ESRF	**Experiment title:** Investigation of the low-T structure of $SrTiO_3$ on a bulk sample	**Experiment number:** HS-983
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Report:
$SrTiO_3$ is a simple cubic perovskite at room-T. It undergoes a cubic-to-tetragonal structural transition at $T_a \simeq 105$ K owing to an instability in the rotation of the TiO_6 octahedra at the R-corner of the Brillouin zone. In the tetragonal phase, the dielectric constants increase considerably on cooling, to saturate at He-temperature but the ferroelectric transition is suppressed. All this was believed to be well understood until K.A. Müller *et al.* discovered an EPR anomaly suggesting another transition, which they thought might be non-structural, at $T_q \approx 37$ K [1]. Experimental indications for this new anomaly are quite numerous (see proposal and report HS-547). Indeed, no clear structural signature had been identified by the time of the present experiment. The purpose of the experiment was to use penetrating x-rays to probe the bulk, in order to obtain information on domains that are not much perturbed by the sample surfaces. Excellent single crystals were available for this experiment.
As we started the experiment, the analyzer was not operational. Thus we decided to proceed with $(\theta, 2\theta)$-scans in order to observe the distribution of ferroic domains below T_a and to determine whether anything unusual occured near T_q. In particular, it is of interest to measure at the R-point of the Brillouin zone, as the structure factor is then most sensitive to oxygen displacements. A typical scan, illustrating the presence of twins below T_a, is shown in Fig. 1. Several such scans were obtained for different values of $[h/2\ k/2\ l/2]$. The main feature is that each reflection breaks up into several peaks corresponding to the ferroic twins, as described for example in [2]. The intensity of these individual peaks does not show anything abnormal on crossing

T_q, as opposed to an earlier report [3]. The spacing of the domain reflections is proportional to the lattice parameter ratio, c/a. Therefore this spacing becomes smaller on approaching T_a, as illustrated in Fig. 1.

As second measurement, we studied the [0 0 6] domains. The associated splitting directly determines the c/a ratio. The result obtained is shown in Fig. 2 (circles), where it is compared to the thin-plate data derived in HS-547 (solid line), and to literature data obtained by dilatometry [4] on a bulk sample (diamonds). In the latter case, the sample had been forced to become a "single tetragonal domain" by application of pressure transverse to the c-axis. The agreement of the present determination with HS-547 is excellent, although the measurement without analyzer is certainly less precise, which could also account for the observed fluctuations. This agreement shows that c/a determined on a polished thin plate is practically the same as that which controls the angle between ferroic domains. The dilatometric data seem to suffer from the difficulty in making a large sample truly single tetragonal domain. This might account for the lower c/a value that is obtained in that case.

In conclusion, $(\theta, 2\theta)$-scans on good quality single crystals do not reveal any obvious anomaly at T_q. It remains to establish whether this is also the case for the much higher precision that could be achieved with three-axis diffractometry.

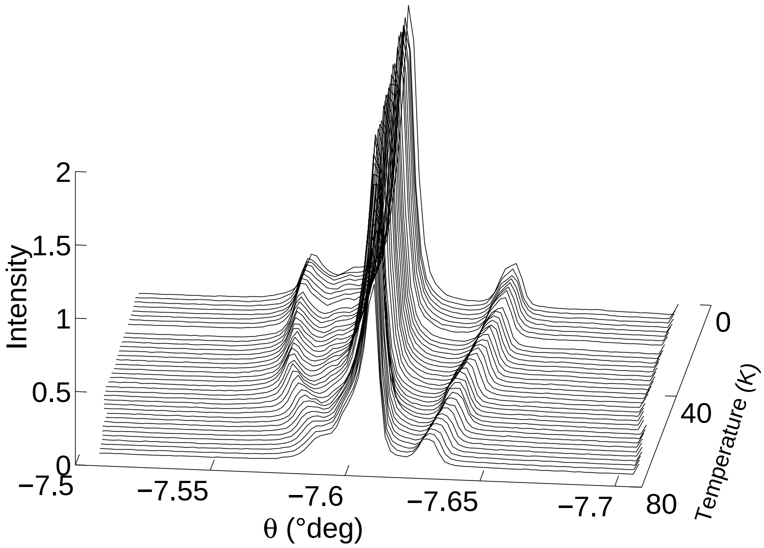

Fig. 1: Scans of the [6.5 5.5 0.5] reflection showing the temperature evolution of twin domains.

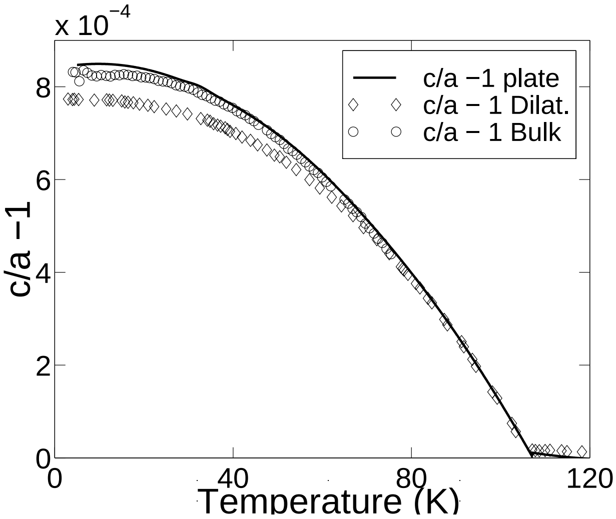

Fig. 2: Different determinations of the c/a ratio as explained in the text.

[1] K.A. Müller, W. Berlinger, and E. Tosatti, *Zeit. Phys. B* **84** (1991) 277.

[2] H.-B. Neumann *et al.*, *Phase Transitions* **55** (1995) 17.

[3] H. Hünnefeld, private communication.

[4] Mao Liu, T.R. Finlayson, and T.F. Smith, *Phys. Rev. B* **55** (1997) 3480.