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 original report should be sent, together with 5 reduced (A4) copies, to the User Office.

In addition, please send a copy of your file as an e-mail attachment to reports@esrf.fr**,** using the number of your experiment to name your file. This will enable us to process your report for the ESRF Annual Report.

Reports accompanying requests for additional beam time

If your report is to support **a new proposal**, the orginal report form should be sent with the new proposal form, and a copy of your report should be attached to each copy of your proposal. 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.

∑ bear in mind that the report will be reduced to 71% of its original size. A type-face such as ìTimesî, 14 points, with a 1.5 line spacing between lines for the text, produces a report which can be read easily.

Postal address: User Office, ESRF, B.P. 220, F-38043 GRENOBLE Cedex, France
Street address: 6 rue Jules Horowitz, F-38043 GRENOBLE Cedex
Telephone: +33 (0)4 7688 2552; Fax: +33 (0)4 7688 2020; e-mail: useroff@esrf.fr

ESRF	**Experiment title:** **Electronic density of carbon nanotubes by Compton scattering**	**Experiment number**: HS 1441
Beamline: ID 15B	**Date of experiment**: from: 20.06.01 15:00 to: 26/06/01 7:00	**Date of report**: 27/08/2001
Shifts: 15	**Local contact(s)**: Veijo HONKIMAKI	*Received at ESRF:*

Names and affiliations of applicants **(* indicates experimentalists)**

Christophe BELLIN* (LMCP, Univ. Paris 6)
Geneviève LOUPIAS (LMCP, Univ. Paris 6)
Jean-Michel GILLET* (École Centrale Paris)
Fabien BRUNEVAL* (École Centrale Paris)
Thomas BUSLAPS* (ESRF, Grenoble)
Philippe POULIN (CRPP, Univ. Bordeaux I)
Alain PÉNICAUD (CRPP, Univ. Bordeaux I)
Patrick BERNIER (GDPC, Univ. Montpellier II)

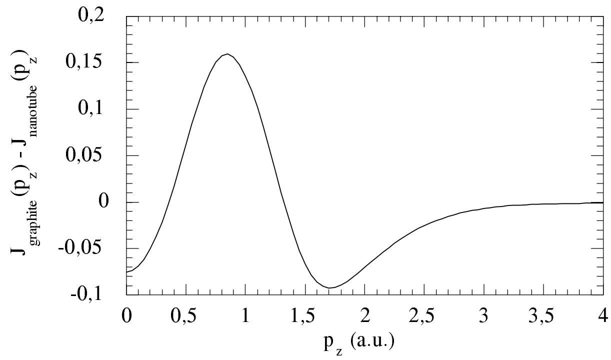
Report:

Single wall carbon nanotubes are made of a graphitic sheet rolled up into a cylinder of several microns length and a few nanometers in diameter. Thus they constitute an excellent model for studying the effect of low dimensionality on electronic density. We used as samples macroscopic fibers of oriented nanotubes. This was the only way to obtain sufficient quantity of matter for getting enough statistics in our measured profiles. Nanotubes used for fibers are produced using the electric-arc technique. As a catalyst, a mixture of 1 at.% Y and 4.2 at.% Ni has been used [1]. The preparation method for fibers « *consists of dispersing the nanotubes in surfactant solutions, and then recondensing the nanotubes in the stream of a polymer solution* ». They are then washed and rinsed for polymer solution to desorb. [2]. The best alignment of nanotubes inside the fibers achieved was ± 20 degrees of desorientation. These samples are the state of art samples available at that present time : best alignment of tubes within the fiber and well caracterized nanotubes (single wall, 14 Å of diameter).

We measured the direction corresponding to the scattering vector **K** perpendicular to the tube axis, with the aim to compare this measured profile both to the Compton profile measured on HOPG graphite with **K** perpendicular to the graphitic sheets [3] and with Compton profile measured on C_{60} powder [4] as well as on a C_{60} monocrystal [5] . The choice of that measured direction will provide us the difference in terms of electronic density delocalization in real space between nanotubes and mentioned above carbon-type materials. Comparison of absolute profiles is of great importance since it carries information on hybridization of π orbitals related to two main caracteristics : 1- the curvature of carbon sheets and 2 - the electronic density behaviour in the case of the nanotubes single wall compared both to the stacking of graphitic sheets in the case of HOPG graphite and to pristine C_{60}.

The experiment on nanotubes has been performed with the scattering angle settled at 175° and the synchrotron radiation has been monochromatized at 29.743 keV. We obtained 3.10^5 counts at the Compton peak. Such a statistics allows a consistent comparison with HOPG graphite as well as C_{60} data. A very accurate determination impurities ratio in the fiber sample is needed : Y and Ni (catalysts) play the role of impurities for Compton scattering measurements since Compton method is sensitive to all electrons in the sample. The data analysis, including the subtraction of yttrium and nickel Compton profiles (taking into account their ratio) from the total fiber measured profile in order to get the absolute nanotube profile, is under progress.

Discussion about experimental results will receive help from theoretical calculations. Preliminary *ab-initio* Hartree-Fock calculation has been performed by us using the code Crystal 95 [6]. The figure below shows the difference between and graphite Compton profile (**K** perpendicular to graphite sheets) and nanotube Compton profile (**K** perpendicular to the tube axis).



One can observe at low p_z that the nanotube profile is higher than graphite profile : we can expect electrons more localized in momentum space for nanotube than for graphite, so that they should be more spatially delocalized perpendicularly to the hexagonal mesh in nanotube than in graphite.

In graphite, *s* bonds are made of the combination between *2s* orbitals and p_x - p_y orbitals lying in graphite plane. The fourth valence electron fills the p_z orbital perpendicular to the plane. The nanotube curvature results in a *2s* and p_z combination : the effective charge supported by p_z electrons should be higher in nanotube than in graphite.

Experimental results with help of theoretical model will enable us to answer the questions about the effect of both unidimensionality and curvature on nanotubes electronic properties.

[1] C. Journet, W.K.Maser, P.Bernier,A.Loiseau, M.Lamy de la Chapelle, S. Lefrant, P. Deniard, R. Lee & J.E. Fischer, Nature **388**, 756 (1998)
[2] B. Vigolo, A. Pénicaud, C. Coulon, C. Sauder, R. Pailler, C. Journet, P. Bernier, P. Poulin, Science **290**, 1331 (2000)
[3] PhD thesis of M. Marangolo, Univ. Paris 6 (1998) - the measurements on graphite were performed at LURE, Orsay
[4] see for example M.Marangolo, Ch.Bellin, G.Loupias, S.Rabii, S.C.Erwin, T.Buslaps - *Phys. Rev. B* **60**, 17084 (1999)
[5] Ch. Bellin, M. Marangolo, J. Moscovici, G. Loupias, Acta Physica Polonica **91**, 681 (1997)
[6] D. Belemlilga, J.M. Gillet, P. Becker, J. Phys. Chem. Solids **61**, 369 (2000)