

# Nitrogen depth profiling of nitrogen-doped multilayer graphene

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Graphene layers grown on SiC have been grown by incorporating N atoms, with the purpose of tailoring their electronic properties. Several related experiments can already be found in the literature [1].

The doping of our samples is achieved after the graphene growth by exposing the samples to a nitrogen radical flux produced by a remote plasma source. The exposure results in the creation of nitrogen-related defects that are mostly simple substitutions of carbon atoms by nitrogen atoms, as seen by STM [2], together with a shift of the Fermi level corresponding to an n-type doping, as seen by STS [2] and ARPES (unpublished results). We can control the level of nitrogen concentration precisely (as seen by STM) by simply varying the exposure time.

XPS is widely used to study both the configuration and the quantity of nitrogen atoms in doped graphene samples. However, several difficulties are met when interpreting the data. Perhaps the most common problem (not always discussed) is that graphene is a one atom thick material whereas XPS probes in any case several nanometers so that the nitrogen signal might come also from nitrogen not embedded in the graphene lattice. This problem is encountered whenever the doping process is not followed by a transfer to another substrate.

In order to supplement our existing results, obtained by soft X-ray photoemission (Al  $K\alpha$  and 600 eV photon energy) as well as by STM, and to improve our understanding of this question, we performed XPS at intermediate photon energies.

From our previous results, we expect our post-growth treatment to dope, if not only the topmost graphene layer of our samples, preferentially the first few graphene layers. To test this hypothesis, we performed photon energy dependent measurements on doped samples at the beamline P04 at PETRAIII. Results from one representative sample are presented in Fig. 1: the C 1s and N 1s spectra recorded at different photon energies are displayed together with the evolution of the intensity ratio (N 1s/C 1s; the C 1s considered is the graphitic one around 284.4 eV) with the photon energy. A typical STM image of the same sample is also displayed. As can be seen from this last graph, we did not observe what we expected: the intensity ratio surprisingly increases with the photon energy (with an exception at  $h\nu=1500$  eV).

We believe that this behaviour is related to the very weak nitrogen signal that invalidates the common view according to which lower photon energy means higher surface sensitivity (a reason could be e.g. that the position of the peaks on the secondary electrons background matters for such small quantities).

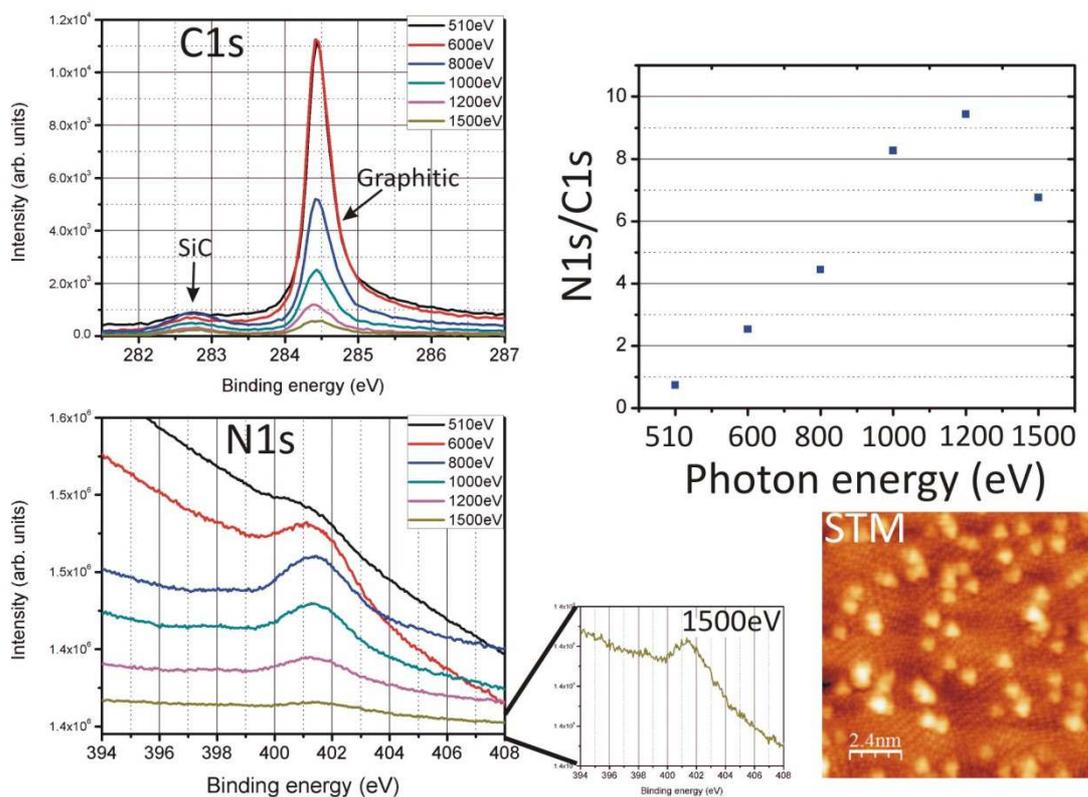


Figure 1: Clockwise, starting at the top left: C 1s spectra, nitrogen / carbon intensity ratio, STM image and N 1s spectra. More details in text.

## References

- [1] Wang *et al.*, ACS catalysis 2, 781 (2012).
- [2] Joucken *et al.*, Phys. Rev. B 85, 161408(R) (2012).