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ESRF	**Experiment title:** Nano-Extreme :Extreme conditions of pressure and temperature for the elaboration of new carbon based nano-materials	**Experiment number**: CH-1704
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Report:

Part II : High pressure & High temperature experiments

In a typical large volume press assembly like the Paris-Edinburgh press, a cylindrical graphite oven allows to reach nearly 1400 Kelvin. However, the maximum pressure is limited to 8 GPa to avoid the graphite-diamond conversion. In order to apply pressure up to 10 GPa and at least 1000 K on our samples we chose to use a metallic heater. For convenient thermal properties, Rhenium was chosen. However, Rhenium is strongly absorbent contrary to Carbon which is a light element. To overcome this problem we used Re thin ribbon in such a way that the beam did not cross the heater. The cell assembly is shown on figure 3. The sample was introduced in a crucible of tantalum (thickness: 25 microns) to avoid diffusion of carbon and iodine in boron nitride. We reached a maximum oil pressure of 530 bars for an electrical power of 300 W corresponding to 15 GPa and 1000°C. Such pressure and temperature are unusual for a Paris- Edinburgh press.

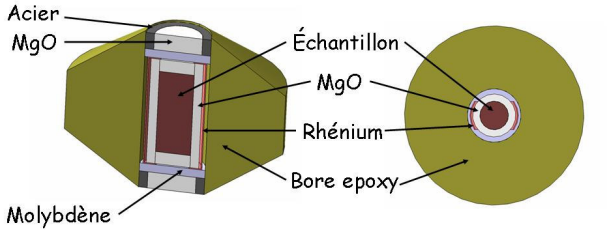

Figure 3 : ***cell assembly for high-pressure and high-temperature experiment***

We performed two x-ray absorption experiments at the iodine K-edge on iodine intercalated Single Wall carbon Nanotubes (SWNT) compounds under high pressure and high temperature conditions.

The EXAFS spectra are shown on figure 4. As reported in Part I, we observed changes in the EXAFS spectra while increasing pressure between ambient pressure and 8 GPa. Heating to 1000°C at 15 GPa leads to additional changes in the spectra. The quality of the spectra does not allow any quantitative information. However, qualitatively, we noticed that the transition is irreversible after high pressure and high temperature cycle.

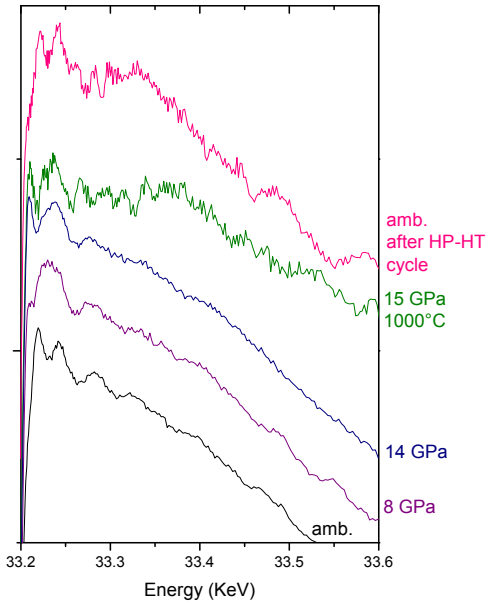

Figure 4. ***EXAFS oscillations at the I K-edge of iodine intercalated single wall carbon nanotubes as a function of pressure and temperature. The signal of the recovered sample was dominated by I-O and I_2***

A second experiment was carried out in the same conditions. Unfortunately, oxidation of Iodine occurred and did not allow any clear conclusion.

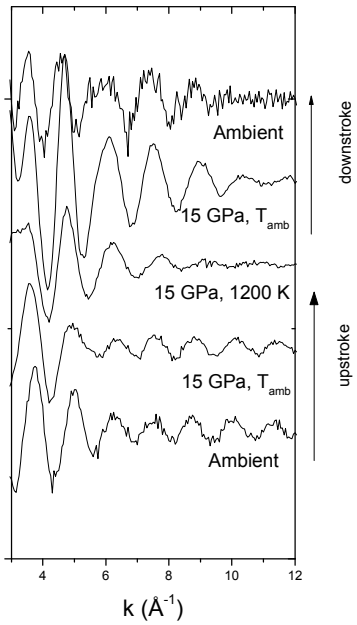

Figure 5. ***EXAFS oscillations at the I K-edge of iodine intercalated single wall carbon nanotubes as a function of pressure and temperature. The signal of the recovered sample was dominated by I-O and I_2***

Complementary experiments were performed in a large volume multi-anvil apparatus[1] at 15 GPa and 1500 C on doped carbon nanotubes in the laboratory. The experiment successfully showed the possibility of

[1] In Laboratoire Magmas et Volcans. This is a French National Facility based in Clermont Ferrand.