

Exploring functional material behaviour with voltage modulated atomic force microscopy modes

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Understanding functional properties of materials is key to a variety of applications in materials science and engineering. Voltage modulated atomic force microscopy modes have enabled insight into functional behaviour and interface phenomena including electromechanical coupling, electrochemical processes and charge distribution, which can be correlated to structural information obtained from topography. In this article, an overview of voltage modulated techniques is provided and their operational principles and capabilities are discussed.

Introduction

The micro- and nanoscale functional behaviour of materials underpins many technological and scientific advances in the fields of electronic and optical engineering, energy harvesting and storage, micro- and nano- electromechanical systems as well as biomedical science. Designing devices that exploit functional materials necessitates profound knowledge on the response to stimuli in the respective operational environment that can range from ultrahigh vacuum and air or other gases to liquids and can comprise a wide span of temperatures and relative humidities. Atomic force microscopy (AFM) is a tool to explore a plethora of material properties at nanoscale resolution in a variety of environmental conditions and has therefore become increasingly important in materials science. In AFM, a sharp tip (~ tens of nm diameter) situated at the end of a cantilever is scanned across a sample surface (Binnig & Quate, 1986). The tip-sample interaction is monitored by reflecting a laser beam from the top coating of the cantilever into a position sensitive photodetector. In order to keep the interaction constant, the vertical

tip-sample distance is adjusted by a feedback loop, thus tracking the sample surface. Dependent on the imaging mode, the cantilever deflection (contact or constant force mode), amplitude damping of an oscillating cantilever (tapping, intermittent contact or amplitude modulation mode) or a shift in cantilever resonance frequency (frequency modulation mode) can be used as feedback signals. Apart from detecting topographic features with up to atomic resolution, AFM is capable of measuring mechanical and functional material properties as also highlighted in recent infocus articles on hybrid photonic-mechanical force microscopy (Passian, Farahi, & Davison, 2015), mechanical measurements (Gunning & Grant, 2016) and scanning microwave microscopy (Kienberger, 2016). This article aims to provide an overview of voltage modulated AFM techniques and to explain how electromechanical coupling, electrochemical phenomena and electrostatic interactions can be probed using these techniques. In addition, recent advances by acquisition of the full cantilever response spectrum and use of data mining algorithms are discussed.

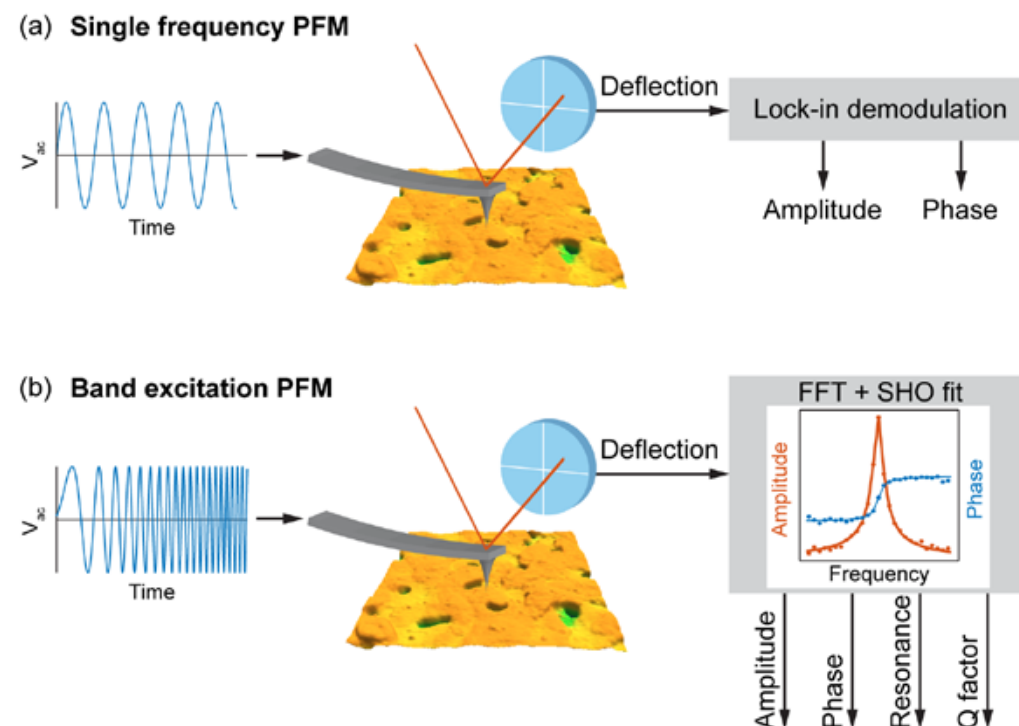


Figure 1. Schematic illustration of (a) single frequency PFM using lock-in demodulation and (b) BE PFM where fast Fourier transformation (FFT) and simple harmonic oscillator (SHO) fitting is applied.

Electromechanical and electrochemical coupling

Many functional material properties can be inferred from periodic cantilever oscillations that arise from interactions between the sample and an alternating current (ac) electric field that is applied via a conductive AFM tip. If the tip is in contact with the sample, the applied excitation voltage can induce periodic sample deformation originating from electrochemical strain or linear electromechanical coupling through the converse piezoelectric effect in some materials (Balke, Bdiin, Kalinin, & Kholkin, 2009). These oscillations are monitored by the photodetector and typically demodulated at excitation or intermodulation frequencies using lock-in techniques (Figure 1 (a)) or fast Fourier transformation and simple harmonic oscillator fitting (Figure 1 (b)) to infer functional material properties. In piezoresponse force microscopy (PFM) (Kalinin & Gruverman, 2010), piezoelectric activity is commonly represented as amplitude and phase signals,

which indicate magnitude of piezoresponse and polarisation orientation, respectively. Alternatively, Cartesian coordinates can be used that combine the available information as mixed PFM signal. Since the material dependent piezoelectric tensor is of third order, the obtained piezoresponse is highly orientation dependent. Vertical or out-of-plane piezoresponse can be detected by demodulating the vertical movement of the tip, whereas in lateral or in-plane PFM shear electromechanical coupling is inferred from tip twisting. Provided appropriate calibration, reconstruction of the complete piezoelectric response vector can be obtained from one vertical and two lateral PFM scans taken after sample rotation by 90° . However, the electric field distribution around the tip is highly non-uniform and is determined by several factors including tip-sample contact, tip geometry, sample material and ambient humidity. Furthermore, cantilever buckling due to in-plane shear response along the long cantilever axis, can falsely be interpreted

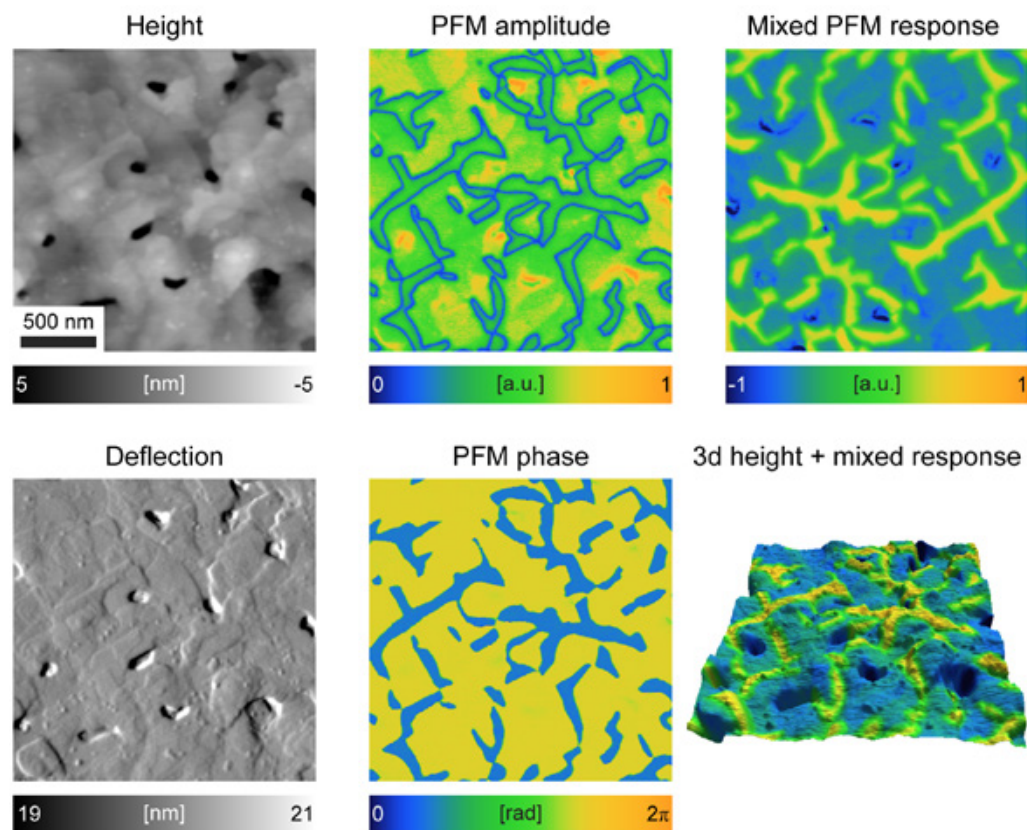


Figure 2. AFM topography and vertical single frequency PFM amplitude, phase and mixed PFM response images of a bismuth ferrite thin film. The image on the bottom right is a 3-dimensional representation of the height image combined with mixed PFM response as colour data.

as out-of-plane signal. PFM can be used to study a variety of materials, as an example vertical PFM amplitude, phase and mixed response measured on ferroelectric bismuth ferrite are shown in Figure 2 where the presence of domains of homogenous polarisation can be inferred from contrast in the phase image accompanied by a decrease in amplitude at domain walls. In addition to classical ferroelectrics, also electromechanical coupling in biomaterials can be probed which might be relevant to biofunctionality and tissue engineering. Shear piezoelectricity of collagen fibrils measured in lateral PFM mode is depicted in Figure 3 where contrast in the phase image relates to the direction of polar bonds in collagen molecules from amine to carboxyl termini (Denning et al., 2012). In general, the piezoresponse amplitude strongly depends on the excitation frequency and follows a simple harmonic oscillator model for contact resonances with small

damping. Therefore, high signal-to-noise ratio can be obtained by operating at or near resonance. However, on-resonance PFM yields high risk for artefacts since the resonance frequency can vary even within one scan as it is subject to numerous parameters, including tip wear, tip geometry, elastic properties of the sample and topography. A shift in resonance frequency can artificially enhance or decrease the measured response or lead to contrast in the phase image without a physical change of polarisation orientation. To overcome these challenges, modes have been implemented that track the resonance frequency and adjust the excitation frequency accordingly using an additional feedback loop (Rodriguez, Callahan, Kalinin, & Proksch, 2007). Another approach is to conduct measurements across a range of continuous frequencies within a band centred on resonance, which is known as band excitation PFM (Figure 1 (b)) (Jesse et al., 2014). Fitting the measured piezoresponse as a

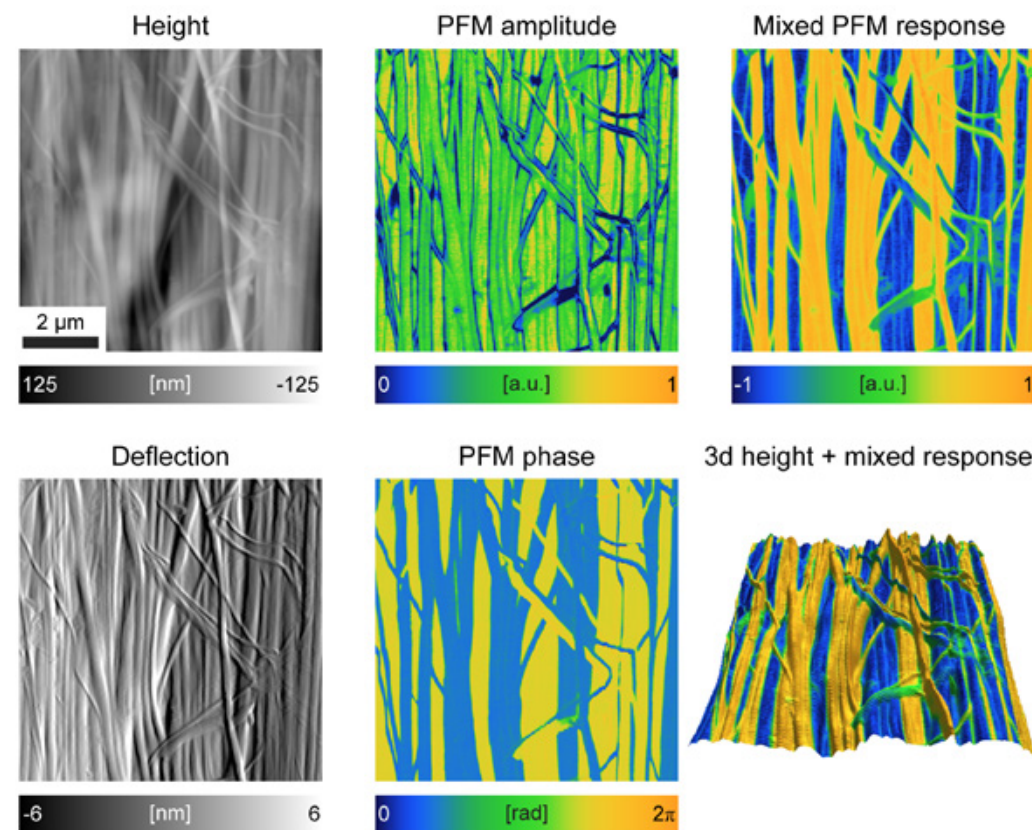


Figure 3. AFM topography and lateral single frequency PFM amplitude, phase and mixed PFM response images of collagen fibrils. The image on the bottom right is a 3-dimensional representation of the height image combined with mixed PFM response as colour data.

function of frequency to a simple harmonic oscillator model allows to extract piezoresponse amplitude and phase but also resonance frequency and quality factor. In addition to preventing PFM resonance artefacts, it is therefore possible to obtain information related to mechanical properties of the sample that manifest themselves in resonance frequency and quality factor due to changes in tip - sample contact stiffness.

In ferroelectric materials, electromechanical coupling can not only be observed but also modified upon application of direct current (dc) voltages. Micro- and nanoscale domain patterns of alternating polarisation orientation can be obtained. Such domains and domain patterns find applications in ferroelectric memory components, domain wall electronics, logic gates and optical devices. The compatibility of AFM with external stimuli like UV light and various ambient conditions including temperature and humidity provides the

opportunity to study the influence of different conditions at the sample surface, which can have significant impact on functional behaviour. Figure 4 (a) shows an example for the effect of relative humidity on size and sample thickness dependence of domains that were switched in Mg doped lithium niobate by the same set of dc pulses (Neumayer et al., 2015). Figure 4 (b) highlights the arbitrary shapes of domains that can be obtained in ferroelectric lithography demonstrated by a domain pattern 3D representation of the University College Dublin logo that was obtained by applying a sequence of positive and negative dc voltage pulses while scanning the surface of a lead zirconate titanate thin film. Precise domain engineering necessitates high resolution ferroelectric characterisation, which can be achieved using spectroscopy techniques based on the application of dc pulses that induce polarisation switching while the electromechanical response is

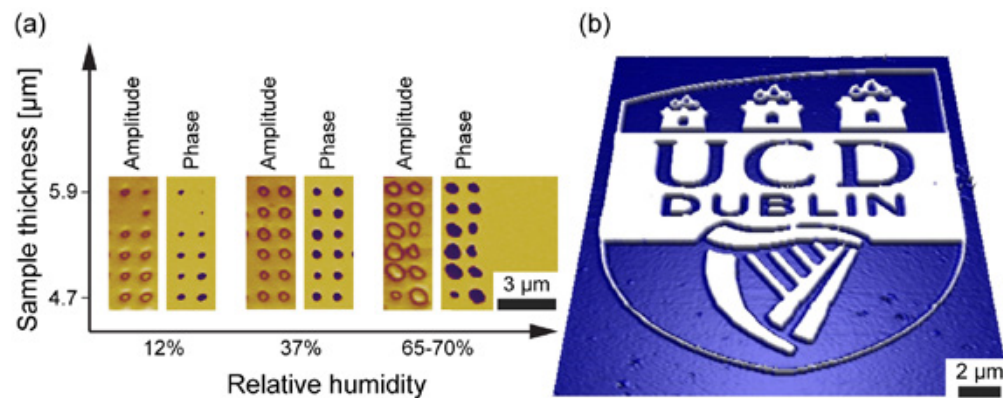


Figure 4. (a) domain patterns switched in initially positively polarised Mg doped lithium niobate using the same waveform at different ambient humidity (colour scales: 0 - 1 [a.u.] PFM amplitude, 0 - π [rad] PFM phase) (adapted from Neumayer et al., 2015 with permission of AIP publishing). (b) 3d representation of polarisation lithography on lead zirconate titanate (colour scale: 0 - π [rad] PFM phase).

monitored during and between these pulses (see Figure 5 (a)). As in continuous PFM scans, switching spectroscopy can be conducted using single frequency excitation on and off resonance as well as band excitation modes. Bipolar triangle envelopes of dc pulses allow to obtain local hysteresis loops (Figure 5 (b)) at each grid point of the sample, which provide fingerprint-like characteristic information on functional behaviour that can be depicted in maps. Different waveform envelopes can be applied

to study local ferroelectric switching dynamics. Examples for BE piezoresponse spectra measured during application of a first-order reversal curve dc pulse waveform are shown in Figure 6. Amplitude and phase can be extracted and fitted at each step and for each pixel and composited into maps. In Figure 7, maps of piezoresponse amplitude, phase, mixed piezoresponse, resonance frequency and quality factor are depicted that were acquired using BE switching spectroscopy. In the shown first-order reversal curve experiment, the applied dc voltage waveform is triangular with progressively increasing amplitude. Unlike in macroscopic poling techniques, single domains are nucleated in PFM spectroscopy modes and the influence of grain boundaries, defects and sample composition in heterogeneous materials can be studied at high spatial resolution. Adding a current amplifier to the electric circuit allows to obtain information on conductivity, which is often polarization dependent and governed by band bending at sample-electrode interfaces.

Techniques based on PFM can also be applied to investigate non-piezoelectric samples where field induced sample deformation originates from electrochemical strain upon field dependent ionic motion. Electrochemical strain microscopy has been used to investigate ion transport in solid electrolytes, battery materials and glasses (Balke et al., 2010; Proksch, 2014). Apart from electromechanical and electrochemical strain, also electrostatic forces

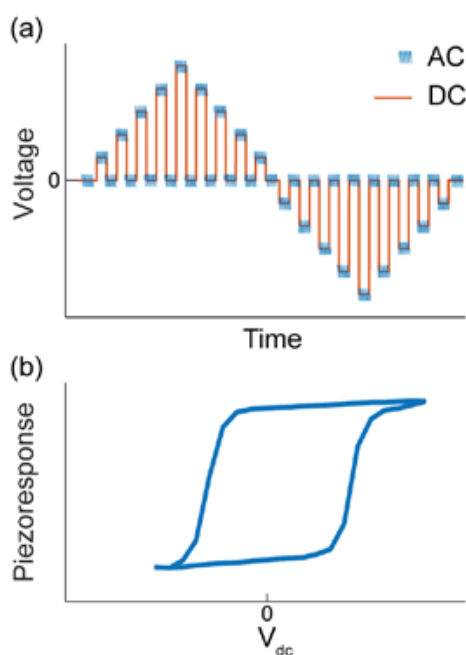


Figure 5. Schematic depiction of examples for (a) a voltage waveform applied in switching spectroscopy experiments and (b) an obtained piezoresponse hysteresis loops.

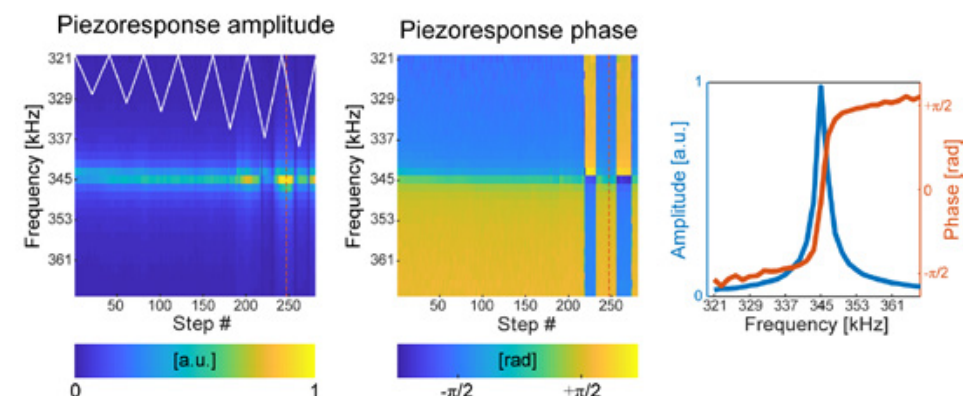


Figure 6. Piezoresponse amplitude and phase spectra extracted from a single pixel during BE first-order-reversal curve spectroscopy on bismuth ferrite. The dc waveform envelope is depicted in white in the amplitude image. Diagram to the right shows amplitude and phase extracted from the spectrograms at step# 247 (as indicated by dashed line).

might act on the tip and contribute to the obtained response. Electrostatic interactions in contact mode can be particularly strong in switching experiments during which dc voltages can cause charge injection. Dependent on material and experimental settings, electrostatic forces might even dominate the measured response. However, electrostatic contributions can be identified by their parabolic ac voltage dependence as opposed to linear piezoelectric behaviour and reduced by using cantilevers of higher force constants.

Electrostatic interactions

While electrostatic interactions can obfuscate genuine electromechanical coupling in PFM experiments, long-range electrostatic forces acting on a tip that is not in contact with the sample surface yield information on local charge density distribution and dielectric properties of the sample and are therefore utilised in electrostatic imaging modes (Kalinin & Gruverman, 2010). As in PFM, electrostatic force microscopy (EFM) is based on extracting the first harmonic component of the dynamic response from the cantilever deflection signal using lock-in demodulation techniques. Qualitative mapping of electrostatic interactions demodulated into amplitude and phase images is typically performed in a two-pass method where the first pass tracks sample topography whereas in the second pass the same line is scanned with the

tip positioned at a set distance of ~tens to hundreds of nm above the surface. However, EFM can also be conducted in a single pass if the electric excitation frequency is different from the tapping mode drive frequency for mechanical excitation. Although EFM measurements of the second harmonic response in conjunction with finite element modelling has been successfully applied to obtain dielectric constants (Fumagalli, Esteban-Ferrer, Cuervo, Carrascosa, & Gomila, 2012), quantitative conclusions are widely impeded due to an unknown capacitance gradient that governs electrostatic forces. Therefore, EFM was further developed into more quantitative techniques like electrostatic force spectroscopy and Kelvin probe force microscopy (KPFM). These modes are based on minimising electrostatic interactions upon application of a dc voltage that corresponds to the contact potential difference (CPD) between tip and sample, as illustrated in Figure 8. Dependent on the studied material and experiment, the CPD can be related to work function, surface potential, surface charge as well as electronic and ionic transport (Collins, Belianinov, Somnath, Rodriguez, et al., 2016). In electrostatic force spectroscopy, a dc voltage sweep is performed at each point of the grid while measuring the electrostatic response, which allows the user to obtain the local CPD from the minimum of the curve. Unlike in force spectroscopy ("open loop"), in KPFM the dc voltage required for nulling the EFM amplitude is automatically adjusted

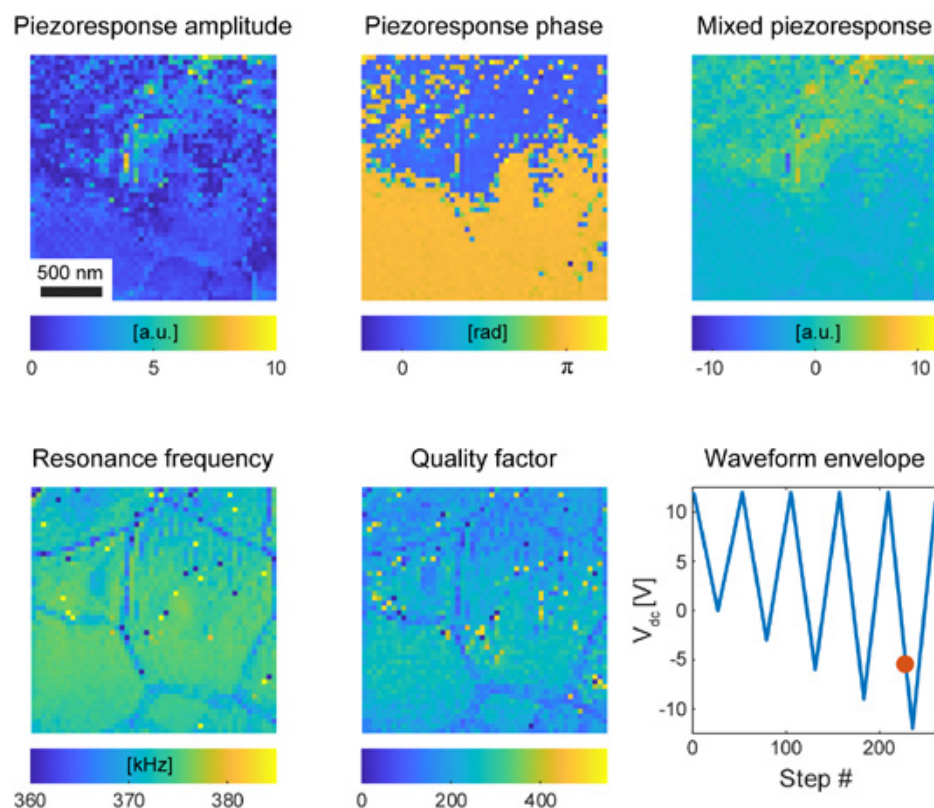


Figure 7. Example of BE first-order-reversal curve spectroscopy maps obtained on a lead zirconate titanate thin film by applying dc voltage pulses of an amplitude as depicted in the waveform envelope at each point of the 50×50 grid. The shown step is marked by the orange circle in the waveform envelope graph.

by a feedback loop (“closed loop”) during scanning and yields the CPD. Quantitative electrostatic force microscopy and KPFM measurements can be obtained by calibrating the system with a sample of known work function, which improves comparability of measured CPD values. However, changes in electrostatic forces due to tip wear, ambient conditions and experimental parameters have to be taken into account. In KPFM, the dc voltage feedback loop is an additional source for systematic errors and topographic crosstalk, which led to the development of dual harmonic KPFM (DH-KPFM) and intermodulation EFM (Borgani et al., 2014; Collins et al., 2013). In DH-KPFM, EFM amplitudes at first and second harmonics of the excitation voltage are recorded. Upon taking into account phase offset and cantilever transfer functions, the CPD can be obtained as the ratio between first and second harmonic responses, where the latter is subject

to capacitance gradients, thus yielding information on topography and dielectric properties of the sample (Collins, Belianinov, Somnath, Rodriguez, et al., 2016). Intermodulation EFM exploits frequency mixing of electrical and mechanical excitation to infer the CPD from the ratio of force components

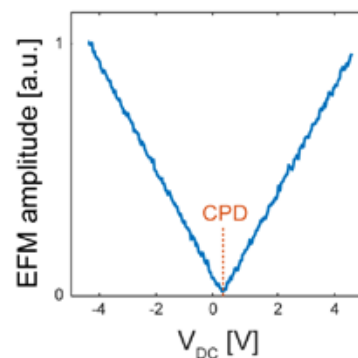


Figure 8. EFM amplitude recorded during dc voltage sweep 50 nm above a bismuth ferrite surface.

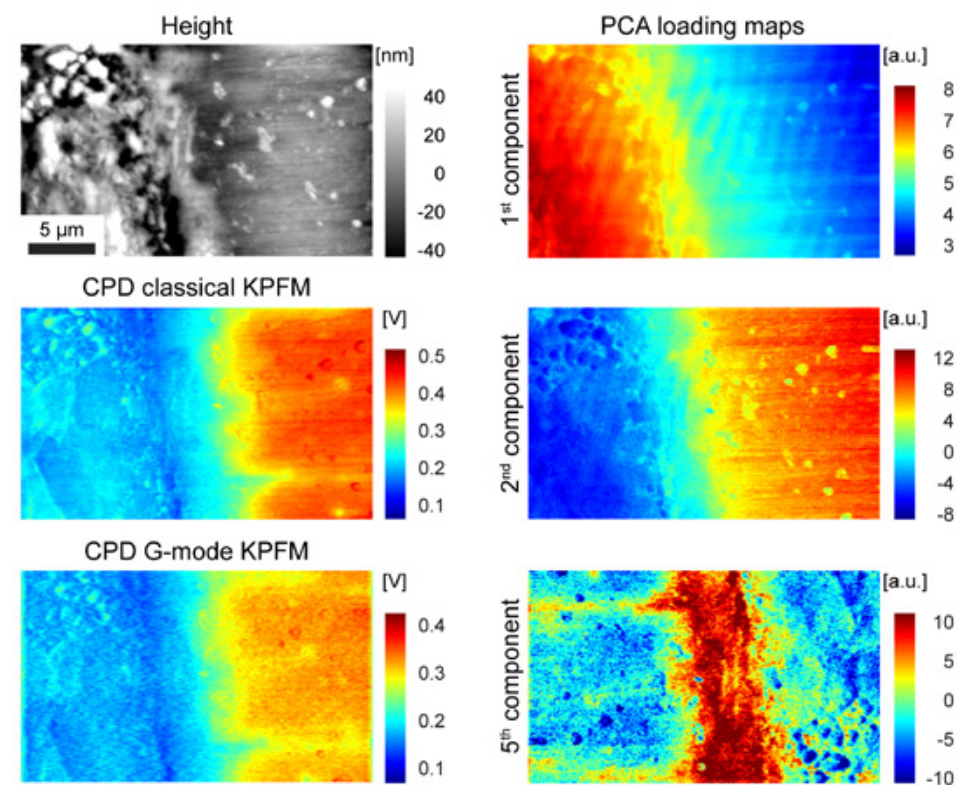


Figure 9. Height, CPD obtained using classical KPFM and G-mode KPFM, PCA loading maps of a Schottky diode (adapted from Collins, Belianinov, Somnath, Balke, et al., 2016).

at intermodulation frequencies in single pass measurements. The absence of dc voltage in these techniques has several advantages, including prevention of feedback artefacts and elimination of feedback loop bandwidth limitations in terms of temporal resolution, which facilitates time resolved studies of electrostatic interactions. Furthermore, DH-KPFM provides a path for measurements in liquids that contain mobile ions, which are crucial to elucidate local electrochemical effects that occur during corrosion, energy storage or biological processes. DH-KPFM has therefore been implemented to study charge dynamics and electrochemical phenomena at the solid - liquid interface. In a related, multidimensional spectroscopic approach called electrochemical force microscopy, electrochemical processes are activated by applying dc voltage pulses upon monitoring charge transport phenomena during

and between these pulses by recording first and second harmonic responses (Collins et al., 2014). As in PFM, excitation signals in EFM and KPFM can be single frequency on or off resonance or multi-frequency with implications for signal to noise ratio and artefacts.

As discussed previously, electrostatic forces are also present when the tip is in contact with the sample, which can lead to artefacts in PFM measurements. KPFM conducted in contact mode allows to differentiate electromechanical and electrostatic phenomena and provides higher lateral resolution than non-contact mode KPFM (Balke et al., 2014).

General acquisition mode

Classical single frequency PFM and EFM modes rely on lock-in based techniques that demodulate the cantilever deflection at a certain frequency and temporally average the measured signal over a time

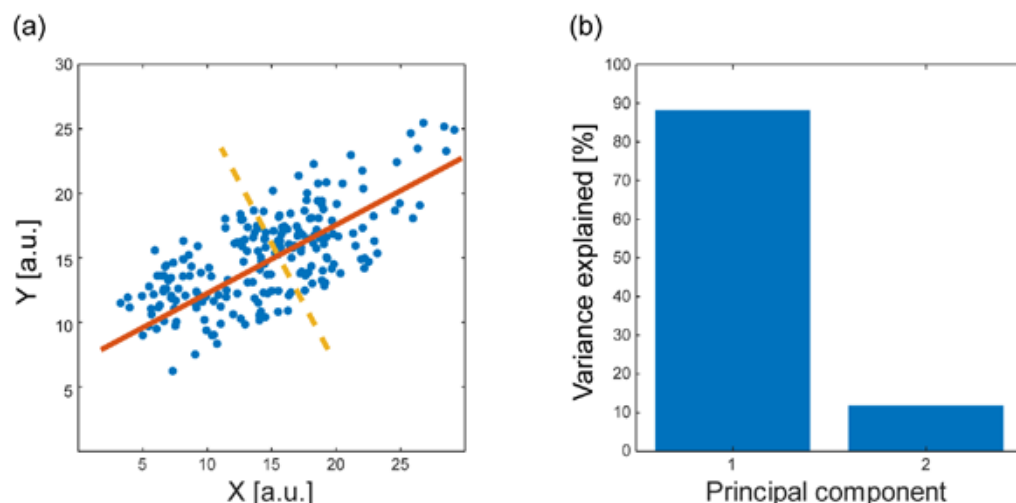


Figure 10. Simple example for PCA: (a) blue dots represent raw data, orange solid line indicates direction of 1st principal component, yellow dotted line direction of 2nd principal component, (b) variance explained by each component.

constant of typically hundreds of μs to several ms. Information at different frequencies or fast time scales is therefore lost. Due to the nonlinear nature of tip-sample interactions and their distribution across several frequency bands that can shift, extraction of quantitative material properties can be challenging. BE techniques give insight into response at eigenmodes, however, other nonlinearities such as transient responses, one-time events and mode-mixing are difficult to assess (Somnath, Collins, et al., 2016; Somnath, Belianinov, Kalinin, & Jesse, 2015). These limitations led to the development of general acquisition mode (G-mode), which is based on capturing the full cantilever response at the photodetector and subsequent processing (Belianinov, Kalinin, & Jesse, 2015). While classical PFM at voltages well below the switching regime can provide high veracity information similar to contrast obtained in G-mode PFM, acquisition of the complete response is particularly insightful in non-linear and switching regimes (Somnath et al., 2015). Furthermore, studies of ferroelectric behaviour based on strain loops obtained in G-mode voltage spectroscopy have been shown to significantly reduce acquisition time as compared to conventional BE switching spectroscopy techniques (Somnath, Belianinov, Kalinin, & Jesse, 2016). G-mode

voltage spectroscopy was first implemented using a sinusoidal single frequency excitation voltage of an amplitude that exceeds the sample's coercive field, however, also multi-frequency excitation voltages can be applied. As the tip is continuously scanned across the sample surface, the cantilever response is recorded, yielding local butterfly-shaped strain hysteresis loops from which switching parameters can be extracted, such as forward and reverse coercive voltages, maximum strains and wing areas. The acquired raw deflection data is processed via fast Fourier transformation and subsequently denoised through noise-floor calculations and a band-pass filter that retains information from the first 11 harmonics of the excitation frequency. By inverse fast Fourier transformation of the spectra into real space, strain loops are obtained that show cantilever deflection as a function of excitation voltage. Functional material properties can be inferred from the shape of these loops, which are explored using statistical methods comprising principal component analysis (PCA), k-means clustering and Bayesian linear unmixing, which are further discussed below. Apart from the capability to extract information on multiple interacting resonances and harmonics, G-mode voltage spectroscopy provides acquisition at a high speed corresponding to the duration

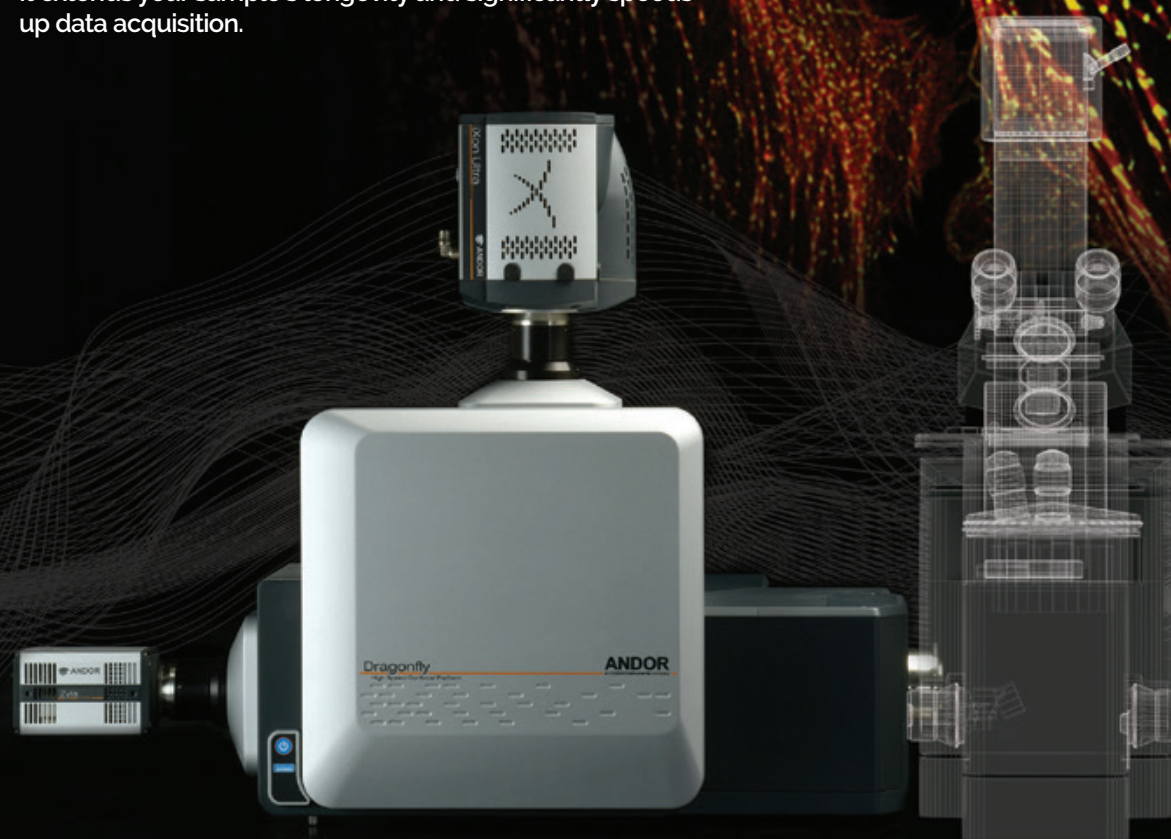
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of a switching cycle, which is determined by the frequency of the excitation voltage (1 kHz - 1 MHz). Tens to thousands of cycles can be performed within the typical waveform of 1-10 ms, which approximately corresponds to the time per scanned pixel. In comparison, a typical switching cycle in conventional BE switching spectroscopy takes >1 s and increases with voltage resolution (i.e. steps per waveform) and step duration (~4-10 ms), which leads to typical acquisition times of several hours for a map of 50×50 pixels. The fast, continuous stream allows for multi-resolution in the fast scanning direction, which means that data obtained from an image of a certain spatial resolution and amount of cycles per pixel can be converted to an image with higher spatial resolution and less cycles per pixel after acquisition, dependent on the feature of interest.

G-mode has also been combined with electrostatic modes to record and analyse the full cantilever response in open loop spectroscopy and continuous scan modes. There are several ways to analyse the obtained data, e.g. by extracting first and second harmonic EFM responses similar to DH-KPFM without the need for dual lock-in amplifier channels (Collins, Belianinov, Somnath, Rodriguez, et al., 2016). Another approach is to fit the parabolic cantilever displacement within each cycle of the sinusoidal excitation signal as obtained from decorrelating the raw data with PCA. From the second order polynomial fit, CPD and capacitance gradient can be derived (Collins, Belianinov, Somnath, Balke, et al., 2016). In Figure 9, CPD maps of a Schottky diode obtained using classical KPFM and G-mode KPFM are shown, which exhibit similar values apart from a small offset that might be related to feedback artefacts. PCA loading maps highlight sample areas that have a different functional response and show contrast between the metal electrode and silicon as well as an interfacial layer. The high temporal resolution, and absence of dc voltages in G-mode KPFM provides a path to study fast ion dynamics and electrochemical phenomena at the interface

between solid samples and conducting liquids. Furthermore, G-mode allows to obtain a full picture of the functional cantilever response and has therefore the power to also reveal unexpected material behaviour. However, data processing and storage as well as extraction and interpretation of information on physical and chemical phenomena can be challenging.

Big data analytics

With the advent of functional AFM techniques that result in large multi-dimensional data sets, extraction and analysis of significant information has become increasingly challenging. In BE switching spectroscopy maps, information on piezoresponse amplitude, piezoresponse phase, resonance frequency and quality factor is obtained for each spatial grid point during and after each voltage pulse of a waveform that usually comprises multiple loops. Therefore, a typical first-order reversal curve experiment of 50×50 pixels and a waveform consisting of 5 loops of 52 voltage steps (as shown in Figure 7) results in 5.2 million data points. Data sets acquired in G-mode are even larger (~1 billion points) and apart from analysis, storage can be non-trivial with raw data file sizes of several GB. In order to aid identification, interpretation and visualisation of functional material behaviour, multivariate statistical methods such as PCA, k-means clustering and Bayesian linear unmixing are used for data mining and discussed below (Belianinov et al., 2015; Gareth, Witten, Hastie, & Tibshirani, 2013; Somnath, Belianinov, et al., 2016; Strelcov et al., 2014). Furthermore, cross-correlation coefficients yield information on how strongly parameters that characterise the obtained response are correlated.

PCA is an unsupervised learning algorithm that has become an important tool for analysis, visualisation and dimensionality reduction of multi-dimensional data sets. In PCA, input data matrix A of size $n \times p$ representing n observations (grid points) on a set of p features (voltage points) is analysed by finding orthogonal directions in feature

space that show most variance within the n data points. These directions are described by principal component loading vectors and correspond to eigenvectors of the matrix ATA . Eigenvectors are sorted in descending order of their eigenvalues that represent variances of the components, thus most variance within the raw data is contained in the first principal component. In a simple geometric interpretation, projecting the n data points onto principal component loading vectors yields PCA scores for each component, which can be used for graphical data representation and further analysis together with eigenvectors. In functional AFM imaging, obtained score values are often plotted in a map of pixels corresponding to a certain sample location, which allows to highlight areas of different functional behaviour (see loading maps in Figure 9). Figure 10 (a) shows a simple, 2-dimensional example for PCA where the two lines indicate 1st and 2nd principal component directions. Scree plots yield the proportion of variance explained by each principal component and thus allows to identify the number of components containing physical relevant information, which can be used for data compression. In the example shown in Figure 10, the scree plot visualises that ~88% of variation is explained by the 1st principal component. While PCA provides an easy, exploratory method to de-correlate and visualise most significant variations within the data, information content is purely qualitative and physical interpretation, especially of higher components can be difficult.

Clustering algorithms can be applied to find subgroups in a data set which contain data points that are similar to each other. A simple and flexible algorithm is k-means clustering where all data points are assigned to one of k clusters upon minimising the total within-cluster variation. A common choice to describe this variation is the squared Euclidean distance between data points. If k is unknown, the optimal number of clusters can be obtained from the number of principal components that contain the majority of relevant information. Otherwise k

can be inferred iteratively by comparing the quality of different k-means results in terms of position of cluster centroids or distance between data points of one cluster to points in neighbouring clusters.

If the number of independent components in the data is known (e.g. from previous PCA or k-means analysis), Bayesian linear unmixing provides a quantitative method to decompose the spectrum of each pixel into a linear combination of position-independent endmembers weighted with relative abundances and corrupted by additive Gaussian noise. Bayesian endmembers are obtained in units of the input data and comparably easy to interpret in terms of physical meaning. Together with loading maps, type and spatial distribution of functional material properties can be revealed that would be obfuscated otherwise (Strelcov et al., 2014).

Summary and outlook

Voltage modulated AFM techniques allow us to explore functional material properties that are of fundamental importance for applications across many fields in materials science. Understanding tip-sample interactions is key to making conclusions on electromechanical, electrochemical and electrostatic phenomena, especially if several functional responses interact. Solutions are developed to differentiate between signal origins and recent technique developments aim to capture an increasingly wide range of these interactions upon post-processing analysis. With advances in AFM techniques and computing, future developments might target on the fly data processing and analysis as well as adaptive data acquisition where the tip could spend more time at regions of interest or image them at higher resolution. Further integration of combined functional AFM imaging and complimentary experimental techniques such as Raman and mass spectroscopy yield rich datasets that could facilitate linking functional behaviour to material structure.

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Heating Stages for EBSD

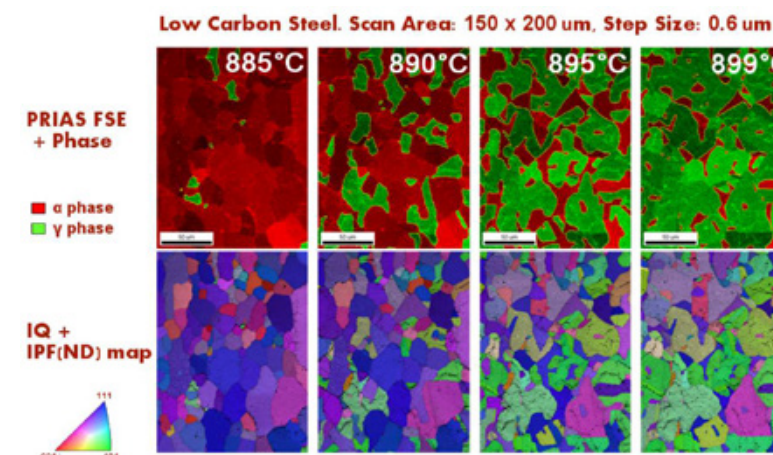


Acutance Scientific brings these high pedigree EBSD Heating stages to the UK

These specimen stages, **designed by EBSD scientists** at TSL Solutions KK for EBSD/OIM, are in situ heating stages which can be combined with EBSD/OIM observation. The HSEA-1000 stage can be heated comfortably and reliably up to 1000°C and the HSEA-500 to 500°C, for direct observation of microstructure changes of the specimen.

Dynamic observation of these phenomena is available by combining with EBSD/OIM.

These stage designs **need no modification for fitting to SEM stages**. They can be set on the SEM stage in the same way as standard specimen holders.



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