Smart Materials: Complexity of Measurement

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1 Abstract

The measurement methods necessary to reliably characterise functional materials for the next generation of smart structure or system must be refined and developed to encompass the materials inherent non-lineariy. This paper addresses the case for piezoelectric materials characterisation and describes one example based on piezoelectric resonance.

2 Introduction

Smart materials are ubiquitous in their application and typically comprise a functional material with integral intelligent control of some kind. This paper describes a set of these functional materials – piezoelectrics – that are the subject of scientific study at the National Physical Laboratory in the UK. With the support of the UK governments Department of Trade and Industry, NPL work with UK companies and research organisations to provide the tools needed to assess new materials or products, processes or models. In this way, the new technologies may be objectively compared to existing ones in a robust, and defendable manner. Ultimately the measurement science developed can be written into new or improved existing standards or provided as services to UK industry. Such activity promotes global trade by reducing barriers to commercial exploitation of new materials or products and enhances competitiveness and company innovation.

The paper first describes the operation of piezoelectric materials and some of their physical properties before the issues of measurement are addressed. The metrology of piezoelectric ceramic materials has become increasingly complex as their application has demanded more in terms of output strain, power, stress and we will demonstrate how this is reflected in their non linear properties.

3 Piezoelectric Concepts

Due to their inherent non-centrosymmetric crystalline structure, piezoelectric crystals possess the ability to become electrically polarised following mechanical pressure, with each surface becoming oppositely charged. Quartz and Rochelle Salt are two early examples. The charge generated is proportional to the pressure exerted. This is called the direct piezoelectric effect. The converse (or indirect) analogue is when the crystal expands (or contracts) along one axis when subjected to an external electric field. It is generally thought that the two effects are synonymous – vis. The direct and indirect effects ere determined by the same physics and described by the same materials parameters.

The mechanical strain, S_j, in response to an applied electric field, E_i, is described by the tensor equation:
$S_j=d_{ij}.E_j$

The d_{ij} is known as either the piezoelectric charge coefficient or (as in this case) the piezoelectric strain coefficient and has units V/m (or C/N).

The dielectric properties of a polarisable material may be described by analysing the charge stored, Q, by the passing of a sinusoidal current, I(t), at any time is given by:
Q(t)=?I(t) dt

The dielectric displacement, D, is defined as the surface charge density:
D(t)=Q(t)/A, with A being the specimens surface area.

A ferroelectric material is a material that exhibits a spontaneous polarisation even in the absence of an applied electric field and shows hysteretic behaviour between D and E. In most cases, for high permittivity ferroelectrics, the polarisation P at a given time is very nearly equal to the dielectric displacement, D. However, a small correction is needed in order to give accurate results for low permittivity dielectrics:
$D(t)=P(t)+\varepsilon_0E(t)$ or $P(t)=D(t)-\varepsilon_0E(t)$

Thus, measurements of current and electric field with time enable the familiar polarisation-field loops to be drawn. The significance of this measurement can be more easily understood by examining the PE-loops for some simple linear dielectrics. The PE 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 leads the voltage by exactly 90° and therefore the charge (or D and P) is in phase with the voltage. For an ideal resistor the current and voltage are in phase and so the PE loop is a circle with its centre at the origin. If these two components are combined in parallel we get the PE loop of in figure 1a, which is in effect a lossy capacitor where the area within the loop is proportional to the loss and the slope proportional to the capacitance. For more complex materials such as non-linear ferroelectric materials we get the PE loop such as figure 1b.

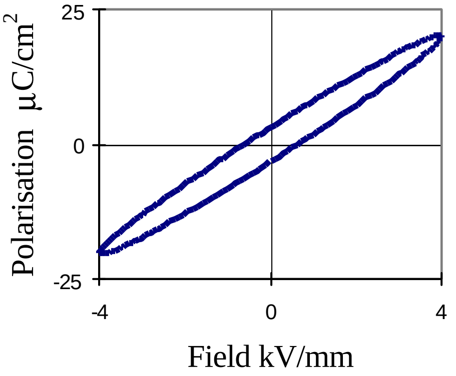

Figure 1a): Lossy capacitor response

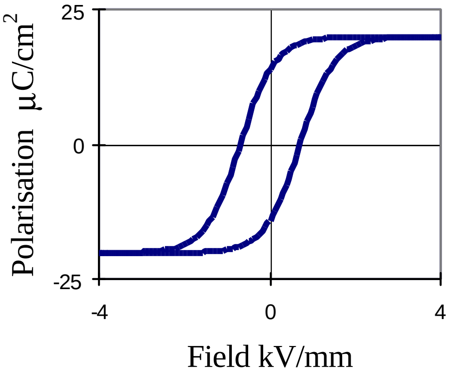

Figure 1b): Non-linear ferroelectric response

The non-linear response, coupled with the sensitive instrumentation necessary for accurate measurement provides us with our challenges for the successful characterisation of functional materials.

4 Piezoelectric Characterisation

The development of robust and reliable measurement techniques will be highlighted using an example of some DTI co-funded work carried out at NPL - that of piezoelectric resonance tests [1].

4.1 Piezoelectric Resonance

Apart from a component's physical size its frequency of free vibration is determined by properties such as its stiffness. This forms the basis for modulus measurements of resonating structures. For piezoelectric materials, since the material can be excited electrically, applying an ac field across the device can induce resonance without the need for external mechanical stimulation. Electrically induced resonance occurs in piezoelectric materials because of the electromechanical coupling that exists between applied field and induced strain and that is defined by the complete set of piezoelectric equations (IEEE standard on piezoelectricity [2]). The electrical impedance of a piezoelectric material exhibits peaks that corresponds to electromechanical resonance in the specimen. The sample response may be measured externally using acoustic or displacement techniques. However, since the current flowing through the sample is proportional to the functional response it is simpler to measure the electric current - or the impedance. Typically, frequency scans using an impedance analyser are used for this function. When an arbitrary piezoelectric sample is made to resonate a complicated superposition of different resonant vibrations exist within the material. These resonance's are made up from the various piezo-electromechanical coupling that occur between applied electric field and boundary conditions arising from the samples fixed surfaces. If samples are manufactured such that only one resonant vibration exists that can be directly linked to just one of the piezoelectric constants then all the components of the piezoelectric tensor may be extracted from measurement data taken from resonance experiments on a set of standard sample geometries. This forms the basis of the IEEE standard method. This enables the piezoelectric equations to be uncoupled to give an equation for the impedance of the sample as a function of frequency – or resonance analysis. These equations have then been solved to yield some of the components of the piezoelectric matrix (and stiffness and dielectric matrix as well).

In studies conducted at this laboratory, it became apparent that unless the experiments were carefully conducted and various test parameters adhered to, then large errors occurred. Many of these parameters included general good experimental design such as interference caused by, for example, the proximity of a human finger to the resonating sample, and compensation methods to eliminate any stray capacitance of test leads. Other issues included the sample holding arrangements and contact fixation method. Figure 2 demonstrated the large effect of clamping the sample off axis. Clearly, this is a gross effect in that it has very markedly changed the resonant features of the sample. Anyone wishing to analyse this spectrum will find that the errors in the various calculated parameters to be unacceptably large. Similar problems exist for the manner of electrical connection – for example using excessive quantities of solder will affect the resonance of the sample quite dramatically. The non-linear effects of piezoelectric activity are manifest in measurements taken at different applied electric fields. The low-field values of the piezoelectric properties are generally measured but it is also possible (and desirable) to measure their high field properties. This is because the materials are very often used at such high fields. For most of the commercial impedance analysers, the maximum applied field is 1 volt and so maximum field levels for typical ceramic samples are restricted to around 1V/mm. The forthcoming CENELEC standard [3] recommends an upper limit of 0.01V/mm. In practice it is usually better to use larger fields because of the increased signal to noise, and it is only when fields go above 1V/mm that it is possible to detect non-linear behaviour in soft piezoelectric materials. Sample quality may also alter the purity of the resonance profile such as when spalled or abraded material and chipped edges exist.

It is relevant to discuss losses prevalent in piezoelectric ceramic compositions since these values are often as important as the functional, dielectric and elastic constants that resonance analysis yields. In reality, a piezoelectric material comprises losses originating from its dielectric response to an electrical field, mechanical response to applied stress or following piezoelectric motion and its piezoelectric (strain) response to an electric field. The significance of loss results in sample heating or noise production and this is why for many applications an understanding of loss mechanisms and absolute values becomes important. Normally, the mechanical loss at resonance is calculated from the width of the resonant peak and is labelled Q or Quality factor. The narrower the resonant peak, the higher its Q. Dielectric losses are normally calculated from the phase angle between capacitance and field and labelled tan δ. Piezoelectric loss may not normally be calculated from resonance data but may be assessed through strain – electric field response whereby any hysteresis present may be ascribed to this loss alone - of course, if strain is produced then mechanical loss may also have an additive effect.

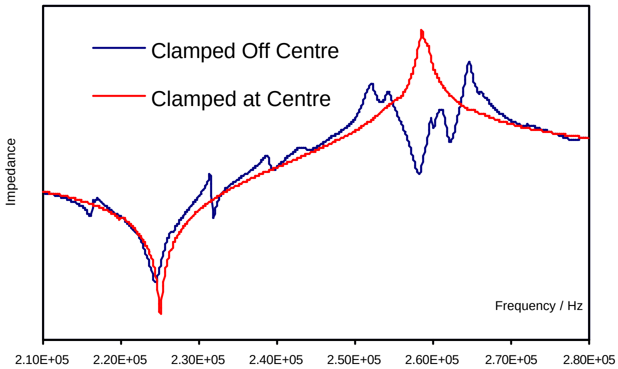

Figure 2: Effects of clamping offset on resonance frequency and spurious peaks.

An alternative method adapted by Sherrit and coworkers [4] from earlier work by Holland [5], Smitts [6] and ultimately commercialised by TASI Technical Software (© PRAP), was to treat the entire piezoelectric matrix as complex. Again, assuming ideal linear piezoelectric behaviour and small field level perturbations, then the same formulation used for ideal loss-less resonators could be utilised, yielding similar equations but of complex form. Here, the impedance, resonant frequencies, piezoelectric, stiffness and dielectric coefficients are all complex quantities now and the forms of the equations may be iteratively fitted to the various shapes of resonance curves for each of the different geometries. PRAP makes extensive use of complex elastic, dielectric, and piezoelectric properties in fitting the resonance curves. This method would yield much more accurate data since the entire resonance curve is being used in the analysis, not just two frequencies used in the IEEE method. Additionally, this method automatically takes into account the losses by virtue of its complex notation.

The losses associated with dielectric constant, piezoelectric constant and elastic constant have been measured independently of one another and most importantly using methods that do not include resonance. This is to try and explore the validity for the use of complex notation – vis. Real and Imaginary components of the parameters with loss given by the ratio real part / imaginary part. Results indicated various trends:

1. Dielectric loss: resonance yielded results ~ 25% higher than standard impedance test.
2. Piezoelectric loss: indirect measure of the piezoelectric coefficient and phase between strain and field yielded results in close agreement with the resonance analysis. Soft materials exhibited better agreement than hard materials. Figure 3 shows the data taken using three methods; 1. resonance analysis, 2. strain-field loop analysis and 3. strain-field phase angle measurement taken using a gain phase analyser. Although the measurement techniques seem to agree quite well with each other the

absolute level clearly depends on applied electric field and so techniques that take measurements at only one field level are less useful that others methods.

3. Mechanical loss: measure of stress / strain response yielded hysteretic loss. Results were consistent with resonance analysis.

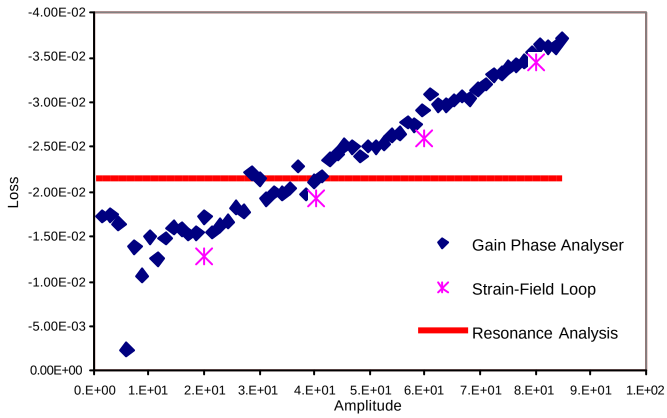

Figure 3: Piezoelectric loss with electric field measured using various techniques.

The results indicated:

a) Resonance analysis yields loss values that are consistent with our alternative measurements but significant differences exist that may be related to operating conditions, internal stress states within the sample and the interdependency of each loss in the resonance analysis algorithms.
b) More work is required to understand the origins of loss.
c) More experimental techniques are required to adequately measure these losses independently and accurately.
d) More theoretical work is needed to understand the dependence of these losses in piezoelectric materials.

5 Conclusions

The complexity of the measurement of the dielectric and piezoelectric coefficients now become clear - the materials exhibit non-linear properties even at modest field levels that makes the development of application-relevant measurement methods absolutely critical.

Similar trends in the need for a sustained development of measurement techniques and standardisation exist for many, if not all, of the other functional materials that are used to construct smart systems. Magnetostrictive materials and electrorheological materials are also inherently non-linear and provide many additional challenges for the metrological community. For piezoelectrics and electrostrictives the electro-mechanical coupling follows complex equations that depend on field amplitudes, frequencies, stress levels among others and measurement techniques need to be refined so that micro-and macroscopic theories that are being developed may be adequately tested in a robust manner.

6 References

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7 Acknowledgements

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