

## Hydrogen Trapping and Stability at the Polycrystalline CVD Diamond Surface and in the Subsurface Layers.

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The interaction of crystalline diamond surface with monoatomic hydrogen, either H or H<sup>+</sup>, results simultaneously in etching and hydrogen in-diffusion. The etching rate is generally more important using rf plasma, compared with micro-wave plasma [1]. Furthermore, the hydrogen present at the diamond surface and/or the subsurface region induces a superficial p-type highly conductive layer [2], the physical origin of which is not clearly understood. It has been shown in a previous work that the hydrogen (deuterium) diffusion profile depends on the nature of the hydrogen (deuterium) plasma treatment. The deuterium profiles analysed by secondary ion mass spectrometry (SIMS) obtained after rf plasma treatment exhibit a higher deuterium accumulation in the subsurface region, compared to micro-wave plasma treatment (figure 1). This accumulation is decreased when using remote plasma [3].

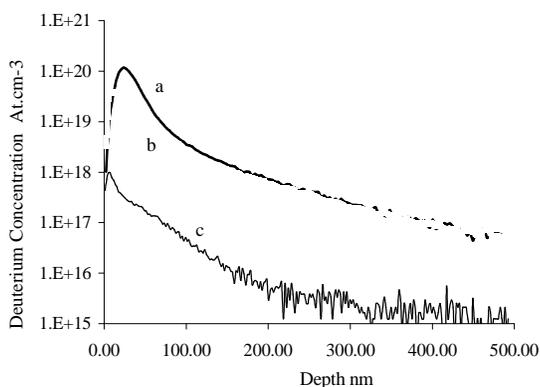
In this paper, three different types of samples are studied: a) as-grown (with deuterium) CVD sample; b) as-grown sample submitted to (direct or remote) rf plasma; c) as-grown sample submitted to micro-wave plasma, using deuterium as tracer to improve the detection limit of the SIMS analysis and effusion measurements. Each type of sample has been submitted to two different treatments: first, annealing at 600°C during 15 minutes in argon atmosphere, then chemical treatments in the three following baths: sulfochromic acid, HCl/HNO<sub>3</sub>, and finally NH<sub>4</sub>OH/H<sub>2</sub>O<sub>2</sub>. It is shown, by SIMS analysis, that the concentration of deuterium accumulated in the subsurface region is slightly modified by the thermal treatment at 600°C, while it is not by the chemical etching, considering the detection limits of the SIMS analysis.

At each step, the effusion spectra of deuterium were measured by the ion current of a quadrupole mass spectrometer coupled to an evacuated quartz tube (10<sup>-9</sup> to 10<sup>-10</sup> mbar) which contained the deuterated samples, and compared. Preliminary deuterium effusion results on deuterated samples are presented in figure 2. Comparison with SIMS results (figure 1) allows to attribute the peak shoulder at 700 °C to the deuterium accumulation in the subsurface region. The effusion spectra obtained after thermal and chemical treatments are presented in figure 3 and show that there is an obvious effect of the thermal annealing at 600°C on the 700°C effusion peak, while the chemical treatment leads to decrease the effusion peak at 880°C, i.e. the deuterium trapped on the surface.

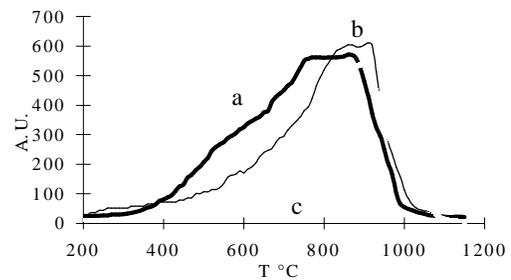
The sample surface has been analysed by X-ray photoelectron spectroscopy (XPS) after plasma deuteration, thermal annealing at 600°C and chemical treatments. XPS spectra are reported in figure 4 and show the evolution of carbon bonding from sp<sup>2</sup> to sp<sup>3</sup>. *In situ* isothermal annealings in the XPS analysis UHV chamber have been performed and related to the effusion of deuterium and the resulting SIMS profiles.

### References

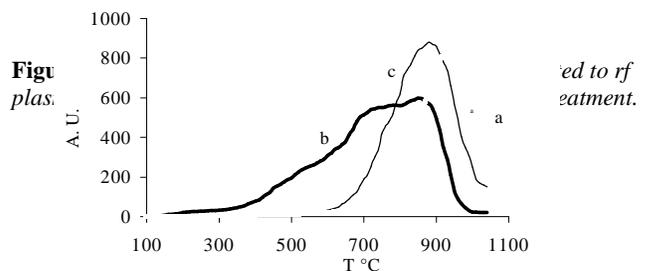
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- [2] K. Hayashi, S. Hamanaka, H. Watanabe, T. Sekigushi, H. Okushi and K. Kajimura, *J. Appl. Phys.* **81** (1997) 744.
- [3] D. Ballutaud, F. Jomard, B. Theys, C. Mer, D. Tromson et P. Bergonzo, , 11<sup>th</sup> European Conference on Diamond-like Materials, Carbon nanotubes, Nitrides and Silicon Carbide, (Porto, Portugal, 3-8 septembre 2000).



**Figure 1:** SIMS deuterium concentration profiles: a) polycrystalline diamond submitted to rf plasma; b) submitted to remote rf plasma; c) submitted to micro-wave plasma.



**Figure 2:** Deuterium effusion spectra: a) as-grown sample; b) submitted to rf plasma; c) submitted to micro-wave plasma.



**Figure 4:** XPS C1s spectra of the sample surface: a) after rf plasma treatment; b) after annealing at 600°C; c) after chemical treatment.

