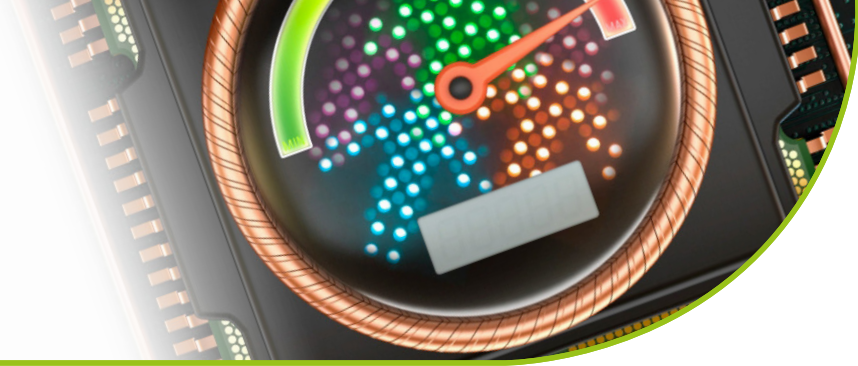




Nanostrain

Providing highly accurate measurement of strain in nanoscale systems to drive innovation



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Welcome

Welcome to the fifth edition of the quarterly EMRP Nanostrain project newsletter. As the project passes its halfway mark, this newsletter will continue to provide you with everything you need to know on the latest project developments, as well as interviews with global industry leaders and a summary of upcoming global events and academic conferences on the exciting technological area of nanoscale piezoelectrics.

Project news

Removing the bend from piezoelectric thin film measurements

Accurately measuring the piezoelectric coefficient of thin films has long been a challenging element of the characterisation process. This is in part owing to the tendency of the supporting substrate to bend, which has on occasions led to vastly different measurements being recorded for essentially similar materials. Intuitively it seems difficult to believe a 1 micrometre thick film will make a 500 micrometre thick silicon wafer bend significantly. The term “significantly” is important here. The bend might be less than a tenth of a micron, but the change in thickness of the piezoelectric film itself will only be a few hundreds of pm.

A solution put forward by Kholkin et al. in 1996 was to use double-beam interferometry where a collinear beam focussing on the top and bottom faces of a sample simultaneously measures the change in thickness, eliminating the bend.

In theory this should work, but the story is complicated by the fact that most researchers want to use small area top electrodes on the sample rather than completely covering it. The reason is twofold, firstly a smaller area will be less likely to short circuit, and secondly so they can measure different regions of

the sample. The effect of reducing top electrode size has been observed to decrease the measured output, but the mechanism has not been understood.

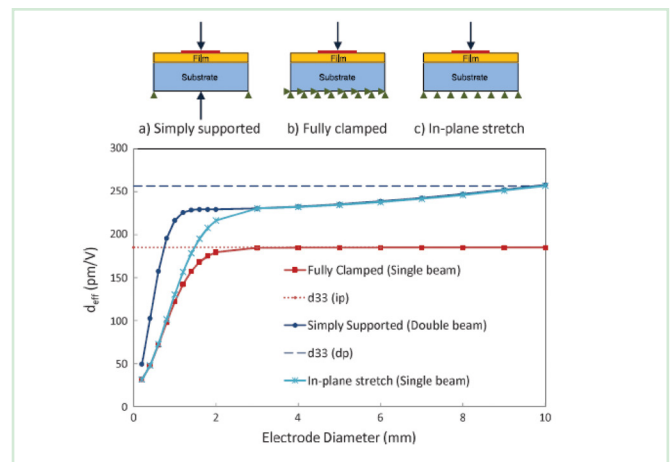


Figure 1

FEA simulations of change in sample thickness along with schematics of the representations of the simply supported (a), fully clamped (b), and in-plane stretch case (c).

In an attempt to investigate the nature of this electrode size effect, members of the Nanostrain community including Mark Stewart, Serban Lepadatu, Neil McCartney, Markys Cain, Jason Crain,

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Dennis Newns and Glenn Martyna conducted FEA simulations of both single and double-beam measurement setups. In results recently reported in APL Materials, the double beam experiment was simulated with a simply supported sample allowing for bending to occur, whilst for the single beam sample, the substrate was fixed at the base both laterally and vertically, Figure 1. In both cases, the electrodes were assumed to be infinitesimally thin, and a voltage of 1V was applied to different sized circular electrodes on a 10mm diameter sample.

For the fully clamped case, the deformation was shown to decrease as the area of the electrode was reduced, with a sharp drop in displacement noticed for electrode diameters below 2mm. The implication is that the measurement of intrinsic piezoelectric response can only be achieved with electrodes greater than 2 mm diameter for a fully clamped sample. In practice as was shown by Kholkin, it is virtually impossible to fully clamp a sample, particularly as the electrode size increases.

A similar situation was observed for the simply supported sample, although here the reduction in thickness change begins as soon as the electrode diameter is reduced, and there is no electrode size independent region.

What this means is that it is almost impossible to measure the piezoelectric coefficient using these methods as the result depends on the size of the top electrode. Whilst investigating these size effects in the simulations the Nanostrain team observed that although the displacement at the centre of the sample always varied, there was a step in displacement at the edge of the electrode that never varied. The significance here is that we can now measure this step height on the top surface, and it doesn't matter that the sample bends, because it is effectively a differential measurement.

In order to put the step height model to the test, an experimental study was carried out using a 1µm thick PZT film deposited on a 500µm thick platinized silicon substrate. Cr/Au electrodes of diameters ranging from 0.1 mm to 1 mm were then evaporated onto 10 mm square samples. Although displacement at the centre of the electrode was shown to vary, the step height at the electrode's outer edge was almost constant as predicted. Figure 2 shows the measured step height for a fully clamped and simply supported sample, for a range of electrode diameters from 0.1 to 1 mm. The step height is relatively constant for all electrode sizes, whereas the centre displacement doubles for the fixed sample, and increases more than fivefold for

the simply supported sample over the same range. The step height for the larger diameters on the simply supported sample decreases slightly.

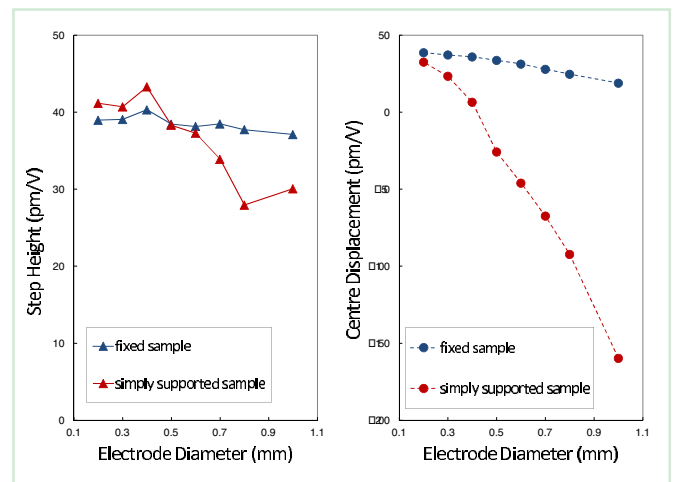


Figure 2

Measured step height (left) and centre displacement (right) at the electrode centre for 1 Vrms applied for the fully clamped and simply supported cases as a function of electrode area.

The fact that the step height is constant is important as far as a consistent measurement is concerned, but what is it that is actually measured? The APL Materials paper also describes an analytical expression for the step height that includes the piezoelectric coefficients and also the elastic properties of the film and the substrate. So the good news is we have a repeatable measurement technique, the not so good news is there is still more work to be done to separate the piezoelectric and elastic properties.

Nanostrain events

Summary of Piezo2015

The Electroceramics for End-users VIII conference held in Maribor (Slovenia) brought together industrial and academic experts in piezoelectric and multi-functional materials from around the world to discuss the latest advances in the development of novel technologies and devices research as well as materials fabrication, measurements and new characterisation techniques. Due to the high participation of representatives from both industry (approximately 30 attendees from 16 companies) and academia (around 60 individuals in attendance), the conference represented an exceptional opportunity for discussions, networking and establishment of new collaborations. During the four days of the conference, several tutorials were also given spanning piezoceramics processing and properties as well as x-rays/neutron scattering techniques for structural characterisation.

The EMRP Nanostrain project was extremely well represented at the conference by three talks and three posters. Mark Stewart, in the first Nanostrain talk, described a new method to measure the clamped indirect piezoelectric effect of thin piezoelectric films; Carlo Vecchini spoke about the new in-situ dynamic electrical and structural measurements on PMN-PT single crystals and epitaxial thin films developed at the XMaS beamline at the ESRF (France); and finally Paul Weaver, on behalf of Anna Kimmel, presented the results on the theoretical work on properties of $\text{Pt/Pb}(\text{Zr}_{0.5}\text{Ti}_{0.5})\text{O}_3$ interfaces.

Oliver Gindele and Jacob Chapman from NPL also presented posters entitled: 'New incommensurate low energy phase of KNbO_3 : ab initio structure searching technique'; 'Development of a Shell Model Force for BaTiO_3 and its Application for Domain Walls and Surfaces'; 'Developing of semi-empirical force field for $\text{Pb}(\text{ZrTi})\text{O}_3$ and its application for the description of mechanism of domain walls motion', covering work performed together with Anna Kimmel.

article contributed by Carlo Vecchini

Nanostrain workshop scheduled for APS Meeting in May

Later this year, the Advanced Photon Source and Centre for Nanoscale Materials will hold its 2015 Users Meeting at the Argonne National Laboratory near Chicago.

The four-day conference will span from May 11th to 15th, and will include a workshop organised jointly by the Advanced Photon Source (APS), NPL and XMaS, featuring contributions from three Nanostrain project members.

Entitled 'Developing Synchrotron Sample Environments to Study Next Generation Field-Driven Device Physics', the full-day session will be held on Tuesday May 12th to bring together experts with a strong emphasis on emerging device physics as well as more fundamental strongly correlated phenomena. The resulting discussions will help to guide the APS to envision cutting-edge sample environments and supplemental in-situ (X-ray) measurements that will augment experimental data and enrich understanding of the underlying physics.

Plans for the workshop are still being finalised, and further details will be made available on the APS website at <https://usersmeeting2015.conference.anl.gov/Program/WK3>.

Paul Thompson, a Nanostrain project partner based at XMaS and one of the workshop's co-organisers, said, "We are employing state of the art synchrotron resources to develop a deeper understanding of how emerging piezo driven devices function. In addition, we hope to showcase how synchrotron radiation is an indispensable tool to characterize this new generation of devices."

Upcoming events

IEEE 35th International Conference on Electronics and Nanotechnology

Kyiv, Ukraine

April 21-24 2015

NanoOstrava 2015 - Nanomaterials and Nanotechnology Meeting

Ostrava-Poruba, Czech Republic

May 18-21 2015

International Conference on Nanotechnology, Nanomaterials & Thin Films for Energy Applications

Manchester, United Kingdom

June 1-3 2015

EuroNanoForum 2015

Riga, Latvia

June 10-12 2015

TechConnect Nanotech 2015

Washington D.C

June 14-17 2015

ICNFA 2015

Barcelona, Spain

July 15-17

IEEE Nano 2015 – International Conference on Nanotechnology

Rome, Italy

July 27-30



Big Interview:

Sandy Cochran - Principal Investigator on the UK EPSRC Sonopill programme

Sandy Cochran is Professor of Biophysical Science and Engineering at the University of Dundee, and Principal Investigator on the UK EPSRC Sonopill programme, a Nanostrain project partner. Sonopill's objective is to develop a capsule capable of providing ultrasound endoscopy of the gastrointestinal tract, through the use of miniature piezoelectric transducers.

What is the SONOPILL project?

Sonopill is primarily a collaboration between Glasgow, Heriot-Watt and Dundee universities with the support of a number of other institutions worldwide and 15 partner companies. In the near future we hope to develop an ingestible pill capable of performing an ultrasound scan of the gut wall throughout its entire length at higher resolutions than traditional endoscopy allows, and with a much reduced negative impact on the patient.

What would be the medical benefits?

As opposed to high-cost, equipment-intensive scanning techniques such as MRI, CT and PET, ultrasound is safe, inexpensive and directly available at the point of care. The Sonopill itself will make use of microultrasound techniques, providing high-resolution images of tissue within a centimetre's depth of the gut wall.

Visualisation at this depth will facilitate the diagnosis of a variety of different conditions, including inflammatory bowel diseases such as Crohn's disease, as well as Barrett's oesophagus which can be a precursor to cancer. It will also considerably simplify diagnoses of coeliac disease, an increasingly common condition which currently requires invasive techniques to identify.

The ultimate challenge, and an extremely important clinical target, is early diagnosis of colon cancer; this poses a variety of unique challenges but if these can be overcome, microultrasound endoscopy may be able to assist.



How can the Nanostrain project facilitate this work?

Capsules for oral ingestion need to be about 10 mm in diameter and no more than 30 mm in length. In order to comfortably fit within these parameters, we are aiming to produce a cylindrical Sonopill 10 mm in diameter – an undertaking that calls for considerable technological expertise.

If we are to build an effective ultrasound capsule, we need to have highly efficient small-scale piezoelectric systems within it that can both transmit and receive ultrasound.

We envisage radial imaging taking place outward from the pill's circumference, involving separate transmitters and receivers composed of smaller array elements. Whereas the transmitter will consist of no more than 8 such elements, our receiver will rely on a much larger number – somewhere in the order of 512.

One of the physical hurdles that needs to be overcome is how to build piezoelectric systems on a scale to allow 512 elements to be fit within the circumference of a circle with a 10 mm diameter. In such circumstances, piezoelectric behaviour at the nanoscale becomes important so we are particularly keen to see the progress made by the Nanostrain project on understanding the properties of nanoscale piezoelectrics in more detail.

What is your envisaged timescale for the Sonopill project?

The project has now been running for 18 months, during which time we have been able to manufacture very early prototypes with connected tethers. Key components of these tethered capsules are currently being tested in vitro, with the aim to further improve performance and autonomy.

Technically advanced clinical devices such as the Sonopill take a long time to develop, and even longer to obtain the necessary regulatory approval.

With a fair wind, we would hope to see prototypes being trialled clinically within 5 - 10 years, but we are also working on a highly simplified prototype which could be in trials by 2017. This simplified system will still involve a tethered capsule, and rather than the 512 piezoelectric elements would involve a single mechanically rotated combining transmission and reception.

In both the high element number arrays and the simplified system, we are working towards the use of new piezoelectric crystal materials made into composites with polymers. These are based on substantial advances in miniaturisation, with features as small as 5 microns in diameter.



Sonopill's Pathfinder Pill

What are your hopes from the Nanostrain project?

As with all our collaborations, working with Nanostrain over the past year has been extremely valuable. Having worked with piezoelectric devices for thirty years now, I find the Nanostrain project tremendously exciting, heading very much in the right direction - downwards. As Richard Feynman said, there is still plenty of room at the bottom – in other words, there is no shortage of potential for innovation on small scales. It has been very exciting to see developments come out of Nanostrain and to look forward to linking them with the Sonopill project as it progresses.



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