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 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:** Study of the structure and arrangement of aligned carbon nanotubes	**Experiment number**: ME-521
Beamline: ID02	**Date of experiment**: from: 09/05/03 to: 11/05/03	**Date of report**: 23/02/04
Shifts: 6	**Local contact(s)**: Peter Boesecke	*Received at ESRF:*

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Report:

Carbon nanotubes are of great interest in nanotechnology because of their unique structural, mechanical and electronic properties. Due to their high aspect ratios, it is difficult to arrange carbon nanotubes into an organised assembly. One way to solve this problem of disorder is to directly prepare well ordered aligned carbon nanotubes. Different methods are used to produce nanotubes. In our work, aligned carbon nanotubes are synthesized by catalytic decomposition from pyrolysis of mixed aerosols containing both the hydrocarbon source and the metallic precursor as catalytic source. This method is based on the simultaneous and homogeneous feeding of the reaction chamber by the carbon and the catalyst sources [1]. The pyrolysis is achieved in the [750-950]°C temperature range. Multi-wall carbon nanotubes almost free of any by-products (amorphous carbon, metallic encapsulated particles,…) are produced. SEM observations show that they look like "carpets". The thickness of the carpets, e.g. the length of nanotubes, can be controlled in the [100-700] microns range. The diameter can vary from 10 to 120nm.
The aim of the experiment at ESRF was to study the structure and the arrangement of aligned nanotubes, e.g. the anisotropy of the carpets.

We used two different aerosol generators for sample synthesis:

- carpets synthesized using toluene and ferrocene 5 wt % at 850°C during 15 or 45 minutes with an ultrasonic aerosol generator[1-2].
- carpets synthesized using cyclohexane and ferrocene 2.5 wt % at 850°C during 15 minutes with a new injection system [3].

Each raw sample has been annealed (at 1700, 2000, 2200, 2500°C under argon during 2h, heating rate of 10°C min^{-1} or at 350 and 500°C under air) in order to improve the structure of nanotubes. Raw samples and annealed one have been studied during the experiment.

Measurements were achieved in transmission across individual carpets of typical width of 1-2 mm. A CCD camera was used to collect the data, the distance sample-detector was 6.5 meters, the wavelength was 0.9951Å.
In a first time, the beam was parallel to the nanotube growth axis and then nanotubes were disoriented by tilting them from this axis to reveal anisotropic signal. Typical isotropic and anisotropic signals obtained on nanotube carpets are shown in figure 1.

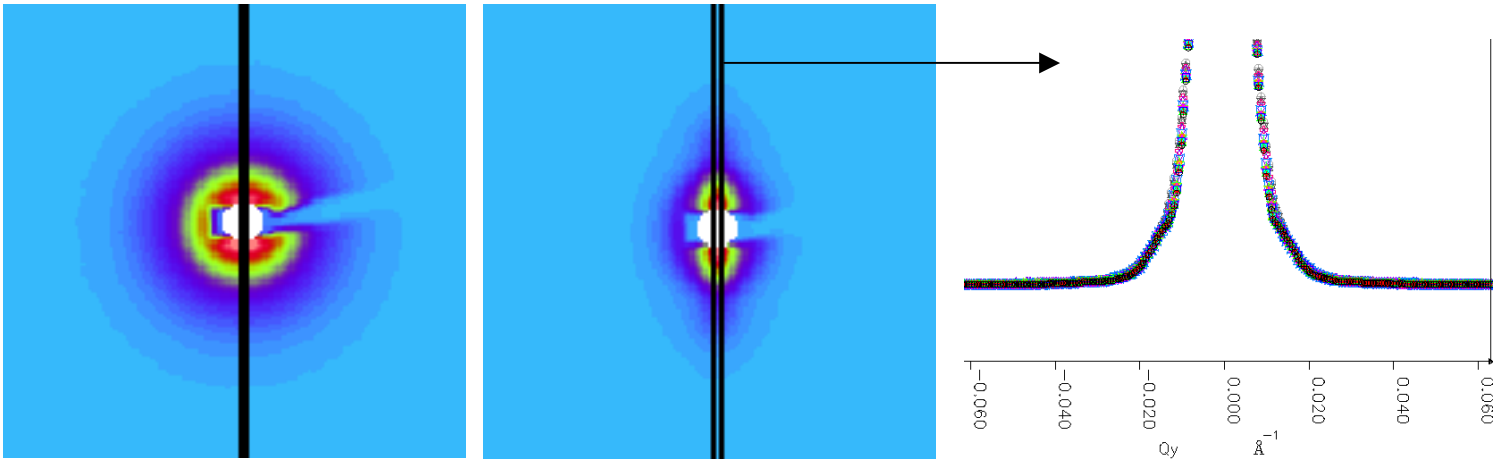

figure1: left :isotropic signal coming from a carpet of nanotubes when the beam is parallel to the nanotube growth axis, middle: anisotropic signal obtained by tilting sample, right: radial scan performed on the anisotropic image.

Sample synthesized with the injection system exhibits a more anisotropic signal than the one coming from the other samples, indicating that the nanotubes have a better alignment degree in this carpet.
Radial scan performed on an anisotropic signal is shown in figure1 (right), shoulders at ±0.015$Å^{-1}$ can be distinguished. A computer program is in progress in order to analyze these data and should allow one to get indications about the inner and outer diameters of the tubes. Comparison between signals coming from raw and annealed carpets should allow to determine the effect of post-annealing temperature on graphitization degree of nanotube walls. We should also get information on the shape of the porosity associated to nanotubes.
In conclusion, the experiment was successful, the high brilliance of the beamline ID02 permitted to obtain new and interesting structural results on aligned nanotubes. This experiment should allow new knwoledge of the structural features of carbon nanotube carpets.

[1] M. Mayne, N. Grobert, M. Terrones, R. Kamalakaran, M. Rühle, H.W. Kroto and D.R.M. Dalton, Chem. Phys. Lett. 338, 101 (2001)
[2] M. Mayne-L'Hermite, X. Armand, D. Porterat, C. Reynaud, Proceeding of the Chemical Vapor Deposition-XVI and EUROCVD-14, edited by M. Allendorf, F. Maury, F. Teyssandier, 2003-08, 549-556.
[3] M. Pinault, M. Mayne-L'Hermite, C. Reynaud, O. Beyssac, J.N. Rouzaud and C. Clinard, to appear in 'Diamond and related materials'