# Sample-Tip Interaction of Piezoresponse Force Microscopy in Ferroelectric Nanostructures

Frank Peter, Andreas Rüdiger, Krzysztof Szot, Rainer Waser, and Bernd Reichenberg

***Abstract*—We report on qualitative and quantitative implications of the sample-tip interaction in piezoresponse force microscopy. Our finite-element analysis of adsorbate effects, sample heterogeneities, and tip asymmetries is in agreement with experimental observation of ferroelectric nanostructures. Qualitative discrepancies arise from locally asymmetric tip-sample interaction. Any quantitative determination of field-related material parameters as required for the verification of semiempirical models of the ferroelectric limit typically relies on an overestimated field across the sample. Our findings indicate that adsorbates reduce the actual field across the nanograin by roughly one order of magnitude.**

## I. Introduction

The electromechanical sensing of ferroelectric nanostructures by atomic force microscopy (AFM)-techniques has been tremendously successful for the last decade [1]–[3]. A big advance in piezoresponse force microscopy (PFM), the detection of elastic deformations under an external electric field applied through the tip, was the introduction of in-plane measurements [4], [5]. The quadrupole photodiode is used such that both laser deflections from bending as well as torsion of the cantilever can be monitored simultaneously as illustrated in Fig. 1. In particular, the amplitude and phase can be interpreted as indicators of the piezoelectric activity and relative orientation of the polarization [6]. As the measurement is fundamentally based on the tip-sample interaction, this system deserves particular attention. The quantification of electromechanical processes relies on the knowledge of local fields. We identify an adsorbate layer and quantify its impact as a voltage divider between tip and sample.

Due to the optical lever method, the in-plane signal is less noisy and, therefore, favorable for many imaging applications of PFM. We report on several contributions to the in-plane piezoresponse that do not originate from the piezoelectric tensor itself but from local asymmetries at and around the tip. Finite-element simulations are presented to model and to quantify the impact of sample topography, tip asymmetry, and local material heterogeneities.

Manuscript received July 29, 2005; accepted January 24, 2006.

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Digital Object Identifier 10.1109/TUFFC.2006.170

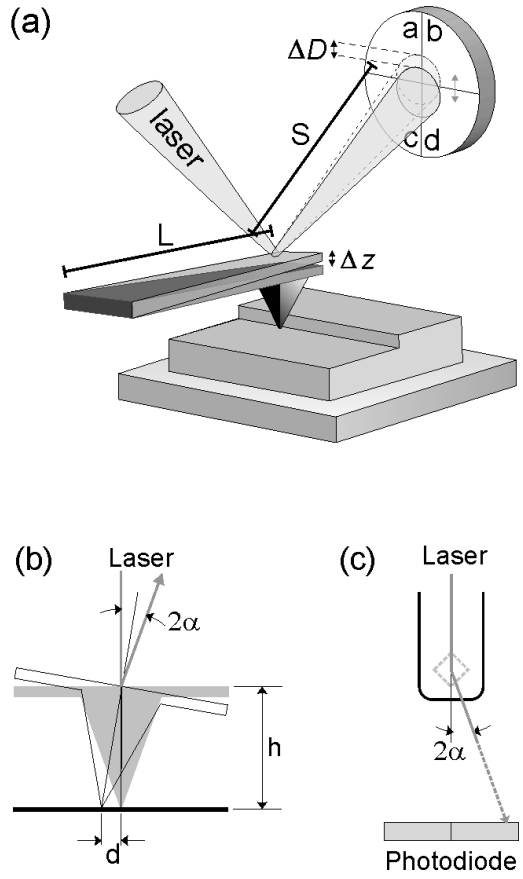


Fig. 1. Optical lever arm method (part a), with $L$ = length of cantilever, $S$ = distance between cantilever and photodiode, $\Delta D$ = movement of laser on photodiode, $\Delta z$ = out-of-plane cantilever movement. Part b shows the front view with $d$ = lateral movement of the cantilever, $h$ = height of tip plus thickness of cantilever, $\alpha$ = tilt of cantilever and part c the top view of the cantilever.

## II. Optical Amplification

In PFM a conducting tip is brought into contact with the sample and serves as a top electrode. An alternating current (AC) voltage is then applied to the tip, and the piezoelectric in-plane and out-of-plane response are optically detected as a deflection of the laser beam reflected from the cantilever. In many cases the in-plane response is substantially larger than the out-of-plane response and, therefore, shows more details and less noise. A typical example of such a measurement is shown in Fig. 2. Details on the growth of the nanoislands by chemical solution deposition (CSD) were reported separately [7]. The PFM measurements are performed on a commercial AFM (JEOL JSPM 4210, Jeol Ltd., Tokyo, Japan) using PtIr-coated

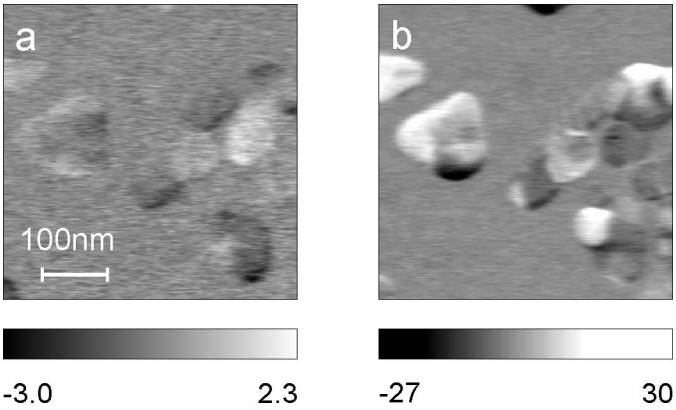


Fig. 2. Out-of-plane (part a) and in-plane (part b) piezoresponse measured on $PbTiO_3$ nanograins. The amplitude of the in-plane image is roughly one order of magnitude larger than the out-of-plane response (adapted from [9].)

cantilevers. In this measurement the in-plane response is roughly one order of magnitude larger than the out-of-plane response. The signal-to-noise ratio is a factor of 3.5 better in the in-plane image. As the piezoelectric coefficients for $PbTiO_3$ vary only by a factor of five for the different directions [8], the question arises, if this effect can be due to a direction-dependent amplification.

The most common method used in AFMs to detect the cantilever deflection is by measuring the position of a reflected laser beam on a position-sensitive detector. A schematic of this optical lever arm method is given in Fig. 1(a). The out-of-plane lever amplification:

$$V_{\text{out-of-plane}} = \frac{\Delta D}{\Delta z} = \frac{3S}{L} \tag{1}$$

is a factor of about 1,000 [10]. In this case the two top quadrants [$a$ and $b$ in Fig. 1(a)] and the two bottom quadrants ($c$ and $d$) have to be regarded as one. This same principle is used to detect the in-plane deflection; here the two left ($a$ and $c$) and right ($b$ and $d$) quadrants of the photodiode have to be considered as one. An in-plane movement of the tip is shown in Fig. 1(b). We assume that the apex of the tip moves a lateral distance $d$, but the middle of the tip base remains stationary. From Fig. 1(b) and (c) it follows for small $\alpha$:

$$\tan\alpha = \frac{d}{h} \approx \alpha, \tag{2}$$

where $h$ is the height of the tip plus the thickness of the cantilever. The change of the irradiated area of the left and right parts of the photodiode is a linear function of the displacement:

$$\Delta D = \sin(2\alpha) \cdot S \approx 2\alpha \cdot S. \tag{3}$$

This results in an amplification factor of:

$$V_{\text{in-plane}} = \frac{\Delta D}{d} = \frac{2S}{h}. \tag{4}$$

The ratio $R$ between the two amplifications is:[1]

$$R = \frac{V_{\text{in-plane}}}{V_{\text{out-of-plane}}} = \frac{\frac{2S}{h}}{\frac{3S}{L}} = \frac{2}{3}\frac{L}{h}. \tag{5}$$

For cantilevers with a length of 450 $\mu$m and a tip height plus cantilever thickness of 12–17 $\mu$m (ContPt-Cantilevers from Nanosensors, Neuchatel, Switzerland.) this ratio is:

$$18 < R < 25.$$

In [9] we presented a dedicated setup to measure the in-plane and out-of-plane optical amplifications. The ratio between the two has been measured to be about 18, in good agreement with the prediction. The higher in-plane piezoresponse presented in Fig. 2 can thus be attributed to the measuring setup and not to a larger piezoelectric expansion of the nanograins. In practice, the in-plane signal provides a considerably better signal-to-noise ratio than the out-of-plane response and, therefore, is more appealing. We will subsequently show that in-plane PFM provides a wealth of additional information that can be used to identify local heterogeneities of a sample.

## III. Tip-Sample Contact

One aspect that often is neglected in AFM measurements is the contact between the tip and the sample. This becomes important when the tip also is used as a movable top-electrode as is the case in PFM measurements. How good is the electrical contact between the tip and the sample? When scanning with a conducting cantilever over a metallic surface, one would expect to have a constantly small contact resistance. Experiments show that this is often not the case [11]. Apart from a poorly coated tip, a contamination layer on top of the sample can be the cause of a bad electrical contact. This raises the question if ferroelectric samples are generally covered by some sort of contamination layer. Answers to this question can be obtained from surface sensitive methods like X-ray photoelectron spectroscopy (XPS) or auger spectroscopy [12].

To obtain a general result, the model material $BaTiO_3$ is chosen. The O 1s core line of this single crystal measured at room temperature by XPS is shown in Fig. 3. In case of an ideally clean surface, only the lattice oxygen with an energy of $E_1 = 529.8$ eV exists. However, two additional components with binding energies of $E_2 = 531.5$ eV and $E_3 = 533.8$ eV are observed. These can be identified according to [13] as chemisorbed CO or $CO_2$ ($E_2$) and as physisorbed OH or $H_2O$ ($E_3$). As the lattice oxygen can be detected, the thickness of the contamination layer can only be a few nanometers. From measurements at different angles, we deduce that the chemisorbate layer is directly on top of the surface of the bulk material and the physisorbates form the top layer. A schematic cross section of a contaminated surface is given in Fig. 4.

[1]This equation is corrected by a factor of 2 as compared to [9].

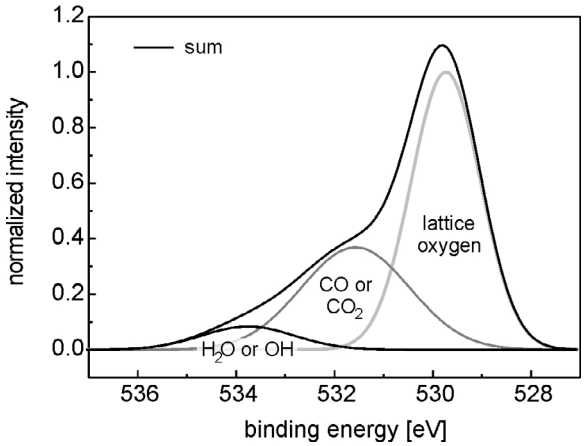


Fig. 3. O 1s core lines of a $BaTiO_3$ single crystal measured by XPS at room temperature (adapted from [14]).

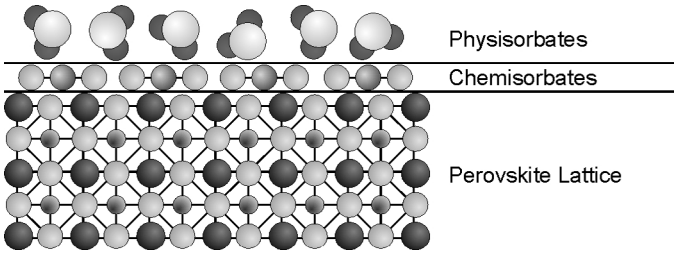


Fig. 4. Model of adsorbate layers on a perovskite-type material (adapted from [14]).

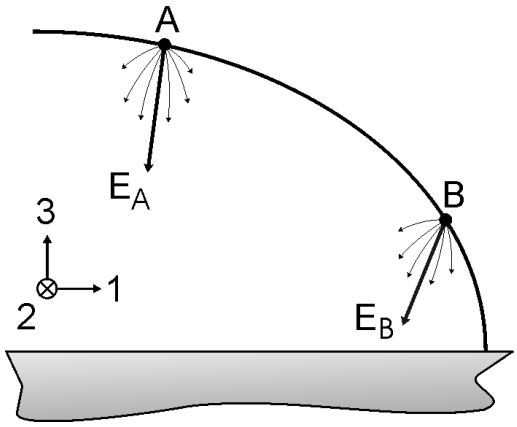


Fig. 5. Electric field distribution inside the ferroelectric nanograin for tip positions A and B showing the in-plane contributions (adapted from [17]).

TABLE I
AVERAGE PIEZOELECTRIC ACTIVITY UNDER DIFFERENT SURFACE CONDITIONS.

| Condition | Piezoresponse |
|---|---|
| Ambient | 100% |
| High vacuum | 115% |
| High vacuum after heating to 350°C | 250% |
| Ambient after heating cycle | 100% |

For the PFM analysis of ferroelectric perovskite materials these adsorbate layers result in a drastic deterioration of the electrical contact between the tip and the actual material. Is there a method to—at least partially—remove these layers?

The effect of ad- and desorption can best be monitored under defined conditions of temperature and ambient pressure. We use the vacuum chamber of a Jeol JSPM 4210 (Jeol Ltd., Tokyo, Japan) and a heatable sample holder to characterize the effect of ubiquitous atmospheric molecules on piezoresponse. A $BaTiO_3$ single crystal is measured in air at room temperature (100% signal). To assure comparable data and to counterbalance drift of the setup, we averaged the piezo-signal over an area of 20 $\mu$m × 20 $\mu$m. Evacuation down to $3 \times 10^{-5}$ mbar improves both in- and out-of-plane signal by 15%. Because our PFM setup only permits room temperature measurements, we heat the sample to 350°C in this vacuum and repeat the measurement at 30°C to find an enhancement of the piezoresponse by 250% on average and 800% at maximum (Table I). Our temperature-dependent XPS measurements up to 800°C indicate that the bulk oxygen remains unaltered, but the chemisorbates can be partially removed. Physisorbates are fully removed already above 350°C [14]. Considering that we desorbed only $H_2O$ or OH, additional heating above the desorption temperature of CO and $CO_2$ may further improve the piezoresponse. We illustrate in Fig. 4 that these adsorbates act as an in-series resistance, thus reducing the actual voltage applied to the sample. As a direct consequence, the in- and out-of-plane piezoresponse are reduced in the same way as confirmed by our experimental findings. Therefore, it should be made clear that a quantitative analysis of di-, piezo-, pyro-, and ferroelectric properties is futile unless special attention is devoted to a preliminary characterization of the surface and its contributions. In the subsequent section we will also discuss the effect of adsorbates on the piezoelectric response of nanograins.

## IV. Geometry Contributions

In contrast to ferroelectric capacitors sandwiched between planar electrodes, the tip-sample interaction of PFM measurements provides a more complex scenario. In PFM investigations of ferroelectric nanograins, the top electrode (i.e., the tip) has a radius comparable to the sample of typically a few tens of nanometers. This gives rise to a nonuniform and possibly nonsymmetric field distribution under the tip and a more complex coupling to the third-rank piezoelectric tensor. This has consequences mainly for the in-plane response. In addition, the symmetry axis of the cantilever (generally parallel to the scanning direction) imposes another constraint to the detectable signal.

### *A. Shape of Nanograins*

Depending on the choice of substrate and deposition method, ferroelectric nanostructures grow single- or poly-

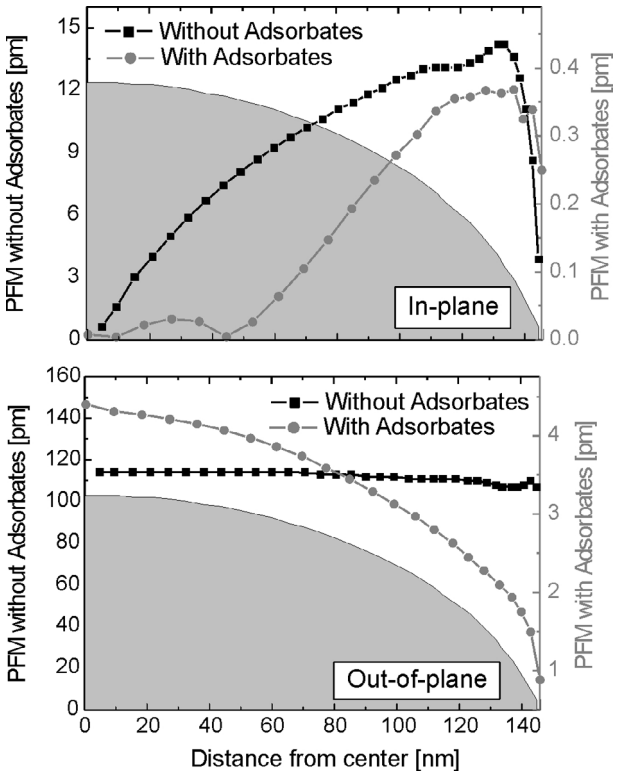


Fig. 6. Simulated in-plane and out-of-plane piezoresponse (amplitudes) as a function of the distance from the center of the nanograin. A drastic increase toward the perimeter of the grain can be seen in the in-plane response (top part). This enhancement is laterally smaller in the presence of adsorbates. The out-of-plane response (bottom part) is constant as expected for the case with no adsorbates and decreases in the presence of adsorbates due to the potential divider created by the contaminants. The shaded regions show the cross section of the topography of the modeled nanograin.

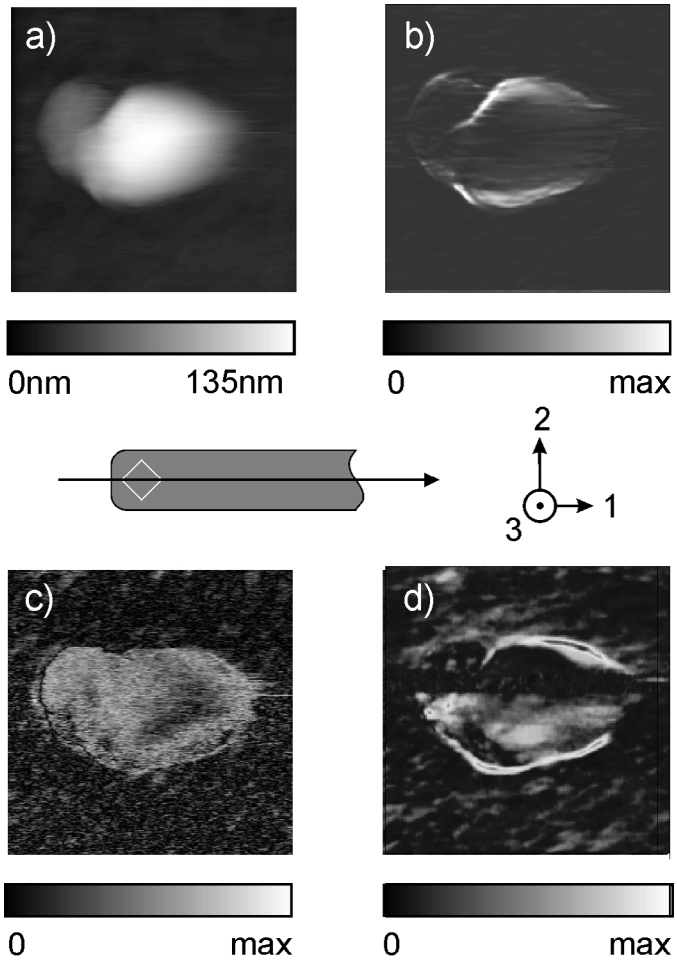


Fig. 7. $BaTiO_3$ sample: (a) topography 500 nm × 500 nm. (b) Derivative of the topography along the 2-direction. (c) Amplitude of the out-of-plane piezoresponse. (d) Amplitude of the in-plane piezoresponse. The out-of-plane response is relatively constant over the total grain, and the in-plane response is very small in the middle and high at the perimeter, an effect caused only by the surface topography. The symmetry axis of the cantilever is shown in the center.

crystalline. Thus edges and slopes will also contribute to the PFM signal. For the sake of simplicity and without loss of generality, we will now discuss a homogeneous, *c*-axis oriented tetragonal sample with an out-of-plane polarization. As the field underneath the tip has in-plane components [15], it becomes evident that the shear elements $d_{15}$ and $d_{24}$ of the piezoelectric tensor need to be considered [16]. Fig. 5 illustrates the dependence of the electric field orientation beneath the tip as a function of its position.

Only for the central position, the electric field will be fully symmetric. A tetragonal sample has fourfold symmetry in-plane—any symmetric field distribution produces a symmetric piezoresponse around the tip. These symmetric movements cancel out at the position of the tip. No in-plane signal, therefore, can be monitored even though the material around the tip is in motion, as the PFM in-plane setup only detects the deflection of the laser beam due to a tilting of the cantilever itself.

Scanning such a nanograin from the center outward has been simulated by finite elements with $BaTiO_3$ as piezoelectric. We start from the center (i.e., the aforementioned symmetrical case) with zero in-plane contribution that increases as we approach the perimeter [see Fig. 6(a)]. The in-plane signal rises to a maximum value close to the perimeter to rapidly drop as the tip leaves the grain. A direct comparison to the (more realstic) situation of an additional adsorbate layer indicates a narrower enhancement of the in-plane signal at the perimeter. As a control, we present the out-of-plane behavior. Because the sample is clamped at the interface, the shear contribution to the out-of-plane signal is minor. The adsorbate-free scenario shows that the out-of-plane signal is thickness independent, just as expected for the converse piezoelectric effect. An adsorbate layer of constant vertical thickness provides a variable thickness fraction underneath the tip (varying potential divider) which is depicted in Fig. 6(b).

Fig. 7 illustrates our PFM results of a pulsed laser deposition fabricated $BaTiO_3$ single crystal on a $SrRuO_3$ bottom electrode. Although the out-of-plane response (*c*) is rather constant over the grain, a pronounced enhancement is visible along the perimeter for the in-plane signal (*d*). Note the absence of an in-plane response at the very left and right end of the grain, which will be discussed in the next section. For more details on the grain topography

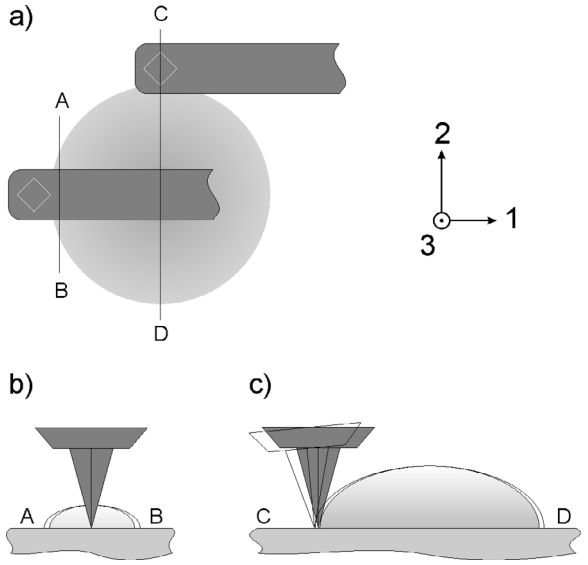


Fig. 8. (a) Top view of the cantilever-grain system for two different positions at the grain perimeter. (b) Side view along the 1-direction for the tip in front of the grain. (c) At a side of the grain, the first case with in-plane symmetry, the latter without.

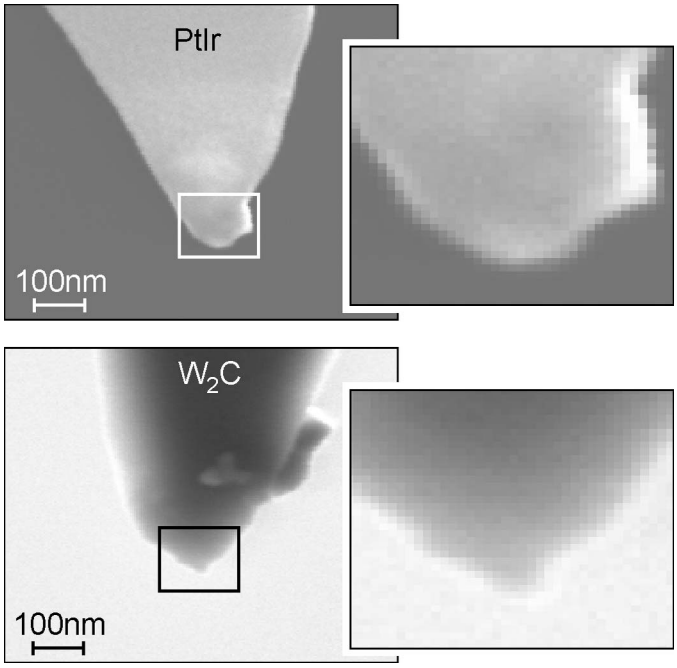


Fig. 9. Scanning electron microscopy images of a used PtIr-coated (top images) and a new $W_2C$-coated (bottom images) AFM tip (adapted from [19]).

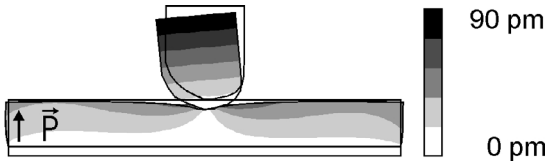


Fig. 10. Simulation of the influence of different tip-radii on the in-plane piezoresponse of a symmetric $BaTiO_3$ single crystal. The piezoelectric deformation has been exaggerated to guide the eye (adapted from [19]).

contribution to piezoresponse and the numerical simulation parameters refer to [17].

### *B. Cantilever Constraints*

Consider the cantilever moving parallel to its symmetry axis along the 1-direction (Fig. 8). Torsion of the cantilever as a consequence of an in-plane motion can occur only perpendicular to this direction (i.e., in 2-direction). The two cases (A-B, C-D) illustrated in Fig. 8(a) are different in their symmetry. Fig. 8(b) shows the cantilever between two equivalent points A and B, and the applied field will cause only a movement in negative 1-direction, a signal that cannot be detected as a torsion. In contrast Fig. 8(c) depicts the situation of the cantilever being shifted sideways as the grain expands and contracts. The difference between the positions C and D is a 180° phase shift of the in-plane signal, also experimentally confirmed. In this respect, the correct correlation between topography and in-plane signal is not the gradient as might be suggested from Fig. 5 but the derivative along the direction perpendicular to the cantilever symmetry axis. Direct comparison between the derivative along the 2-direction and the in-plane piezoelectric response for a ferroelectric $BaTiO_3$ grain as illustrated in Figs. 7(b) and (d) shows excellent agreement.

## V. Local Asymmetries

We have seen that an uneven topography causes an in-plane signal. From a global point of view, the topography introduces an asymmetry to the tip-sample system and, therefore, causes a tilting of the cantilever and a deflection of the laser beam. In a real system, there are additional ways to create such an asymmetry. In the previous section we considered a perfectly spherical tip apex and a homogeneous tetragonal sample with out-of-plane polarization. What if the tip is anything but spherical or if the sample is heterogeneous? For our PFM measurements we use commercially available PtIr-coated silicon tips.[2] Even under virgin conditions, these tips are not perfectly spherical as illustrated in the scanning electron microscopy (SEM) images of Fig. 9.

The situation typically deteriorates with every measurement and even complete delamination of the platinum may occur [18]. Whenever the tip radius varies with the in-plane orientation, the electric field will be highest at the highest tip curvature. This field asymmetry directly causes a corresponding in-plane piezoresponse that gives rise to a tilting of the cantilever. Even without a real in-plane polarization, we will monitor a constant in-plane response on a totally flat and homogeneous sample. This effect is considerable as indicated by our finite-element analysis [19] displayed in Fig. 10. For our simulation we chose a metallic tip with

[2]ContPt-Cantilevers from Nanosensors, Neuchatel, Switzerland

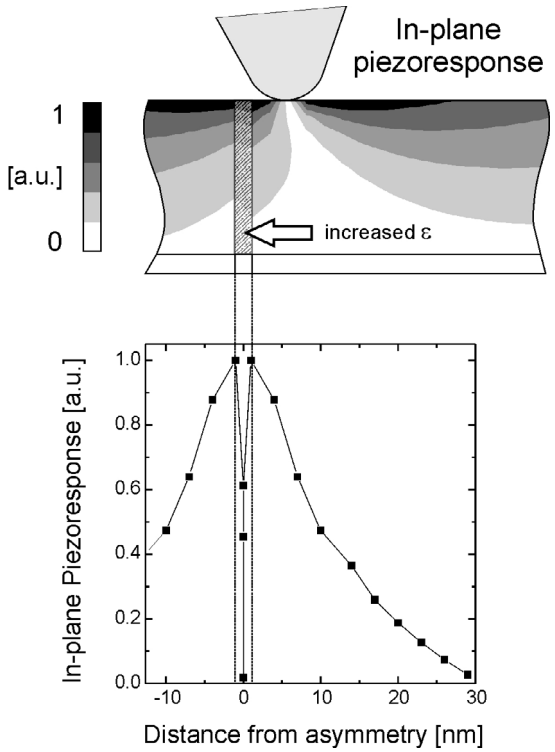


Fig. 11. Simulation of the influence of an increased dielectric constant on the in-plane piezoresponse. The top part shows the piezoresponse amplitude distribution at the indicated tip position. In the bottom part, the detectable in-plane response depending on the tip-position is depicted (adapted from [19]).

a steady and smooth surface but with different tip radii for the left and the right side. Although the left side has a radius of 24 nm, the right side has only 16 nm. Considering the impression from the SEM images, this rather moderate asymmetry gives rise to a considerable in-plane signal. However, as this signal is omnipresent, it can be easily subtracted as a background.

Material heterogeneities can possibly arise from local nonstoichiometries at the nanoscale and will result in a variation of the local electric properties. An otherwise existing electric-field symmetry will break and give rise to an asymmetric in-plane piezoelectric response that no longer cancels out at the position of the tip. Due to the high sensitivity of the in-plane piezoelectric signal, even small local variations (as small as a few percent in the static dielectric permittivity according to our finite-element analysis) will be sufficient to cause a detectable signal. This holds true irrespective of a variation in the local polarization that may go hand in hand with the heterogeneities.

The upper part of Fig. 11 shows the modified electric potential distribution in the presence of a local material heterogeneity. Here we increase the dielectric permittivity by 10% in all directions as compared to the bulk values. As a consequence, the in-plane piezoelectric response is asymmetric and the tip is significantly tilted to the left side. In the lower part of Fig. 11, we illustrate the cross section of the numerically simulated piezoelectric signal of a $BaTiO_3$ sample in the vicinity of a perturbation along

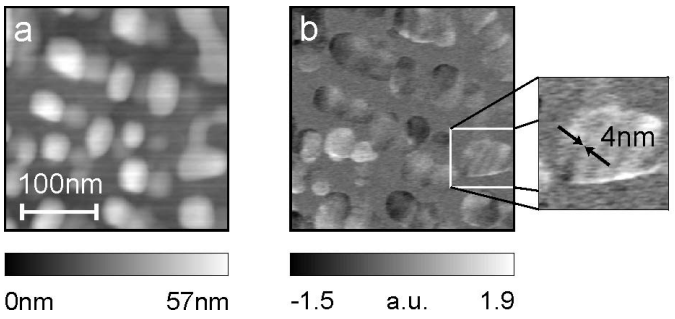


Fig. 12. Topography (part a) and in-plane (part b) piezoresponse measured on $PbTiO_3$ nanograins under UHV. Note the domain width of only 4 nm.

the polar axis. The symmetry remains undisturbed as long as the tip is sufficiently far away from the respective site (about 30 nm in our scenario). As the tip approaches the inhomogeneity, the in-plane signal increases and should rapidly drop to zero at the very center of the distortion as the symmetry is again restored. If this drop in the in-plane response can be experimentally resolved depends on the size of the variation and the experimental resolution.

## VI. Conclusions

As long as samples are prepared, stored, and transferred in an ambient atmosphere, adsorbates are an omnipresent nuisance. Especially the quantification of electromechanical coupling factors at the nanoscale is impeded unless precaution is taken. Our experimental findings indicate that the actual electric field can be overestimated by up to one order of magnitude as long as adsorbates are present. But, even for a qualitative analysis and imaging, the removal of adsorbates has an immediate consequence as the screening of the depolarization field also depends on the surface conditions. Fig. 12 shows recent in-plane PFM measurements of $PbTiO_3$ nanograins at $10^{-9}$ mbar after in-situ annealing up to 350°C. We observe a previously unachieved domain width of 4 nm (i.e., only 10 unit cells). These defined conditions of a ultra high vacuum (UHV) setup pave a reliable way to circumvent artifacts from adsorbates and to quantify measurements to compare them to theoretical predictions at the nanoscale.

Because in-plane measurements provide a significantly better signal-to-noise ratio for our cantilever geometry, we investigated a couple of specific issues of asymmetry in the tip-sample system. Radially asymmetric tips with a respective electric field cause a constant offset in the in-plane response on any sample. For the simplest case of a *c*-axis oriented sample, we used finite elements to explain the experimentally observed perimeter-enhanced, in-plane piezoresponse on the basis of sample topography without need for a modified strain distribution along the grain perimeter. In a subsequent step, we generalized this observation to other asymmetries such as imperfect tips and inhomogeneous sample properties. Calculations for lower-symmetry materials are on the way.

## Acknowledgments

The authors would like to thank Sven Clemens and Regina Dittmann for providing the $PbTiO_3$ and $BaTiO_3$ samples.

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