

*Measurement
Good Practice Guide*

**High field dielectric
properties of
piezoelectric
materials**

Mark Stewart & Markys Cain

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Measurement Good Practice Guide No. 25

High field dielectric properties of piezoelectric materials

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Abstract:

These guidelines are intended to enable a user to perform high field dielectric measurements on piezoelectric ceramic materials such as PZT (lead zirconium titanate). Many of the properties of piezoelectric ceramics such as PZT are highly dependant on the applied field, and therefore to make intelligent design choices, the dielectric properties are required at these field levels. These guidelines cover measurements at a fixed frequency of 1 kHz, to enable comparison with measurements made at low field. The measurement methods could all safely be extended from line frequency up to several tens of kHz, to cover a broad range of applications. However, for frequencies in the MHz range and above different factors need to be considered which are not covered in this guide.

The guidelines give some general advice on high field dielectric measurements followed by a detailed description of three different measurement methods: Schering bridge; impedance analysis; and PE hysteresis loop methods.

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High field dielectric properties of piezoelectric materials

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1. Scope

These guidelines are intended to enable a user to perform high field dielectric measurements on piezoelectric ceramic materials such as PZT (lead zirconium titanate). Many of the properties of piezoelectric ceramics such as PZT are highly dependant on the applied field, and therefore to make intelligent design choices, the dielectric properties are required at these field levels. These guidelines cover measurements at a fixed frequency of 1 kHz¹, to enable comparison with measurements made at low field. The measurement methods could all safely be extended from line frequency (mains frequency 50 Hz) up to several tens of kHz, to cover a broad range of applications. However, for frequencies in the MHz range and above different factors need to be considered which are not covered in this guide.

The guidelines give some general advice on high field dielectric measurements followed by a detailed description of three different measurement methods: Schering bridge; impedance analysis; and PE hysteresis loop methods.

2. Introduction

The dielectric properties add to a set of critical design parameters when selecting piezoelectric materials for active applications. Depending on the application, it may be desirable for the real value of permittivity (related to capacitance) to have a low value for use as an insulator for example, or to have a high value if the material is to be used simply as a capacitor (and physically as small as possible). Although piezoelectric materials and other ferroelectric materials are commonly used as capacitor materials, this guide is mainly concerned with their use as active materials. In this case the samples need to be 'poled'.

Considering the dielectric properties of the material alone, the designer needs to know how much current the power supply will need to provide and if the material is likely to undergo thermal runaway and thus possible failure. These questions can be answered with a knowledge of the relative permittivity, the dielectric loss and the thermal loss of the material in question. For piezoelectric materials the added complication is that these properties are

¹ Hall, Stevenson and Mullins [1] investigated the effect of electric stress on the dielectric loss factor ($\epsilon_r \tan \delta$) of a hard piezoelectric ceramic PZ26. Two methods of measurement were used, a high voltage Schering Bridge, operating at a frequency of 1kHz with the sample in air, and a low frequency, 'loop tracing' method operating at 1Hz with the sample in a temperature controlled oil bath. The two methods gave results which differed by more than the expected uncertainties of measurement. A recent NPL paper [2] resolves the situation by consideration of the thermal conditions in the two experiments.

dependent on the applied field, as well as the frequency. For many material selection purposes the effect of frequency is bypassed by standardising measurements at 1kHz and assuming that the materials will roughly obey the same frequency dispersion characteristics, bearing in mind that the heat generated is proportional to the frequency (equation 7).

The dielectric breakdown strength is also an important design consideration, but for most practical applications using piezoelectric materials the material will have lost much of its piezoelectric activity before breakdown occurs. A forthcoming CENELEC standard will cover issues concerning dielectric breakdown strength of high permittivity materials.

3. Capacitance, relative permittivity and dielectric loss

The term capacitance is the property of a device rather than a material, and is used to quantify the amount of charge (q) that can be stored when a potential difference (V) is formed across that device. The SI unit for capacitance is Farads, F.

$$C = \frac{q}{V} \quad \text{Equation 1}$$

Permittivity is the material-dependant parameter which can be used to predict the capacitance of devices of different geometries. For a parallel plate capacitor the capacitance is

$$C = \frac{\epsilon A}{t} \quad \text{Equation 2}$$

where ϵ is the permittivity in F/m, A the plate area (m^2) and t the thickness (m) of the dielectric. Because of the large units involved in permittivity, and for ease of measurement, it is more generally broken down into a dimensionless constant called the relative permittivity, ϵ_r , such that

$$\epsilon_r = \frac{\epsilon}{\epsilon_0} \quad \text{Equation 3}$$

where ϵ_0 is the absolute permittivity of free space, 8.854×10^{-12} F/m. The relative permittivity, ϵ_r is also referred to as the 'dielectric constant', and in common usage the word relative is often dropped.

The previous argument describes the properties of an ideal capacitor where the current is exactly 90° out of phase with the applied voltage, and all the power delivered to the capacitor is recovered. That is to say there is no loss. In real capacitor materials there is always some loss in power which can best be understood by imagining this loss as an ideal resistor in parallel with the ideal capacitor. Figure 1 represents this equivalent circuit along with its Argand (phasor) diagram. The R represents the lossy component of the dielectric. The permittivity must take into account the real (capacitance) and imaginary (loss) and is thus represented as a complex addition as:

$$\epsilon = \epsilon' - i\epsilon'' \quad \text{Equation 4}$$

The dielectric dissipation factor $\tan\delta$ is the ratio of imaginary and real parts of the complex permittivity where the arctangent, θ , is the phase angle between the voltage and the current. The dissipation factor can be evaluated by assessing the resistive leakage and capacitive values of the total current yielding:

$$\tan\delta = \frac{\epsilon''}{\epsilon'} \quad \text{Equation 5}$$

In terms of the equivalent circuit the dissipation factor can be expressed as:

$$\tan\delta = \frac{1}{\omega RC} \quad \text{Equation 6}$$

and the power generated within the material is found to be:

$$W = \omega CV^2 \tan\delta \quad \text{Equation 7}$$

It is important to remember that $\tan\delta$ and the capacitance can, and often do, vary with frequency and applied field which have implications on the power equation above.

For the preceding discussion it is assumed that the dielectric is an ideal linear lossy capacitor. This means that the capacitance and loss varies linearly with electrical field. This is not the case for ferroelectric materials but can be used as a first approximation and is useful in describing the meaning of loss and capacitance. For real piezoelectric materials the

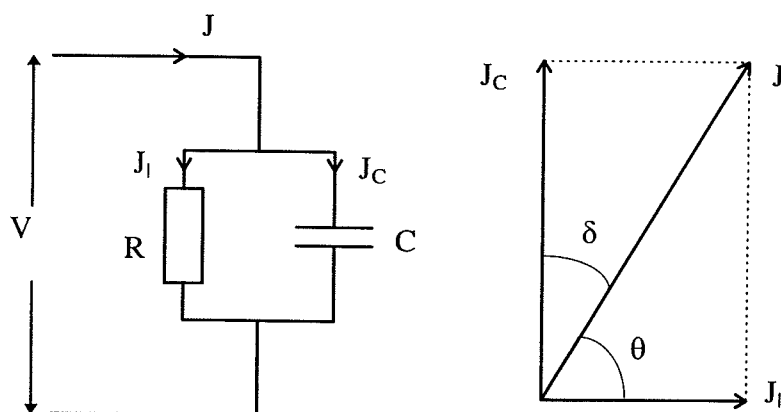


Figure 1: Phasor (Argand) diagram for a parallel RC equivalent circuit

capacitance and loss will increase roughly linearly with increasing applied a.c. field, and at higher fields will become very non linear. That is the series RC will no longer be a satisfactory model and additional non linear resistance terms are needed to describe the behaviour. Currently, few practical applications operate at these field levels, although recently there has been a drive to increase operational output with electric fields approaching the materials saturation level.

Please refer to the glossary for standard terms and definitions, taken from IEEE standards.

4. Definition of low and high field properties

It is difficult to give exact definitions of the difference between low field (sometimes termed small signal measurements) and high field in relation to properties of piezoelectric materials. Initially it was meant to distinguish between two different type of behaviour, low field, where the properties are independent of applied field, and high field, where a change in the applied field causes different material behaviour and consequent changed material property values. The low field measurements are typically measured using an instrument such as an impedance analyser or LCR meter that applies a maximum voltage of 1V. Using typical sample sizes this translates to fields of about 1V/mm. The high field measurement would be typified by experiments using a 1000 times amplifier giving fields of anything over 50V/mm up to several kV/mm. As measurement systems have become more refined it has been found that the piezoelectric properties are almost always dependent on applied field, however it is only at high field that changes of many percent are evident. The nature of this changeover and its exact field level is difficult to determine and define so a more pragmatic approach is to divide the behaviour based on the measurement systems. Thus we recommend that low field measurements would encompass measurements performed on systems that have a maximum applied voltage of less than 10 volts a.c. peak to peak (p-p), and high field measurements on a system that has a minimum applied voltage of 10 volts a.c. p-p.

5. Sample dimensions

Although the measurement of dielectric properties of piezoelectric material can be carried out on almost any sample shape or dimension *it is recommended that discs or cylinders are used*, as this makes for simpler permittivity calculations. For thin samples, it is easier to generate high fields, but for very thin samples porosity can become a problem so *it is recommended that monolithic samples not less than 0.5mm thick are used (0.2mm thick samples of high density and homogeneity may be used as a lower limit).*

For permittivity calculations, sample thickness measurements should be taken as the average of four measurements, one at the centre and three around the outside. Similarly, the diameter should be taken at the centre of the length of the disc sample as an average of four measurements taken at approximately 45° intervals around the circumference.

For high voltage measurements, it is preferable to use the manufacturers electrodes which have been optimised for the purpose as usually fired-on silver/glass frit. For a more detailed discussion on other electrode materials and geometries see [6]. It is possible to correct for the losses in electrodes if needed.

6. Poled samples

Most commercial PZT samples will have been poled, and the +ve direction marked with either a dot or a cross. Although it does not matter how the sample is oriented, it is good practice to decide on the field direction that these should orient with and maintain this for all experiments. This is particularly relevant when a *bias field* is applied. It may be wise to check the polarity of the sample with the manufacturer before applying bias voltages.

7. Recommended driving levels

In order to produce repeatable measurements it is desirable that the measurement process itself does not change the sample. A simple method to detect any changes in the sample, brought about by the application of a high voltage, is to measure the low field capacitance and loss, normally at 1V/mm at 1kHz, *before* and *after* the experiment. The level of change obviously depends on the type of material but, in general, there will be a small *increase* in capacitance following the application of a high a.c. electric field. This change depends on the aged condition of the sample, and for conditions which cause depoling - such as high fields or temperatures - a much larger *decrease* in the sample capacitance will be detected. Changes in the materials quiescent characteristics may also be modified by the application of strong d.c. electrical fields.

There are three main causes of sample 'degradation' brought about by high electric field exposure. Firstly, quasi static fields high enough to cause depoling, secondly sample self-heating arising from the dielectric losses in the material and thirdly changes in properties brought about by accelerated ageing. In practice, for typical PZT materials, there will be little change in the measured properties of the sample if the peak to peak voltage is kept to below approximately 1kV/mm. This, of course, would be halved when using uni-polar voltages (i.e. with D.C. offset), and for hard materials this value could be doubled to 2kV/mm. For the soft

materials, the dielectric losses can lead to significant sample heating at frequencies of 1kHz and above. In a recent round robin exercise on the evaluation of dielectric properties of various compositions of PZT ceramic, measurements were made at 1kHz up to 400V/mm rms on a hard and a soft material. There was no permanent change in the hard PZT materials properties. Depending on the sample's thermal surrounding - heat sink or still air - the soft composition measured at 400V/mm rms exhibited significant thermal heating, whereas at 200V/mm the measurements taken with or without thermal heat-sinks agreed to within 5%.

8. Frequency

The measurement of dielectric properties at a frequency of 1kHz is an industry standard used mainly for material comparison purposes as it is easily measurable and covers a broad range of applications. In order to maintain comparability, high field dielectric measurements are usually performed at 1kHz, as the higher currents associated with higher frequencies cause amplifier problems, whilst lower frequencies create difficulties in measurement of the current. 1kHz is also far from resonance for most practical monolithic materials, although this could be a concern for very large multilayer or stack actuators.

9. Temperature and humidity

It is always good practice to measure temperature and humidity in piezoceramic testing since some of the properties are known to be affected by variations in these parameters. However, in practice, changes in humidity have little effect on the *piezoelectric* properties but mainly affects the *breakdown strength* of the surrounding air. This will influence 'surface flash over'. Surface stresses should not exceed 2kV/mm in order to avoid this phenomenon. The change in ambient laboratory air temperature has *less* effect on the measured values of piezoelectric activity than that caused by *sample self heating*, often occurring when the materials are 'driven' hard. Measurement of the sample surface temperature is thus desirable, but the attachment of thermocouples is often incompatible with the high voltage associated with these tests.

The effects of dielectric heating of the sample can to some extent be calculated, for example see [3]. However, the paucity of thermal and electrical data for these materials means that the predictions can vary wildly. In addition, some of the data needed for the calculations can only be determined by performing the very high field experiments that are being simulated! The effects of dielectric heating can be minimised by increasing the thermal loading of the sample, such as making the electrical contacts in the form of large brass heat sinks, using forced air cooling, or even placing the sample in liquid such as xylene. If the permittivity changes with

time this is a good indication that self heating is occurring. Although the converse is true it does not necessarily mean that the whole of the sample is at ambient.

A forthcoming standard [4] on high power measurements of piezoelectric materials proposes that the field should be applied for 1 minute before measurements are made, and should be turned off for at least 2 minutes before successive measurements. This is an attempt to stabilise the temperature before readings are made.

10. Drive waveforms

The type and duration of the applied electrical waveform can often affect the materials property values that are measured. For example, many methods utilise a continuous sine waveform for the applied electrical field. As discussed in section 9, this may cause the sample to self heat, thus changing its piezoelectric and dielectric properties. Methods to minimise this effect are primarily based on ways to remove the thermal energy before the sample has time to significantly heat up. An alternative method, that is used in the PE-loop measurement method, applies a single or double pulse of continuous wave signal; that is, one or two complete cycles of electrical field. The sample responds to the electrical field and material property data can be determined, but the sample does not have the opportunity to heat up since the integrated driving energy is now much less. There are other issues that need to be addressed when using such pulse type techniques including the polarisation state of the piezo ceramic. These and other matters are discussed in a separate publication*.

11. High voltage amplifier requirements

The choice of high voltage amplifier must be made based on several factors:

- high voltage specification
- maximum drive frequency that can be maintained at the power amplification stated (thus the transfer characteristics are important)
- maximum current that can be delivered into a resistive and capacitive load
- amplification linearity and distortion
- input and output impedance
- circuitry protection
- cost

The typical operating fields of ceramic piezoelectric materials can go up to 500V/mm and even 1kV/mm for some hard compositions, and so depending on the sample thickness voltages in excess of several kV are necessary. Typical voltage gains for amplifiers used for these types of experiments are 100, and 1000 times, and are usually controlled by a function

* Ferroelectric Hysteresis Measurement & Analysis, NPL Publication CMMT(A)152, 1999.

generator capable of only 10 volt outputs. Depending on the capacitance of the device and the required frequency the maximum current of the amplifier can quickly become a limiting factor. The gain of the amplifiers are often fixed, however when the sample begins to draw too much current the gain is reduced. Normally this is not a problem, as the actual voltage is measured by means of a resistor divider network incorporated in the amplifier, which is fixed at the amplifier gain setting. However the clipping of the amplifier as the device pulls too much current introduces unwanted harmonics, and is sometimes difficult to spot. A divide by 10 or 100 oscilloscope probe can be used to double check the voltage at the sample. This can also highlight poor connections to the sample, since electrical breakdown can readily occur in narrow air gaps between electrodes at high voltages.

Since the measurement operating frequency is typically much lower than the response dynamics of the amplifier and the limiting drive factor is usually the current limit, there is a possibility of introducing high frequency noise from driving the saturated amplifier. In acoustic emission experiments on piezoelectrics this noise can be a problem as the high bandwidth detector can pass these frequencies on to the measurement system. This high frequency component is removed by adding a resistor in series with the sample (capacitor) [5], with a value chosen such that the time constant is much longer than the driving frequency. Care must be taken to exclude the effect of this additional component in the measurements, i.e. the voltage drop is measured across the sample only.

A list of suitable amplifier manufacturers is given in the appendix to this guide.

12. Sample breakdown/ flashover

When dealing with thick monolithic materials it is often necessary to apply several kilovolts to obtain even modest applied fields. Sample breakdown is always a possibility, however this is unlikely, since most PZT materials (depending on material porosity) will have a breakdown strength in excess of 5kV/mm, and driving the materials at these levels will produce very non linear hysteretic behaviour. A more likely scenario is flashover due to air breakdown around the sample. Air breakdown can occur around at fields of about 2kV/mm, dependent on the humidity level, and depending where the flashover occurs could damage sensitive measuring equipment. When designing the sample holding arrangement it is important to keep this in mind, so that there are no gaps between the high voltage and ground small enough to cause air breakdown. Sometimes even with careful design it is easy to overlook things such as poorly defined electrodes on ceramic samples.

13. Circuit protection

In most high field measurements systems the current measurement circuitry is highly sensitive, designed to measure mA down to pA. If a fault condition develops, such as described above, the consequent voltage spike will almost certainly destroy parts of the current measurement circuit. The damage is usually very local, often an IC will act as a fuse, protecting further damage. The cost of replacement components may be small, but the system will require recalibration.

In order to prevent this, a protection circuit consisting of two back to back diodes connected between the input and ground, and an optional current limiting resistor (figure 2) is utilised. The critical characteristics of these diodes is that they should be able to withstand the high voltage and that the reverse current leakage should be as small as possible so as not to effect the measured current. Suitable diodes, which have been used successfully are 1N4007. It is always good practice to compare measurements with and without protection to establish the effect of the additional components.

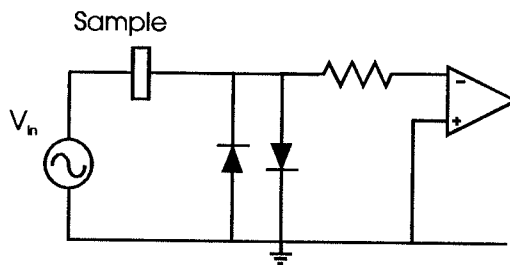


Figure 2 Protection circuitry for current amplifiers

14. Safety

An area which can cause a potential equipment and personal safety hazard is applying a much higher voltage than intended, which can occur when adding *1000 volt amplification to systems designed for low voltage operation. Potential pitfalls are incorrect sequencing of switching equipment causing momentary application of high voltages, or breakage in the feedback system of a the high voltage generation. To avoid these instances it is good practice to run the test sequence without the high voltage amplification in place and to monitor the signal to the high voltage generator with an oscilloscope set to trigger just above the maximum applied field. This will highlight any untoward applied voltages.

It is recommended, and depending on the local regulations may be a legal requirement, that some form of safety interlock is incorporated into the

measurement system that prevents any possible handling of the high voltage components whilst the high voltage is applied.

15. Dielectric measurement under stress/load

Since many piezoelectric materials are typically operated under some external stress it is often useful to know the dielectric properties under stress. Two problems can arise during these measurements. Firstly the application of the 1kHz high field to the piezoelectric sample causes the sample to vibrate and, depending on the stiffness of the loading system, the resonance of the complete measurement system can interfere with the results. It is advisable to carry out a frequency sweep over the frequency range of interest to determine the extent of any resonant behaviour. One proposed method of negating this effect is to mount two identical samples back to back so that there is no net displacement of the sample.

The other problem associated with dielectric measurements under load is maintaining a uniform stress level throughout the sample. For most practical purposes piezoelectric samples are usually thin discs - as thicker samples will need higher voltages to give the same field levels. However, uniform uniaxial loading of thin discs is difficult due to *end effects* and it has been found that longer cylindrical samples give a more reliable result which can be extrapolated to other geometries. Standard piezoelectric solid rods 15 mm long, 6.35 mm diameter are suitable for this purpose. If the dielectric properties of thin discs are required, then carrying out the tests on thin discs is certainly easier than finding the true stress level in the disc using finite element analysis and determining the dielectric properties in a truly uniaxial sample.

16. Calibration and verification of measurement system

Calibration of the equipment obviously depends on the particular equipment used. Problems can arise because, although the equipment may be calibrated at low voltages, this may not be valid for the high voltages being considered here. High voltage Schering bridge manufacturers often supply reference capacitors with certification valid for high voltages, but these are usually expensive and bulky air gap capacitors. A more cost effective solution may be to use polypropylene capacitors, or even a very hard and stable PZT material, which has been traceably calibrated. NPL is currently investigating this option.

One disadvantage of voltage-independent reference materials is that it is difficult to know if the field application and measurement systems are functioning correctly. This is because the capacitance will be the same for all applied voltages and so measurements will reveal nothing

about the actual applied field. To remedy this, the applied voltage could be measured independently (generally good practice) or a more pragmatic method of verification would be to examine the behaviour of a *soft piezoceramic*. For soft PZT materials there could be a 2 or 3 fold increase in the permittivity at fields of 400V/mm rms compared with the low field value. The increases are roughly linear with applied field, with the response of the soft 5A type being very linear. For hard PZT materials the increase in permittivity will be around 10%, but for these materials the increase in dielectric loss is likely to be more significant. A decrease in permittivity with increasing field or any massively *non-linear* behaviour is likely to be due to measurement error.

17. Reporting measurement results

The amplitude of the applied field and the resultant current waveform can be expressed as either rms or peak to peak (p-p). The capacitance and loss can be determined using either method providing the signals are sinusoidal. However, if there is significant non linearity then the values determined by the two methods can start to differ. As the properties of piezoelectric materials are field dependent, it is important to list the applied field and whether this is an rms or p-p value. Conversion between the two is simply -

$$V_{pp} = 2\sqrt{2} V_{rms} \quad \text{Equation 8}$$

In summary, the following table gives a minimum requirement for reporting the results of the measurement of high field dielectric properties.

Test Details for Disk Samples

Sample Details			
Sample History			
Sample dimensions		Poling Date	
thickness (mm)		Date Tested	
diameter (mm)		Temperature (°C)	
Frequency (Hz)		Relative Humidity (%)	
Applied Field (p-p) (V/m)		Capacitance pF	
relative permittivity		Dielectric loss (tan.δ)	

18. Dielectric measurements using a Schering Bridge

It has been known that dielectric self heating (loss) and permittivity are functions of temperature, frequency and field strength since the early '30's, with much of the pioneering work being conducted by Wien culminating in the development of bridge techniques that bear his name. During time the Wien bridge was replaced by the generally more relevant, precise and adaptable Schering type bridge for measurements at both low and high voltages.

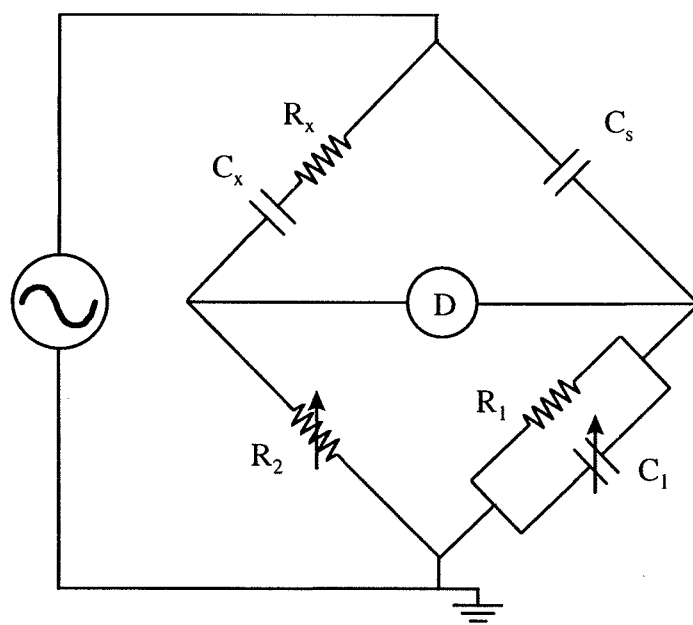


Figure 3: High Voltage Schering Bridge Circuit

There are many implementations of the Schering bridge and its derivatives such as the transformer ratio arm (TRA) type bridges for measurements of the dielectric properties of materials at high voltages. Advantages that exist for the TRA bridge include lower cost compared to the Schering bridge, wide range using one standard and no effect of leads on the measurements. Figure 3 gives a schematic of a high voltage version of the Schering bridge. The capacitive upper arms of the bridge represent a low impedance to the mains power source compared to the (essentially) resistive lower arms. Consequently, most of the voltage is dropped on the upper arms leaving the lower arms at virtually ground potential. This confers a certain degree of safety to the operator since the balancing of the variable components forms part of the lower arm. Usually some form of protection is needed in case the sample breaks down, in which case the high voltage can appear across the balancing circuitry.

The bridge is balanced by varying C_1 and R_2 . The standard capacitor, C_s , is of very low loss and typically air filled (or vacuum) parallel plate type. The balance point of the bridge is produced by equating impedance's of opposite arms in the usual way, yielding values for capacitance and $\tan \delta$ of:

$$C_x = \frac{C_s R_1}{R_2}$$

Equation 9

and

$$\tan \delta = \omega R_1 C_1$$

Equation 10

The fact that the Schering bridge is subject to certain measurement errors - due in large part to stray capacitance - other bridge techniques may be regarded as preferable in certain applications (although using three terminal guard techniques does provide correct compensation of stray capacitance). Examples include the Poleck type universal capacitance and $\tan \delta$ bridge, the modified transformer ratio arm bridge and the current comparator bridge (which can measure $\tan \delta$ values as low as 10^{-6}).

Advantages of the Schering Bridge measurement method are that it is a simple robust technique, the major disadvantages apply for any bridge technique - notably long balancing times, cost of ownership and purchasing costs (which includes a large number of precision components). Typically commercial equipment is made to operate at a fixed frequency, either mains frequency or 1kHz, so the opportunity of exploring the variation with frequency is not available. A list of possible equipment suppliers is given in the appendix.

19. Dielectric measurements using an impedance analyser

Measuring systems based on non-bridge techniques have become more widely available and are typically based on computer controlled integrators and phase sensitive detectors coupled with high precision Analogue to Digital (A/D) converters. For example, the dielectric properties in the near DC (~100Hz) to several kHz range may be measured with an electric impedance analyser such as the Hewlett Packard HP4194A (or HP4294A). This analyser applies a sinusoidal voltage (up to a maximum voltage of around 1V rms.) to the Device Under Test (DUT) and measures the current flowing through it. The sample impedance may be determined by the measured current and applied voltage. The dielectric constant (permittivity) and its loss factor ($\tan \delta$) can be determined from the admittance (1/impedance) of the DUT.

In principle these systems could be made to operate at much higher voltages, however the commercialisation of this type of instrument is probably not viable. As users have expressed an interest in performing the measurements, manufacturers have described how to modify the standard impedance analysers to operate at higher voltages by using a high voltage amplifier.

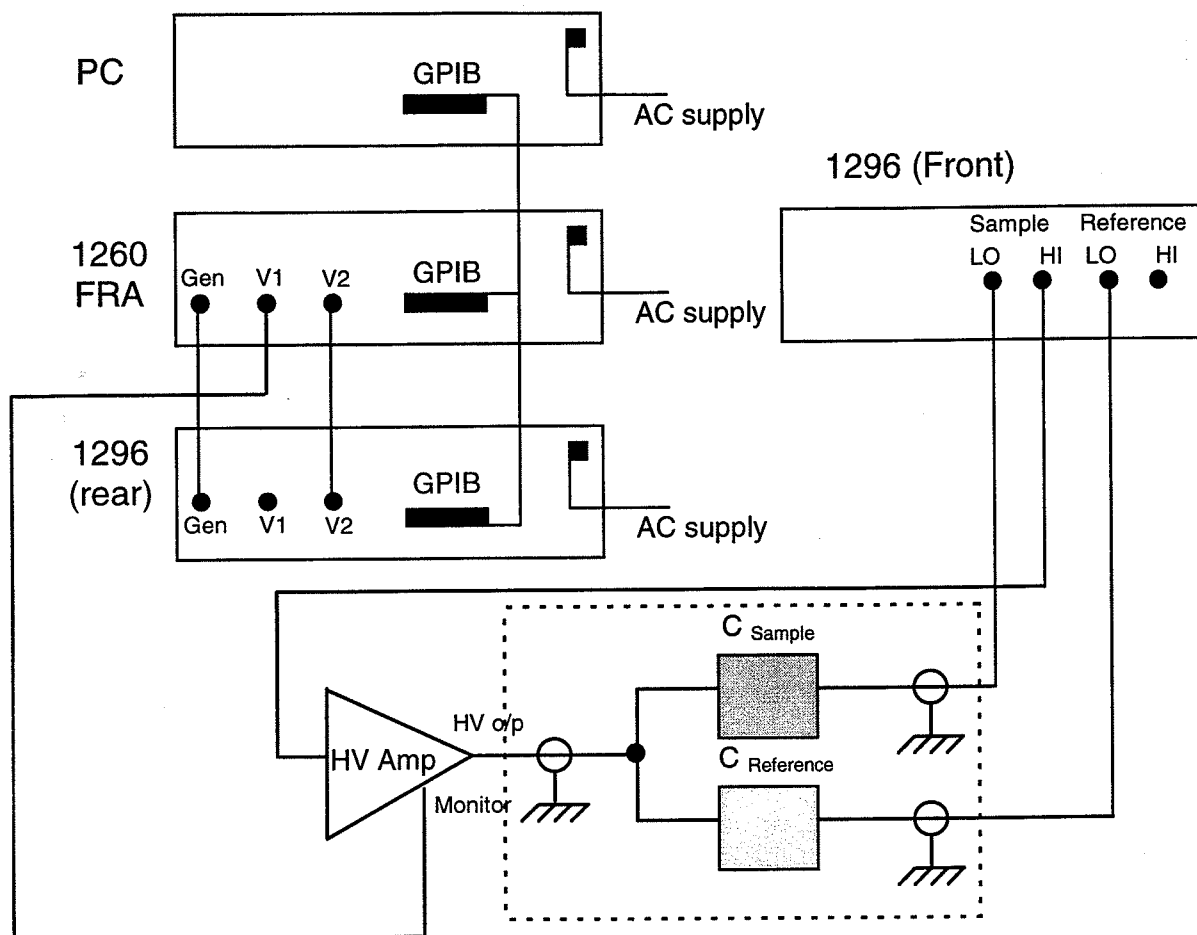


Figure 4: Impedance analyser with additional high voltage ac source circuit diagram - from 1296 Dielectric Interface Manual (Solartron).

Figure 4 shows such a set up for a Solartron impedance analyser and dielectric interface. The Monitor output from the HV Amplifier contains circuitry which includes a resistor divider network to drop the typically 1000 fold voltage gain (i.e. for a 1000 times gain high voltage amplifier), and a current limiting resistor. The measurement of capacitance is usually not a problem with these systems, but, in order to accurately measure the dielectric loss, a reference capacitor of known capacitance and loss is needed. By keeping similar lead lengths on the low voltage side of the capacitors means the phase angle can be accurately measured with respect to the reference capacitor. One minor drawback with this system is that a reference capacitor that has similar capacitance value to the DUT is needed, that is insensitive to applied voltage, and has been independently calibrated at these voltages. In commercial Schering bridges these capacitors are usually highly accurate and stable air gap capacitors, but these tend to be expensive and bulky. Current work at NPL is investigating the use of inexpensive polypropylene capacitors for use as reference materials.

The main advantage of using impedance analysers to perform high voltage dielectric measurements is that there is no manual balancing involved, and the systems are easily automated to perform many measurements. The frequency range of the equipment is now

dependent on the bandwidth of the high voltage amplifier, and using a number of amplifiers a suitably large range of frequencies can be covered.

20. Dielectric measurements using P-E hysteresis loops

A P-E loop for a device is a plot of the charge or polarisation (P) developed, against the field applied (E) across that device at a given frequency. The significance of this measurement can be more easily understood by considering the P-E loops for some simple linear devices. The P-E loop for an ideal linear capacitor is a straight line whose gradient is proportional to the capacitance. This is because for an ideal capacitor the current waveform leads the voltage waveform by 90 degrees, and therefore the charge (the integral of the current with time) is in phase with the voltage. The P-E loop for an ideal lossy capacitor - represented by a parallel RC network - is shown in figure 5, where the area within the loop is proportional to the loss tangent of the device, and the slope proportional to the capacitance.

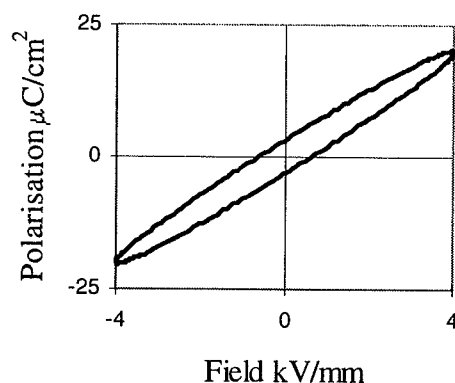


Figure 5 PE loop for an ideal lossy capacitor (parallel RC circuit).

PE loops studies are usually concerned with very high field behaviour, near saturation of the piezoceramic. However, for most practical applications the operating field is well below that which would cause ferroelectric domain switching and hysteresis. PE loop experiments can still be used at these lower field levels, and quantitative measurements of the dielectric permittivity and loss can be derived.

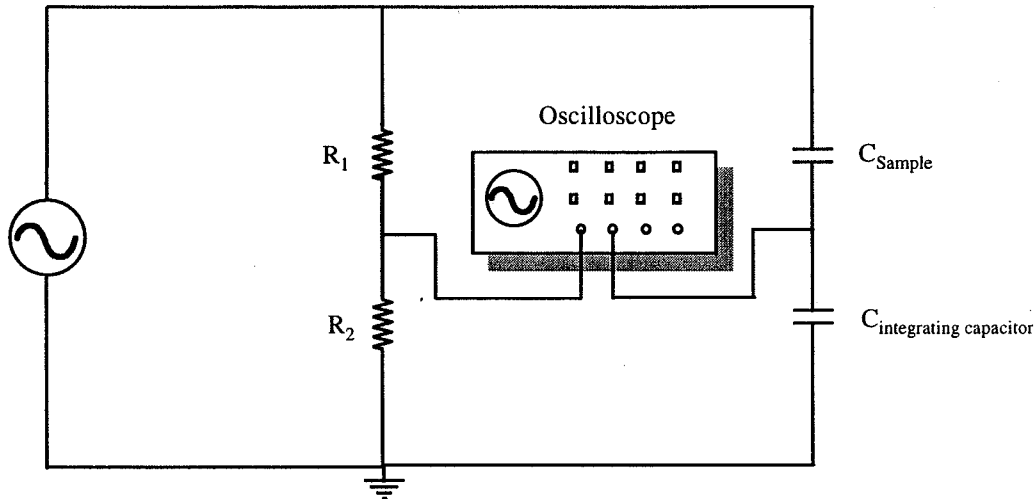


Figure 6 Schematic of a Sawyer Tower circuit for P-E loop measurements

The hardware for performing PE loops is very simple, the classic example is the Sawyer Tower circuit of figure 6. Here, the PE loop is displayed on an oscilloscope by using a resistor divider to monitor the voltage, and an integrating capacitor to measure the charge. Using modern data acquisition and computing methods these PE loops can be digitised for subsequent calculations. An advantage using computer aided data acquisition is that the integrating capacitor can be replaced by a current amplifier and the integration to determine charge performed by software.

In most cases, for high permittivity ferroelectrics, the polarisation P at a given time is very nearly equal to the total charge density or dielectric displacement D . However, a small correction is needed in order to give accurate results for low permittivity dielectrics such that:

$$P(t) = D(t) - \epsilon_0 E(t) \quad \text{Equation 11}$$

The *hysteresis loss* U_H is determined as the area enclosed within the P - E loop by numerical integration:

$$U_H = \int_{t1}^{t2} P dE \quad \text{Equation 12}$$

P - E loops such as in figure 5 can be analysed to yield the high field dielectric coefficients ϵ_r' , ϵ_r'' and the loss tangent $\tan\delta$. Although linear dielectric theory cannot strictly be applied to a non-linear ferroelectric material at high field strengths, it is possible to calculate effective dielectric coefficients which represent the total charge stored and power dissipated within the dielectric. It has been found in practice that the deviations from linearity are usually small in the working range of most piezoelectric transducers, and so the dielectric coefficients determined in this manner do not deviate significantly from those found by other measurement methods (e.g. Schering bridge measurements).

In order to calculate the dielectric coefficients ϵ_r' and ϵ_r'' for a lossy dielectric, it must be recognised that the magnitude of the polarisation waveform will be influenced to some extent by the dielectric loss

$$\text{i.e. } P^* = \epsilon_0 \epsilon_r^* E^* \quad \text{Equation 13}$$

(where * indicates a complex quantity).

The magnitude of ϵ_r^* is found from the amplitude of the polarisation and electric field waveforms as follows:

$$|\epsilon_r^*| = \frac{|P^*|}{\epsilon_0 |E^*|} \quad \text{Equation 14}$$

By introducing the specimen dimensions and substituting the electric field E for the applied voltage V , into equation 7 the power dissipated can be expressed as:

$$\text{Power dissipated} = \pi f \epsilon_0 \epsilon_r'' E_0^2 \quad \text{Equation 15}$$

where f is the frequency of the applied AC signal.

This equation yields the energy loss per cycle, which is the hysteresis loss U_H :

$$U_H = \pi \epsilon_0 \epsilon_r'' E_0^2 \quad \text{Equation 16}$$

Subsequently, the real part of permittivity ϵ_r' is found from these two values :

$$\epsilon_r' = \sqrt{(|\epsilon_r^*|)^2 - (\epsilon_r'')^2} \quad \text{Equation 17}$$

The dielectric loss tangent $\tan \delta$ is simply the ratio of ϵ_r'' to ϵ_r' as detailed in equation 5.

The accuracy of using PE loops for dielectric measurements is very poor in comparison to the Schering bridge and impedance analyser, but it can still give valuable information. If there is any non-linearity present then this will be evident as a distortion of the PE loop. At present, there are no techniques to quantify these deviations, but it should be possible to do some kind of difference between ideal parallel RC and experimental PE loops. Another advantage of PE loop analysis is based on the result having been measured during just one single cycle of the applied field at the selected frequency. This means there is very little chance of the sample self heating.

The software to perform the calculations described here are not generally available in commercial hysteresis packages, but there is sufficient information given here to implement them. Alternatively, software to perform these calculations has been jointly developed by NPL in conjunction with the University of Manchester, and its implementation to suit your own requirements may be available. Please contact NPL for further information (refer to leading pages of this document for contact details).

21. Advantages and disadvantages of the various methods

Advantages and disadvantages of the various measurement systems, described in previous sections, are summarised below:

- *Schering bridge methods*: generally, very sensitive, usually limited frequency and limited voltage ranges but is the best method for extremely low loss measurements (< 0.001). The main drawback is the need for manual balancing, thus hard to automate, good for high voltages, & can have Wagner earth for guarded measurements. Equipment can be expensive. (*The Transformer Ratio Arm* systems offers the highest accuracy (can get better than 1ppm), is good choice for long leads, not easy to automate, and good for guarded measurements.)

The various disadvantages of bridge techniques, viz. time consuming (for Schering type bridges), low frequency loss in accuracy (for transformer type bridges) and high voltage limitations and lack of easily integrated automation, has led to the development of time domain type systems. Here, the ac characteristics are evaluated by analysing the charging and discharging currents flowing after the application of the imposed sinusoidal signal.

- *Impedance analyser*: general purpose instrument which can readily be adapted for high voltage measurements. Not as sensitive for loss measurements. Errors in low loss measurements due to the fact that the minor component is in digital form, and thus may have a 100% error if a small value (< 0.005). Can cover a large frequency range but is dependant on performance of high voltage amplifier. Measurements can be readily automated. For the Solartron Impedance Analyser (1260) the frequency range is extended down to 0.001 Hz with the use of a high impedance interface (*Dielectric Interface 1296*) which can then provide the user with a low loss, fully automated, dielectric measurement system.
- *Hysteresis measurements*: the only method by which you can actually record and view the voltage and current signals and the nature of the P-E relationship. Non-linear effects can be readily identified and quantified. Very wide voltage and frequency ranges can be covered once the basic system is set up (limited by the specifications of instruments making up the system). This is also the only method in which you can easily depart from sine wave drive and still expect to get useful results. The method allows very low frequency measurement and/or pulse technique thus minimising effects of sample self-heating. Determination of the loss tangent becomes unreliable below 0.01 (this could be improved somewhat by taking care over the current measurement method and noise suppression).

22. Suppliers of equipment

This list is intended to give a novice possible sources of instrumentation for performing the tests described in this guide, and is not intended to be exhaustive. The inclusion of a manufacturer does not imply recommendation by NPL, neither does the exclusion imply a product is unsuitable.

22.1 Impedance Analysers

Solartron

Victoria Road, Farnborough, Hampshire GU14 7PW, UK.

<http://www.solartron.co.uk>

Tel. +44 (0) 1252 376 666

Fax. +44 (0) 1252 543 854

Hewlett-Packard Ltd.

Cain Road, Bracknell, Berkshire RG12 1HN, UK.

<http://www.hp.com>

Tel. +44 (0) 1344 36 00 00

22.2 High Voltage Amplifiers

Trek, Inc.

3932 Salt Works Road, P.O. Box 231, Medina, N.Y. 14103 USA.

[Http://www.trekinc.com](http://www.trekinc.com)

Tel. +1 716 798 3140

Fax. +1 716 798 3106

22.3 Schering Bridge Type Hardware

Ceast S.p.A.

Via Airauda 12

10044 Pianezza (TO)

Italy

Tel: 39-011-966-4038 (10 lines)

Fax: 39-011-966-2902

Web: <http://www.ceast.com>

Lemke Diagnostics GmbH

Radeburger Straße 47
01468 Volkersdorf/Dresden
Germany
Tel.: +49 35207 863-0
Fax: +49 35207 863-11
Web: <http://www.ldic.de/>

Tettex

Trench Switzerland AG
Tettex & Haefely EMC Division
Bernstrasse 90 / P.O. Box CH-4028
Dietikon-Zurich / Switzerland
Phone +41.1.744 74 74
Fax +41.1.744 74 84
Web: <http://www.tettex.com/>

Take Control

The Institute of Research
University of Birmingham Research Park
Vincent Drive
Birmingham B15 2SQ
ENGLAND
Tel: +44 121 415 4155
Fax: +44 121 415 4156
Email: admin@take-control.demon.co.uk

Appareils Vettiner S.A.

Z.I. 18 bis, rue Pierre Semard
69 007 Lyon
France
tel +33 47872 3232
fax +33 47872 8066

22.4 PE Loop Measurement Hardware

Radiant Technologies

2021 Girard SE, Suite 100

Albuquerque, NM 87106

Tel: 505-842-8007

Fax: 505-842-0366

e-mail: radiant@ferrodevices.com

Web: <http://www.ferrodevices.com>

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- [3] M G Cain, M Stewart, M G Gee, G J Hill and D A Hall, NPL Report CMMT(A98), March 1998.
- [4] CENELEC working group BTTF 63-2 "Piezoelectric properties of ceramic materials and components. Part 3: Methods of measurement and properties - High power". Draft version of standard.
- [5] H. Aburatami and K Uchino, Jpn. J. Appl. Phys. Vol. 35 (1996) L516-518.
- [6] ASTM Designation D 150-95. Standard test methods for AC loss characteristics and permittivity (dielectric constant) of solid electrical insulation.

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Engineering Dielectrics Volume 11A, 'Electrical Properties of Solid Insulating Materials: Molecular Structure and Electrical Behaviour', ed. R. Bartnikas, R. M. Eichhorn, ASTM Special Technical Publication 783, (1983).

ANSI/IEEE Std 180-1986. IEEE Standard Definitions of Primary Ferroelectric Terms. 1996.

ANSI/IEEE Std 176-1978. IEEE Standard on Piezoelectricity. 1978.

'Coaxial AC Bridges', B P Kibble, & G H Rayner, Adam Hilger Ltd., Bristol 1984.

'Complex Permittivity', Compiled by B K P Scaife, The English University Press Ltd, London 1971.

'AC Bridge Methods', B Hague, Sir Isaac Pitman & Sons Ltd, London 1962.

25. Glossary

This list of standard terms and notations have been taken from the IEEE Standard Dictionary of Electrical and Electronics terms (IEEE Centennial Edition 1984).

Loss Angle

'Loss Angle' is the smallest possible angle, 'delta', of which the loss tangent ('tan delta') is the tangent (i.e. it is the arctangent of the loss tangent). The term is usually used for materials with very low loss, having a loss tangent less than 0.001. For such materials the loss angle, expressed in radians, is invariably numerically equivalent to the loss tangent to within practical measurement uncertainty limits. The use of 'loss angle' instead of 'loss tangent' allows the loss to be expressed in terms of the convenient unit 'microradian' which has the value 1×10^{-6} or 1E^{-6} radians. Expression of such low losses in terms of loss tangents (which cannot be expressed in terms of radians) is often less convenient. Abbreviated in places to 'Loss Ang.'

Loss Factor

Loss Factor is equivalent to 'imaginary permittivity' or 'the imaginary part of the relative complex permittivity'. Abbreviated in places to 'Loss Fac.' Or 'Imag. Perm.'

NB. A distinction of common usage should be made between 'loss factor' used as a term in the physics of dielectric relaxation, and its phenomenological use to quantify the total measured electrical loss of a dielectric – including the conduction loss. Likewise 'conductivity' may refer to just the physical conductivity due to free charge carriers drifting steadily through the material in an applied electric field, or it may be an 'apparent conductivity' which is derived phenomenologically from the total measured electrical loss – including the dielectric relaxation loss.

Loss Tangent

'Loss tangent' is the ratio of the imaginary permittivity to the real permittivity of a material. Loss tangent is often referred to as 'tan delta' where 'delta' is the loss angle.

Abbreviated in places to 'Loss tan'.

Tan delta

'Tan delta' is a commonly used term for the loss tangent. Its use can be accepted because it is not ambiguous. 'Tan delta' is the tangent of the loss angle 'delta'.

Abbreviated in places to 'Tan del.'

(This is an informal term. 'Loss tangent' is more formally correct.)

Dielectric Constant

'Dielectric constant' is equivalent to 'real permittivity' or 'the real part of the relative complex permittivity'. But this term is also used by some authors for low frequency or 'static' permittivity in contradistinction to 'permittivity' which is used to describe RF and Microwave real permittivities. Though still in wide use, 'dielectric constant' is not a preferred term, partly because of this ambiguity but also because permittivity is not constant – it changes with frequency and temperature.

Abbreviated in places to 'Diel. Const.'

Conductivity

'Conductivity': in physics the formal definition is: *'a factor such that the conduction-current density is equal to the electric field intensity in the material multiplied by the conductivity'*. However in dielectric metrology the term is often used phenomenologically.

Abbreviated in places to 'Cond.'

Milliradian

'Milliradian' is a unit of angle equivalent to 1×10^{-3} or 1E^{-3} of a radian. It is often used to quantify low dielectric loss in terms of loss angle.

Microradian

'Microradian' is a unit of angle equivalent to 1×10^{-6} or 1E^{-6} of a radian. It is often used to quantify low dielectric loss in terms of loss angle.

Dissipation factor

'Dissipation factor' is equivalent to 'loss tangent'. This is a widely used term for describing dielectric properties but is not a preferred term because it can have a very different meaning in other disciplines.

Abbreviated in places to 'Disp. Fact.'

Power factor

'Power Factor' is the sine of the dielectric loss angle, i.e. 'sin delta' rather than 'tan delta'. It is not a preferred term because it can have a very different meaning in other disciplines.

Abbreviated in places to 'Pow. Fact.'

Q-factor

'Q-factor' is assumed here to mean the reciprocal of the loss tangent of a material. Its use in this sense is not preferred because there is ambiguity between this expression of an intrinsic property of a material (i.e. its loss tangent) and the non-intrinsic quality factor – Q-factor of a circuit or cavity in which it may be measured.

Abbreviated in places to 'Q-fact.'

Permittivity

When used alone 'permittivity' is equivalent to 'real permittivity' which is equivalent to 'the real part of the complex relative permittivity'.

Abbreviated in places to 'Perm.'

Real Permittivity

'Real permittivity', in some cases shortened to just 'permittivity', is equivalent to 'the real part of the complex relative permittivity'.

Abbreviated in places to 'Real Perm.' Or just 'Perm.'

Complex Permittivity

'Complex permittivity' is used here as being equivalent to 'relative complex permittivity'. It has real and imaginary parts and is defined in such a way as to make the imaginary part (the loss factor) always positive for passive dielectric materials.

Imaginary Permittivity

'Imaginary permittivity' is equivalent to 'loss factor' or 'the imaginary part of the relative complex permittivity'.

Abbreviated in places to 'Imag. Perm.'

ENERGY

