EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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.

<table>
<tr><td>ESRF</td><td>Experiment title:
Structural investigation of 0.4nm sized nanotubes aligned in the channels of $AlPO_4$-5 single crystals</td><td>Experiment number:
HS1396</td></tr>
<tr><td>Beamline:</td><td>Date of experiment:
from: April 25, 2000 to: May 2, 2000</td><td rowspan="2">Date of report:
August 30, 2000
Received at ESRF:</td></tr>
<tr><td>Shifts:
21</td><td>Local contact(s):
HAMILTON Miles</td></tr>
<tr><td colspan="3">Names and affiliations of applicants (* indicates experimentalists):
LAUNOIS Pascale*, Laboratoire de Physique des Solides (LPS), Orsay
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MARUCCI Alessandra*, LPS, Orsay
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</table>

Report:

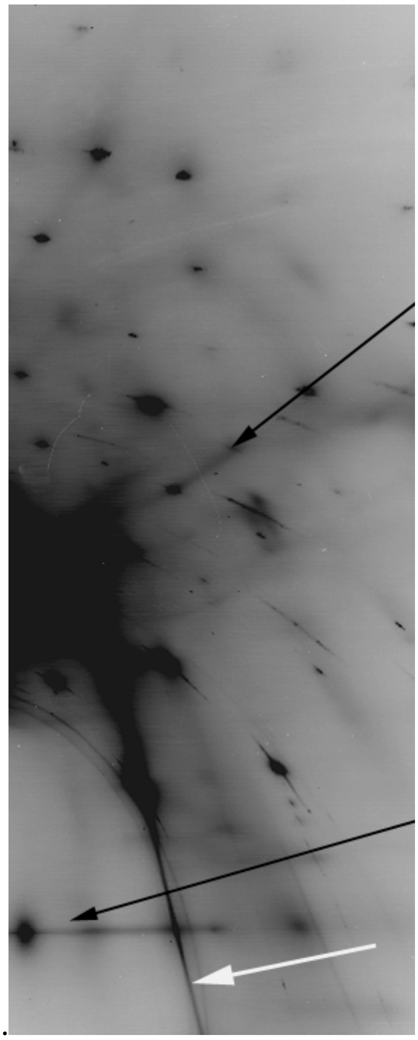
The interesting physical properties of single-wall carbon nanotubes (SWCNT) are strongly linked to their structure, which makes structural analyses on nanotubes particularly desirable. We had thus proposed to study SWCNTs confined in the channels of a zeolite single crystal. To our knowledge, these samples are the only ones that contain macroscopic quantities of aligned SWCNTs and they thus afford us unique possibilities to investigate in details the structural properties of the nanotubes. Recent experiments performed in lab. on standard sources and feasibility tests at ESRF on ID1 (2 days of in-house beam time) have allowed us to determine the nanotube diameter, equal to 4Å [1,2]. This diameter is the smallest ever found and it is controlled by the zeolite host. One can speculate that interactions with the zeolite may also lead to the selection of only one nanotube helicity, which would make our samples absolutely unique. Remarkably, superconducting properties have also been evidenced very recently, at low temperature [3]. The structural determination of the SWCNT helicity(ies) is fundamental for the understanding of zeolite-nanotube interactions and of superconductivity. The subject of the experiment reported here was the **study of nanotube helicity(ies).**

Sample elaboration was performed by collaborators at Hong Kong. Tripropylamine molecules are encapsulated in the channels of $AlPO_4$-5 (AFI) single crystals during the crystal growth. The crystals are subsequently pyrolysed at 350-450°C and the carbon nanotubes are formed at 500-800°C. Samples with empty channels are prepared by heating under oxygen atmosphere. Crystal volume is typically 10^{-2}-10^{-3} mm^3. During the experiment, **four crystals have been studied**: three crystals with nanotubes inside the channels (named 'AFI-nanotubes'), and one with hollow channels ('AFI-hollow'). The experimental procedure consists in the comparative study of AFI-hollow and AFI-nanotube samples, to determine which scattering features are characteristic of the nanotubes.

Due to the low intensities of the signals expected from the nanotubes (small sample volumes and small diffusion factor of carbon), the background has to be extremely small. The experiments have thus been performed **under vacuum**. Incident wave-length was λ=1.77Å.

For nanotubes with orientational or positional disorder inside the zeolite channels, one expects to measure **diffuse scattering planes** l=constant, whose position is directly related to the nanotube periodicity and thus to their helicity [1]. The distribution of intensity inside the planes should also give valuable information, for instance, about nanotube orientations within the channels. In order to look for such diffuse scattering planes, experiments have been performed with large **image plates**, allowing quantitative investigations of large areas of reciprocal space. Unusual configurations such as image plate inclinations were used to avoid peak spreading for high level reciprocal planes. Comparison between image plates taken on AFI-hollow and AFI-nanotube samples allowed us to determine which ***scattering features*** are due to the zeolite host crystal and which may be ***due to the nanotubes***, as is illustrated in the figure. The detailed analysis of the measured data is in progress.

X-ray scattering Image Plate (ID1, ESRF) on a fixed AFI-nanotube single crystal (vertical axis **c**). Black arrows point toward the trace of diffuse planes attributed to nanotube disorder. The white arrow indicates a diffuse scattering streak due to the zeolite host crystal.



[1] P. Launois, R. Moret, D. Le Bolloc'h, P.A. Albouy, Z.K. Tang, G. Li, J. Chen, Solid State Comm. 116 (2000) 99-103

[2] P. Launois, R. Moret, D. Le Bolloc'h, P.A. Albouy, Z.K. Tang, G. Li and J. Chen, ESRF Highlights 2000, p. 67

[3] Z.K. Tang, L. Zhang, N. Wang, X.X. Zhang, G.H. Wen, G.D. Li, J.N. Wang, C.T. Chan and P. Sheng, Science 292, 2462, juin 2001