	Experiment title: <i>Fluctuations of the Central peak phenomenon in SrTiO₃ studied by XPCS</i>	Experiment number: hs2196
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Shifts: 15	Local contact(s): Abdellatif Moussaid	
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Report:

The cubic-to-tetragonal displacive phase transition in SrTiO_3 has been extensively studied by X-ray and inelastic neutron scattering, leading to the observation of two unexpected features: respectively, the Lorentzian-squared *narrow component* (second length-scale) and the *central peak*. While far above T_c , the fluctuation time-scale is governed by the inverse soft-phonon frequency, a few K above T_c , a new feature appears in the inelastic neutron scattering data: a narrow *central* (i.e. zero-frequency) line, whose weight grows critically on approaching T_c . This component, unlike the narrow component seen in high-resolution X-ray diffraction, is a *bulk* feature and EPR measurements have set an upper bound of $\Delta\nu < 0.6$ MHz for its frequency. These results lend strong support to a defect-induced mechanism, such as vacancies or interstitials for which the relaxation process is coupled linearly with the soft-phonon oscillatory motion. A direct measurement of the characteristic *collective* relaxation time associated with these defects is however still missing and photon-correlation techniques appear particularly well-suited for such type of investigation: 15 shifts on Troika have been obtained to measure those relaxations.

Results:

A SrTiO_3 single-crystal, grown by the top-seeded technique, with a $4 \times 4 \text{ mm}^2$ polished [110] face, was mounted in an Orange Cryostat and aligned with a $(3/2 \ 1/2 \ 1/2)$ superstructure reflection in the horizontal scattering plane. The conditions to get a coherent beam at 8 keV were obtained by limiting the beam divergence and by using a $10 \text{ }\mu\text{m}$ pinhole just before the sample. The overall degree of coherence at low scattering angle, was estimated at $\beta = 0.04\%$, from a test silica aerogel sample. We used a point detector coupled to an auto-correlator as well as a directly illuminated CCD camera. With a $22 \text{ }\mu\text{m}^2$ pixel size, the Q-resolution of the CCD set-up was better than $\Delta Q/Q = 4 \cdot 10^{-5}$. The beam and temperature stability of the Orange cryostat were excellent.

In order to pre-orient the sample inside the cryostat a sample holder was designed and machined from a copper block, in our laboratory (see figure 1).

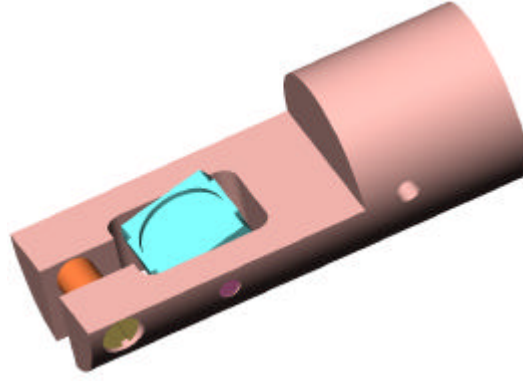


Figure 1: Sample holder mounted at the end of the cryostat stick used to preorient the sample.

The first goal of the experiment was to identify the contributions from the narrow and broad components. With 8 keV photons, the beam penetration depth $\sin\theta_i/\mu = 3.8 \text{ } \mu\text{m}$ ($\mu^{-1} = 17 \text{ } \mu\text{m}$; $\theta_i = 13^\circ$). Hence the contribution from the narrow component, which originates from a defect-rich, a few 100 μm -thick, near-surface layer is expected to be dominant in our case. However a radial cut across the CCD image in Fig. 2b at $T_c + 3 \text{ K}$ ($T_c = 106 \text{ K}$), shows an intensity profile intermediate between Lorentzian and Lorentzian-squared. The side maximum observed to the right of the main peak may arise from a weak contribution from the bulk, off-centered due to elastic strains in the skin-layer. Fig. 2a at $T_c - 3 \text{ K}$, shows a very anisotropic intensity distribution, with a sharp streak to the left of the main peak, and possibly a second streak oriented at 90° from the first one. The streaks are oriented along the $\langle 100 \rangle$ directions and are probably due to the incomplete orientational ordering of the oxygen octahedra along the staggered rotation axis (platelet-like anti-phase domains). The origin of the intensity distribution along the streak cannot be ascertained for the time being. In particular its decay rate with distance Δq from the main peak ($\Delta q_{1/2} = 0.003 \text{ rlu}$) may be partly due to the fixed-crystal diffraction technique used.

The grainy aspect of the CDD-camera pictures is characteristic of a speckle-like pattern which proves the feasibility of performing speckle experiments on this system. Nevertheless, the weak degree of beam coherence obtained at $\theta_i = 13^\circ$, with a $20 \mu\text{m}$ pinhole ($\beta = 1\%$) do not allow to draw definite conclusions concerning the dynamics of the fluctuations. At the start of the experiment, one beam-day has been spent working on the beam quality, without succeeding in obtaining a clean focussed beam profile. The poor quality of the Be lenses is likely to be at the origin of the low degree of coherence achieved. Furthermore, the experiment has been long to set up (work on the beam quality, installation and cooling of the cryostat, sample alignment) and it was not possible to systematically study the relaxation time with temperature (only 15 shifts have been obtained).

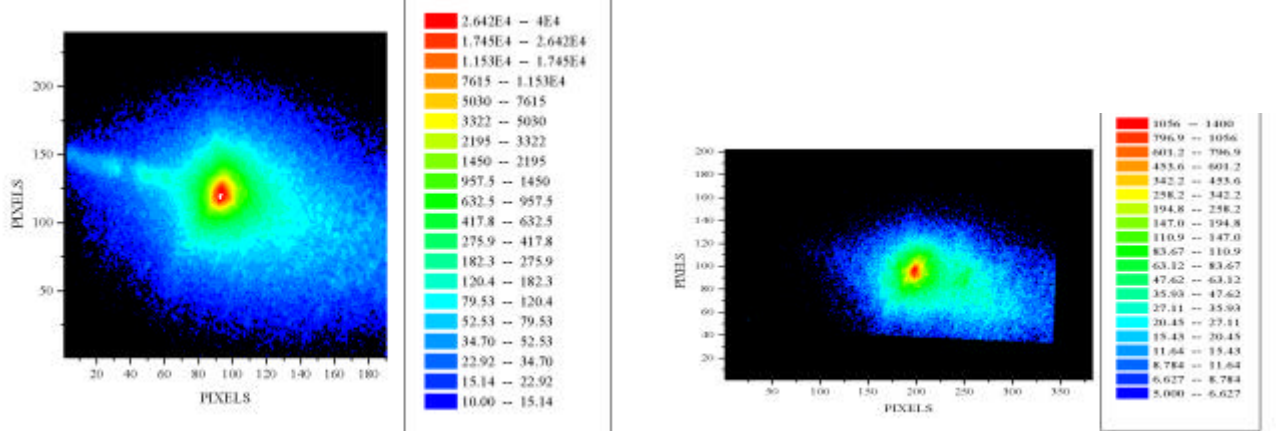


Figure 2: $(3/2 \ 1/2 \ 1/2)$ superstructure reflection at $T = 103 \text{ K}$ ($T_c - 3 \text{ K}$) (left) and $T = T_c + 3 \text{ K}$ (right) with 1000s of acquisition time.

