

Structural Study of Acetylene Thin Films Adsorbed on Exfoliated Graphite (0001)

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Abstract: *The structures of deuterated acetylene [C₂D₂] thin films adsorbed on the (0001) surfaces of exfoliated graphite have been investigated by elastic neutron diffraction. The aim of this study was to assess the structures of the condensed phases. Neutron diffraction spectra have been recorded at temperature ranging from 10K to 141K and for coverages corresponding to the monolayer completion 0.7 layer $\leq \theta \leq 1.23$ layers. Two bidimensional solid phases S₁ and S₂ have been identified. Their structures present commensurability with the (0001) graphite surface. At an intermediate coverage ($\theta = 1.0$ layer) there is coexistence of the two solid phases. The evolution of the recorded spectra with the temperature suggests the fusion of the S₁ solid to occur at $T_m(S_1) = 135 \pm 1K$. The fusion of S₂ solid happened at higher temperature: $T_m(S_2) > T_m(S_1)$.*

Keywords: neutron diffraction, physisorbed phases, commensurate phases

1. Introduction

Graphite, with its well defined and uniform surface, is still considered as a basic model for investigating adsorption mechanisms on carbonaceous substrates. In fact, a sheet of carbon atoms arranged in a hexagonal framework constitute the principal piece of graphene or carbon nanotubes. These later are known do to their scientific and technological interest and do to the particular properties they induce within sorbed phases. Besides noble gases and simple molecules, the adsorption of hydrocarbon ones on uniform substrates has been widely undertaken [1-8].

It has been inferred that the structures of alkanes molecules investigated at sub-monolayer coverage's and low temperature are isomorphous with rectangular unit cells of plane group pgg containing two molecules per cell [1].

Many studies have been devoted to the adsorption of acetylene on uniform surface substrates. If we limited our bibliographic study to graphite, MgO and NaCl as substrates we can quote [9-19] among a large list. However, only three groups attempted to determine the structures of condensed phases of C₂H₂ on graphite [13-15].

In the first study [13], Menaucourt & al. have measured adsorption isotherms of C₂H₂ on exfoliated graphite in the temperature range 110 K -160K. Based on the area occupied by a C₂H₂ molecule σ when adsorbed on graphite and at the various steps observed in an isotherm and by comparing σ to the surface of one molecule determined from Van Der Waals radius, they conclude about the existence of three (2D) dense phases. But they have no sufficient matter to establish these structures.

In the second one [14], Thorel & al. have measured neutron diffraction spectra by the film of acetylene adsorbed on graphite at low temperature T=57K. Two bidimensional (2D) solid phases have been reported respectively S₁ for the

coverage 0.65 to 0.89 layers and S₂ for the coverage 1.24 to 1.54 layers. At a coverage corresponding to 0.99 layer, the measured spectrum was attributed to a mixture of the previous S₁ and S₂ phases.

In the last study [15], Peters & al. have measured adsorption isotherms of C₂H₂ on graphite at the temperature range 109 to 184K. But since they used a compressed graphite as a substrate, their conclusion about the (2D) critical temperature T_C (2D) was incorrect and this point has been discussed elsewhere [16]. Concerning the structures of the (2D) solid phases, they tried to reproduce the neutron diffraction spectra by acetylene adsorbed on graphite and presented in [14]. Using Monte Carlo simulations, their investigation has improved the structures of the S₁ and S₂ solid phases by proposing for each one of the two (2D) solids a unit cell containing two molecules instead of those containing one molecule and proposed by Thorel [14].

Firstly, to complete a previous paper [18] where thermodynamic characterisation of acetylene thin films adsorbed on graphite has been exposed, and secondly to assess the structures of the two (2D) dense phases observed at the completion of the monolayer, we present here a detailed study of the structural investigation of C₂D₂ adsorbed on exfoliated graphite. The structures retained for the two bidimensional solid phases were supported by the thermodynamic characterization mentioned above.

2. Experimental Techniques

Neutron diffraction spectra by the two-dimensional acetylene phases condensed on graphite were measured at the Atomic Study Center in Saclay, France. We used the instrument G6-1 equipped with a multi-detector formed of 400 cells and covering an angular range $2\theta = 80^\circ$, as well as a cryostat allowing to regulate between 2K and 300K to better than 0.05K, and on rather long times (the counting of a spectrum can last up to 12 hours), the wavelength used is λ

= 4.725Å. The spectra giving the intensity (number of hits) as a function of the angle are recorded during the manipulation.

In this type of manipulation, a background noise spectrum is recorded (bare substrate), then spectra (substrate + adsorbate) are recorded, the spectra corresponding to the condensed phase are therefore difference spectra. So that the signal due to the condensed matter is sufficiently intense and as in all neutron diffraction experiments we used deuterated acetylene C_2D_2 because the coherent diffusion coefficient of deuterium is greater than that of hydrogen.

In such experiments, the structural parameters (a, b, γ) of the unit cell of the (2D) solid phase are deduced from the peaks positions, whereas the peaks intensities are correlated to the orientation of the molecules in the unit cell. on the other hand, the width at mid-height of a peak of Miller indices hk is an indication of the extent of the order in the direction hk i.e. short range order or long range order

In what follows, we first give the spectra measured as a function of the recovery rate by keeping the temperature constant and from which we have determined the structures of the different condensed phases.

Then we will present the evolution of the neutron diffraction spectra by the determined (2D) solids as a function of temperature in order to locate their melting temperature and / or loss of the orientational order within.

3. Results and Discussion

Neutron diffraction spectra have been recorded for coverages $0.7 \text{ layer} \leq \theta \leq 1.23 \text{ layer}$ at temperatures $10K \leq T \leq 141K$. In figure-1 are shown the spectra measured at $T=121K$ for coverages $\theta_1=0.7 \text{ layer}$; $\theta_2=1.1 \text{ layer}$ and at an intermediate coverage $\theta_{12}=1.0 \text{ layer}$. Such spectra are difference ones (with and without adsorbate). The saw-tooth line shape of the observed peaks is the signature of two dimensional (2D) diffracting matters. The features at $1.778 \text{ \AA}^{-1} \leq Q \leq 1.913 \text{ \AA}^{-1}$ in the neutron pattern arise from imperfect subtraction of the graphite background. As it can be point out from figure-1, the spectrum measured at the intermediate coverage (1.0 layer) includes peaks observed respectively at 0.7 layer and 1.1 layer. And this will constitute a primary indication that the corresponding phase can be interpreted as a combination of those corresponding to 0.7 and 1.1 layers.

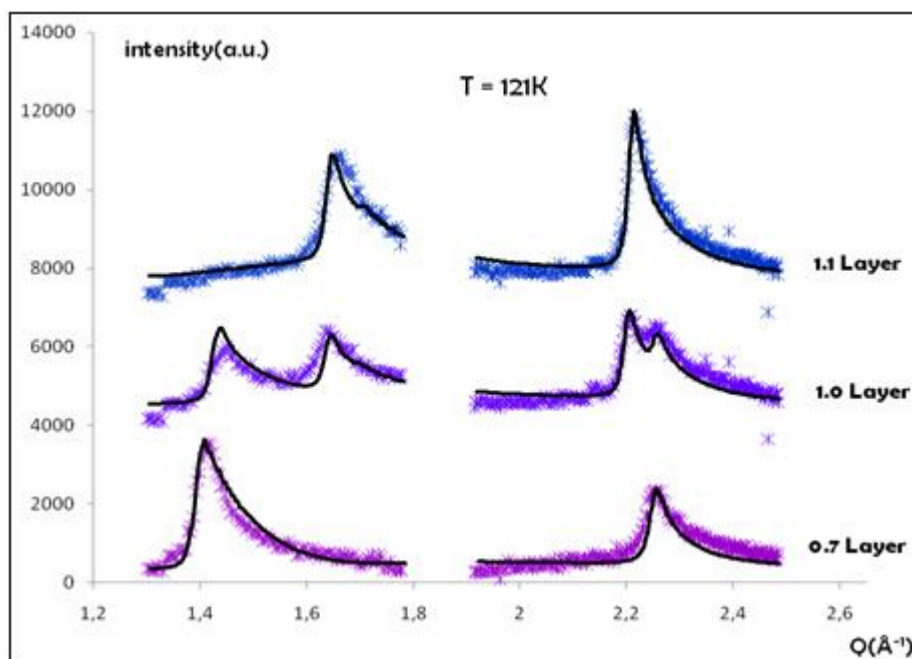


Figure 1: Neutron diffraction spectra by solids S_1 and S_2 measured respectively at coverage rates $\theta_1 = 0.7 \text{ layer}$ and $\theta_2 = 1.1 \text{ layer}$. The intermediate spectrum includes the peaks observed in S_1 and S_2 . Fits calculated from the parameters given in Table-2 are shown in solid lines

Identification of the various (2D) solids

Neutron diffraction spectra by acetylene adsorbed films on graphite were recorded for three different coverages, respectively $\theta_1=0.7 \text{ layer}$, $\theta_{12}=1.0 \text{ layer}$ and $\theta_2=1.1 \text{ layers}$. They are differentiated by the positions of the peaks and their intensities. In Figure-1 we give the three spectra measured at 121K.

In the following table (Table-1) we give the positions of the peaks deduced from the measured spectra as well as the corresponding inter-reticular distances.

Peak positions given by Thorel et al. are in agreement with those deduced from our spectra. We will see later that the interpretation proposed by Thorel et al. is in agreement with the one we will present although the structures they have proposed seem to us to be incorrect as it will be demonstrated.

θ (layer)	$Q_i (\text{\AA}^{-1})$	$Q_i (\text{\AA}^{-1})$ [14]	d (\AA)
0.7	1.416	1.449	4.437
	2.257	2.291	2.784
1.0	1.451		4.330
	1.636		3.804
	2.209		2.844
	2.255		2.786
1.1	1.662	1.648	3.780
	2.217	2.244	2.834

Table 1: Peaks position and corresponding inter-reticular distances observed in neutron diffraction spectra by two-dimensional C_2D_2 films adsorbed on graphite at 121K. The positions of the main peaks observed by Thorel [14] at 57K and for coverage rates close to ours are also given to make comparison easier.

To determine, unambiguously, the parameters (a, b, γ) of the unit cell, at least, three peaks are needed. Unfortunately, in the spectra measured at θ_1 and θ_2 coverages, only two peaks are observed and additional information has to be considered to determine the parameters of the unit cells. The first information comes from the area occupied by a C_2H_2 molecule in the (2D) condensed phase ($\sigma_{C_2H_2} \approx 16 \text{\AA}^2$), determined by comparison of a krypton isotherm to a C_2H_2 one, measured separately on the same sample of graphite at

Θ (layer)	a (\AA), b (\AA), γ ($^\circ$)	$Q_{hk} (\text{\AA}^{-1})$	$d_{hk} (\text{\AA})$	$\sigma_{C_2H_2} (\text{\AA}^2)$	N mol/uc	Position and orientation of molecules (x, y, z, θ , φ)
0.7	9.00; 3.56; 90	$Q_{20}=1.396$ $Q_{21}=2.250$	$d_{20}=4.50$ $d_{21}=2.792$	16.02	2	0; 0; 4.55; 60; -70 4.50; 1.78; 4.55; 60; 70
1.0	$S_{12} = 0.53S_1 + 0.47S_2$					
1.1	7.36; 4.46; 90	$Q_{20}=1.707$ $Q_{21}=2.213$	$d_{20}=3.680$ $d_{21}=2.839$	16.41	2	0; 0; 2.50; 90; 45 3.68; 2.23; 3.75; 90; 135

Table 2: Parameters (a, b, γ) of the unit cells proposed for the C_2D_2 (2D) solids formed at 121K and for the coverages respectively equal to 0.7 layer and 1.1 layers. These meshes allow indexation of the observed peaks. The positions and orientations of the molecules in the elementary cells are also given: θ being the angle that the C=C axis of the C_2D_2 molecule form with the vertical to the graphite surface and φ the angle between the projection of the C=C axis on the plane (x, y) and the x-axis.

In figure-1, in addition to the recorded spectra (symbols) we give the corresponding fits (continuous line) obtained from the parameters summarized in table-2. For the spectrum measured at a coverage rate equal to 1.0 layer, we noticed that it is formed of the peaks observed at $\theta_1 = 0.7$ layer and those observed at $\theta_2 = 1.1$ layers. This spectrum therefore corresponds to a solid where the two phases S_1 and S_2 coexist. We then reproduced this spectrum by combining the spectra corresponding to S_1 and S_2 . The solid S_{12} is a mixture of S_1 and S_2 ($S_{12} = 0.53S_1 + 0.47S_2$). It is important, at this level, to note that Thorel et al. have reported that the phase observed for a coverage rate $\theta = 0.99$ layer, is the coexistence of the two phases (phase α observed for coverage rates ranging from 0.65 to 0.89 layer and β phase for coverage rates ranging from 1.24 to 1.54 layers). Their phase α corresponds to what we denote by S_1 , while their phase β corresponds to what we call S_2 . We preferred S_1 and S_2 to avoid confusion with the ordered (α phase) and partially disordered (β phase) phases of bulk acetylene (3D).

approximately equal reduced temperature T/T_{3C} where T_{3C} is the bulk critical temperature. The second information considered here is the quadrupolar interaction which induces a T shaped arrangement of the molecules since the quadrupole moment of C_2H_2 is important ($Q=7.2D\text{\AA}^{-1}$). So, only unit meshes containing two molecules and allowing indexation of the observed peaks are retained.

The elementary meshes, whose parameters are given in the table-2, are rectangular centered, they each contain two molecules: one placed at the origin and the other at the center. The positions and orientations of the molecules in the elementary meshes and which made it possible to calculate the best fit of the experimental spectra are summarized in the same table.

For the solid S_1 , the molecules are slightly straightened and their C=C axis are at an angle of 30° with respect to the surface and are oriented symmetrically with respect to the x-axis.

For the solid S_2 the molecules are flat on the surface and make an angle of 90° between them.

In agreement with the study conducted by Thorel et al., the solid S_1 is stable at least between 0.7 and 0.85 layer which are the two coverage rates at which neutron diffraction spectra were recorded. For the solid S_2 , the corresponding spectra were recorded for $\theta = 1.1$ layers and $\theta = 1.23$ layers. What seemed to us inconsistent in the interpretation of Thorel et al. are the structures proposed for solids S_1 and S_2 where they have considered a simple single-molecule mesh which means that the quadrupole moments will be aligned, and this seems to be in disagreement with the fact that the C_2H_2 molecules are endowed with a strong quadrupole moment $Q = 7.2D\text{\AA}^{-1}$. Moreover, by energetic calculations, Anne Terlain [20] has shown that an almost rectangular centered structure is, from energetic point of view, more stable than that proposed by Thorel.

The spectra measured at $T = 133.5K$ and for coverages equal to 0.85 layer and 1.1 layers, corresponding respectively to solids S_1 and S_2 , have been reproduced by considering the structures mentioned in table-2 with a slight modification in the parameters (figure-2). And this constitutes additional proof of the validity of the structures proposed for the solids S_1 and S_2 on the one hand and of the invariance of the structures when the temperature increases on the other hand, ie a strong index of the commensurability of each of the structures (2D) with the surface (0001) of graphite.

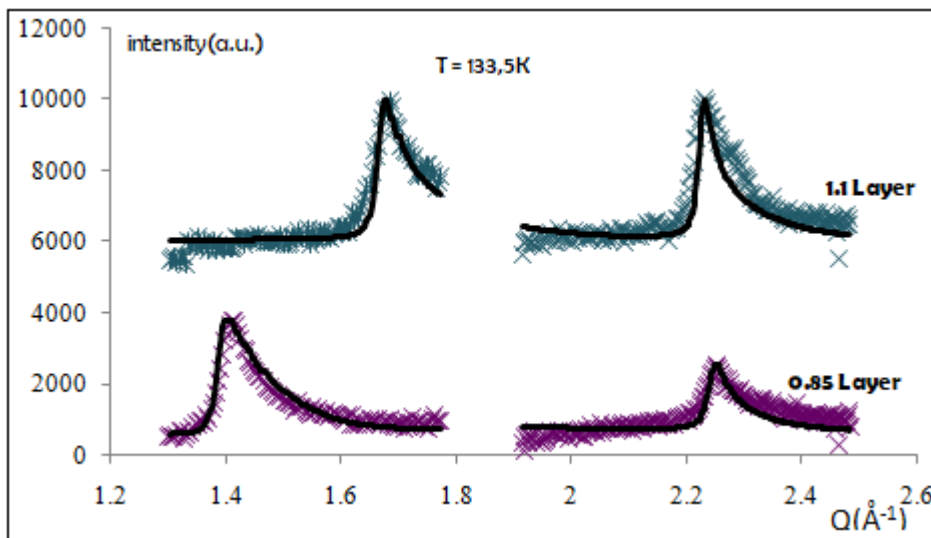


Figure 2: Neutron diffraction spectra by two different coverages and corresponding to S_1 and S_2 phases, measured at $T = 133.5K$

A schematic representation of the elementary cells is given in Figure-3, below.

Figure 3 (b)

Figure 3: Schematic representation of the two elementary cells at $T = 121K$ (a): solid S_1 , (b) solid S_2 .

The reproduction of the elementary cell of the solid S_1 (figure-4) shows that this solid phase has commensurability at a great distance with the (0001) surface of the graphite. In matrix notation, this commensurability is given by

$$\begin{pmatrix} a_{ads} \\ b_{ads} \end{pmatrix} = \begin{pmatrix} 33 & 0 \\ 5 & 10 \end{pmatrix} \begin{pmatrix} a_{sub} \\ b_{sub} \end{pmatrix} \text{ where } a_{sub} = b_{sub} = 2.46\text{\AA}.$$

The commensurable cell contains 108 molecules of C_2D_2 . This commensurability is supported on the one hand by the fact that the solid S_1 is stable for a coverage rate ranging from 0.7 to 0.85 layer or even lower coverage 0.65 layer [14] and secondly in that this phase is stable up to 133.5K (see evolution of the spectra as a function of temperature).

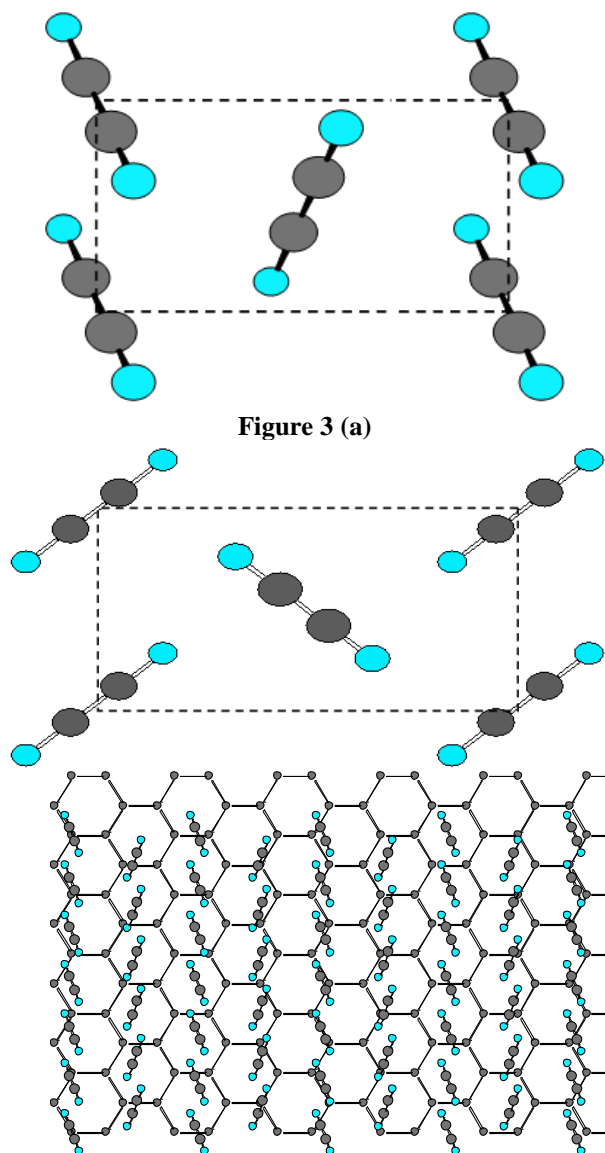


Figure 4: Solid S_1 : commensurable mesh containing 108 molecules of C_2D_2

In matrix notation, it presents the commensurability

$$\begin{pmatrix} a_{ads} \\ b_{ads} \end{pmatrix} = \begin{pmatrix} 33 & 0 \\ 5 & 10 \end{pmatrix} \begin{pmatrix} a_{sub} \\ b_{sub} \end{pmatrix}$$

The location of the C₂D₂ molecules centers on the (0001) graphite surface, as in the following figure, is arbitrary. Low energy electron diffraction manipulations as well as energy minimization calculations will give the epitaxy of the films with the graphite surface.

Similarly, the reproduction of the elementary cell of the solid S₂ (figure-5) shows that this solid phase present an original commensurability with the (0001) face of the graphite. In matrix notation, this commensurability is given

as $\begin{pmatrix} a_{ads} \\ b_{ads} \end{pmatrix} = \begin{pmatrix} 7 & 2 \\ 4 & 11 \end{pmatrix} \begin{pmatrix} a_{sub} \\ b_{sub} \end{pmatrix}$, the commensurable cell

contains 22 molecules of C₂D₂. This commensurability is consolidated on the one hand by the fact that the solid S₂ is stable for a coverage rate ranging from 1.1 to 1.23 layers or even 1.54 layers [14] and secondly by the fact that this phase is stable up to 141K, the highest temperature at which we recorded a diffraction spectrum for this coverage rate (see evolution of the spectra as a function of temperature).

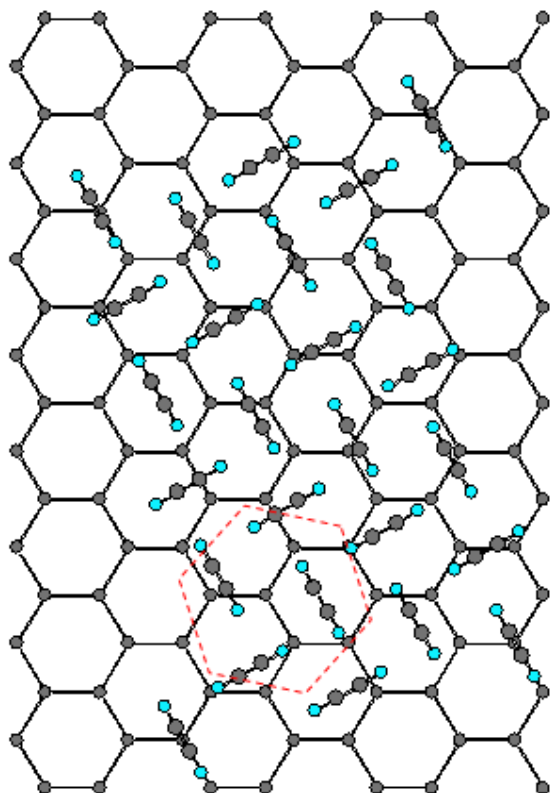


Figure 5: Solid S₂: commensurable cell containing 22 molecules of C₂D₂. In matrix notation, it presents the

commensurability: $\begin{pmatrix} a_{ads} \\ b_{ads} \end{pmatrix} = \begin{pmatrix} 7 & 2 \\ 4 & 11 \end{pmatrix} \begin{pmatrix} a_{sub} \\ b_{sub} \end{pmatrix}$

In this phase, S₂, and as it can be shown in figure-5, the centers of mass of the admolecules are organized in an almost bidimensional (2D) hexagonal structure (4.40Å, 4.40Å, 117.57 °). A slight increase in the coverage (from 1.1 to 1.23 layers) does not change the structure of the solid but the parameters of the mesh are slightly modified, leading the centers of mass of the admolecules to organize themselves in an even closer (2D) hexagonal structure (4.29Å, 4.29Å, 118.62 °). This compartment is illustrated in table-3 where the evolution of the parameters of the rectangular centered cells according to the coverage rate is given. It seems that this evolution will continuous until the structure of the S₂ solid reaches a strong commensurability $\sqrt{3} \times \sqrt{3}$ with the (0001) surface of graphite. But, an increase in the coverage must be accompanied by at least partial overlap of the molecules so that they keep the same structure. The calculated spectra of the S₂ solid show that the adsorbed molecules remain parallel to the surface independently of the coverage and the compression of the film is manifested at the level of the admolecules height by a slight variation of their position along the z-axis.

Table 3: Evolution of the parameters of the elementary meshes: for T = 121K and for T = 133.5K. Solids S₁, S₂ are stable in a broad overlap rates.

Temperature (K)	solid	Coverage (layer)	Parameters of the unit mesh			average area occupied by a molecule σ (Å ²)
			a (Å)	b (Å)	γ (°)	
133.5	S ₁	0.7	9.04	3.56	90	16.09
		0.85	9.02	3.56	90	16.05
121	S ₂	1.1	7.36	4.46	90	16.41
		1.23	7.38	4.38	90	16.16

Evolution of neutron diffraction spectra as a function of temperature:

a) Solid S₁ (0.7 ≤ θ ≤ 0.85 layer)

The evolution of the diffractograms recorded at θ = 0.7 layer and θ = 0.85 layer as a function of the temperature are represented in Figs. 6 and 7 respectively. These spectra are attributed to the solid S₁, they have two main peaks which undergo a slight displacement when the temperature increases. This displacement reflects a small expansion of the mesh when the temperature increases. They disappear beyond 133,50K, leaving their place to two rather large bumps, which are the signature of a liquid phase. It follows that the solid S₁ has a melting temperature T_m (S₁) of the order of 135 ± 1K.

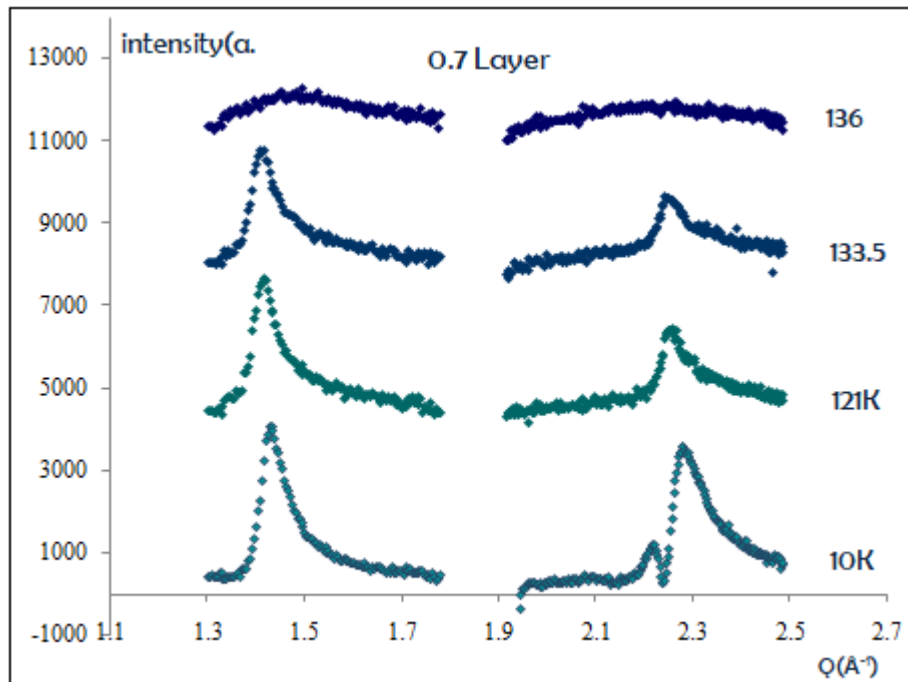


Figure 6: Evolution of the neutron diffraction spectra by 0.7 layer of C_2D_2 adsorbed on graphite as a function of temperature. The fusion temperature of solid phase S_1 was estimated to be about $135 \pm 1K$.

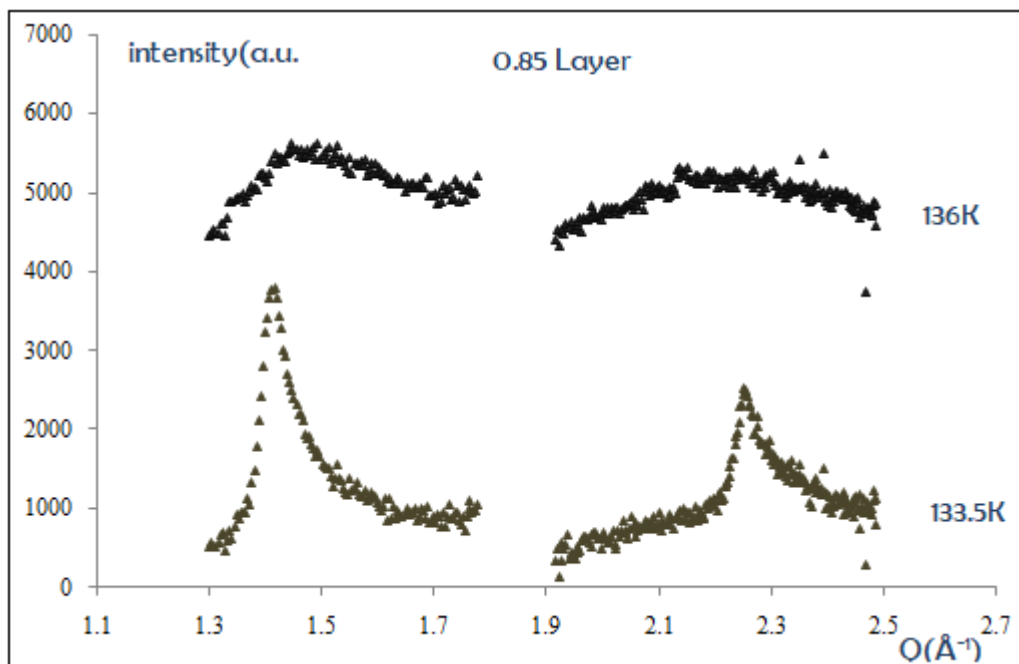


Figure 7: Evolution of the neutron diffraction spectra by 0.85 layer of C_2D_2 adsorbed on graphite as a function of temperature

b) Solid S_2 ($1.1 \leq \theta \leq 1.23$ layers)

The evolution of the neutron diffraction spectra as a function of temperature by the coverage rates of 1,1 layers and 1,23 layers is shown in Figs. 8 and 9 respectively. The peaks keep their dissymmetric forms even at 141K ($\theta = 1.23$ layers), thus indicating that this solid S_2 has a melting point $T_m(S_2)$

greater than 141K. It is found that $T_m(S_2) > T_m(S_1)$ and this is in agreement with the fact that S_2 present a stronger commensurability with the surface (0001) of the graphite compared to that of the solid S_1 .

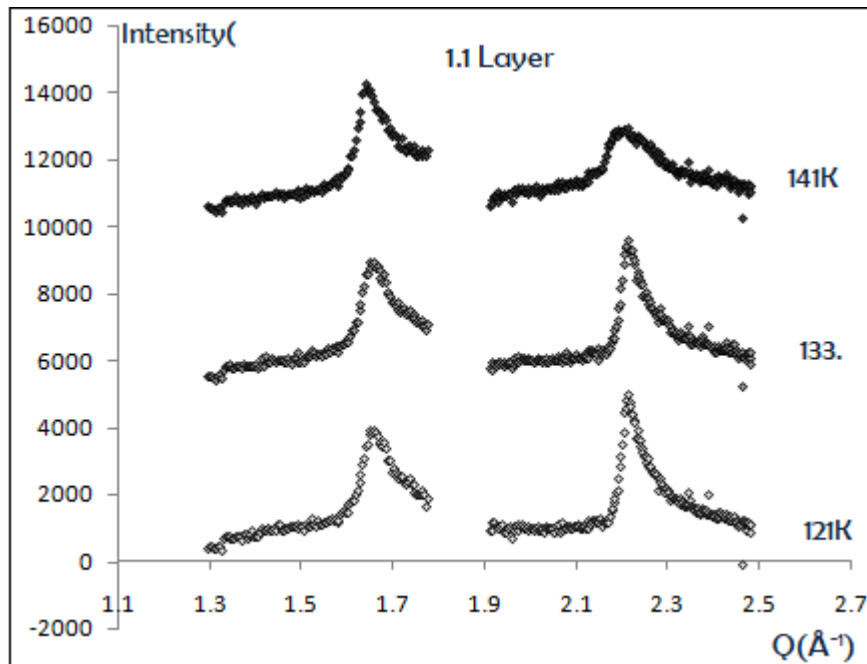


Figure 8: Evolution of the neutron diffraction spectra by 1.1 layer of C_2D_2 adsorbed on graphite as a function of temperature. Even at 141K, the peaks are well defined suggesting the condensed phase to be solid rather than liquid. The solid S_2 melts at $T > 141K$.

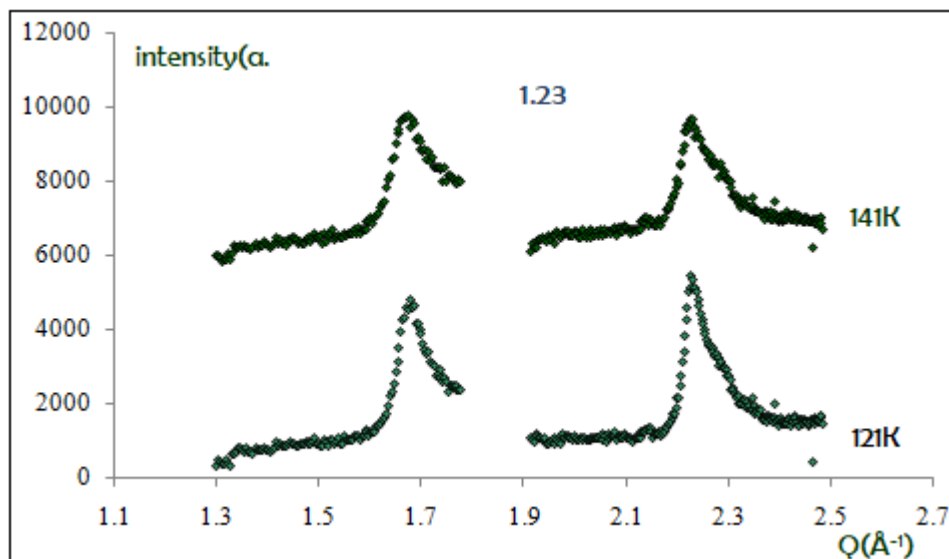


Figure 9: Evolution of the neutron diffraction spectra by 1.23 layer of C_2D_2 adsorbed on graphite as a function of temperature. The solid character of the adsorbed phase is evidenced even at 141K.

c) Solid S_{12} ($\theta = 1.0$ layer)

For this coverage rate, the evolution of the spectra as a function of temperature is given in Figure-10. The spectra measured at 121K and 133.50K show four peaks where each pair characterizes one of the two solids S_1 and S_2 . Beyond 133.5K, only the characteristic peaks of S_2 persist. Indeed, the peaks relative to the solid S_1 disappear beyond the melting temperature $T_m(S_1)$. The fact that in the spectra

measured at 136K and 141K, the peaks are less intense and poorly defined does not involve the melting of the S_2 solid at a temperature below 141K. In fact, once the domains corresponding to the solid S_1 are melted, the molecules that constituted them will find a mobility leading them to partially cover the domains corresponding to the solid S_2 thus blurring their signal.

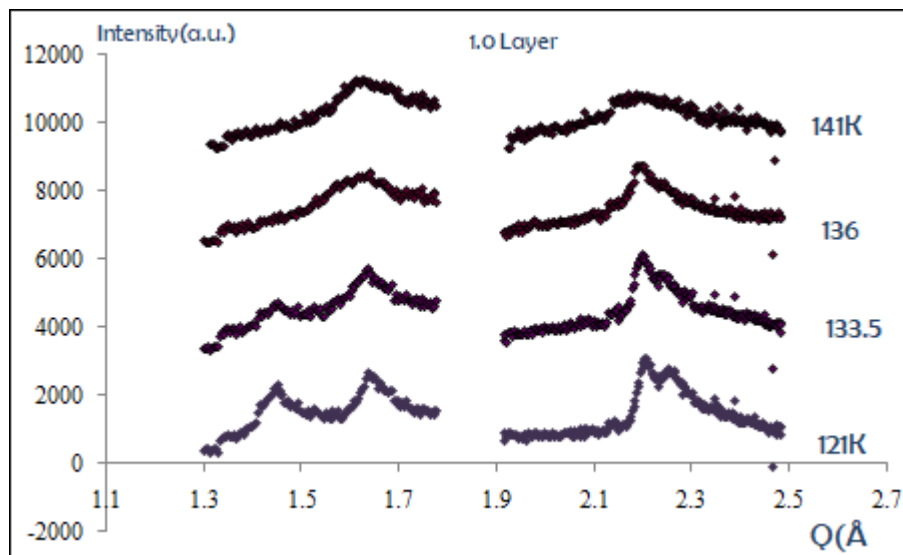


Figure 10: Evolution of the neutron diffraction spectra by 1.0 layer of C_2D_2 adsorbed on graphite as a function of temperature. The peaks relatif to the S_1 solid phase disappear between 133.5 K and 136K.

4. Conclusion

Neutron diffraction technique has been used to determine structures of the (2D) deuterated acetylene adsorbed phases on graphite. Spectra have been measured at coverages ranging from 0.7 to 1.23 layers. Two bidimensional (2D) solid phases have been identified. The structure of the solid S_1 which is stable at a wide range of coverage (0.65 to 0.85layer) present long range commensurability with the (0001) face of graphite. This commensurability is also supported by the fact that the corresponding structure was stable from 10K to 133.5K. The fusion of S_1 occurs at $T_m(S_1) = 135 \pm 1K$. The structure of the solid S_2 corresponding to a coverage ≥ 1.1 layer present an original commensurability with the (0001) surface of graphite. This commensurability was supported on one hand by the fact that it is stable from 1.1 layer to 1.54 layer and on the other hand by the fact that is stable at temperature $> 141K$, the upper temperature at which a spectrum has been measured. So $T_m(S_2) > 141K$. An other originality, characterizing the C_2D_2 /graphite system comes from the (2D) solid phase corresponding to a coverage equal to 1.0 layer and which was identified as a mixture of S_1 and S_2 solids. A similar situation has been encountered when studying the adsorption of C_2D_6 on MgO (100) [21]. Finally we can conclude that this structural characterization, coupled to a previous thermodynamic one constitute an important data to elaborate phase diagram of the two dimensional matter of C_2D_2 adsorbed on graphite.

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