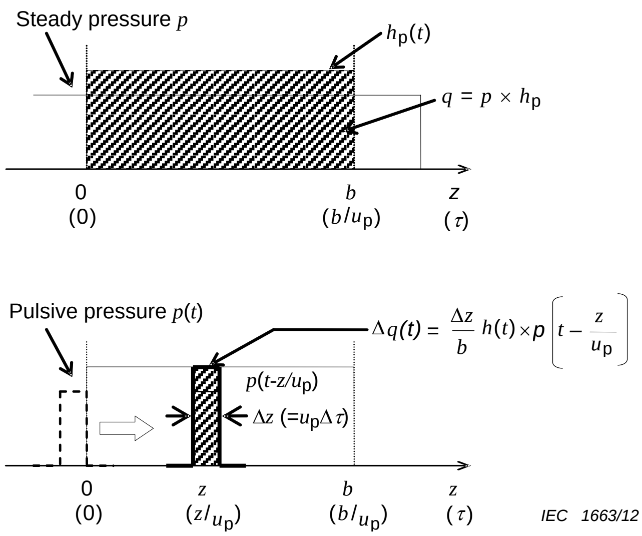


Figure A.3 – Response of piezo-transducer

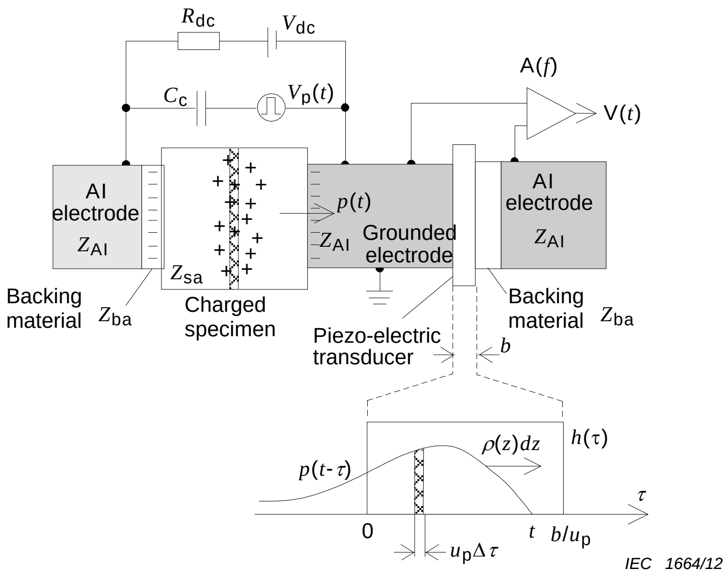


Figure A.4 – Transform from pressure to amount of charge induced on piezo-transducer

A.1.3 Spatial resolution of PEA measurement

In the PEA measurement system, the time-variable induced charge $q(t)$ is measured as voltage signal $v_{\mathrm{s}}(t)$ using external detecting circuit. The minimum width of $v_{\mathrm{s}}(t)$ is the width of signal derived from the induced charge $\sigma(0)$ on the grounded electrode because it is formed as a very thin layer on the electrode. In other words, the pressure wave from the induced charge $\sigma(0)$ can be treated as a delta function $\delta(t)$. When the pulse voltage with duration of

Δt_{vp} applied to the sample, the width of the pulse pressure wave is equal to Δt_{vp} if $\sigma(0)$ is treated as a delta function. When the pressure wave is travelling through the piezo-electric transducer with time-duration of Δt_p, the amount of charge $q(t)$ is described as a convolution of the $h_p(t)$ and the $p(t)$ as shown in Figure A.5. In the case of $\Delta t_{vp} < \Delta t_p$, the obtained $q(t)$ is a trapezium shape, as shown in Figure A.5, with a rising and falling time with Δt_{vp} and half-value width with Δt_p, respectively. In this case, the half-value width is defined as the "spatial resolution" in time domain. On the other hand, in the case of $\Delta t_{vp} > \Delta t_p$, the obtained $q(t)$ is of a trapezium shape, as shown in Figure A.5, with a rising and falling time with $\Delta t_p$and a half-value width with Δt_{vp}, respectively.

This means that the larger one of them is the "spatial resolution" in time domain. In the case of $\Delta t_{vp} = \Delta t_p$, since the obtained $q(t)$ has a shape of triangle with a high peak, a high sensitive measurement is expected. The "spatial resolution" in length is obtained by product of the time resolution and the acoustic velocity in sample. Relative resolution in percentage is calculated as the ratio of the resolution in length to sample thickness. Figure A.6 shows a relationship between the relative spatial resolution and observed wave form. The observed wave form includes a signal derived from induced surface charges with the width of the spatial resolution. It means that the higher peaks are observed in the higher resolution measurement. When the higher peak is included in observed distribution, the space charge distribution in the bulk becomes relatively small, as shown in Figure A.6. On the other hand, the wide peaks of the induced charges must overlap with space charge distribution in the bulk of the sample. Therefore, adequate resolution should be chosen.

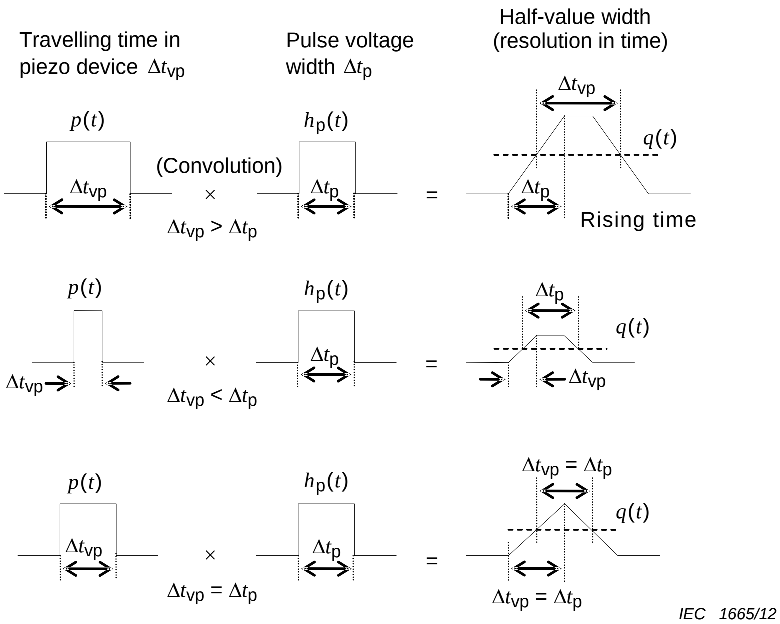


Figure A.5 – Relationship between the pulse width and thickness of piezo-transducer

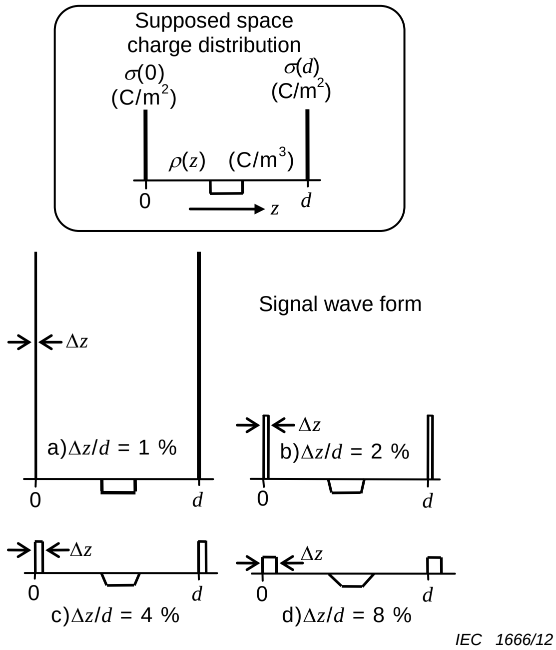


Figure A.6 – Adequate spatial resolution

A.2 Deconvolution

The actual observed voltage signal of the PEA measurement is affected by acoustic reflection at the interface between the piezo-transducer and the backing material and/or frequency response characteristics of the detecting circuit as shown in Figure A.7. Since the distortion of the signal by them is a kind of systematic error of the measurement system, the obtained voltage signal $v_S(t)$ is described as the following convolution of the impulse response of the system $h(t)$ and the charge distribution $\rho(t)$:

$$v_S(t) = h(t) \times \rho(t) = \int_{-\infty}^{+\infty} h(t-\tau)\rho(\tau)d\tau \tag{A.11}$$

Therefore, by obtaining the impulse response $h(t)$, the charge distribution is calculated using the so-called "deconvolution" technique. The convoluted functions are described as a simple product of them:

$$V_S(f) = R(f) \times H(f) \tag{A.12}$$

where $V_S(f)$, $R(f)$ and $H(f)$ are Fourier transforms of $v_S(t)$, $\rho(t)$ and $h(t)$ in frequency domain, respectively. In general, $H(f)$ is called a "transfer function" of the measurement system. To obtain the transfer function, an impulse response must be observed. Since, in the PEA measurement, the signal derived from the induced charge on the electrode is treated as an impulse (delta function), the signal itself is the impulse response of the system. However, if space charge is accumulated in the bulk of the sample, the induced charge on the electrode may be deformed. Therefore, it is necessary to measure the signal without space charge in the bulk to obtain the impulse response of the system. Under a relatively low d.c. stress, the induced charges are observed by the PEA measurement as shown in Figure A.7(a). Since such voltage application doesn't cause any damage to the sample, the signal is obtained before the actual measurement. Furthermore, since the induced charges are theoretically calculated as described in Equation (1), the previously obtained signal should be taken before the actual measurement. The data is called a "reference" signal for measurement. The reference signal should be composed of two same shape peaks. However, the pressure wave generated at the upper (far from the piezo-device) electrode is sometimes distorted during propagation through the sample by attenuation and/or dispersion, the signal derived from lower grounded electrode is adequate for calibration. The induced charge on grounded electrode $\sigma(0)$ is originally surface charge density in units of Coulomb per unit area. However, when the signal is observed using digital oscilloscope, the observed signal has a width of at least one digit τ_s (sampling time) of the time resolution (sampling time) of the oscilloscope. Therefore, the induced charge $\sigma(0)$ is described as the volume charge density $\rho(0)$ in units of Coulomb per unit volume:

$$\rho(0) = \sigma(0) \times \frac{1}{\tau_s \times u_{sa}} \tag{A.13}$$

The signal from the induced charge $V_r(f)$ in frequency domain is described as

$$V_r(f) = R_0(f) \times H(f) \tag{A.14}$$

where $R_0(f)$ is Fourier transformed induced charge $\rho(t)$ in frequency domain.

Since the $\sigma(0)$ is an impulse, the Fourier transformed $R_0(f)$ is a constant value described as

$$R_0(f) = \rho(0) = \sigma(0) \times \frac{1}{\tau_s \times u_{sa}} = \frac{\varepsilon_0 \varepsilon_r}{d} V_{dc} \times \frac{1}{\tau_s \times u_{sa}} \tag{A.15}$$

When the sample including the charge density distribution $\rho(t)$ is measured using the PEA system, the signal $v_s(t)$ shown in Figure A.7(b) is obtained. Using Equations (A.12) and (A.14), the charge density in frequency domain $R(f)$ is obtained as

$$R(f) = R_0(f) \times \frac{V_s(f)}{V_r(f)} \tag{A.16}$$

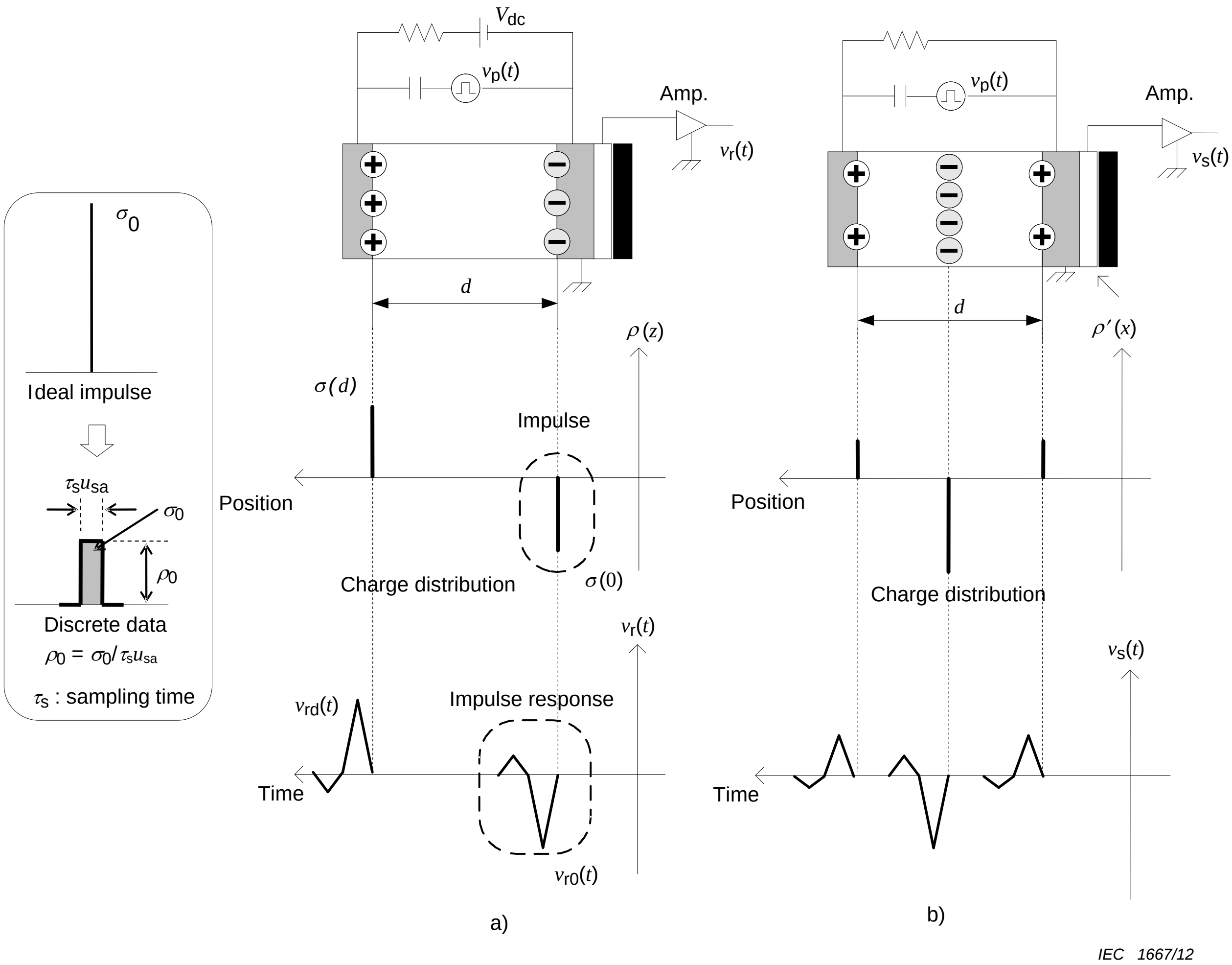


Figure A.7(a) – Reference signal **Figure A.7(b) – Measurement signal**

Figure A.7 – Example of two types of signal

Figure A.8 shows a calculation flow to obtain the deconvoluted signal. In the process of deconvolution, the division is carried out in the frequency domain. However, the division by small values occur in high frequency components resulting in a divergence of the calculated value. Therefore a certain kind of low pass filter is used to reduce the divergent value. Since the shape of first peak in reference signal depends on the filter characteristics, the so-called Gaussian filter is preferably used in many cases because it makes the shape of peak symmetry. Figure A.9 shows the effect of Gaussian filtering. When a cut-off frequency of Gaussian filter is chosen over the frequency corresponding to the resolution of measurement, the results of calculation include an oscillating waveform as shown in Figure A.9. Therefore, the cut-off frequency must be chosen approximately within the frequency corresponding to the measurement resolution.

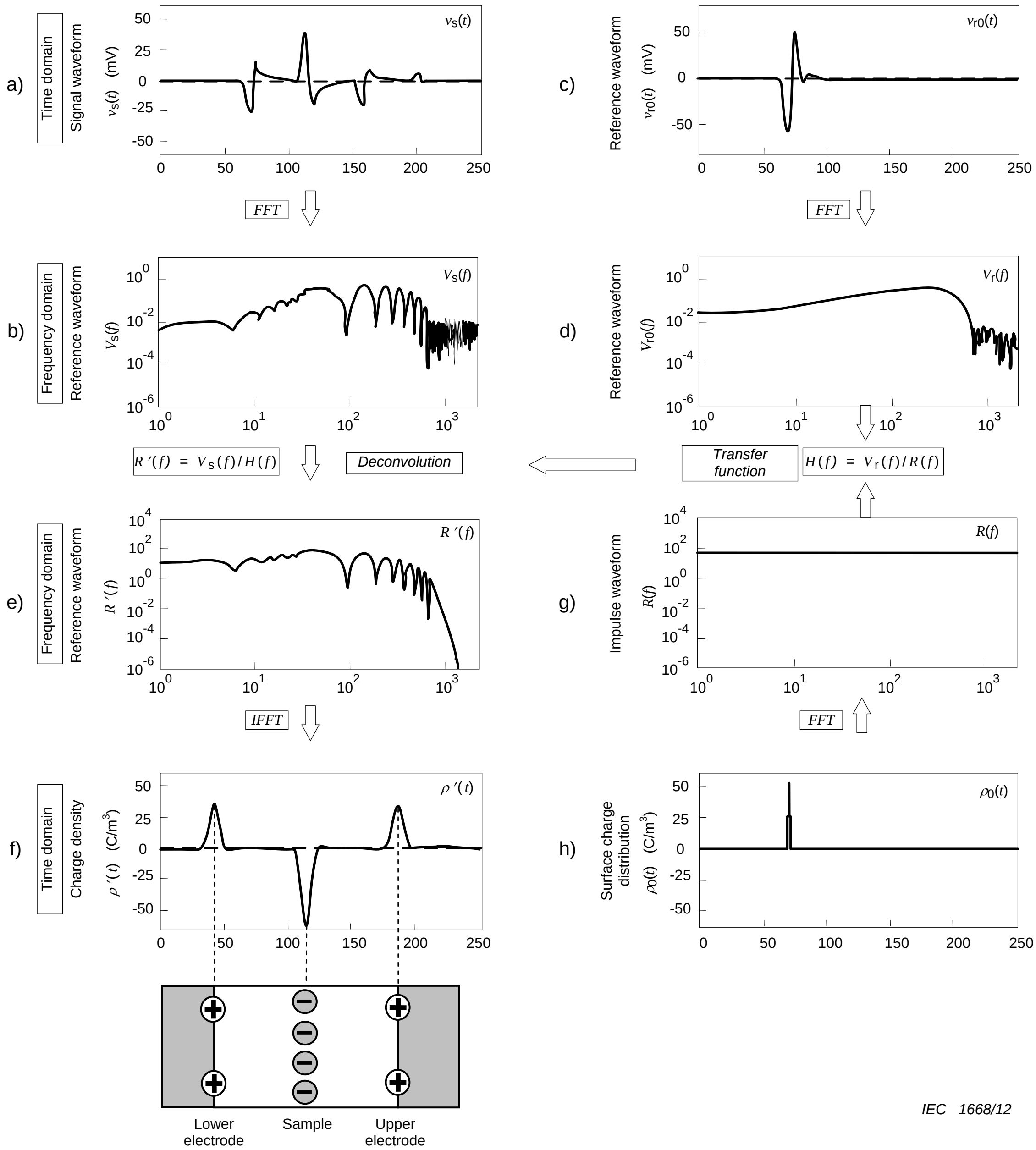
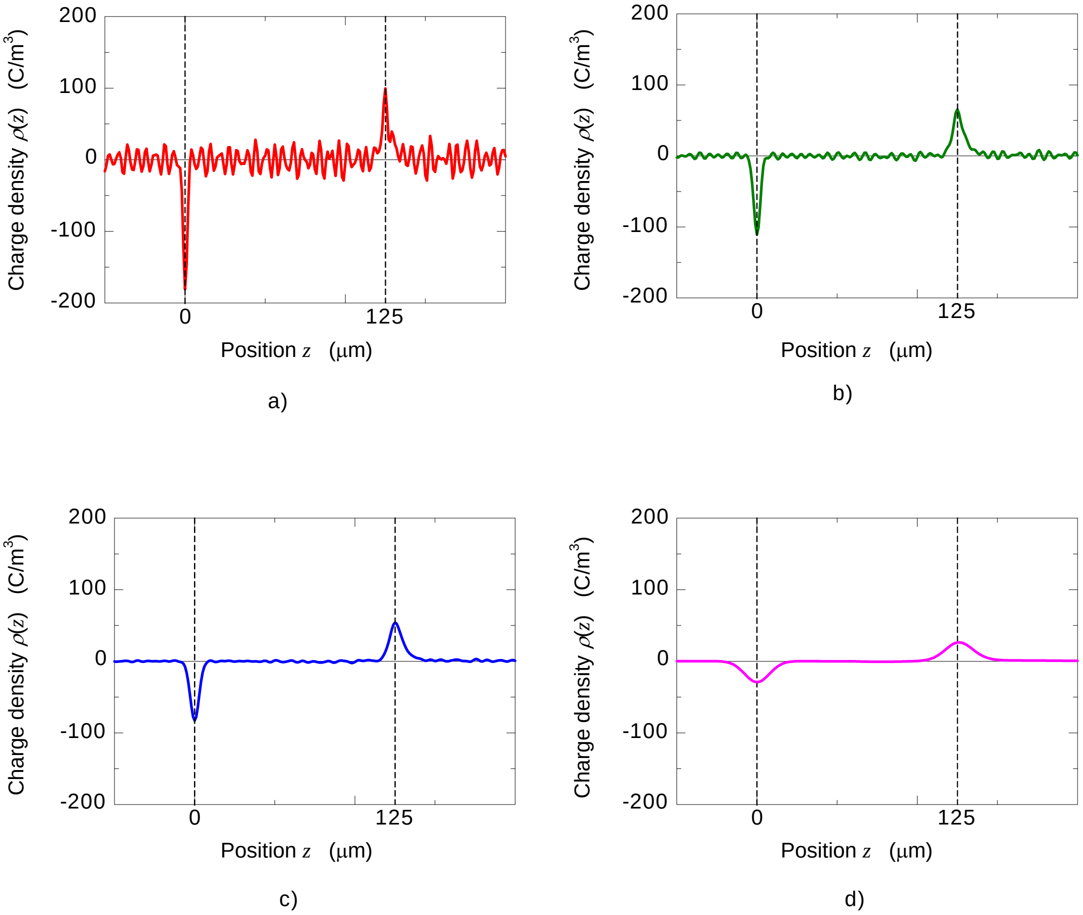


Figure A.8 – Calculation flow for deconvolution



(Sample : PMMA, thickness : 125 µm)

IEC 1669/12

Figure A.9(a) – Waveform using an inadequate Gaussian filter (cut-off frequency corresponding to resolution of 2 µm)

Figure A.9(b) – Waveform using an adequate Gaussian filter (cut-off frequency corresponding to resolution of 5 µm)

Figure A.9(c) – Waveform using an adequate Gaussian filter (cut-off frequency corresponding to resolution of 6,5 µm)

Figure A.9(d) – Waveform using an inadequate Gaussian filter (cut-off frequency corresponding to resolution of 20 µm)

Figure A.9 – Effect of Gaussian filter

A.3 Measurement system

A.3.1 Measurement apparatus

Figure A.10 shows a typical measurement apparatus of the PEA system. The apparatus is composed of following parts.

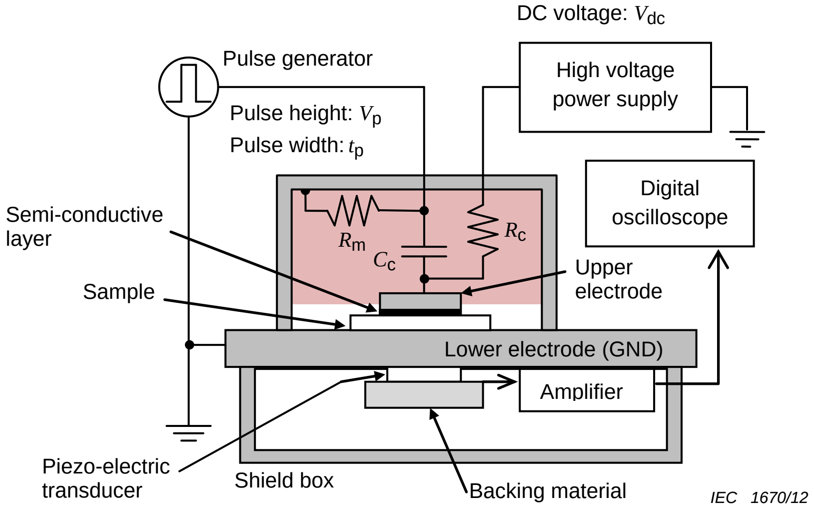


Figure A.10 – PEA measurement apparatus

A.3.1.1 Electrodes

When a d.c. and a pulse voltage are applied to the sample, surface discharge should be avoided. Furthermore, adequate pressure is applied to the sample homogeneously to avoid a reflection of acoustic waves at air gaps between the sample and electrodes. Therefore, the electrodes should be adequately parallel to each other and the apparatus should have a mechanism to give some pressure to the sample. Especially in the PEA system, the fitness between the sample and electrode is important to make a good condition for propagation of the pressure wave. If the electrodes have a dent, the mounted sample must be deflected. Such deflection would cause a lack of reproducibility because the gap condition between the sample and electrodes strongly depends on the pressure applied to the sample in each measurement. Therefore flatness and smoothness of them are also important. When the d.c. and the pulse voltages are applied to the sample, a semi-conductive layer is usually inserted between the sample and upper electrode to improve the acoustic impedance matching (see A.1.2). The semi-conducting layer should be shaped with a round edge to prevent a flashover.

A.3.1.2 Bias resistor

To apply a d.c. voltage to the capacitive sample, a bias resistor R_{dc} is needed. As the equivalent circuit shown in Figure A.11, the bias resistor R_{dc} is connected in series to d.c. power supply and in parallel to the capacitance of the sample C_{sa}. Since the capacitance of the sample C_{sa} (in the order of pF) is usually very small compared with the capacitance of power supply (in the order of mF), the pulse voltage is almost applied to a coupling capacitor C_c (in the order of nF) if there is no R_{dc} in the circuit. Since the current by the pulse voltage application mainly flows through the sample, the value of R_{dc} should be much larger than the high frequency impedance of C_{sa}. For example, when the frequency is supposed to be in range between 10 MHz – 200 MHz, the resistance of more than 10 kΩ is required. The resistance is also used as a protective resistor for power supply. When a breakdown occurs in the sample, then all currents flow through the resistance. If a flashover occurs on the surface of the resistance or breakdown also occurs in the resistance, the excess current flows through the power supply and the power supply may get a serious damage. Therefore, the resistance with large size for high voltage shall be used.

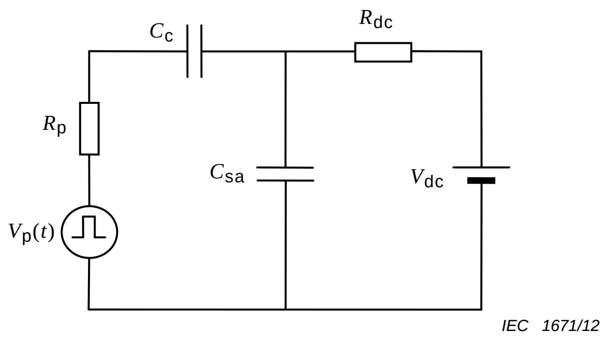


Figure A.11 – Equivalent circuit for voltage application

A.3.1.3 Coupling capacitor

The coupling capacitor C_c is necessary to apply a d.c. voltage to the sample. If there is no coupling capacitor C_c in the circuit, the d.c. voltage is separately applied to the bias resistor R_{dc} (> 10 kΩ) and an internal resistance of pulse generator R_p (ca. 50 Ω), and consequently the voltage is not applied to R_p placed in parallel with the sample. When the coupling capacitor (in the order of nF) is connected to the circuit, the impedances of both the coupling capacitor C_c and the sample capacitance C_{sa} for d.c. voltage become much larger (> 10^{12} Ω) than R_{dc} (> 10 kΩ). Therefore, the d.c. voltage is adequately applied to the sample using the coupling capacitor. In addition, when the capacitance is decided as much larger than the sample capacitance ($C_c >> C_{sa}$), the pulse voltage v_p is correctly applied to the sample. Since most of the sample capacitance is expected as several pF, the capacitance of a few nF is adequate for the coupling capacitor.

A.3.1.4 Piezo-transducer

The piezo-transducer is an acoustic sensor to detect the pressure waves in the PEA method. A thin PVDF (polyvinylidene difluoride) film is usually used for the piezo-transducer in the PEA system for room temperature measurement. In the case of high temperature measurement, a crystal sensor of $LiNbO_3$ (Lithium niobate) is preferably used because of its stability against change of temperature. Since the thickness and the acoustic velocity of piezo-transducer are important to estimate the spatial resolution of the PEA measurement, the information about them should be obtained. Furthermore, the capacitance C_p of the piezo-transducer is important to estimate the frequency characteristics of the detecting circuit (shown in Figure A.11) including the piezo-transducer. Therefore, the capacitance of the piezo-transducer should be approximately estimated.

A.3.1.5 Amplifier

Since the output voltage signal from the piezo-transducer is usually very small, an amplifier is used. The detecting external circuit is composed of output impedance of amplifier Z_a and capacitance of piezo-transducer C_p as shown in Figure A.12. Therefore the output impedance Z_a should be known. In addition, since the signal voltage read out from piezo-transducer is very weak and affected by noise, the amplifier is located as close as possible to the piezo-transducer to make the lead line between them shorter. It means that the amplifier should be put in a shield box. A secondary amplifier is put outside the shield box as necessary.

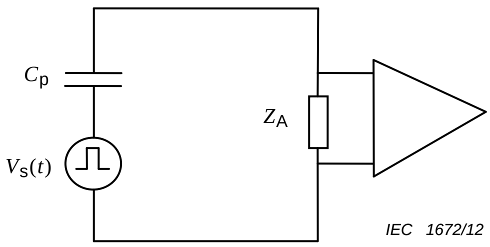


Figure A.12 – Equivalent circuit for signal detection

A.3.1.6 Shield box

One of the remarkable advantages of the PEA measurement is electric independence of the detecting circuit from the high voltage application unit. In other words, since the detecting circuit is independently composed in an electrically shielded box, the measurement is able to be carried out under the severe condition including electric breakdown of the sample. Therefore, a shield for electric noise is required. Especially since the application of pulse voltage to the sample generates a large noise, the shield is essential to reduce the noise.

A.3.2 Pulse generator

In the PEA measurement, the pulse voltage is used to generate the signal pressure wave. Since the measurement is usually carried out using the averaging technique, a repeatable pulse generation is required. The spatial resolution of the measurement depends on the pulse width. Therefore the width is decided as per the necessity of the spatial resolution. In general, the width is close to the travelling time of the pressure wave through the piezo-transducer (see A.1.4). The sensitivity of the measurement depends on the height of pulse voltage. The height is decided as large as possible at the same time it should be negligible in the measurement (see A.3.1 and A.1.1).

A.3.3 DC power supply

In the PEA measurement, d.c. voltage is applied to the sample to obtain a “reference” data (see 4.1.2 and Clause A.2). In this calibration procedure, the d.c. power supply for ca. 20 kV is required.

A.3.4 Oscilloscope

The output signal from the amplifier is observed using an oscilloscope. The oscilloscope is required to have a time resolution less than the measurement resolution in time domain (see A.1.4).

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