ESRF	**Experiment title:** PRESSURE-TEMPERATURE PHASE DIAGRAM OF IRON NANOWIRES CONFINED INSIDE CARBON NANOTUBES	**Experiment number**: HS3257
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Report:

We report on our high pressure-high temperature (HPHT) x-ray diffraction (XRD) experiments on multiwalled carbon nanotubes (MWNT) filled with iron nanowires (Fe@MWNT).

MWNT produced by Catalytic Vapor Deposition are partially filled with iron nanowires, which present a great interest for applications such as high density magnetic storage. It has been previously shown that most of the iron contained in the tubes was under γ-Fe phase, although only the α-Fe phase is supposed to be stable under ambient conditions. This observation can be explained by the confinement due to the nanotube wall, which prevents the phase transition between the high temperature stable γ-Fe phase and the low temperature stable α-Fe from occurring during the temperature cooling down. Our main objectives were i) to investigate the structural behaviour and the phase transitions of the encapsulated γ-Fe nanowires when submitted to HPHT conditions, and ii) to study the effects of extreme conditions on the layered structure of MWNT.

We used diamond anvil cells as high pressure device, and high temperatures were reached using a resistive heating, which allowed us to reach a maximum temperature of 700K. The pressure medium was Ar for ambient temperature measurements, and Ne for high temperature measurements. A ruby was used as pressure indicator. The X-ray wavelength

was set to 0.3738 Å and sample to CCD detector distance was 229.6144 mm. The beam size on the sample was 4 µm * 4µm.

It must be pointed out that according to the very small mass of nanotubes inserted in the pressure cells, it has proven very hard to get enough signal to perform significant measurements. As a matter of facts, we first aimed at positioning the nanotubes carpets as well-oriented as possible in the cell, and we consequently put a rather weak mass of nanotubes in the pressure cell. After several unfruitful attempts, we finally decided to put much more nanotubes in the pressure cell. This cell loading gave much better results, as can be seen on figure 1.

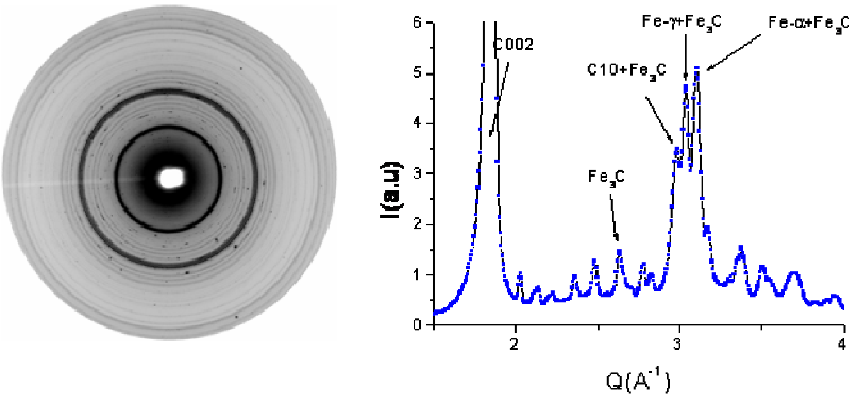

Figure 1. *Left* : XRD pattern recorded on 2-D CCD detector for Fe@MWNT under ambient pressure and ambient temperature. *Right* : Integrated radial scan of the pattern.

In the experimentally accessible Q range, it is possible to observe the diffraction signal coming from the inter-shell periodicity (denoted as C002) of carbon nanotubes walls located at ~1.8 $Å^{-1}$. Some other important features were identified in the diffraction pattern, namely diffraction peaks coming from Fe_3C, α-Fe and γ-Fe.
The evolution of these diffraction features was followed as a function of the pressure for 2 temperatures, namely 300K and 573K. Pressures up to 40 GPa were reached in the series of measurements performed at 300K, and the maximum pressure reached at 573K was 30 GPa.
The results of these experiments are in contrast with the previous HPHT studies performed by Karmakar et al. (*Phys. Rev. B* **69**, 165414 (2004)), and they are the main topic of an article currently in preparation.