High-resolution ferroelectric domain imaging and hysteresis measurement by piezoresponse force microscopy with the SmartSPM™ 1000

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Piezoresponse force microscopy (PFM) is the primary tool for studying size effects of piezoelectrics at nanometer-resolution. The AFM tip acts as mobile top electrode to apply an AC voltage to the sample. The piezoelectric response of the sample, typically of the order of picometers, leads to a deflection of the AFM cantilever at the frequency of the applied AC voltage. SmartSPM™ senses this deflection in the vertical and lateral directions through built-in lock-in amplifiers and generates a map of the piezoelectricity while scanning the sample. Ideally, this allows for the reconstruction of the local piezoelectric tensor and thus the orientation of the ferroelectric polarization vector [1,2]. The high sensitivity of the SmartSPM™ for vertical deflection of the cantilever enables AFM contact mode scanning with cantilever deflections of a few nanometers which leads to low mechanical force on the sample even with stiff cantilevers. This reduces the mechanical impact on the sample and prolongs the life of the tip while assuring a stable electrical contact between tip and sample.

Figures 1 through 5 show the different PFM signals acquired simultaneously in a 1 by 1 µm contact-mode scan on a polycrystalline lead titanate sample ($PbTiO_3$) grown by chemical solution deposition on a platinized silicon wafer [3].

Settings of the PFM scan:

- 1x1 µm @ 256x256 points
- Contact force: ~10 nN
- Excitation frequency: 20 kHz
- Excitation voltage amplitude: 3 V_{pp}
- Scanning-speed: 2.5 s/line

AFM tip:

- AppNano ANSCM-PT
- Material: Silicon, Pt-Ir coated
- Tip-radius: < 40 nm
- Spring constant: 3 N/m
- Length: 225 µm

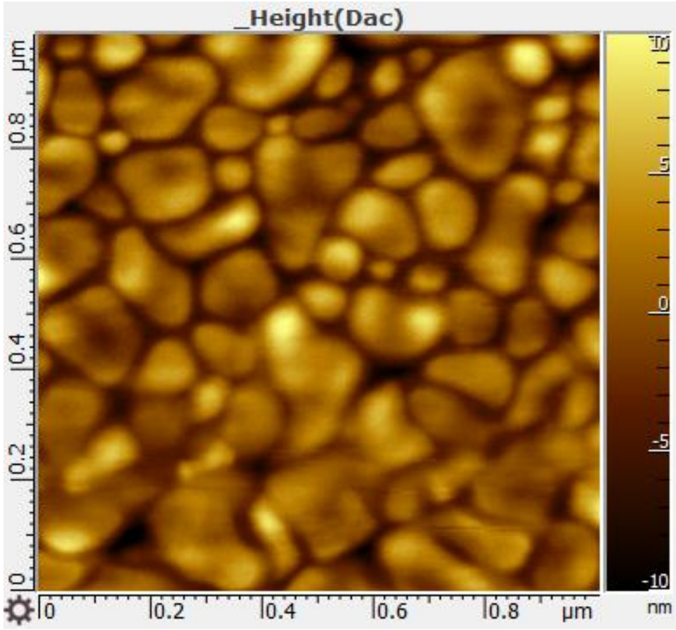

Figure 1: Contact mode topography scan of a $PbTiO_3$ sample simultaneously acquired during the PFM scan.

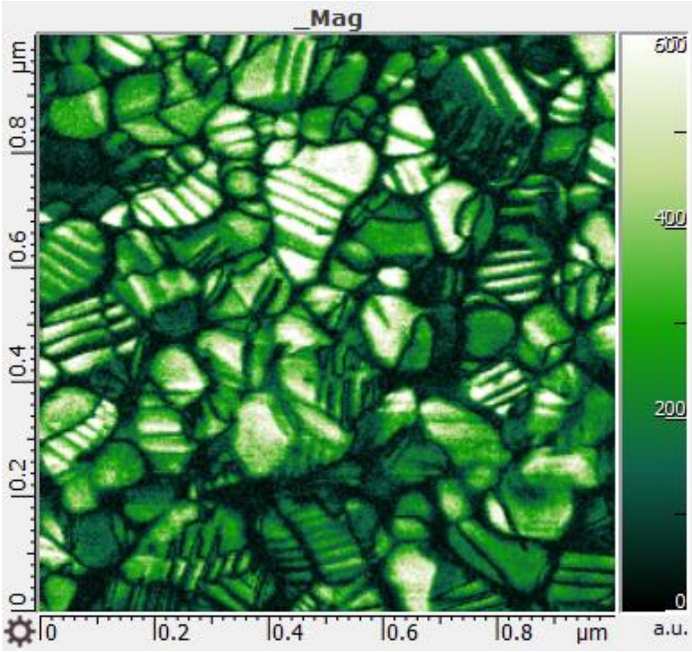

Figure 2: Amplitude of the vertical piezoelectric response.

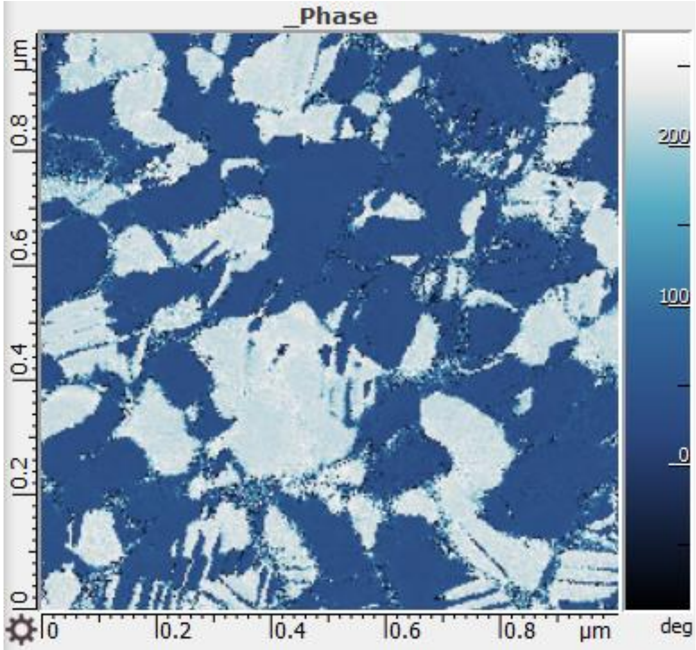

Figure 3: Phase of the vertical piezoelectric response.

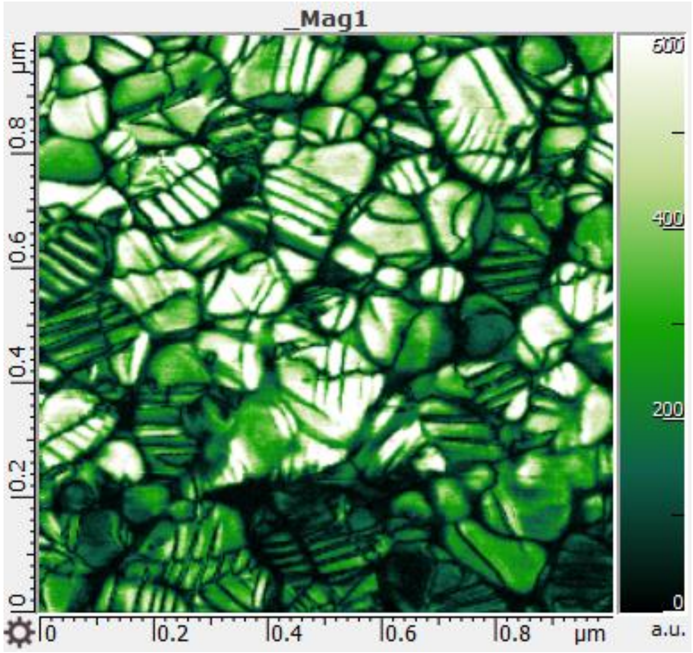

Figure 4: Amplitude of the lateral piezoelectric response.

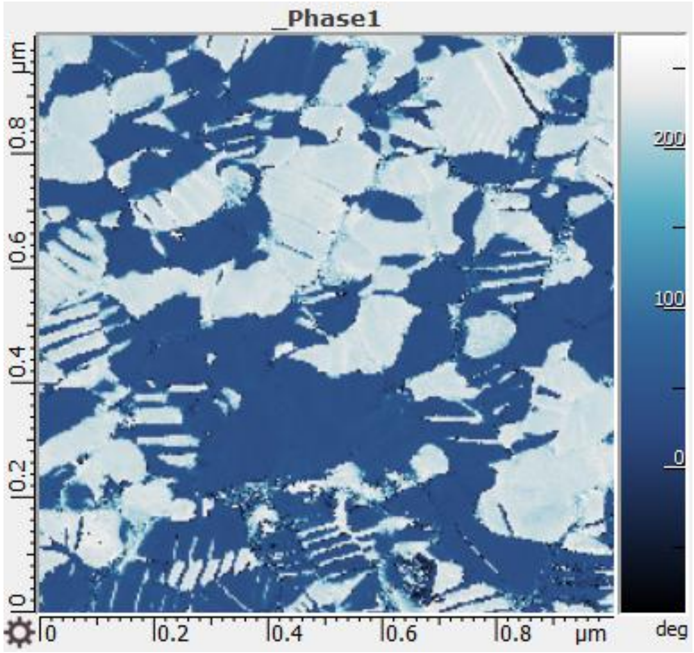

Figure 5: Phase of the lateral piezoelectric response.

The PFM scan clearly reveals a wealth of information on the complex 180 degree domain structure including examples of electrostatic domain coupling across grain boundaries. The lateral resolution of the PFM scan lies below 10 nm.

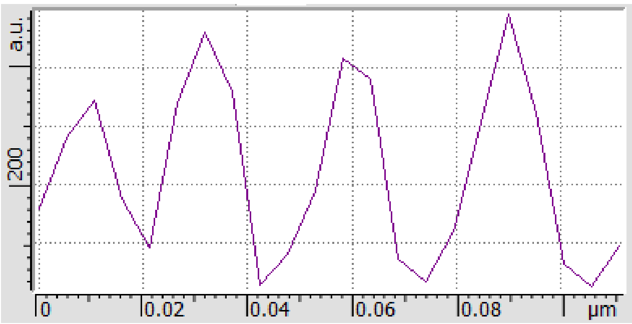

Figure 6: High resolution: Cross-section through the lateral amplitude map of a series of stripe domains with an average width of 12 nm.

In ferroelectric materials, a subgroup of piezoelectric materials, the direction of the polarization can be changed by an electric field. SmartSPM^{TM} can switch the polarization at any position of the sample by using the tip as a local top-electrode. Figure 7 shows a hysteresis curve on a domain of the above characterized $PbTiO_3$ sample. The hysteresis was measured by applying a series of DC voltage pulses and sensing the phase of the vertical piezoelectric response after every pulse. Figure 8 describes the voltage during the measurement.

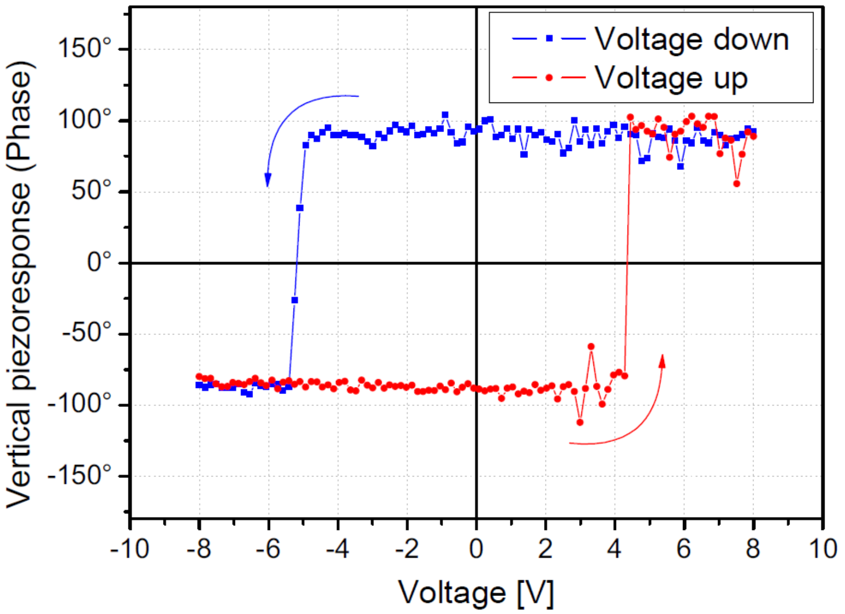

Figure 7: Hysteresis curve of $PbTiO_3$. The phase signal changes by 180 degrees when the voltage exceeds a threshold value (coercive field) at about 4 V applied to the tip. This effect is reversible at the negative coercive field.

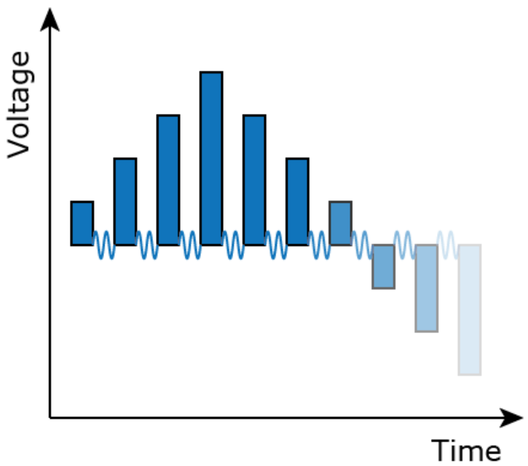

Figure 8: Sketch of the voltage during the hysteresis cycle. The DC pulses are applied to switch the domain while the PFM mode detects the phase after every pulse to measure the orientation.

The property of ferroelectrics to permanently switch polarization can also be used in nano-lithography to “write” on samples by applying a DC voltage. We show an example of reversibly switching of the polarization in a small area of the $PbTiO_3$ sample. Three slightly overlapping squares of a size of 1x1 µm were written in contact mode with 1024x1024 points at 1 second per line under a tip bias of 7 V and -7 V respectively. Figure 9 shows the vertical phase signal of a 3x3 µm PFM scan of this area revealing the previously switched area. The PFM scan was achieved with the settings described on page 1.

The overlapping of the squares shows that previously switched areas can be switched back again without loss of signal. The reason that not all domains were switched is based on the crystallographic order of the sample and the piezoelectric tensor. The applied electric field can only change the polarization of out-of-plane domains. In-plane domains are not affected by this method and remain in their original state.

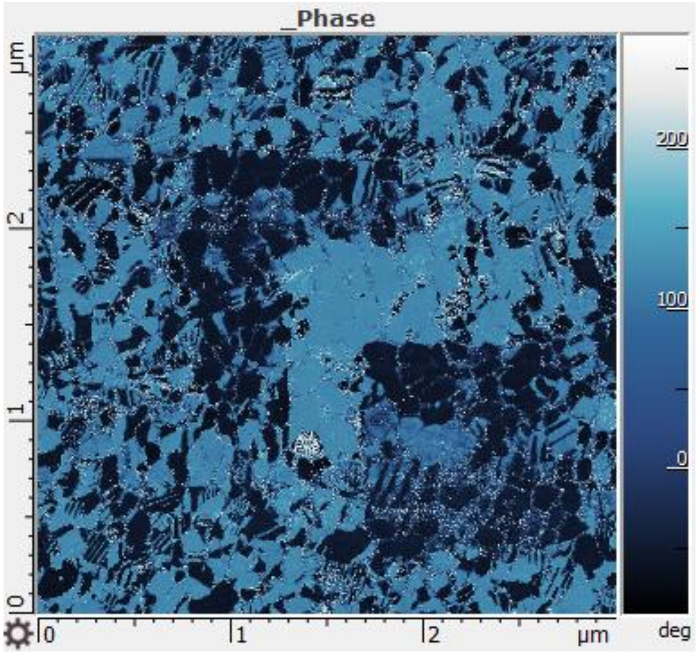

Figure 9: Vertical phase map of a PFM scan on a previously switched area. Three overlapping squares of different domain polarization are visible in the middle.

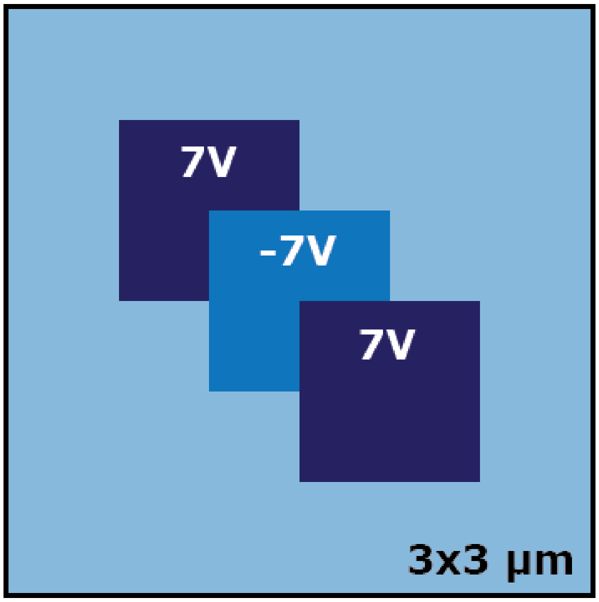

Figure 10: Sketch of the switched area with the respective DC voltages that were applied.

All scans shown in this application note were conducted with one single tip without observing any tip damage or loss of signal strength throughout the scans.

LITERATURE

[1] S.V. Kalinin and A. Gruverman (eds): "Scanning Probe Microscopy of Functional Materials: Nanoscale Imaging and Spectroscopy", Springer 2010.

[2] F. Peter, A. Ruediger, R. Waser, K. Szot, and B. Reichenberg, Review of Scientific Instruments 76, 106108 (2005)

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