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ASYLUM RESEARCH

Application Note

The Vero Interferometric AFM - Advances in Accuracy for Piezoresponse Force Microscopy

Introduction

Aptly named from the Latin root *ver*, meaning "truth," the Vero atomic force microscope (AFM) uses a patented[1] interferometer-based design to directly measure tip displacement, bringing unprecedented accuracy to the field of AFM.

Interestingly, almost all AFMs are unable to measure tip displacement directly and must rely on a proxy measure instead. Specifically, most AFMs can only infer this through a calibrated measurement of the change in cantilever deflection or angle. Herein lies the basis for possible sources of error: any change in the bend or angle of the cantilever can only be interpreted as a change in tip displacement, regardless of whether this is true or not. Indeed, many phenomena can affect the cantilever deflection or angle even in the absence of tip displacement, such as electrostatics, in-plane sample forces, and forces between the tip and sample during scanning, and will therefore be misinterpreted. Piezoresponse force microscopy[2,3] (PFM) is a technique that is plagued by all of these artifacts. This note discusses each of these artifacts in more detail, thereby showing the benefits of the Vero AFM within the specific context of PFM measurements.

PFM Background

PFM enables the characterization of a sample's nanoscale electromechanical response. In this mode, a piezoelectric material under an applied electric field will exhibit mechanical strain, a phenomenon known as the converse piezoelectric effect. Typically, the electric field is applied across the sample through a conductive AFM tip in contact with the sample and the resulting strain is measured by detecting cantilever deflection to infer tip displacement.

And yet ironically, the very application of this electric field, which is so fundamental to the PFM measurement itself, can cause errors. The cantilever bias, meant to induce a piezoresponse from the sample, also causes electrostatic bending of the cantilever, which most AFMs will misinterpret as tip displacement. In samples known to be piezoelectric, this has resulted in inconsistent values of the measured effective piezoelectric coupling coefficient. Additionally, there have also been many reports of "strange" ferroelectricity observed in various materials that have no physical basis for such phenomena.[4]

These uncertainties in PFM measurements continue to persist because with most AFMs, tip displacement can only be interpreted through the cantilever's angle, and a sample's piezoresponse cannot be distinguished from other phenomena that also cause the cantilever to bend. In contrast, the Vero AFM is immune to these artifacts because tip displacement is measured directly.

Vero AFM Design Avoids PFM Artifacts

At the heart of any AFM is its capability to measure tip displacement. Until now, almost all AFMs achieved this through a detection system known as "optical lever" or "optical beam deflection" (OBD).[5,6] In this type of system, the cantilever is the optical lever, converting angular changes in its deflection into indirect measures of tip displacement. Simply put, when there is a change in tip displacement, there is a corresponding change in cantilever angle. However, the converse of this is not necessarily the case – artifacts arise with OBD-based optical lever AFMs when there are changes in cantilever angle that do not correspond to changes in tip displacement.

An alternative to measuring cantilever angle through an optical lever detector is to measure the cantilever displacement directly through interferometry. Interferometric detectors were proposed in the early days of AFM[7] but largely abandoned in favor of OBD-based detectors due to cost and complexity. In 2015, the Cypher-IDS became the first commercially available AFM coupled with an external interferometer[8] and has since provided accurate crosstalk-free nanoscale functional measurements in a variety of areas ranging from memory and computing beyond Moore's law[9], photonic computing[10,11], energy storage and production, MEMs devices such as bulk acoustic resonators, and 2D materials.[12,13]

The Vero AFM marks another step forward in the evolution of interferometric AFMs with the first built-in quadrature phase differential interferometer (QPDI)[14] to accurately measure tip displacement directly rather than having to instead infer this from a change in cantilever angle. While unwanted cantilever bending from various sources can and do still occur, the Vero AFM is simply immune to these effects by design as shown in Figure 1.

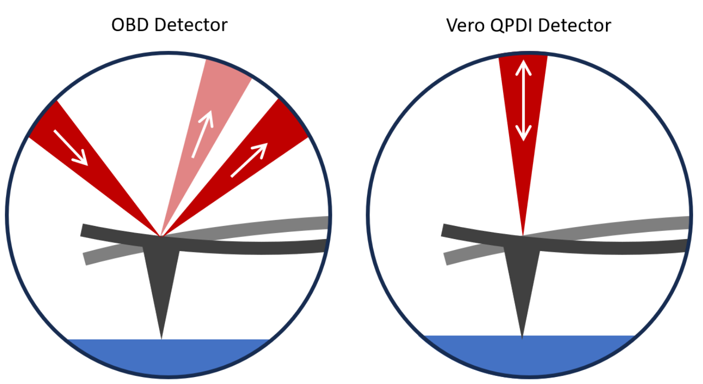

Figure 1: During a PFM measurement on a sample with no piezoresponse, the biased cantilever can bend due to electrostatics. The OBD-based AFM would register a false change in tip displacement due to the cantilever angle changing, whereas the QPDI-based Vero AFM would be insensitive to bending when the interferometric spot is positioned directly over the tip.

Specifically, the measurement becomes insensitive to cantilever bending when the Vero AFM's interferometric spot is positioned directly over the cantilever tip itself, with the tip acting like a fulcrum about which the cantilever can bend.

Electrostatic Artifacts in PFM

Electrostatic interactions between the cantilever and sample can be a significant source of error in PFM measurements.[15] One particularly salient consequence of this has been erroneous reports of switching spectroscopy PFM[16] (SS-PFM) showing purported ferroelectric switching in non-ferroelectric samples.[4]

SS-PFM is used to characterize a sample's hysteretic polarization switching, or lack thereof as the case might be. In this technique, a DC bias is applied to the sample in the form of a square wave that ramps in magnitude over time while the sample's PFM response is simultaneously measured. If the sample is ferroelectric, this changing DC bias should induce polarization switching resulting in a signature "butterfly" hysteresis loop. However, nonlocalized electrostatic interactions between the tip and sample can complicate this interpretation.

When SS-PFM is applied to soda lime glass, a non-ferroelectric sample, an apparent hysteretic switching response is observed. A similar SS-PFM response can be observed when the tip is not even in contact with the soda lime glass surface indicating that this effect is due to nonlocalized electrostatic interactions between the cantilever and sample and is not actually polarization switching that would be the hallmark of a ferroelectric material.[17]

Figure 2 compares two SS-PFM measurements taken with the Vero AFM with the only difference being in the placement of the interferometric spot position. When the spot is positioned far away from the cantilever tip, the measurement is sensitive to the electrostatic bending of the cantilever. Despite Vero's QPDI detector being insensitive to cantilever angle, it can measure cantilever displacement resulting from electrostatic bending if the spot is positioned far away from the fulcrum of the cantilever tip. This results in a false switching spectroscopy response where cantilever deflection changes caused by electrostatics get misconstrued as actual piezoresponse. This false response is similar to what would be observed with previous-generation AFMs based on optical lever detection. However, when the Vero interferometric spot is positioned directly over the cantilever tip, the measurement becomes immune to this electrostatic cantilever bending artifact, and the response is flat as would be expected for soda lime glass, a non-ferroelectric sample. In this way, the Vero AFM can unambiguously determine whether there is a ferroelectric response.

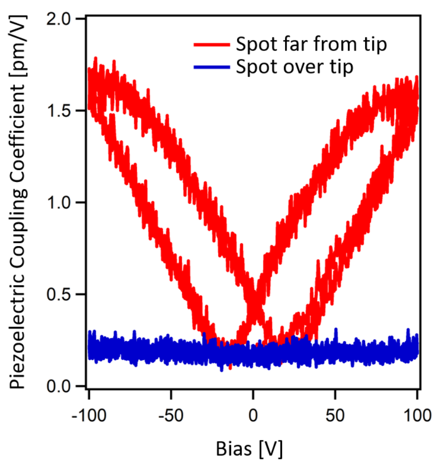

Figure 2: SS-PFM on soda lime glass results in a false "butterfly" loop when the Vero interferometric spot is positioned far away from the cantilever tip. However, the SS-PFM response is flat, as would be expected, when the Vero interferometric spot is positioned directly over the cantilever tip.

Artifacts from In-Plane Piezoresponse

The cantilever deflection angle can also be affected by any in-plane forces acting on the tip and causing buckling or longitudinal bending of the cantilever beam. This is particularly relevant for PFM measurements where signals are generally small, and samples may have some in-plane piezoresponse. Components of this in-plane response oriented along the long axis of the cantilever can cause the cantilever to bend and this can add to or subtract from the actual measured vertical piezoresponse.

This signal cross-coupling can be seen in comparative PFM measurements between an OBD-based AFM and the QPDI-based Vero AFM on bismuth ferrite (BFO), a multiferroic sample that has both in-plane and out-of-plane piezoresponse components. The same cantilever and scan settings were used with both AFM types, and the same sample location was imaged, as shown in Figure 3. The OBD-based measurements were taken on resonance as the preferred approach for optimal PFM signal amplification, whereas the QPDI-based Vero measurements were taken at a fixed drive frequency of 30 kHz.

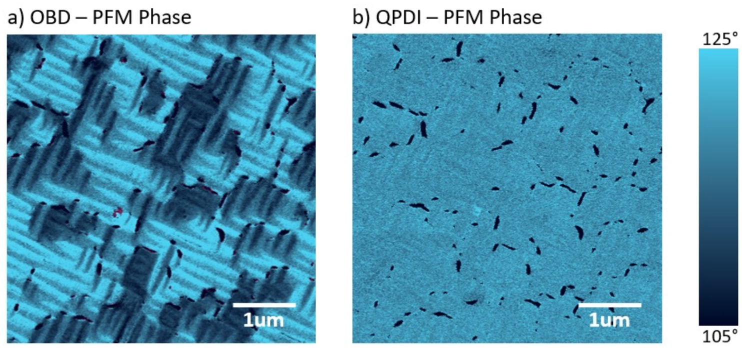

Figure 3: BFO's in-plane piezoresponse can cause the cantilever to bend during the PFM measurement, and an OBD-based AFM will falsely interpret this as vertical piezoresponse. This artifact does not appear with the QPDI-based Vero AFM since tip displacement is measured directly and is independent of cantilever angle.

This BFO (001) sample is not expected to have much out-of-plane contrast, however there is significant signal contrast in the phase channel taken with an OBD-based optical lever AFM, as shown in Figure 3a. However, with the QPDI-based Vero AFM, this contrast is no longer present when the interferometric spot is positioned directly over the cantilever's tip, as shown in Figure 3b. This indicates that the supposedly vertical piezoresponse signal shown in Figure 3a is an artifact and results from the BFO's in-plane piezoresponse cross-coupling in. Any component of the sample's in-plane piezoresponse aligned with the cantilever will cause the cantilever to bend and this gets misattributed as vertical piezoresponse. In contrast, since the Vero AFM measures tip displacement directly, it is insensitive to the BFO's in-plane response and only shows the true vertical response.

Artifacts from Tip-Sample Forces During Scan

A cantilever can also bend during a PFM scan simply from the in-plane lateral and longitudinal forces acting between the tip and the sample. The resulting cantilever shape can introduce error in PFM measurements taken with an OBD-based optical lever AFM. Furthermore, this error varies with scan angle since this affects the direction of the tip-sample force vector, which in turn affects cantilever shape.

PFM scans were taken at different angles on periodically poled lithium niobate (PPLN), a ferroelectric sample with striped domains of alternating polarization orientations of up and down. The amplitude response of these domains is expected to be identical with the only difference being in the domain polarization. However, when scanned with an OBD-based AFM, not only is there a difference in amplitude response between adjacent domains, but this response also varies as a function of scan angle as shown in the left column of images in Figure 4.

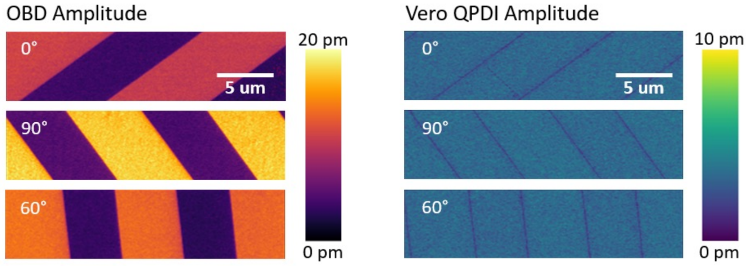

Figure 4: Changing the scan angle changes the amount of longitudinal and lateral in-plane forces acting on the scanning tip, which affects the cantilever bending. This results in an artifact for PFM measurements with an OBD-based AFM, where the piezoresponse Amplitude range and offset vary with scan angle. In contrast, the piezoresponse Amplitude is constant and independent of scan angle when measured with the QPDI-based Vero AFM.

In contrast, the corresponding series of images shown in the right column of Figure 4 were taken with the QPDI-based Vero AFM and shows accuracy and consistency in the amplitude response regardless of domain orientation and scan angle. These images were taken with the same cantilever and at the same sample location. With the interferometric spot positioned directly over the cantilever tip, the Vero AFM shows the true vertical PFM response, again being immune to other effects causing cantilever bending.

Improved PFM Measurement Repeatability

Each of the aforementioned artifacts add together along with the sample's actual vertical piezoresponse because they all affect the cantilever's deflection angle. OBD-based optical lever AFMs are unable to distinguish between actual tip displacement and these other artifacts, resulting in measurement uncertainty. Consequently, it is not surprising that PFM with OBD-based AFMs has resulted in a wide range of reported effective piezoelectric coupling coefficients for known piezoelectric materials.

To illustrate this, five PFM scans were taken on aluminum scandium nitride each with a different probe at different drive amplitudes and scan angles with an OBD-based AFM and repeated under these same conditions with the QPDI-based interferometric Vero AFM. Figure 5 shows histograms of the effective piezoelectric coupling coefficient as measured from the optical lever based AFM and the Vero AFM, shown in red and blue, respectively. All five histograms of the Vero AFM measurements are narrow and tightly clustered despite all being taken under different scan conditions. In contrast, the corresponding histograms of the OBD-based optical lever AFM measurements are broad and scattered.

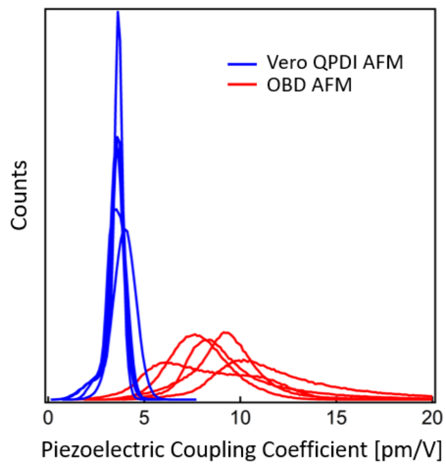

Figure 5: The piezoelectric coupling coefficient of AlScN was measured with both an OBD-based AFM and the QPDI-based Vero AFM at various scanning conditions and the corresponding histograms are shown. The measurement repeatability is much better with the Vero AFM compared to the OBD-based AFM as can be seen from the spread of the distributions.

Conclusion

The Vero AFM delivers results with a degree of accuracy that has been largely missing from the PFM field until now. Seemingly simple measurements like establishing whether a given material is ferroelectric or measuring the effective piezoelectric coupling coefficient become difficult if not impossible to answer accurately with an OBD-based optical lever AFM. With the Vero AFM, these simple measurements become just that – simple.

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