EUROPEAN SYNCHROTRON RADIATION FACILITY

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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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal 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:** The charge density wave in superconducting CaC_6	**Experiment number**: 28-01 898
Beamline: BM28	**Date of experiment**: from: 01 April 2010 to: 06 April 2010	**Date of report**: 10 December 2010
Shifts: 18	**Local contact(s)**: Peter Normile	*Received at ESRF:*

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Report:

The discovery of superconductivity at 11.5 K in CaC_6, a graphite intercalation compound, has motivated a wealth of research into understanding the superconducting mechanism in this material. Another electronic ground state which manifests within this system is that of a charge density wave (CDW) as evidenced by recent STM and STS data. The co-existence of charge ordering and superconductivity within graphitic materials strengthens the already existing analogy with layered high-T_c materials. In order to further characterise the CDW state in CaC_6 we intended to utilise single-crystal x-ray diffraction to observe the charge superlattice and compare its periodicity with that of STM data. Additionally we aimed to measure the temperature dependence of the CDW peaks and the CaC_6 lattice parameters.

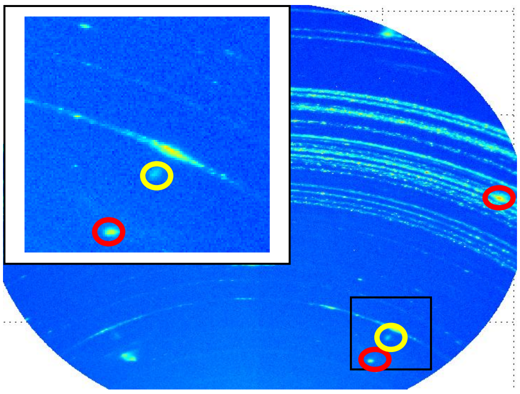

Figure 1 – shown above is a MAR CCD image taken at 15 K on a CaC_6 sample. The peaks highlighted in red are the (100) and (200), respectively, from bottom to top. The peak highlighted in yellow has yet to be identified. The insert shows this peak in more detail, accompanied by the (100).

CaC_6 samples were grown via a molten alloy method at University College London. The samples were characterised using in-house x-ray diffraction and transported to the ESRF in an argon atmosphere to inhibit oxidation. The samples were loaded onto copper stubs, specific for transmission geometry, i.e. plate-like single crystal samples were mounted vertically in order for the scattering vector to probe the in-plane charge structure. The stubs were then mounted onto the end of a displex, encapsulated by two Beryllium domes.

We attempted to measure the temperature dependent, in-plane scattering from CaC_6, using the newly acquired MAR CCD camera. However, the FIT 2D software required to automatically calculate reciprocal space positions highlighted on the 2D images was not working as yet, so these had to be calculated a posteriori. Figure 1 shows a very recent result from the MAR CCD camera, the (100) and (200) peaks are highlighted in red and the (100) is shown in the insert. The peak that is highlighted in yellow is yet to be identified and lies at the (1.16 0 0) position. Two very weak satellites accompany this peak and lie either side of the [100] direction. This image was taken at 15 K, however the peak has been seen up to 300 K. The precise temperature dependence could not be achieved due to stability problems with the PIDs of the displex. However, these initial results and the fact they these unusual peaks are present at low T and at 300 K are of considerable interest, since extra peaks are expected from the charge structure seen using STM. Further experiments to more precisely determine the in-plane lattice parameters as a function of temperature and to measure up to 800 K in a cryofurnace would be of great benefit, as would a comparison with other GIC systems.

In summary, we have observed some unusual diffraction peaks in unexpected regions of reciprocal space, but need further measurement to probe the temperature dependence.