



	Experiment title: Observation of the structure of ferroelectric SrTi ¹⁸ O ₃ using ID31 High Resolution Powder Diffractometer	Experiment number: HS-4538
Beamline: ID-31	Date of experiment: from: 02/09/2011 to: 06/09/2011	Date of report: 18/01/2012
Shifts: 12	Local contact(s): Caroline Curfs	<i>Received at ESRF:</i>
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Report:

The experiment was aimed at verifying the structure of the ferroelectric phase of SrTiO₃. Since the discovery[1] of the isotope-induced ferroelectric phase transition in SrTi¹⁸O₃ (STO18) neutron diffraction experiments[2] determined that the space group of the resulting ferroelectric phase is orthorhombic. Our density-functional theory (DFT) calculations[3] indicated that STO18 assumes *Ima2* symmetry, but could not produce an accurate estimate of the lattice parameters of the new structure. We decided to use ID-31 to collect high-resolution x-ray diffraction data to determine the lattice parameters of STO18 in its ferroelectric phase.

We used a powder sample of SrTiO₃ that contains approximately 60% ¹⁸O (estimate based on room-temperature Raman measurements). This concentration of ¹⁸O corresponds to the ferroelectric transition temperature of 15 K[4]. The sample was placed in a helium cryostat to allow us to reach the required temperature range. The powder patterns were collected using 0.4Å wavelength.

The powder patterns were collected over a range of temperatures:

1. 3.6 K (base temperature)
2. 4 - 23 K at 1 K intervals
3. 32 - 60 K at 3 K intervals
4. 100 - 300 K at 50 K intervals

The lattice parameters obtained from Rietveld refinement of the pattern collected at the base temperature (3.6 K) were 0.1% (c) and 0.05% (a) smaller than for regular SrTiO₃ [5]. At 300K the lattice parameter is 3.9056 and is 0.06% larger than in regular SrTiO₃.

The attempts to fit the powder pattern at the base temperature to the orthorhombic $Ima2$ space group did not result in an improvement in R-factor compared to the tetragonal $I4/mcm$ space group that is present in regular SrTiO₃. The best R-factor achieved for each of the structures was 14.6%. However, in a sequential fitting of the powder patterns, if the tetragonal symmetry is assumed throughout the entire temperature range, a change is visible in the fitted parameters such as the isotropic thermal parameters U_{iso} . Figure 1 shows that the linearity of the fit of U_{iso} of Ti is broken at 8 K.

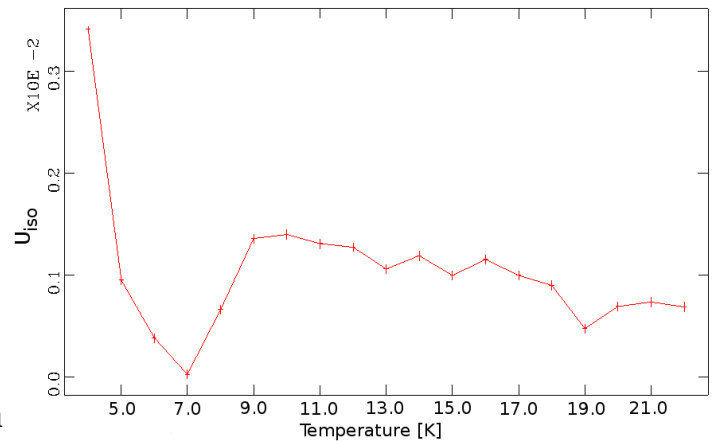


Figure 1: Isotropic thermal parameter of Ti in $I4/mcm$ tetragonal structure of STO plotted against temperature. The fit breaks down at 8K.

The refinement of the powder patterns shows that the distortion into the orthorhombic phase is very subtle (that is, the difference between a and b is smaller than 0.001 \AA). The effect of the isotopic substitution on the overall volume of the unit cell is clearly visible, but determination of the structure of the ferroelectric phase of STO18 is not conclusive. Possibly the concentration of ¹⁸O in the sample is lower than originally established, as there is change in the sample structure at 8 K and not at 15 K as expected. It is possible that the experiment would produce conclusive results if repeated on a sample with a higher content of ¹⁸O.

References:

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