Characterisation of thin film piezoelectric materials by differential interferometric techniques

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ABSTRACT

Piezoelectric thin films are considered emergent materials for integration within Micro Systems Technology (MST) or MEMS devices. The development of suitable measurement facilities to characterise the materials functional properties is complicated by the fact that the film is often attached to a substrate which acts to clamp the film thus affecting the system performance. This paper describes a new differential interferometer system based on common path Jamin optics and measurement lock-in techniques that is able to analyse the displacements of the thin film to 1/10's of pm resolution. The film/substrate acts as a natural bending element and this effect means that the displacement measurements must be carefully controlled to minimise any substrate bending. The system designed at NPL is able to measure the degree of substrate bend so that an 'effective' piezoelectric coefficient may be calculated. Results will be shown for ceramic monolithic materials, quartz single crystal material and sol-gel derived thin and thick films of PZT on Si substrates.

Keywords: piezoelectric, thin films, interferometer, functional

1 INTRODUCTION

Piezoelectric materials can be used as either sensors, where the application of a stress will induce a charge, or as actuators, where an applied voltage induces a strain in the material. The piezoelectric coefficient, d, is a measure of this effect and is thought to be identical for both the direct effect (stress to charge conversion) and the indirect effect (field to strain conversion). The measurement of the indirect effect necessitates the measurements of small displacements, with the maximum piezoelectric coefficient of currently known materials being around 2000pm/V, but many practical materials having far smaller coefficients. Additionally as systems become smaller the measurement method must be non-contact, in order to avoid any mechanical interference between the measurement probe and sample. Laser interferometer based systems are the ideal tool to satisfy this measurement need, and coupled with the knowledge of the illumination wavelength provide a traceable measurement.

The resolution of interferometer systems can be as low as 10^{-15}m $Hz^{-0.5}$, which is close to the shot noise expected in the photodiode, and depends on the filtering that is used to separate the signal form the noise. Zhang [1] have reported a resolution of the order 10^{-13}m, using time constants of the order 100s, at frequencies below 2kHz, whilst more recently Li [2] developed a system capable of 0.1pm resolution using integration times of 10s. Commonly these systems are large, and need highly stable environments with respect to thermal and acoustic perturbations. In this work a modular interferometer system has been developed in collaboration with UK company Interferomet Ltd, where by reducing the optical components, and careful mechanical design, resolutions equal to that previously found are achieved, but in a smaller and more vibration tolerant system. The purpose of this study is to investigate the use of this modified Jamin common path system to measure the piezoelectric coefficients of materials in the bulk and thin film form, to investigate the sensitivity, resolution, and practical aspects of measurements on real systems.

2 INTERFEROMETER SYSTEM

The interferometer system consists of three distinct parts, figure 1, the unstabilised He-Ne laser, a double-pass Jamin type interferometer beam-splitter block, and a reference retro-reflector block. The system is common path between the beamsplitter and the reference retro-reflector, and the measurement beam exits from the centre of the retro-reflector block, with the measurement beam and its return path spaced at 5mm. The system can be used to measure the displacement of a corner cube reflector, or plane mirror attached to a sample, or in this work an achromatic lens with a 25mm focal length was used to focus the measurement point to a single 6mm diameter spot on a sample.

The reference retro-reflector block incorporates a PZT ring actuator that provides external control of the optical path length in the interferometer by controlling the spacing between the measuring and reference retro-reflectors. The use of this is two fold, firstly to scan the retro-reflector through a complete path length to generate a circle on the lissajous figure for ease of alignment. Secondly, through the use of appropriate feedback, the scanner can be used to servo the sine/cosine outputs to maintain the outputs at the $\lambda/4$ path difference position [3]. When the path difference between the reference and sample is equal to $\lambda/4$ then one of the quadrature signals will be zero, and the other at a maximum. Since the signals are sine waves, the displacement sensitivity and linearity of the signal around zero is greatest, whilst the signal at maximum will have very low sensitivity. When the interferometer is held in this condition then for small displacements where the approximation sin(x)=x is valid, the errors are less than 1% for displacements of less than 13nm.

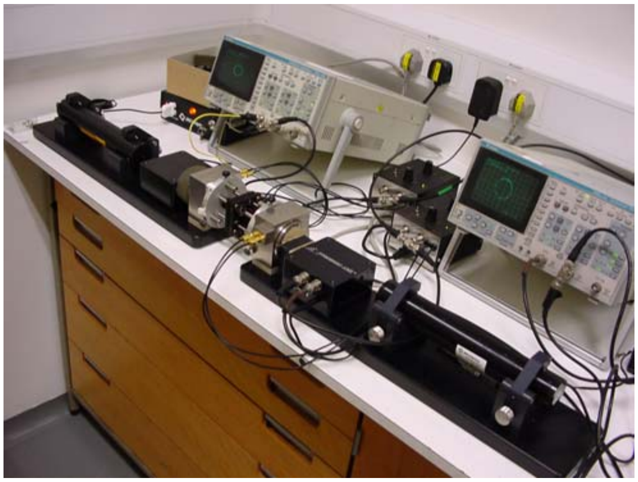

Figure 1: The two interferometer arms set up at NPL for a differential measurement.

2.1 Mechanics/optics

If a corner cube reflector is used on the sample then alignment of the system is simple. However, in the non contact mode, using a lens, the sample must be positioned at the focal plane, and the sample tilt must also be adjusted so that the return path is aligned. To facilitate this the samples were positioned using a standard opto-mechanical 30mm cage assembly. The tilt and fine focus is achieved by a kinematic mount, and the coarse focal position is realised by moving this stage along the rod assembly. The key features of the opto-mechanical path is to keep the path between the sample and the reference retro-reflector as short and as stiff as possible. The system as it was used in this work was mounted on a wooden bench with only four rubber feet as anti vibration mounting. There was no need to provide complex anti-vibration tables or to isolate the system from thermal and acoustic noise, since the operation of the system was at frequencies far above those where they would present a problem. Obviously the lower frequency performance of the system could be improved by adequate isolation from these factors.

When two systems are used back to back to measure differential displacements the alignment becomes more difficult. In theory, if the sample were plane parallel and the two systems collinear then if the sample were aligned on one system, the second system would merely need to be focussed. In practice, the samples are rarely plane parallel and some form of tilt adjustment is needed on the second system. This can be achieved by incorporating an X-Y scan of the lens, as this will effectively create a tilt, at the expense of moving the spot away from the collinear point.

2.2 Calibration

The output from the systems lock-in amplifier is a signal magnitude, V_{RMS} (volts RMS) and phase, and this voltage can be converted to a displacement using the following equation 1.

$$displacement = \frac{\lambda}{2} \cdot \frac{V_{RMS}}{\pi \cdot V_{pp}} \quad (1)$$

where λ is the wavelength of the laser light (632.81nm), and V_{PP} is the diameter of the circle on the lissajous figure. For cost reasons an unstabilised laser was used, which meant that the circle diameter, and hence the displacement output calibration varied over the timescale of the experiments. In order to account for this variation in circle diameter the DC level of the non-zero sine/cosine signal was measured during the experiment and used to adjust the displacement calibration. Ideally the complete circle would be measured during the experiment, however because of the available control system this was not practical. Separate measurement of the circle showed that the DC level is a good measure of the circle diameter, provided that the circle is centred about zero, and the centre does not drift. Measurement of the stability of the circle showed that the diameter varies by up to 20%, but the drift of the centre is negligible.

When the system is used in the non contact mode, with a focussing lens, there is an additional obliquity factor introduced, since the light path travels a greater distance than the actual horizontal displacement. This is obviously dependent on the system, which in this case for a 6mm beam spacing and a 25mm focal lens introduces an additional multiplication factor of 0.9928.

2.3 Sample Fixing

Three types of piezoelectric samples were measured in this work, X-cut quartz of various shapes, bulk PZT ceramic, and thin film PZT on a silicon substrate. The samples need a reflective surface in order to get sufficient contrast to make a measurement. This can be done either through polishing the sample and gold coating, as in the case of the quartz samples, attachment of small mirrors made from pieces of glass cover slip, as for the bulk PZT, or in the case of the thin films, through gold electrodes deposited on the samples. The samples were then fixed on aluminium stubs, using either cyanoacrylate or conducting epoxy in the case of single sided measurements, or silicone adhesive for the double-sided experiments. The premise is that in the single sided measurements there is negligible motion of the back face into the glue, and for the double-sided measurements the silicone adhesive is flexible enough to allow the samples to move freely.

2.4 Sensitivity/Resolution

The resolution of the system is dominated by many noise sources, mechanical, acoustic and electrical, and the system performance is ultimately dominated by the ability of the lock in amplifier in detecting the relevant signal. In order to examine the ultimate performance of the system a quartz sample was measured at 20kHz, with a 1V RMS signal applied, and the displacement signal recorded over time. The measured signal was of the order 25mV, giving a displacement of roughly 2pm. Over the period of the experiment this signal drifted over 0.3pm due to the thermal drift of the unstabilised laser, figure 2. Once the data was corrected to account for the circle diameter change then the response is much more linear, and the standard deviation of the corrected results is 0.012pm. Obviously the system could be improved by the addition of a stabilised laser, however the performance with the unstabilised laser is satisfactory.

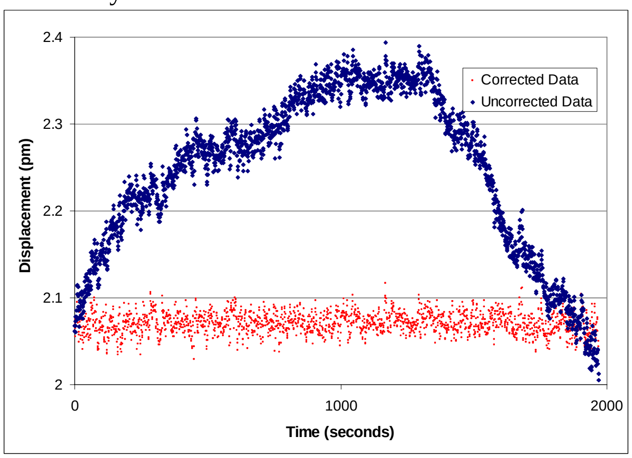

Figure 2: Corrected and uncorrected laser drift measurements taken from quartz

3 RESULTS

3.1 Single sided operation

In order to obtain meaningful values for a piezoelectric coefficient it is necessary to perform two experiments, firstly a frequency sweep to prove that the measurements are performed away from any resonant maxima, and secondly an applied voltage sweep at the desired frequency. Initial experiments on quartz discs gave unreliable results because of the many resonant peaks over the measurement range, and it was not until thicker more cubic shaped samples were used that improved results were obtained. The piezoelectric coefficient of quartz is known to be linear with applied field, and frequency independent, and the resonant frequency of unclamped samples of the size used in these experiments is far above those used in the measurements. However in order to perform the single sided measurements the back face of the sample must be fixed, which can generate unwanted modes and clamp the piezoelectric response.

An amplitude sweep for the quartz cube shows very linear behaviour, and the piezoelectric coefficient at 20kHz is 2.11pm/V, which is within 10% of the accepted value of 2.3pm/V. Several quartz samples were made in order to verify the calibration of the system, and most of these samples gave values between 2.00 and 2.10 pm/V depending on the sample and the position on the sample surface that was measured. The difference between the measured and 'standard' value is thought to be due to sample clamping, and loss of displacement into the backing materials.

3.2 Thin Films

The system was also used to measure the piezoelectric displacement of PZT thin films on silicon substrates. A variety of film thicknesses from 100 to 600nm thick were examined, made by spin coating onto platinised silicon substrates, 0.5mm thick. Top electrodes were sputtered gold, patterned into 2mm diameter circles, centres spaced 4mm apart. Contacts were made to the individual gold pads by wire bonding, and this in turn bonded to strain relief pads, placed well away from the interferometer beam. The gold electrodes were sufficient to produce a good reflected signal, however in order to measure a signal from the rear surface, the silicon substrate was covered with an aluminised Mylar film fixed using cyanoacrylate.

The films will behave as unimorphs, causing the substrate to bend, so in order to determine the piezoelectric coefficient of the film, the change in thickness of the film must be measured. This means, either the substrate must be held rigidly, or truly measure the change in thickness using a dual beam system. Initially small squares of film, roughly 1cm square were bonded to an aluminium stub using cyanoacrylate. This does not provide ideal clamping, but was tried for later comparisons. This sample gave a flat frequency response, with a large peak around 78kHz, however the response at 20kHz gave an effective d_{33} of about 11pm/V, (figure 3). The response of thin films is obviously limited by the clamping of the silicon substrate, however the 11pm/V value is an overestimate, as this includes any bending motion allowed by the imperfect clamping of the adhesive. In comparison a similar sample was fixed to a similar aluminium stub, this time with a 6mm diameter through hole, with the adhesive bonding the edges. The frequency response is similar to the fixed back surface sample, although the main resonant peak is somewhat lower, however the displacement output is an order of magnitude higher, giving a d_{33} at 20kHz of 182pm/V.

In the preceding test the active part of the film is a 2mm diameter circle under the activated electrode, whilst the rest is inactive. The active electrode was positioned, by eye, so that it was in the centre of the 6mm hole in the aluminium holder. As discussed in this form the film will bend, and the displacement of the film will depend on the X-Y position of the beam. The system was modified to include a manual X-Y lens translating stage with +-1mm travel and 0.25mm/revolution screw adjusters, to enable the beam to scan across the electrode. The beam was positioned close to the edge of the electrode, and measurements taken every 0.125mm as the beam was moved to the centre of the electrode. Figure 4 shows the scan across the free 500nm film, plotting the effective d_{33} at 20kHz, which is the value including the bending of the substrate. It can be seen that the displacement reaches a small plateau around a 0.5mm diameter in the centre, but drops away rapidly outside of this zone.

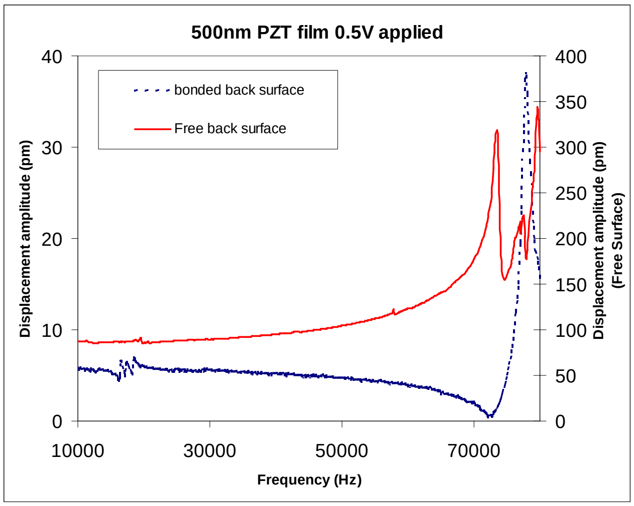

Figure 3: Frequency response from PZT thin film showing effect of free verses fixed back face

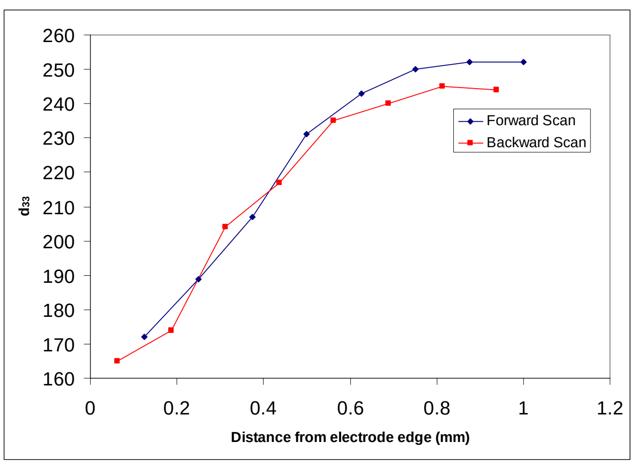

Figure 4: Measurement scan across the face of a piezoelectric thin film.

3.3 Double sided operation

In order to use the double-sided operation to determine the change in thickness of a sample it is useful to consider two cases:

1. A hard PZT rod, 10mm long, 3mm diameter, held in the centre using silicone rubber adhesive, was measured using the double sided system measured at 20kHz. The results are very linear, and the displacement outputs for the front and the back face are very similar with effective piezoelectric coefficients of 134pm/V and 126pm/V. As both signals were in phase, the piezoelectric coefficients for the two sides must be *added* to yield a true piezoelectric coefficient of 260pm/V, which compares favourably with a value of 263pC/N measured for this sample using the Berlincourt method.

2. To test the response of bending type actuators the 500nm thin film with the backface free was measured. The signals were 180º out of phase, and so the two signals should be *subtracted* from each other, with the active face amplitude being the larger of the two. Unfortunately, the amplitude for the front face was 183pm/V whilst the signal from the rear face was higher at 259pm/V, giving rise to a negative piezoelectric coefficient.

This experiment was carried out before the position scan of the thin film, which showed the importance of the probe beam position on the sample. Because of the difficulty in system alignment beam on the front face was not positioned in the centre of the electrode, and because it was difficult to see the beam on the rear face its position was unknown. However it appears from the results that the beams were not collinear. In order to produce sensible results additional alignment adjustments are needed in order that the beams are collinear and aligned with respect to focus and tilt, such that the natural bending of the film/substrate can be accurately measured [4].

4 CONCLUSIONS

A Jamin-optics common path interferometer system has been developed that enables sub pm measurements of the displacement of piezoelectric thin films. The system has been designed to be compact and modular that can operate without an expensive stabilised laser source, and works in environments with minimal thermal and vibration isolation.

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