Experimental Report

Proposal: **1-04-75** **Council:** 10/2012

Title: Stress dependence of the domain kinetics in Strontium Titanate

This proposal is continuation of: 1-04-58

Researh Area: Materials

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Experimental Team: SIDORUK Jakob

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Samples: SrTiO3

Instrument	Req. Days	All. Days	From	To
D10	6	6	14/03/2013	20/03/2013

Abstract:

Strontium titanate (SrTiO3) is a well studied substance widely used in multilayer
ferroelectric thin films and as a substrate for high temperature superconductors. However, fundamental properties like the kinetic behaviour of the domain structure at low temperatures and the influence of an electric field are not yet fully understood.
Real-time scattering experiments at D10 have shown that the time constant of the electric field induced domain redistribution can dramatically be changed if mechanical stresses are applied.
As a continuation of the previous experiment, the present proposal aims to investigate influence of the stress dependence of the switching behaviour in detail using stroboscopic single crystal diffraction.

Stress dependence of the domain kinetics in Strontium Titanate

Scientific Background

Strontium titanate ($SrTiO_3$) is a well-known member of the perovskite family. It exhibits an antiferrodistortive phase transition at 105 K leading to a tetragonal paraelectric phase with three different structural domains (x-,y- and z-domains). At low temperatures a polar phase can be induced by the application of electric field [1].
Surprisingly, the domain distribution can be manipulated only by applying uniaxial mechanical stresses but also by electric fields, although a direct coupling between the order parameter and the electric field is forbidden by symmetry in the paraelectric regime [2].

Aim of the experiment

The aim of the experiment was to study the kinetics of the field induced domain switching in strontium titanate ($SrTiO_3$) and its variation if a competing mechanical load is applied.
Preliminary investigations showed that the domains were redistributed by pulsed electric fields along [001]-direction on a time-scale of microseconds if simultaneously a (competing) uniaxial stress of 15 MPa is applied along [110].
If the stress is, however, released the back-relaxation process becomes extremely sluggish and the corresponding time constant increases by five to six orders of magnitude.
While the previous experiments were restricted to only a single level of applied mechanical stress, the aim of the experiment was to study in detail the influence of the stress on the domain redistribution kinetics.

Experimental Details

For real-time scattering experiments stroboscopic techniques as well as a portable data acquisition system were developed in our group [3] which allows the synchronization with different neutron instruments.
For the simultaneous application of uniaxial stress, electric field and low temperature we used a CCR-cryostat which was equipped with a specially designed sample stage including a pressure cell with force sensor and vacuum feedthrough, to allow the variation of stress at low temperature. The cryostat was adapted for the use with the Eularian cradle of D10. Hence, the domain distribution could be determined

quantitatively from the intensities of three superlattice reflections ((0.5 0.5 3.5) and permutations) under various conditions. The stroboscopic neutron diffraction measurements were performed for an electric field of 4 kV/cm along [001]-direction and applying stress along [110] up to 10 MPa between 20 K and 50 K.

Results

In the as prepared state, the crystal consisted of a mixture of tetragonal x-, y-, z-domains with the respective volume fractions 21%, 36%, 43%. The application of mechanical stress along [110] favours the z-domain and at 10 MPa the z-domain fraction reached already 80%. The application of electric fields along [001] transforms z-domains back into x- and y-domains.

Figure 1 shows the time evolution of the z-domain volume under the influence of a pulsed electric field in the [001]-direction (4kV/cm/500Hz) and various applied mechanical stresses along [110] (1MPa, 5MPa, 10MPa) at 20 K. Due to the fact that stress along [110] and field along [001] represent competing perturbations, it is not surprising that the total amount of domains that can be switched by the electric field decreases with increasing stress. But the kinetics show clearly the remarkable effect that even under high mechanical load a good part of the z-domains are still able to transform on a time scale of microseconds leading to an intermediate stage after about 200 µs.

The characteristic relaxation times for the field-on and field-off phase are shown in figure 2. At 1 MPa, the driving force is rather small and, hence, the transformation after switching off the field is rather slow. If the bias stress is completely removed, the back transformation takes minutes or hours as established in previous experiments.

At higher mechanical stresses, however, the relaxation time is reduced to about 20 µs and becomes smaller than the corresponding value for the field-on phase. Even if the field induced redistribution remains rather fast, not all of the domains are participating in this process. Rather, there remain strongly pinned domains that need much longer time to approach the true equilibrium distribution which is indicated by the arrows at the right hand side of figure 1. The fraction of these pinned domains increase with increasing mechanical stress. We were also able to determine the field amplitude and temperature dependence of the kinetic behavior and observed that the

proximity of the field induced ferroelectric phase strongly affect the relaxation process.

The D10-data clearly show that electric fields strongly affect the domain structure of $SrTiO_3$ with non-trivial transformation kinetics.

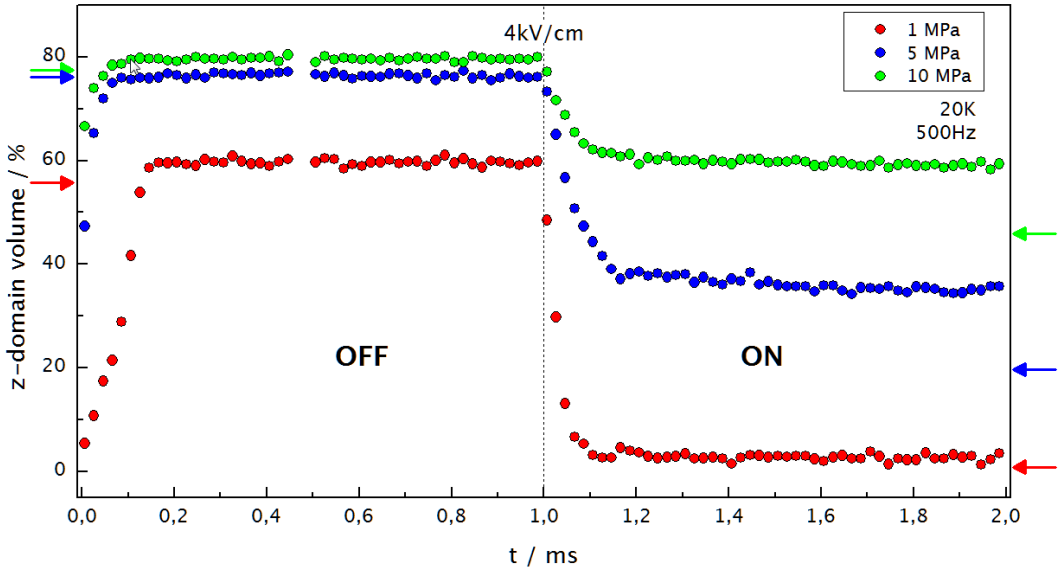

Figure 1. *Time evolution of the z-domain volume influenced by pulsed electric fields [001] 4kV/cm (500 Hz) and various mechanical stresses [110] (1MPa, 5MPa, 10MPa) at 20 K.*

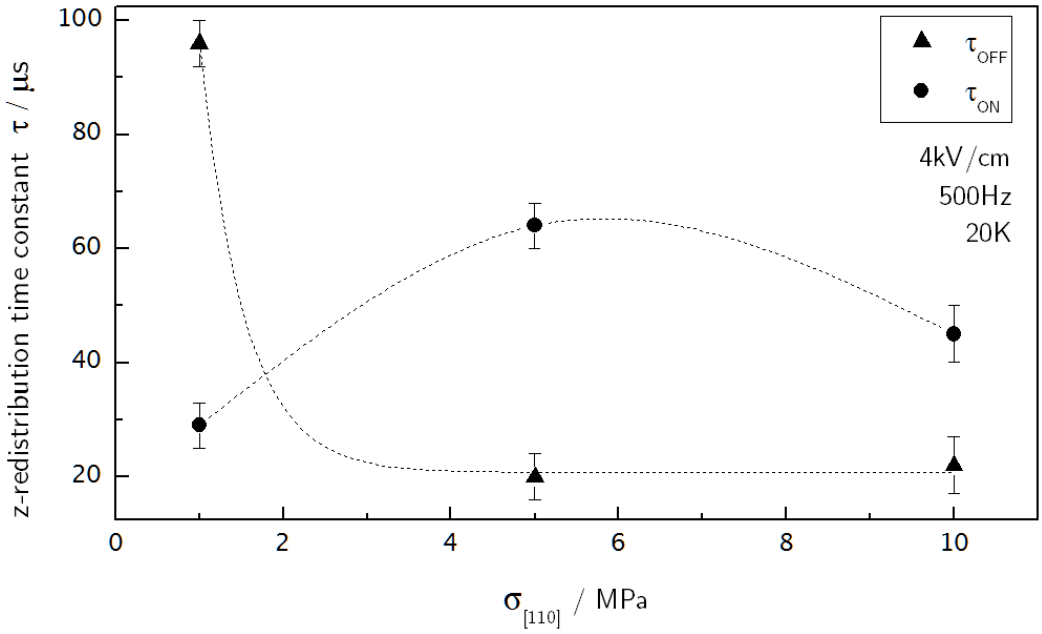

Figure 2. *Time constant of the z-redistribution volume as a function of applied mechanical stress along [110]-direction influenced by pulsed electric field [001] 4kV/cm (500 Hz) at 20 K.*

[1] J. Hemberger et al., *J. Phys. Condens. Matter* **8**, 4673-4690 (1996).

[2] J. Sidoruk, J. Leist, H. Gibhardt, M. Meven, K. Hradil and G Eckold, *J. Phys. Condens. Matter* **22**, 235903 (2010).

[3] G. Eckold, *Nucl. Instr. & Methods* **A289**, 221 (1990).