ESRF

Experiment title: Investigation of the low-T structure of $SrTiO_3$ at high x-ray energy	**Experiment number:** HS-547

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Report:

$SrTiO_3$ is a cubic perovskite at room temperature. It undergoes a cubic-to-tetragonal transition at $T_a \simeq$ 105 K. The order parameter is the staggered rotation angle ϕ of the TiO_6 octahedra around one of the four-fold axes. On further cooling, the dielectric constants increase considerably to level off at T -10 K owing to quantum fluctuations. To these high values correspond two zone center optic phonons, TO-E, and TO-A_{2u}, whose frequencies are very small at low T. The situation looked clear until recently, when K.A. Mueller et *al.*[1] reported that another transition might occur at $T_q \simeq$ 37 K. Reconsidering early as well as recent experiments, indeed weak transition-like features are often seen around T_q. This prompted us to reinvestigate the low frequency (w) and wave vector (Q) dynamics of $SrTiO_3$. Brillouin spectroscopy combined with inelastic neutron scattering suggest now that an instability of a transverse acoustic phonon might occur at a Q-value located in the inaccessible gap between both techniques, (6 x $10^{-3} <$ Q $\leq$ 2 x 10^{-2} $Å^{-1}$).[2] If so, T_q would correspond to an incommensurate (IC) transition, most likely in the c-direction of the tetragonal phase.[2] A structural analysis of $SrTiO_3$ at low-T is crucial to settle this issue.
As the effects are likely to depend greatly on sample quality, we proposed to experiment on two different samples. One polished thin plate of 2 x .2 x 8 mm^3, and a bulk sample of 3 x 5 x 3 mm^3 (the former dimensions are along [ll0], the last along [00l]). The platelet is similar to the EPR samples, which become monodomain below T_a owing to *built-in stresses.* Time only allowed to investigate-the plate, and a continuation is needed to perform the second part of the proposal.
We first measured the T-dependence of the lattice parameters a and c, using the high resolution configuration of the spectrometer. The d-spacings of the monochromator, the sample, and the analyser were selected as close as possible: we used perfect Si-(711) as monochromator and analyser, and measured the (005) and (333) reflections of the plate. The incident energy was fixed at 116.858 KeV to match the angular range of the spectrometer. The Bragg condition is then fulfilled at small diffraction angles, $\theta \simeq$ 4 deg. The optical interferometer was used to control the analyser rotation. This setup has a precision of .1" for the rotation step, corresponding to a resolution of $\Delta d/d$ = 3.5 x 10^{-5}. Although

the mosaic of the sample was found space dependent (certainly owing to inhomogenous stresses), it was easy to find regions were $\Delta\theta_{sample} \leq 2$". Before each point, θ_{sample} was re-centered, to compensate for a possible small rotation. Then, a θ-scan of the analyser (θ_{an}) was performed. Exemples are shown in Fig. la. The lines correspond to fits with a Gaussian. The resulting values for d_{001} = c and $d_{111}/\sqrt{3}$ are presented in Fig. lb. The data were normalized with the value $a(110K)$ = 3.898475 A. Fig. lc, zooms on the low-T region for two sets of d_{001} measurements. The difference between the curves could originate from spectrometer drifts, but could also be real, depending on either the position in the sample or the sample history. These data constitute the first high resolution X-ray results obtained inside monodomain platelets of $SrTiO_3$. These results seem to confirm anomalies found in the EPR experiments, where ϕ might have a similar behaviour. It is interesting to notice that an anomaly does not seem to occur on d_{111}.

We also attempted to see IC-reflections on image plates. To this effect the sample was rocked around [1$\bar{1}$0], with q either along [110] or [00l], the plates being exposed for a fraction of a minute. Although nice images of the reciprocal space could be obtained, the set up was not sufficiently reliable to draw definite conclusions. Similarly, we made θ-scans around [111] at 5K, 20K, 40K, and 50K, using an elongated resolution ellipsoid. This did not reveal IC reflections either. It is definitely possible that IC-reflections are too diffuse on a polished platelet to allow for their observation, should they exist. Therefore, similar measurement on the high quality bulk sample are needed to be conclusive.,

[l] K.A. Mueller, W. Berlinger, and E. Tosatti, Z. Phys. B 84, 277 (1991).

[2] B. Hehlen, L. Arzel, A.K. Taganstev, E. Courtens, Y. Inaba, A. Yamanaka, and K. Inoue, Phys. Rev. B 57, R13989 (1998).

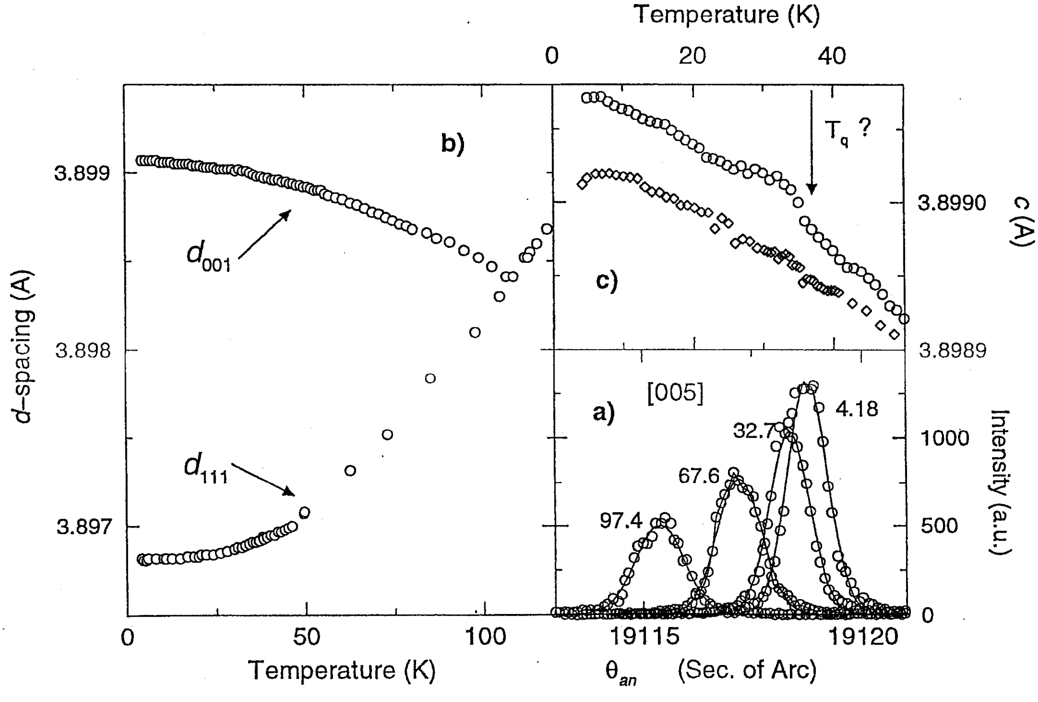

Figure 1: a) θ_{an}-scans of the [005]-reflection at four values of T. b) d-spacings, and c) a zoom on two measurements of d_{001}, one of them showing a possible anomaly near T_q.