ESRF	**Structural evolution of the C_{70} fullerenes peapods under high pressure and high temperature**	**Experiment number**: HS-3531
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Report:

Context of the experiment

C_{70} peapods are carbon hybrid nanostructures formed by C_{70} fullerenes encapsulated inside carbon nanotube. It has been shown that the fullerenes can adopt various orientations according to the nanotube diameter. In thin carbon nanotubes, the molecule long axis is parallel to the nanotube axis; the orientation is called “lying”. In thick carbon nanotubes, the molecule long axis is perpendicular to the nanotube axis; the orientation is called "standing" [1]. Electronic properties are predicted to be dependent on the fullerene orientation [2].

In a previous work, we showed that X-ray diffraction is a powerful method to determine the structure of a macroscopic sample of C_{70} peapods. We estimated the critical nanotube diameter which allows the changing of orientation from the lying to the standing orientation of about 14.2Å [3]. In recent papers, we demonstrated that polymerization of C_{60} fullerenes is achievable inside carbon nanotube for moderated conditions of pressure and temperature. Typically we used pressure around 1.5GPa and temperature around 500°C [4,5]. Concerning the C_{70} fullerenes, we showed that formation of long polymerized chains does not occur inside carbon nanotubes. We explained this result with geometrical arguments [4]. The diameter of the nanotube which allows the presence of the C_{70} zigzag chains (typical polymer chain observed in the crystal phase) is about 18Å. But the mean nanotube diameter of our sample is about 14.2Å with a distribution of diameter of about 2Å. The thick nanotubes of 18Å do not exist in our sample. We concluded that confinement inside the nanotube prevents the formation of long polymer zigzag chains.

On the other hand, we proposed the hypothesis that C_{70} dimers could be synthesized inside carbon nanotubes with a minimum diameter of 13.5Å: it could occur in our samples. The aim of the present experiment was to study the possible formation of dimers. We used different thermodynamic pathways from previous study [4,5,6] where no dimerization was evidenced.

Experimental part

Experiments were done on C_{70} peapods buckypapers for which the signal from the fullerenes chains is enhanced due to orientation effect. As our experiments a priori only necessitate moderate conditions of pressure and temperature, we used the carbide anvil setup and the resistive setup on the Paris-Edinburgh press.
Figure 1 presents the main result of the experiment.

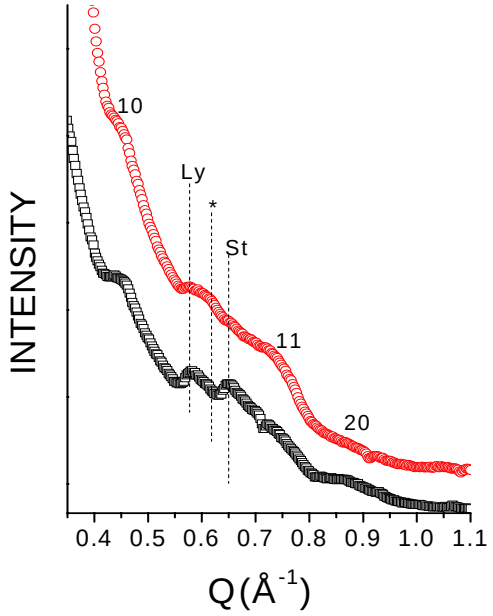

Fig1:Comparison of the diffraction diagrams obtained before and after the HTHP treatment. Symbol Ly and St refer to the peak of the periodicity of the lying and standing chains respectively. The star refers to the peak from the new synthesized structure. Indices hk are relative to the organization of the peapods on a two-dimensional hexagonal lattice. Diffractions patterns have been translated for the sake of clarity.

The lower diffraction diagram (black open squares) is related to the C_{70} peapods before treatment. The diffraction diagram presents two peaks related to the periodicity of the fullerene chains inside the carbon nanotubes. The first one is situated at $0.57Å^{-1}$ and is related to the lying periodicity. The second one situated at $0.64Å^{-1}$ is related to the standing one. One can observe peaks from the hexagonal lattice of the peapods bundles. For more information about the analysis of the C_{70} peapods diffraction diagram, one can refer to [3].
The second diffraction diagram (open red circles) is the one obtained after HTHP treatment. The standing peak situated at $0.64Å^{-1}$ has mainly disappeared during the treatment. The lying peak seems to be at the same position and with similar intensity. Moreover, a new contribution has appeared around $0.62Å^{-1}$. This peak is related to another structure of fullerenes inside the carbon nanotubes. Analyses are in progress to determine this new structure.

Conclusion

Experiments allowed us to synthesize a new phase in C_{70} peapods under pressure (possibly a dimer phase). The analysis is presently in progress and will be published soon.

Bibliography

[1] Hirahara et al. Phys. Rev. B, **64**, 115420, 2001
[2] Hornbaker et al. Science, **295**, 828, 2002
[3] Chorro et al. Phys. Rev. B, **75**, 035416, 2007
[4] Chorro et al. Eur. Phys. Lett., **79**, 56003, 2007
[5] Chorro et al. Phys. Rev. B, **74**, 205425, 2006
[6] Kawasaki et al. Chem. Phys. Lett, **418**, 260, 2006