ESRF	**Experiment title:** Anisotropy of CaC6 electron momentum density	**Experiment number**: HE 2135
Beamline:	**Date of experiment**: from: 3 May to: 9 May 2006	**Date of report**: 01/09/2006
Shifts:	**Local contact(s)**: Thomas Buslaps	*Received at ESRF:*

Names and affiliations of experimentalist **applicants**:

N. Emery, C. Hérold and P. Lagrange, LCSM, University of Nancy, Fance

Ch. Bellin and G. Loupias, IMPMC, University of Paris 6, France

Report

Summary

CaC_6 is a new superconducting Graphite Intercalation Compound (GIC) [1] with a *Tc* = 11.5K. Superconductivity in GICs was subject of intensive studies during the late 80's and early 90's, due to their quasi-2D structure [2]. 40 years ago, some of GICs have be found superconducting, but at very low temperature, like KC_8 with T_c=0.14K [3]. **CaC_6** *Tc* is **almost 2 orders of magnitude larger** than the *Tc* in the well-known KC_8.

Recent *ab initio* calculations indicate the crucial role played by the charge in the intercalated Ca plane [4, 5]. These calculations [4] point at a BCS behaviour with a moderate electron-phonon coupling. The carriers are mostly electrons in Ca Fermi surface coupled with C atoms perpendicular to the graphene plane phonons, leading to **superconductivity of Ca intercalant planes**. The role played by the intercalant non-complete ionization is pointed out by this **new scenario** and even **higher T_c** seem to be possible.

In order to understand the electronic density of **CaC_6** (electron transfer from Ca atom to graphene layer and distortion of the graphite host electronic density, we have performed Compton measurements on CaC_6 at ESRF, on ID 15B, using the high resolution spectrometer.

We have measured **2 Directional Compton profiles of CaC_6** (with ***K*** respectively parallel and perpendicular to **c**-axis) and **1 Directional Compton profile of pristine HOPG,** with the scattering vector ***K*** parallel to **c**-axis.

Experimental details

- The incident energy was 56,28 keV and the diffusion angle equal to 172°, leading to a Compton tranfer of 10.18 keV. Raman departures (K of Ca (4038 eV, i.e. - 9 a.u) and C (284 eV i.e. -14.6 a.u.) are far from the momentum range of interest, surronding q=0 a.u.
- The beam was focused on the sample with a focus size of 0.3 mm horizontal. In the vertical plane, we have used 6 mm of beam on our bulk intercalated sample.

To achieve the same experimental conditions for the 2 directional measurements, we needed a sample with a square section (containing the c-axis). We have succeeded in synthesizing large size CaC_6**bulk samples**, from highly oriented pyrolytic graphite (HOPG) [1]. The resulting compound is made of randomly oriented crystallites in the planes perpendicular to the **c**-axis axis (i.e. in a graphite plane) while they are highly oriented along their ***c***-axis, allowing us to define its angle (0 or π/2) with the scattering vector ***K***. The sample

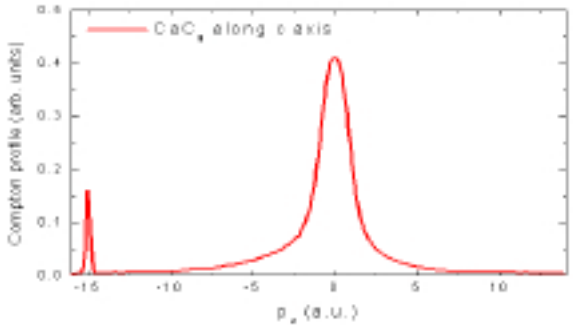
was constituted of 2 plaques, perpendicular to the c-axis, 400µm thick, 0.8 mm large and 7 mm high, in order to obtain a square section (0.8 x 0.8 mm2) for our sample of 7 mm high. One can notice the sample thickness is limited to achieve the intercalation down to the sample core.
- These GIC samples are reactive with air and have to be kept in dry argon all the time. The experiments have been performed on a celled Lindman tube. For comparaison, HOPG sample was positionned in a same Lindman tube.

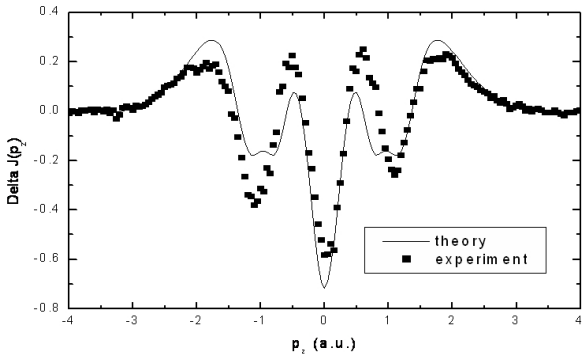
Experimental Results

Data:

- We have reached a resolution of 0.19 a.u.of measured profile, at the Compton peak.
- We have obtained three measured profiles of high quality due, in particula, to a high statistic at Compton peak: with ***K***// graphene **c**-axis: up to 625.000 counts for CaC6,
with ***K*** in graphene **plane :** up to 900.000 counts for CaC6 , and up to 1.000.000 counts for graphite

An experimental profile, as well as the anistropy obtained by the difference between the two directionnal profiles raw data, obtained on **CaC_6**, are shown below. One can notice the good symetry, around q=0 a. u., of the signals.





Anisotropy:

The anisotropy of measured Directional Compton profiles of CaC_6 is compared to the calculated anisotropy, using **new *ab initio* calculations** [4]. One can notice that the features of measured and calculated anisotropies present a good overall agreement.

Difference between **CaC_6 and graphite** *Directional Compton profiles, measured with the same direction of the scattering* vector ***K***.

We have found that each directional measured differenceprofile is narrower than the calculated one, supporting a **larger delocalisation of conduction electrons in CaC_6** than expected by the theory. The comparison with theory is still in progress (*very recent experimental results obtained in* May 2006).

In such a study, we will be soon able to evaluate the contributions of both the electron conduction profile and the valence profile **distortion** (as we have already done for alkali intercalated graphite). In particular, we wish to elucidate the Ca electron hybridization with the graphene electrons, and the crucial role played by the **charge in the intercalated Ca plane**, due to a partial ionization of Ca atoms, as predicted by the different *ab initio* calculations.

[1] N. Emery, C. Hérold, M. d'Astuto, V. Garcia, Ch. Bellin, J.F. Marêché, P. Lagrange and G. Loupias, Phys. Rev. Lett. **95**, 87003 (2005), selected for August 29, 2005, issue of Virtual J. of Nanoscale Science & Technology and for comments in Physics World magazine.
[2] R. A. Jishi and M. S. Dresselhaus, Phys. Rev. B **45**, 12 465 (1992).
[3] Y. Koike, S-I Tanuma, H. Suematsu and K. Higuchi, J. Phys. Chem. Solids, **41** 1111 (1980)
[4] M. Calandra and F. Mauri, Phys. Rev. Lett. **95**, 237002 (2005),
[5] G. Csnayi et al., Nature Physics **1** (2005)