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Quantitative Piezoresponse Force Microscopy Informed by Cantilever Vibrations

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Piezoresponse force microscopy (PFM) is a ubiquitous technique for the characterization of ferroelectrics and related materials exhibiting electromechanical strain. By applying an AC bias between an atomic force microscope (AFM) tip and substrate, resultant local strains can be detected via the AFM cantilever's bending. While the method has seen considerable development in the past 3 decades, the quantitative and artifact-free nature of the resultant data have been subject to considerable scrutiny. Researchers have sought to understand the influence of AC frequency, tip and distributed electrostatic force, sample stiffness, and myriad other parameters on the interpretation of PFM maps and point-spectroscopy data. Here, we show that understanding and control of cantilever dynamics can allow for elucidation of many of these effects. In particular, we explore PFM measurements wherein the AC frequency is either at a sub-resonance frequency or at a contact resonance frequency (i.e. the resonance frequency of the cantilever when the tip is in repulsive contact with the substrate). We present recent findings on how either modality can be used to inform quantitative measurements of piezoelectric coupling coefficients, with eliminated or greatly reduced distributed electrostatic force artifacts.

Sub-resonance PFM, defined as operation far below the contact resonance frequency where inertial effects can be neglected, is attractive because it allows for straightforward quantification of the coupling coefficient based on the same cantilever sensitivity calibration used in force vs distance spectroscopy. Despite its apparent simplicity, quantitative sub-resonance PFM requires that the measured signal not exhibit significant contribution from distributed electrostatic forces that arise due to the capacitive gradient between sample and cantilever body. In the presence of distributed electrostatic forces, the cantilever bends in a manner that is a superposition of the sample strain directly displacing the tip and the distributed forces which impart an effective point load set back from tip. Owing to the fact that conventional optical beam deflection (OBD) detection of the cantilever bending is intrinsically sensitive to the local bending slope of the cantilever rather than displacement, placement of the detection laser at the location of the distributed load's equivalent point load results in a maximum displacement of the cantilever, and hence a zero slope. Notably, at this same laser location, the cantilever slope induced by the sample strain is still detectable, and can be quantified using the conventional OBD sensitivity calibration. We refer to PFM performed with this specific laser position as electrostatic blind spot PFM (ESBS-PFM). We have demonstrated on materials such as periodically poled lithium niobate and copper indium thiophosphate how ESBS-PFM allows for quantitative measurements and maps of piezoelectric coupling coefficients in excellent agreement with complementary methods employing interferometric sensing of cantilever displacement [1]. Here we expand our investigation of ESBS-PFM to consider specific effects of parameters such as detection spot size, tip-sample contact stiffness, cantilever shape and more to establish clear guidelines for the implementation of ESBS-PFM in routine ferroelectric characterization.

Complementing the use of sub-resonance PFM, contact resonance PFM (CR-PFM) allows for amplification of electromechanical strains roughly proportional to the quality factor Q of the resonance

(50 < Q < 500 typical). Thus, for weakly coupled signals CR-PFM provides one of the only pathways for detecting local piezoresponse and related phenomena. Nonetheless, CR-PFM presents considerable challenges with quantification of electromechanical displacements and identification of distributed electrostatic force artifacts. We have developed a number of CR-PFM tools to enable quantitative measurement with reduced or appropriately-considered electrostatic artifacts. For quantification, we demonstrate how the specific vibrational shape of the cantilever for a given boundary condition can be experimentally calibrated [2]. This "shape factor" provides a scale factor to correct for the difference in cantilever sensitivity between the contact resonance eigenmode and the static force-distance bending shape of the cantilever. To mitigate the influence of distributed electrostatic force on observed PFM amplitude, we investigate operation at higher-order resonance modes [3]. At higher-order modes the cantilever is increasingly dynamically stiffened, lessening the influence of the electrostatic force on the resultant amplitude. Finally, we address elimination of artifacts induced by stiffness heterogeneity in the sample. As the sample surface changes stiffness, the eigenmode and the modal damping of the cantilever both change. By introducing isomorphic contact resonance PFM, we have developed a method wherein tip-sample interaction force is varied to achieve a constant contact stiffness at all pixels in the image [4]. The result is that any remaining amplitude contrast can by directly attributed to local piezoelectric coupling coefficient (assuming electrostatic forces have been adequately suppressed). Finally, we show how CR-PFM and ESBS-PFM can be used in conjunction to quantify contact stiffness and correct for it in observed displacements, as well as for ESBS-PFM to inform shape factor for higher order CR-PFM modes of compliant, soft-matter-compatible cantilevers. Overall, the methods discussed can and should be used in conjunction to ensure self-consistency of observed electromechanical phenomena.

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