EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal (“relevant report”)

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *“preliminary report”),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as “relevant report(s)” in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** **Lattice dynamics and phason modes of the incommensurate composite [Sr]1+x[TiS3]**	**Experiment number**: HC-4743
Beamline:	**Date of experiment**: from: 10/11/2021 to: 16/11/2021	**Date of report**: 17/10/2022
Shifts:	**Local contact(s)**: L. Paolosini	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

* Geoffroy de Laitre, UGA, CNRS , Grenoble-INP-UGA, SIMaP, Grenoble France
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Report:

Aperiodic crystals are long range ordered structure lacking lattice translation. They are generally classified in three categories: incommensurately modulated phases, incommensurate composites and quasicrystals. Their structure is now well understood and best described using the superspace approach [1].

The long-range aperiodic order brings in new modes in the excitation spectrum named phasons modes. They are related to the degree of freedom resulting from the high dimensional space description. Phason modes have been predicted to be diffuse excitations in the long wavelength limit and have been observed as damped phonon modes in a few incommensurately modulated phases and as purely diffusive excitations in quasicrystals [1; 2].

The existence of phason modes and their characterisation in incommensurate composites remains controversial, and we propose to carry out a detailed lattice dynamics study in a well-characterised incommensurate composite crystal. Two sublattices, whose period is incommensurate, and that might interact with each other, characterise composites. We have chosen the Sr1+xTiS3 system. The TiS3 network forms host channels in which the guest Sr atoms are located [3]. The two sublattices have strong interactions leading to an inter-modulation and characteristic satellites reflections. In this case the diffraction pattern is characterised by the common a and b lattice parameter and the c1 and c2 parameter of the host and of the guest respectively (c1/c2 is an irrational number). Four indices h, k m1, m2 are thus necessary for indexing the diffraction pattern. Bragg peak with indices of the form (h k m1 0) are related to the host lattice, with (h k 0 m2) to the guest lattice, and when the four indices are non zero, this corresponds to the so called satellite reflections.

Phason modes in the case of composites, correspond to the relative 'sliding' of the two sublattices (the phase of the modulation can vary continuously) and are sometime refer to as sliding modes. Few theoretical predictions are available (see for instance [4; 5]).

New single crystals in the form of needles a few hundred mm in length and with a diameter of the order 50 mm have been grown in the crystallography laboratory, University of Bayreuth, by the group of Sander van Smaalen. They have been characterised by x-ray diffraction on the ID28 side station, with strong satellites visible and a small amount of diffuse scattering. Their quality matches the previous samples, but their size are about 20 times bigger. They have been mounted inside a quartz capillary for temperature studies up to 900 K.

Severall samples have been tested in the temperature range from RT to 600°C. A clear transformations occurs above 300°C, with most eof the weak satellites vanishing. However the different anisotropy of the diffuse scattering is still preserved at high temperature and does not seems to change as illustrated in the Figure 1. When cooling down the sample we oberved some extra peaks together with the initial RT state. Heating up to 400°C results in a fully revesible transition.

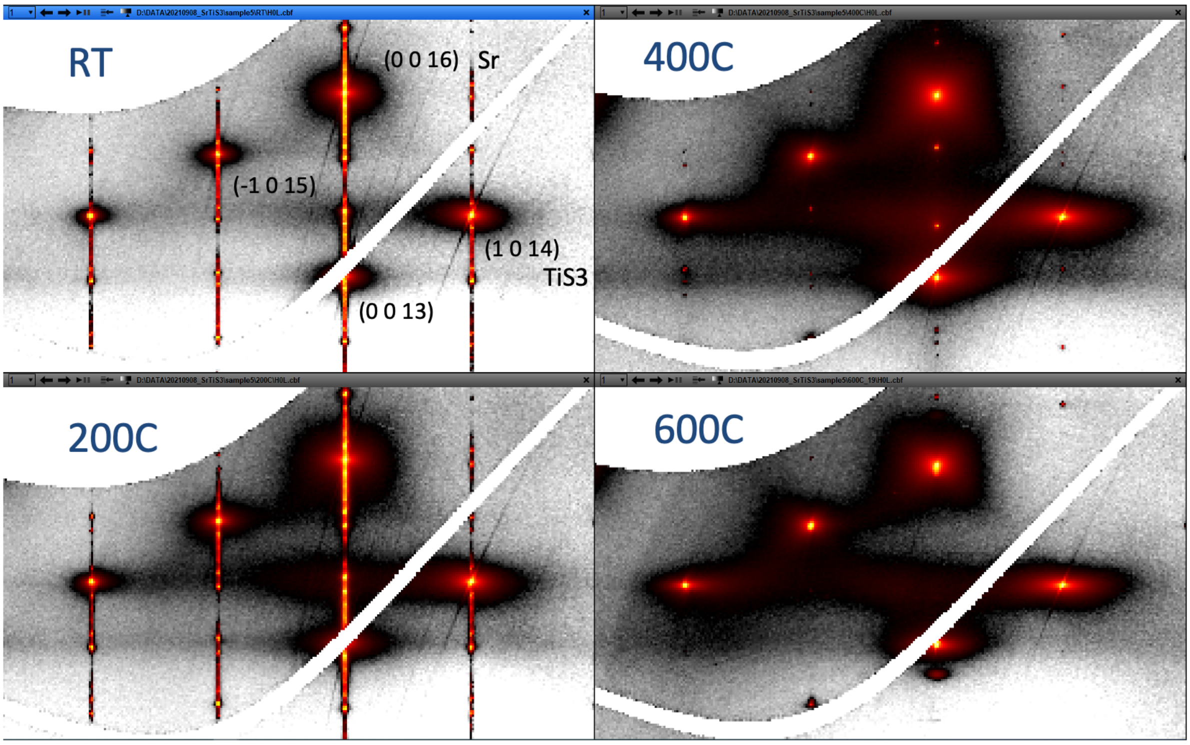

Figure 1 : Evolution of the Bragg and diffuse scattering in the the Sr(1+x)TiS3 phase. Most of he weak incommensurate satellites have disapperaed at 400°C

The same sample has then been mounted on the ID28 inelastic scattering spectrometer. Reference measurements have been carried out at RT. Following carefully the intensity distribution untill the weak satellite disapeared, measurement where carried out again at 300°C. We have not observed any significant differences between the two températures . It has to be noticed however that a low energy mode at about 3-4 meev seems to be present at both T.

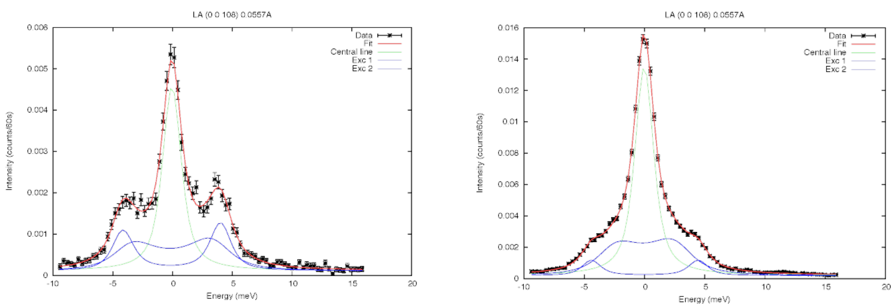

Figure 2 : comparison of the signal measured at RT (left) and 300°C (right). The lo energy excitation is still present.

References

1. Janssen T, Chapuis G and de Boissieu M, Aperiodic Crystals. From modulated phases to quasicrystals (second edition), 560 pages (Oxford University Press, Oxford,2018)
2. de Boissieu M, Currat R and Francoual S 2008 in Handbook of Metal Physics: Quasicrystals (eds. T. Fujiwara and Y. Ishii) 107 (Elsevier Science)
3. van Smaalen S, Incommensurate Crystallography, pages (Oxford University Press, 2012)
4. Janssen T, Radulescu O and Rubtsov 2002 European Physical Journal B 29 85.
5. Radulescu O and Janssen T 1999 Phys. Rev. B **60** 12737.