NANONIS APPLICATION NOTE

EXPLORING NANOELECTROMECHANIC OF FERROELECTRICS: PFM WITH DUAL-OC4

Piezoresponse Force Microscopy (PFM) is now the primary technique for imaging, spectroscopy, and domain patterning in ferroelectric materials. Piezoresponse (PR) studies of ferroelectric materials has started in the beginning of 90s [1], and today undergo exponential growth due to rapidly emerging applications of ferroelectric and multiferroic materials for nonvolatile memories and data storage [2, 3]. These applications have stimulated extensive efforts toward understanding the mechanisms for polarization reversal in ferroelectrics on the nanometer scale.

1. Vertical and Lateral PFM

In the following we use the Nanonis controller to operate a commercial Veeco MultiMode microscope in ambient, for the investigation of thin films of ferroelectric materials.

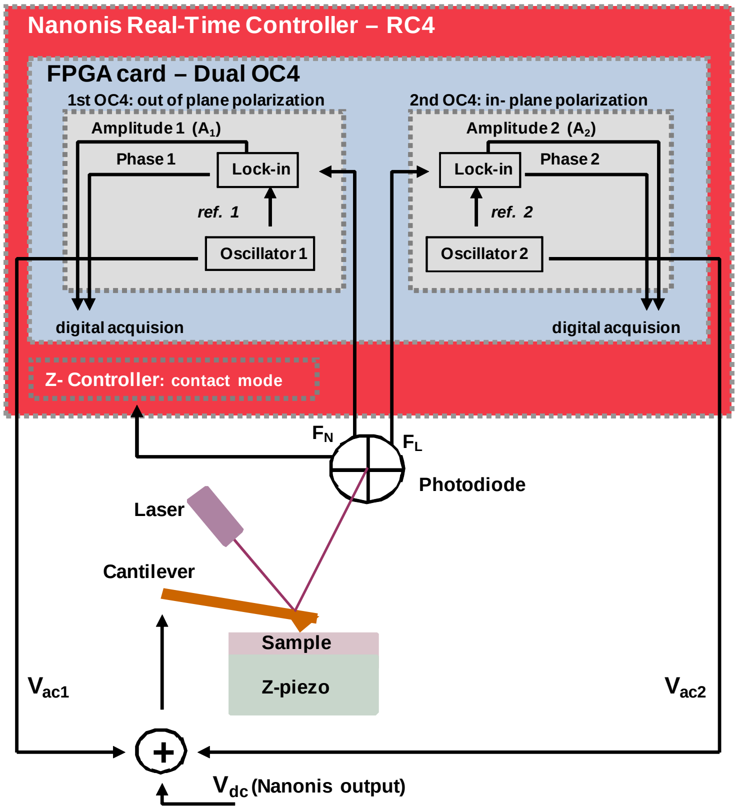

Figure 1. Schematic of the experimental setup. The vertical deflection signal is fed simultaneously into z-controller for the control of the tip-sample distance; and first OC4 for the detection of the out-of-plane PR. The horizontal deflection is the input of the second OC4, such that the in-plane polarization can be detected. The total bias voltage applied to the tip (or sample) is $V=V_{dc}(t)+V_{ac1}$ cos $(w_1 t)+V_{ac2}$ cos $(w_2 t)$.

A metallic tip is scanned over the surface in contact mode by keeping the vertical deflection constant. AC voltages at two different frequencies are produced by two OC4 devices, summed and applied to

Measurements performed during the 2nd International Workshop on PFM,EPF-Lausanne, Switzerland.

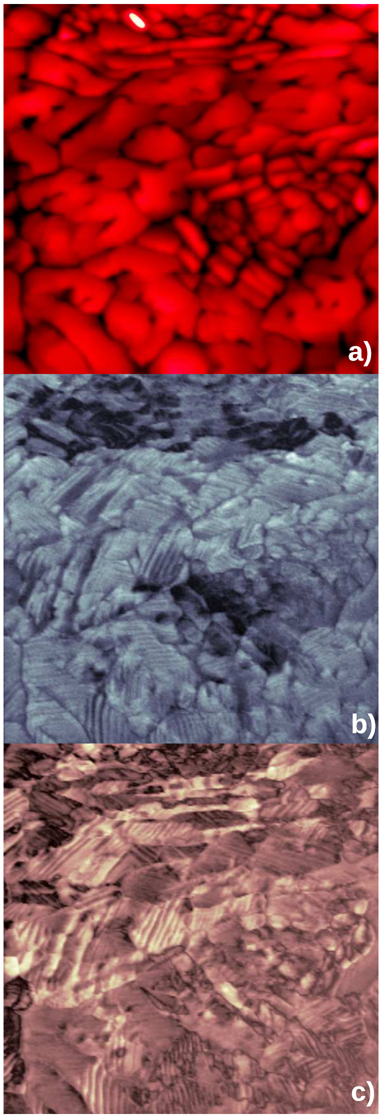

Figure 2. a)Topography, b)normal c) lateral PR images (8x8μm^2) on 1μm thin PZT thin film. f_1=31kHz and f_2=13kHz, V_{ac1}=2.8V, V_{ac2} =2V. The time constant of the two lock-ins was τ_1=1ms, lower than the pixel rate τ_2=3ms.

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the sample (or tip). Both OC4s are used as lock-in amplifiers in this experiment. Appropriate frequencies have to be chosen for optimal detection of out-of-plane and in-plane piezoelectric responses. V_{ac1} and V_{ac2} induce oscillatory vertical (F_N) and lateral (F_L) deflections of the surface due to inverse piezoelectric effect that are transferred to the tip and detected through standard beam deflection system. F_N and F_L are then fed in as inputs for the two lock-ins. The amplitude and phase of both deflection signals are monitored as direct measurements of the magnitude and orientation of the polarization in the thin film. The concept is illustrated in Figure 1. All signals of the lock-ins are easy accessible in the Nanonis controller since the OC4 devices are digitally integrated in the control system.

2. Piezoresponse Spectroscopy and Switching Spectroscopy PFM

Polarization switching in ferroelectrics exhibits hysteretic behavior that can be quantitatively studied on the nanoscale by piezoresponse spectroscopy, i.e. measurements of piezoresponse signal as function of voltage, $PR=PR(V_{dc})$ (Figure 3a). The hysteretic curves are obtained by applying a probing dc voltage to the sample superimposed on detection ac wave. The shown waveform of the V_{dc} is enabled by the Nanonis *LabVIEW Programming Interface* (Figure 3b), and can be modified to enable more complex spectroscopic measurements. The detection is performed both in the in-field and remanent states. In many ferroelectric materials including thick films and ceramics, coercive and nucleation voltages are of the order of tens of volt. For that purpose, the AUX output of the Nanonis HV-Amplifier can provide V_{dc} bias voltages up to ±400V.

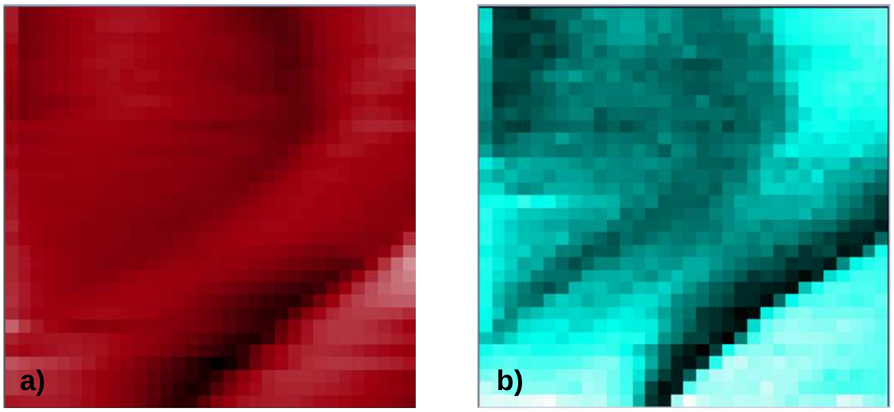

Figure 4. Topography and SS-PFM images of PZT film take on a grid of 32x32 points. SS-PFM on the same location obtained by applying V_{ac1}=3.6V at f_1=111 kHz while sweeping V_{dc} between ±40V. a) Topography of 1.3x1.3µm^2, b) slice through the 3D data for V_{dc}=0V.

The hysteresis loops can be acquired on the 2D grid of points, constituting switching spectroscopy SS-PFM [4, 5]. The implemented *Measurements on*

Hysteresis loops:

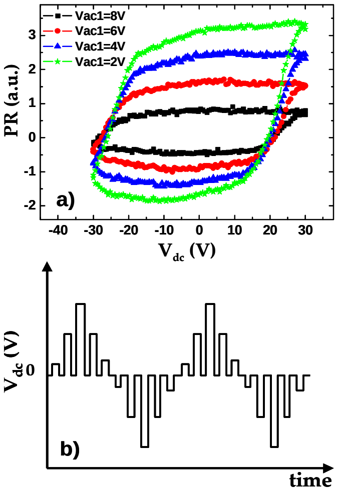

Figure 3. a) Local hysteresis curves (vertical PR) obtained for different values of V_{ac1}, b) $V_{dc}(t)$ waveform used for hysteresis curves.

a Grid module collects hysteretic curves on each point of a user definable N x M grid, see Figure 4. From the 3D hysteresis curve array parameters such as coercive bias, saturation and remanent polarization, and nucleation values are extracted and plotted as 2D maps.

3. Lithography

If the applied V_{dc} bias is large enough, it can completely reverse the orientation of the polarization. In such a way, by applying pulses of defined width and voltage along a predefined spatial pattern, the corresponding domain pattern can be "written" in the material. Notably, this property of ferroelectrics is directly utilized in ferroelectric data storage, where ferroelectric nanodomains serve as physical storage elements. The pattern can be defined by mouse click in *Lithography* module, or alternatively, the *LabVIEW Programming Interface* can be used to design complex and precise geometrical paths that will be followed by the tip when writing, see Figure 5.

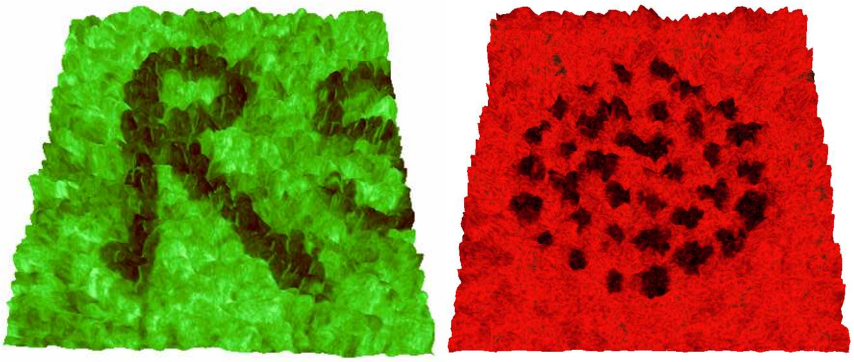

Figure 5. Writing on PZT thin films. The two images show the topography (5x5 μm^2) superimposed with the piezoresponce image (amplitude of the first lock-in) obtained after applying large pulses of -30V along a pattern drawn with the mouse (left) and one obtained using the *LabVIEW Programming Interface* (right).

Nanonis controller, together with two digitally integrated lock-ins, allow an extremely flexible, powerful, and at the same time user friendly experimental setup. Predefined software modules like *Bias Spectroscopy*, *Measurements on a Grid*, *Lithography*, together with the *LabVIEW Programming Interface* give the users the freedom to choose and customize their experiments.

All presented measurements were performed during the 2nd Workshop on Piezoresponse Force Microscopy in Lausanne, 26-29 May 2008. We thank Sergei Kalinin (ORNL, USA) for the invitation to the workshop, Nava Setter (EPFL) for the extraordinary organization, Igor Stolichnov (EPFL, Lausanne) for supplying us with appropriate samples, and all participants for their interest and active feedback.

Nanonis Modules in Use:

- Base Package
- OC4-Dual Add-on
- Veeco adaptation kit
- HV-Amp with AUX fast output: 1MHz and factor of amplification up to 40
- Nanonis LabVIEW Programming Interface

System:

- Veeco MultiMode AFM

References:

[1] O. Kolosov et al., *Nanoscale Visualization and Control of Ferroelectric Domains by Atomic Force Microscopy*, Phys. Rev. Lett. **74,** 4309 (1995).

[2] A. Gruverman et al., *Nanoscale Ferroelectrics: Processing, Characterization and Future Trends, Reports on Progress in Physics*, **69** (8) (2006).

[3] Sergei V. Kalinin et al., *A Decade of Piezoresponse Force Microscopy: Progress, Challenges, and Opportunities, IEEE Transactions on Ultrasonics and Ferroelectric Materials* **53**, 2226 (2006).

[4] S. Jesse et al., *Quantitative Mapping of Switching Behavior in Piezoresponse Force Microscopy*, Rev. Sci. Instr. **77**, 073702 (2006).

[5] S. Jesse et al., *Direct imaging of Spatial and Energy distribution of Nucleation Centers in Ferroelectric Materials*, Nature Materials **7**, 209 (2008).