EUROPEAN SYNCHROTRON RADIATION FACILITY
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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**: 28-01 923
Beamline: BM28	**Date of experiment**: from: 08 September 2010 to: 14 September 2010	**Date of report**: 10 December 2010
Shifts: 18	**Local contact(s)**: Laurence Bouchenoire	*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.

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: oxidation was prevented via the use of kapton and mylar shrouds. This eliminated the problem of scattering from a beryllium dome which had obfuscated images in previous experiments. A cryofurnace was used to enable access to a wide temperature range (from ~10K up to ~800K where the samples are expected to decompose).

We have made the first measurement of the temperature dependent, in-plane scattering from CaC_6. Detailed diffraction images were taken using a MAR CCD camera around the (100), (110) and (200) Bragg peaks. These images demonstrated the presence of satellites surrounding the Bragg peaks which are not expected from the ideal CaC_6 structure and are approximately 100 times weaker in intensity than the primary peaks. The position of these peaks can not yet be explained from the periodicity of the charge ordering observed via STM. Images of these peaks were taken over a wide temperature range from 10K up to 800K; interestingly the satellite peaks are present at all temperatures measured.

Monitoring the lattice parameters over such a wide temperature range was performed to search for a structural transition indicative of a CDW condensation. However, no such transition was found: instead only a slight decrease in the in-plane lattice parameter was observed as the temperature was raised as is expected for most GICs.

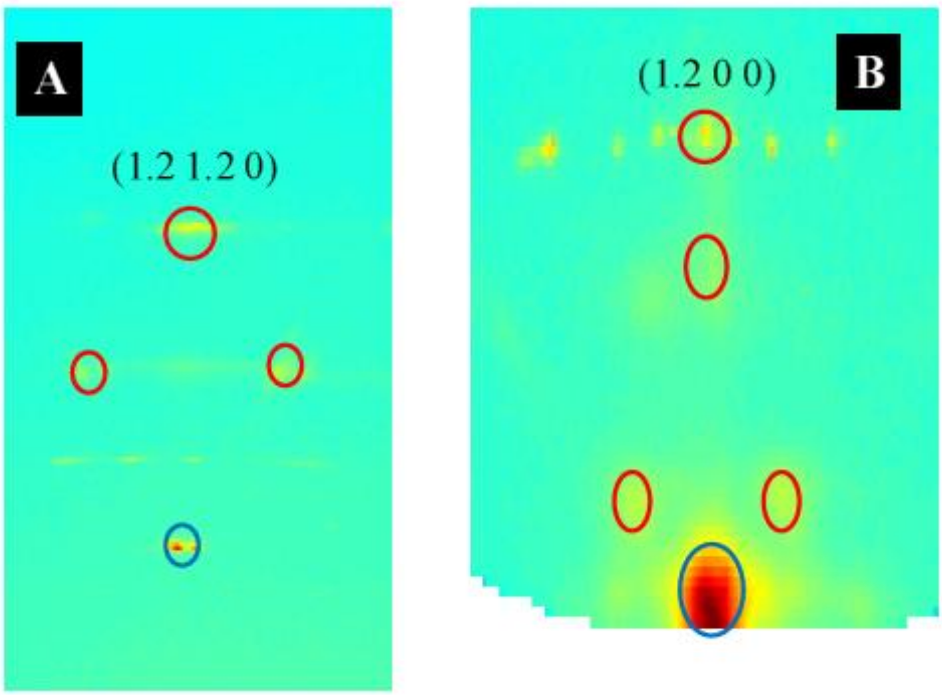

Figure 1: Panel A shows the (110) peak (blue) as seen on a MAR image with (1.2 1 0), (1 1.2 0) and (1.2 1.2 0) peaks, almost two orders of magnitude weaker in intensity. Panel B shows the (1 0 0) peak (blue) decorated by three incommensurate peaks and the (1.2 0 0).

The presence of additional peaks accompanying the Bragg peaks and their persistence at high temperatures raises many questions about their physical origin. In the family of graphite intercalation compounds both CaC_6 and YbC_6 have been shown to exhibit satellite peaks at (1 1.2 0), (1.2 1 0) and (1.2 1.2 0) without explanation. In future work we intend to extend the x-ray diffraction study to other divalent GICs. Whilst SrC_6 has a critical temperature of 1.65 K, no superconductivity has been observed in BaC_6 down to temperatures of 80 mK. Furthering the investigation to these other GICs will allow us to determine whether the presence and intensity of these peaks correlates with superconductivity or the magnitude of T_c.