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STM Studies of $CrCl_3$-Graphite Intercalation Compounds

Intercalation at 800 °C of $CrCl_3$ into Kish Graphite leads to well defined stage 2 intercalation compounds. The eventual existence of Daumas-Herold domain in these compounds is represented schematically *figure 1*. An STM image [1] of such a surface will distinguish between domain (I) and (II), since this image is in fact the result of a convolution of the signals of the atoms of the first layers. Actually, on *figure 2*, we distinguish clearly these two domains, one in the upper part which images domain (II) while domain (I), in the lower part, can be considered as the image of one sheet of graphite rid of the influence of the other sheets. An enlarged view of this domain, *figure 3*, represents an hexagon of graphite with a measured bond length of 1.4 Å. *Figure 4* shows that the boundaries between the two domains are in fact intricated indicating that the Daumas-Herold domains have no definite shape.

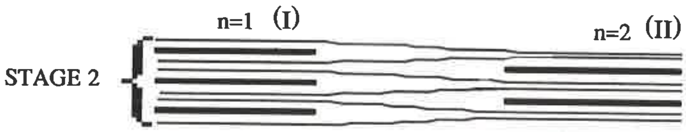

FIGURE 1. - *Representation of the surface of a second stage GIC, after it has been cleaved, according to the Daumas-Herold structure.*

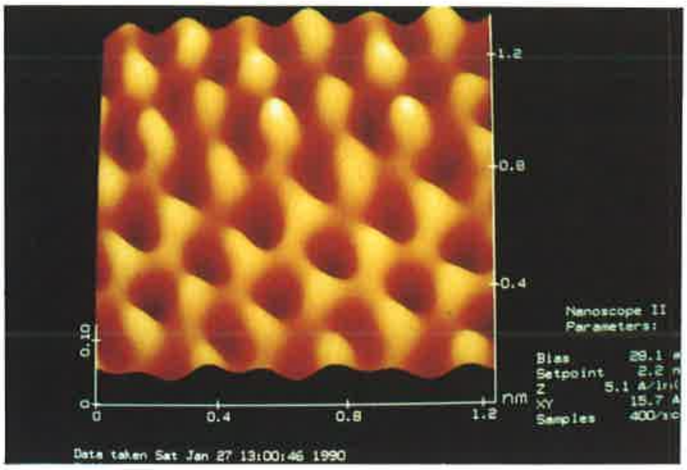

FIGURE 2. - *Transition zone between the two types of domains. One domain gives profiles similar to those of graphite (upper part), while the other (lower part) gives hexagonal arrangement as in* figure 2. *Experimental conditions : current 2.2 nA, bias voltage 28.1 mV, 8.7 Hz.*

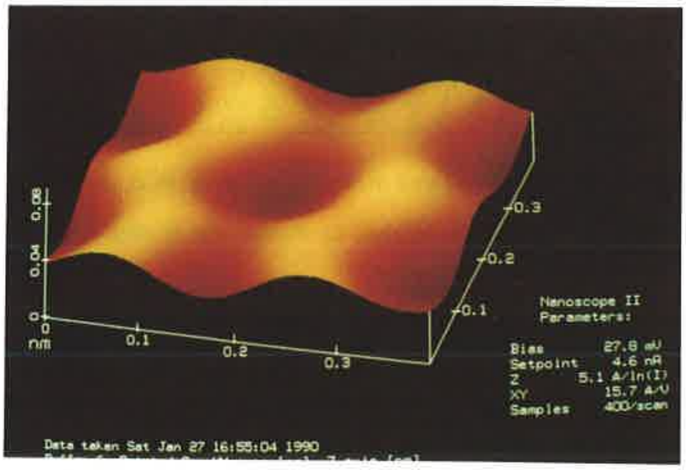

FIGURE 3. - *Close up on a part of image given in* figure 2. *The six atoms of the carbon hexagon appear clearly and are all identical.*

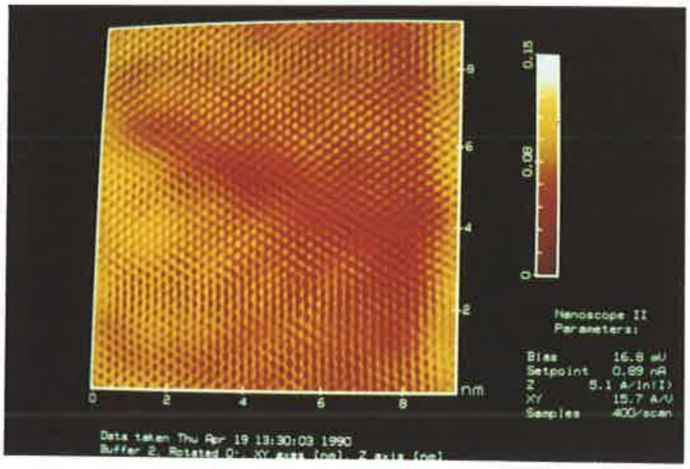

FIGURE 4. - *Two kinds of domains appear closely mixed. Experimental conditions : current 0.89 nA, bias voltage 16.8 mV, scan rate 8.7 Hz.*

Thus the intercalation allows us to study the surface of graphite with no interferences from the atoms of the underlying atoms. Therefore, the identification of the eventual in-plane defects will

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be easier on the GIC than on pure graphite. *Figure 5* and *6* show such defects [2] : from time to time one extra carbon atom enters a graphite hexagon ring producing an heptagon, and this heptagon is connected to the regular hexagonal lattice by a triangle. This structure, evidenced *figure 7* and *8* corresponding respectively to the images 5 and 6, is certainly not plane because the apparent bond length measured are unrealistic. The surface concentration of these defects is about one over 6 000 atoms. This order of magnitude is in good agreement with recent magnetic susceptibility measurements [3] which can be understood by a shift in Fermi level from − 0.026 eV for HOPG to + 0.006 eV in Kish graphite. In addition such a shift could explain the ability of Kish graphite to yield stage 2 GIC with $CrCl_3$ whereas only stage 3 is obtained in other graphite materials.

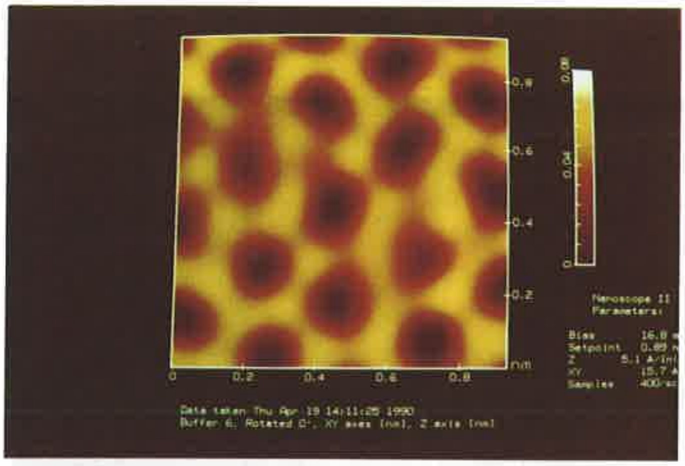

FIGURE 5. - *Two isolated defects.*

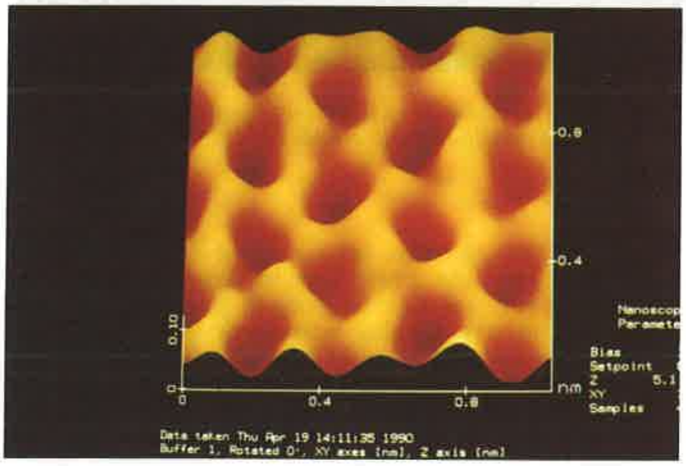

FIGURE 6. - *Two groups of defects ; one at the top left, the other in the lower-middle of the image. In this image the extra atoms are closer to each other and the deformations are more important than* figure 5.

Furthermore this shift in the Fermi level, modifying the charge density, will in turn modify the apparent corrugation of carbon atoms. Actually, using the same tips and the same experimental conditions (bias 40 mV set point 1nA) we observed *figure 9* a decrease of 40 % in the corrugation going from HOPG to Kish graphite.

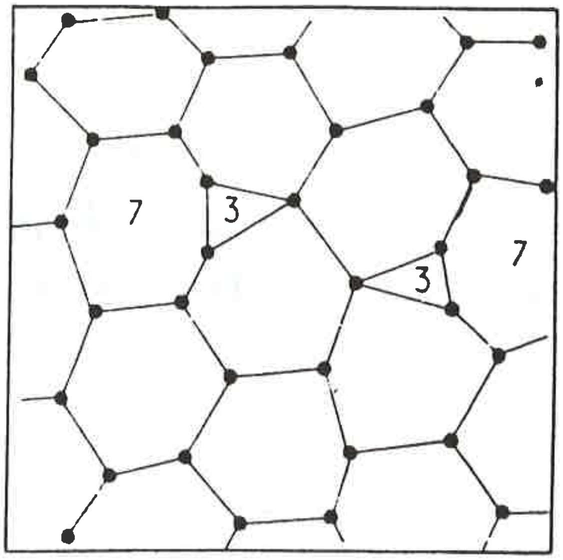

FIGURE 7. - *Schematic representation of the carbon atom lattice shown in* figure 5.

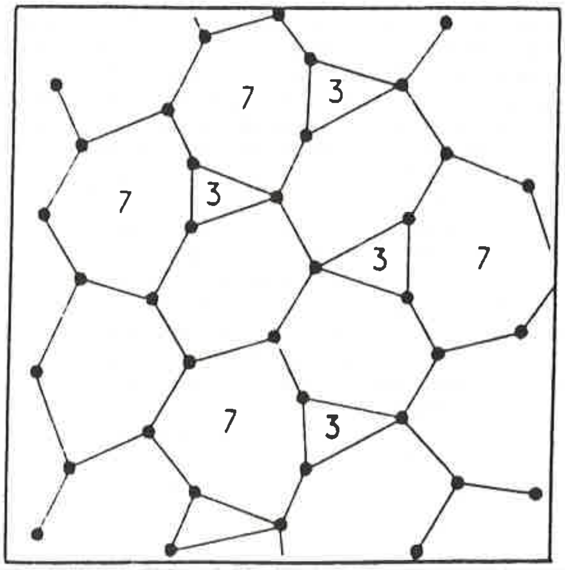

FIGURE 8. - *Schematic representation of the carbon atom lattice shown* figure 6.

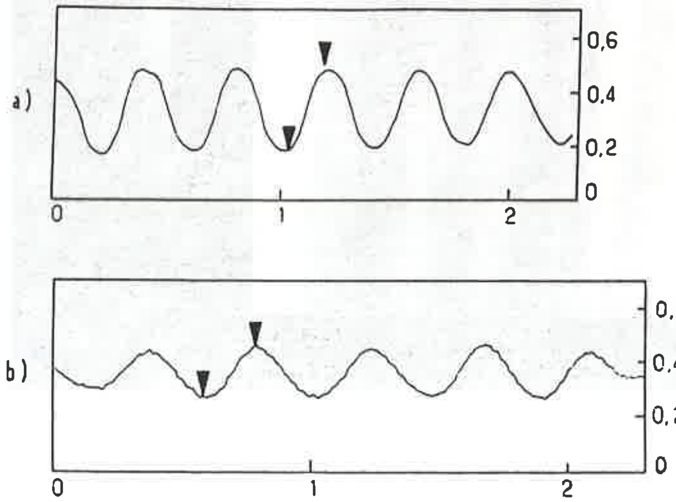

FIGURE 9. - *Corrugation amplitude of pure HOPG (a) and Kish (b) in the same tunneling conditions. Scales are in nanometer.*

References

[1] P. Biensan, J.C. Roux, H. Saadaoui, S. Flandrois, *Microsc. Microanal. Microstruct.*, **1990**, *1*, 103.
[2] P. Biensan, J.C. Roux, H. Saadaoui, S. Flandrois, *Microsc. Microanal. Microstruct.*, **1991**, *2*, 465.
[3] P. Biensan, thèse, Université de Bordeaux I, **1991**.