Experimental report

28/01/2016

Proposal: 5-32-821 **Council:** 4/2015

Title: Structure of Fe-Phthalocyanine encapsulated inside SWCNT

Research area: Physics

This proposal is a new proposal

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Samples: C32H16FeN8

Instrument	Requested days	Allocated days	From	To
D16	3	3	09/12/2015	12/12/2015

Abstract:

FePc@SWCNT are peapods-like nanostructures composed Fe-phthalocyanine encapsulated inside Single-Walled Carbon Nanotube. This metallo-organic macrocycle (FePc) molecule exhibits not only optical but also structure depending magnetic properties. Through confinement inside the holow cavity of carbon nanotube, 1D chains of these molecules are created, with new confomationnal order and that are thus expected to exhibit specific properties (optical, magnetic). Preliminary XRD experiment have proven that FePc@SWCNT are filled with the guest molecules but also show a signature of confined
1D phase. We now aim at performing high resolution neutron diffraction (D16 instrument) on FePc@SWCNT in the range 2-320K, in order to fully identify the 1D phase, possible order/disorder transitions, and the associated magnetic properties.

Experimental report D16

The aim of this experience is to characterize the one-dimensional chain of Iron-Phthalocyanine (FePc) which is encapsulated inside single-walled carbon nanotubes. In order to do so, two different nanotube diameters were chosen: 14Å and 21Å, hereafter referred as NT14 and NT21.

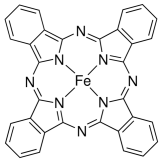

Figure01. Sketch of the Iron phthalocyanine molecule.

The diameter of the encapsulated molecule is around 15Å. The van der Waals distance between it and the inner walls of the nanotube is around 3.2 Å. Therefore the angle between the stacking of the molecule along the nanotube axis depends on the diameter of the nanotube. Therefore our interest is in the 1D chain of FePc.
At the instrument D16, the wavelength used was 4.5Å. Three different positions of the 2D detector were used in order have access to the range of momentum transfer up to 1.3 $Å^{-1}$. The measurements were done in function of temperature for the hybrid nanotubes (Hereafter called: FePC@NT14 and FePc@NT21), the respective empty nanotubes were measured for room temperature only. Below we show the small angle diffraction of the sample NT21. The two features correspond to the hexagonal lattice of the bundles of NT21.

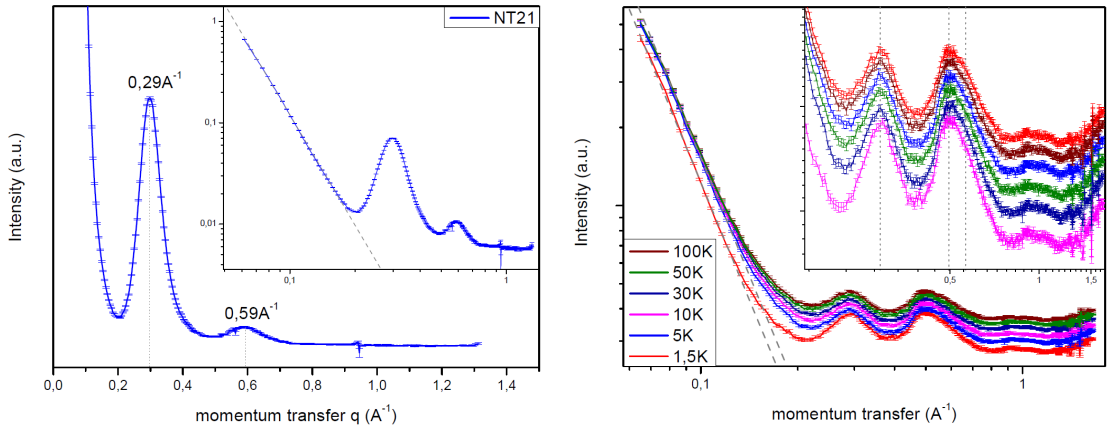

Figure02. On the left: Diffractogram of the sample NT21. On the right: Experimental data from the temperature-dependence measurements for the sample FePc@NT21.

The second series of measurements, under pressure, were performed only for the sample FePc@NT21. The powdered sample was inserted inside an aluminum capsule along with a liquid mixture (at room temperature, room pressure) of 4:1 ethanol methanol, both deuterated, used as the transmitting medium of pressure. The aluminum capsule goes inside a TiZr Clamp cell. The choice for this specific transmitting medium was the possibility of recovering the samples after the measurement.

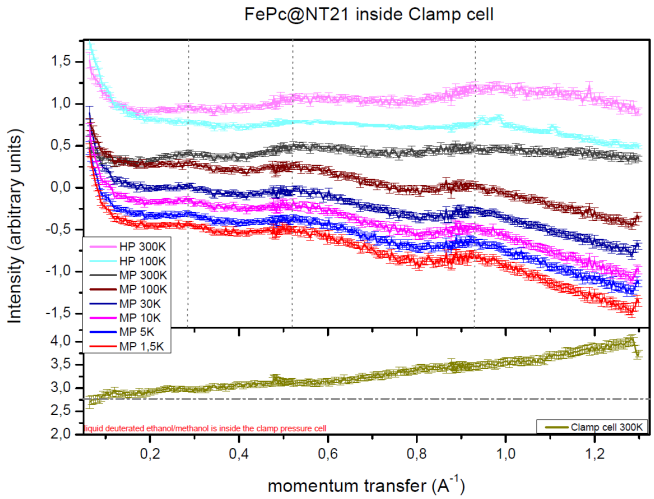

Figure 03. For a constant pressure measurement, MP=7.5Kbar and HP=14kbar, temperature dependence measurements. On the graphic on the bottom, measurement for the TiZr pressure cell with aluminum container and liquid transmitting medium.

At the moment of writing this report, the obtained data is still under interpretation. The preliminary conclusions are:

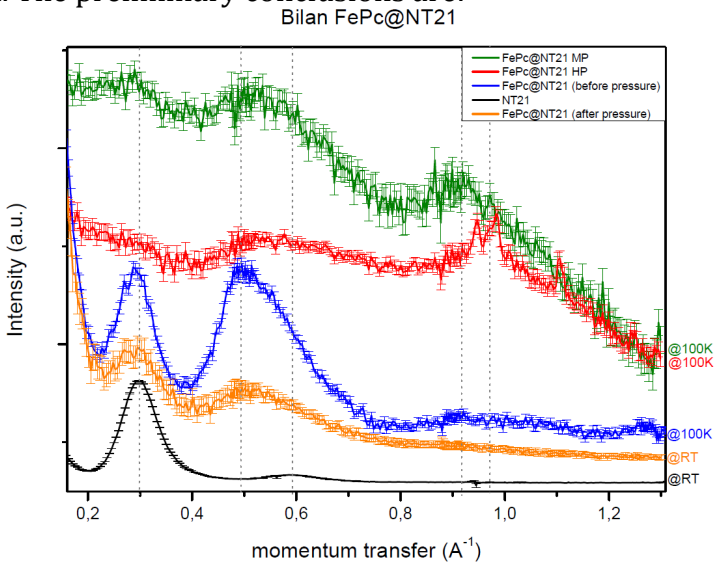

Figure 04. black curve: NT21; orange curve: FePc@NT21 after pressure measurements; blue curve: FePc@NT21 before pressure; red curve: FePc@NT21 under medium pressure and green curve: FePc@NT21 under high pressure.

1) Under medium (dark green) and high (red) pressure the hexagonal lattice of the nanotubes loses part of its symmetry, due to ovalization of the nanotubes.
2) Under medium (dark green) and high (red) pressure the 1D chain of the encapsulated FePc is compressed: The 1D peak seems to be shifted from 0.45 up to about 0.95 $Å^{-1}$
3) On releasing the pressure (orange curve) the (10) peak as well as 1D chain of FePc peaks come back to the initial position (blue curve). However part of the intensity of the 1D chain of FePc seems to be lost.