Contact electrostatic force microscopy of polarization domains in thin ferroelectric films.

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Contact electrostatic force microscopy (EFM) is a promising technique for studying ferroelectric films on the nanometer scale. Of particular interest is the potentiality of the technique to probe the spatial distribution of ferroelectric properties at the film surface, namely, the orientation and magnitude of polarization and the coercive field [1,2,3]. To that end, a voltage difference should be induced between the conductive probe and the film bottom electrode. Then electromechanical response (EMR) of a ferroelectric film could be measured with a nanometer resolution, and the local hysteresis loops may be recorded. In this work, we develop this technique on the base of P47 microscope for studying thin singlecrystalline $PbZr_{1-x}Ti_xO_3$ (001) and polycrystalline $PbZr_{1-x}Ti_xO_3$ (111) films. Analyzing the polarization states in the films we take into account the parasitic capacitance contribution that is determined via measurements of local pulse and dynamic EMR dependencies on the tip-sample voltage. To collect EMR dependencies, we use the raster-lithography mode of the software of NTMDT devices. We demonstrate that more precise information on the polarization domain structure may be extracted from the measurements, when in addition to the EMR images a signal characterizing the mechanical stiffness is acquired simultaneously.

Using the developed approach we conduct the polarization domain engineering experiments. The study on threshold times and voltages to create stable in time nanodomains in $PbZr_{0.47}Ti_{0.53}O_3$ (001) films of different thickness are presented as well. Fig.1 shows a set of experimental data used in our study. In Fig.1a the image of EMR signal reveals the periodical grating of polarization domains. The grating was created by the local application of 10V in height and 1ms long impulses between the conductive NSG11/W_2C probe and the bottom electrode of a ferroelectric film. The bright contrast corresponds to the native state of the film polarization, the areas with the polarization reversal are characterized by the dark contrast. We prove this as follows. The local hysteresis loop shown in Fig.1d demonstrate two evident states of the EMR signal, they correspond to the levels of +0.4 nA and -0.4 nA. At the same time a profile taken along a white line in Fig.1a rather inambiguously manifests the very states in the signal. It should be noted, however, that not every black spot in the EMR image in Fig.1a is a domain of the opposite polarization. For example, the peculiarities of a kind marked in Fig.1a by an arrow arise due to coupling of EMR signal with the surface relief, because the topography image in Fig.1b reveals the elevation at the location of the arrow. We have found, however, that more convenient and informative way to analyze the parasitic coupling of the EMR signal with the surface relief is to simultaneously collect the signal of the local stiffness excited at the same frequency as the EMR signal. Indeed, in the region marked by the arrow in the image on Fig.1c the stiffness signal behaves the same way as the EMR signal in Fig.1a. Finally, we would like to underline that the real domain structure demonstrated by EMR signal does not correlate at all with the topography and stiffness data, as one may be convinced comparing the corresponding cross-sections in Fig1.e, f and g.

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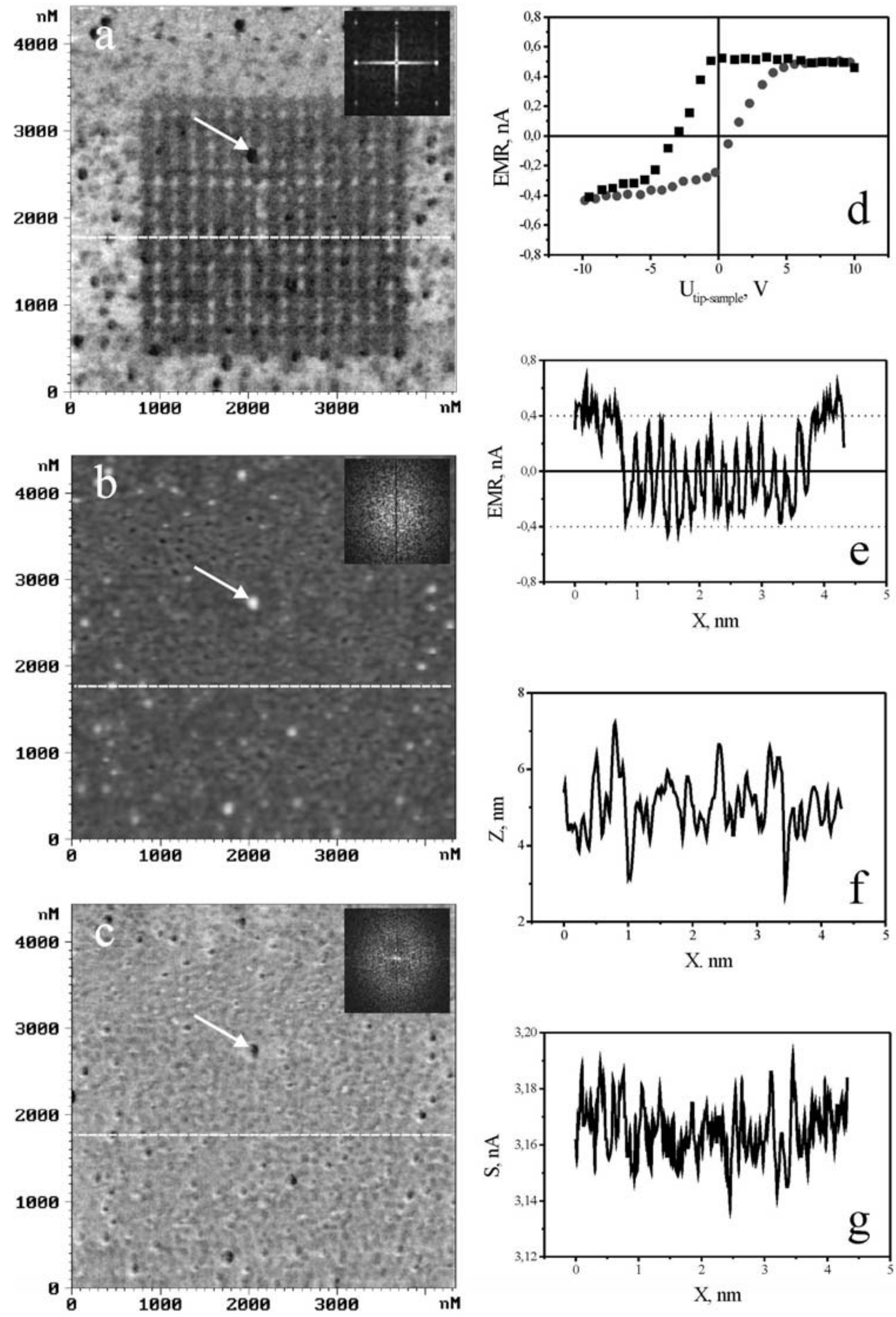

Fig.1. Contact EFM study of $PbZr_{0.47}Ti_{0.53}O_3$ (001) films of 30 nm thickness. EMR signal image **a**; parameters of signal excitation are 3V ac at 50 kHz. Topography **b** and stiffness signal **c** images were measured simultaneously with the EMR signal. Typical local pulse hysteresis dependence **d**; EMR, topography and stiffness signal profiles **e**, **f** and **g** taken along the white lines of the corresponding images **a**, **b** and **c**.

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