Time-resolved synchrotron x-ray microdiffraction for studying ferroelectric and multiferroic thin films

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We have developed an approach to studying structural dynamics in solid crystalline systems by combining the sub-micrometer spatial resolution of x-ray microdiffraction and the sub-nanosecond temporal resolution of time-resolved techniques using synchrotron radiation. The dynamics of polarization switching and piezoelectric response in ferroelectric and multiferroic thin crystalline films was studied using this approach.

The time-resolved piezoelectric response of thin 300 nm ferroelectric film of $Pb(Zr,Ti)O_3$ (PZT) is shown in Fig. 1. When the electric field is applied to a ferroelectric material the lattice spacing of the material responds to the field and this response can be measured using corresponding x-ray reflections. For the PZT films in [001] surface orientation the (002) Bragg reflection position will change if the film experiences piezoelectric distortion due to the electric field applied along [001] direction. The upper panel of Fig. 1 shows temporal changes in the lattice spacing along the [001] direction of the PZT film while applying triangular electric field pulses to the PZT film along the [001] direction. The time step here was 20 µs. The difference of the lattice parameter before and after the electric pulse is due to charging effects. The electric field dependence of the PZT lattice parameter is shown in the bottom panel of Fig. 1. Using this dependence one can determine important physical parameters including piezoelectric coefficient d_{33} that was in this case 53 pm/V. By probing other reflections such as (103) Bragg peak, which has an in-surface plane component, we have measured the lateral piezoelectric response of the thin PZT film. Using the spatial resolution of focused x-rays of ~100 nm we could apply the time-resolved technique to measuring the piezoelectric response of individual sub-micron size structural domains in multiferroic thin film of $BiFeO_3$.

To address the problem of structural transformations during polarization switching in ferroelectric and multiferroic materials we have triggered the switching using square pulses of electric field and synchronized these pulses with pulses of x-rays coming from synchrotron. The piezoelectric response of the film to the switching pulse results in a smaller unit cell length along the c-axis until the structural transformation of polarization switching propagates to the measurement spot. The piezoelectric response sign changes some time after applied electric field pulse indicating the time when the polarization switching domain wall arrives to the point of the measurement as in Fig. 2. Using this structural signature of the polarization switching we could make a map of the switching time across the sample and study such important physical parameters as the domain nucleation density, the polarization domain wall propagation directions and velocities (A. Grigoriev *et al.*, Phys. Rev. Lett. **96**, 187601 (2006)).

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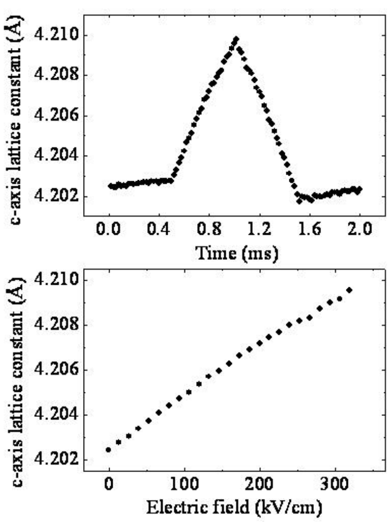

Fig. 1: *Upper panel:* Time-resolved piezoelectric response of thin ferroelectric PZT film during applied triangular electric field pulse. The c-axis paramter is derived from the position of the PZT (002) Bragg peak. *Bottom panel:* The dependence of the c-axis lattice parameter on the electric field magnitude, the data shown correspond to the piezoelectric coefficient d_{33} = 53 pm/V.

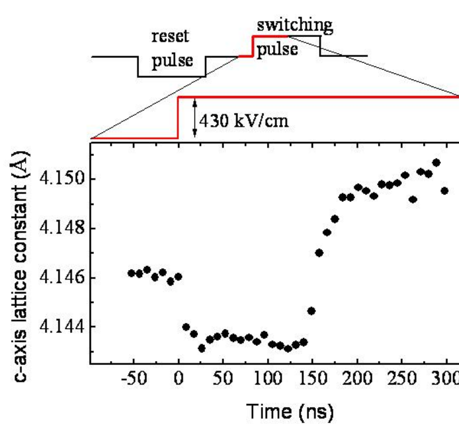

Fig. 2: Time-resolved piezoelectric response of the lattice of the thin ferroelectric PZT film during the structural transformation of polarization switching. The sign of piezoelectric effect is changed after ~150 ns when the polarization switching domain wall propagates through the measurement spot.