Effects of electron exposure on graphene with intercalated oxygen

AUTHOR JONATHAN WIDOV

SUPERVISOR JAN KNUDSEN
CO-SUPERVISOR VIRGINIA BOIX

DEGREE B.SC
PROJECT DURATION 3 MONTHS
DEPARTMENT PHYSICS

May 2021

LUNDS
UNIVERSITET

1   Abstract

New 2D materials with high electron mobility can potentially be used for small and energy efficient electronic devices in the future. One such material that has been studied intensely is graphene. Despite the large amount of studies, the question of how to create useful shapes of graphene remains unanswered. In this work I studied how graphene may be spatially etched by intercalating oxygen before exposing the graphene to an electron beam. Using scanning tunneling microscopy I studied the morphology of electron exposed graphene without intercalated oxygen. I studied graphene with intercalated oxygen using x-ray photoelectron spectroscopy to determine the effects of electron exposure on such a surface. I find that it is possible to etch the graphene by intercalating oxygen before exposing the surface to an electron beam. When the graphene lacks the intercalated oxygen it shows no changes in its overall structure. My studies provide insight into the requirements for electron induced etching of graphene.

Acknowledgments

I would like to thank my supervisor Jan Knudsen. Thank you for working with me and teaching me this spring. I do not believe that I would gotten anywhere without your patience and motivation. I would also like to thank Virginia Boix for helping me in the lab. Even if things did not always work out you always seemed to have the will to start over. As a final note I would like to thank my family as they always remained supportive even as my ramblings became ever more incoherent.

CONTENTS

List of Abbreviations

CVD....Chemical Vapour Deposition

STM....Scanning Tunneling Microscope

XPS.....X-ray Photoelectron Spectroscopy

LDOS..Local Density of States

MFP....Mean Free Path

FCC.....Face Centered Cubic

TPG....Temperature Programmed growth

BE........Binding Energy

R0........Rotation 0

2 INTRODUCTION

The carbon allotrope graphene is one of the most studied 2D materials due to its several unique properties. Graphene is, for example an excellent electrical conductor. If combined with other 2D materials, having band gaps one can use this property to build electronic devices. Graphene is also a good heat conductor. This property also makes graphene a useful material for device fabrication, since the heat conductivity helps to dissipate heat from devices and keep them cold. This is important since small components are prone to break down if overheated. To produce electronic devices from graphene it is necessary to shape or modify its macro structure. One way to achieve this is by spatially controlled etching. This can for example be done by electron-induced etchings in an oxygen atmosphere using a spatially confined electron beam [1].

Such methods have become more prevalent in recent years showing a stark contrast to some of the first experiments dealing with graphene. Graphene is a relatively new discovery and was only isolated for the first time in 2004 by Novoselov *et al.* [2]. Novoselov and his group used the so-called Scotch tape method to peel layers off graphene of graphite. Graphite consists of closely packed carbon layers held together by weak Van der Waals forces. The scotch tape method works since it breaks these bonds using the adhesive force between the scotch tape and the topmost graphene layer.

In most modern experiments, graphene is not fabricated using the scotch tape method. Instead, it is fabricated by so-called chemical vapor deposition (CVD) of a carbon containing pre-cursor dosed on a hot metal surface. Alternatively, it may be fabricated by what is known as temperature programmed growth where the metal is heated after the surface has been saturated with the pre-cursor. Metals upon which graphene has successfully been grown includes for example, Cu, Ni, Pt, Ru, and Ir [3].

Beyond simply growing or etching of graphene there have been many studies focused on how we may alter or combine graphene with other materials. A simple way of doing this is a variation the already mentioned CVD where the pre-cursors of both the graphene and other material would be dosed sequentially or simultaneously. CVD does however come with the limitation of only producing mono-layers of both materials.

Because of this, alternative methods that allow multilayered structures to be grown on graphene or other similar materials are needed. Some examples of such methods include high-temperature high-pressure direct CVD growth, plasma-enhanced CVD, molecular-beam epitaxy (MBE) and co-segregation from the substrate [4]. A problem with these methods is that they are limited to specific material combinations.

One technique that has been commonly used to deposit materials with high spatial accuracy is called Focused Electron-Beam Induced Deposition (FEBID). This technique has been used to alter the 2D structure of graphene by introducing localized defects as well as to create 3D carbon structure by dissociating molecules previously adsorbed to the surface.

With the ability to deposit materials onto the surface of graphene comes the question if this can be used to spatially etch graphene. As mentioned, it is possible to etch graphene using electrons in an oxygen environment. To build on these ideas the question becomes whether it is possible to place oxygen below graphene and then expose it to the electron beam. However, since oxygen is a gas this cannot be done using deposition. Instead, intercalation may be used to trap oxygen atoms underneath the surface of the graphene

In this bachelor thesis an etching method using intercalated oxygen and electron exposure is investigated. In my work I characterize pristine and O-intercalated graphene flakes supported by Ir(111) before and after electron exposure. A scanning tunneling microscope (STM) was used to characterize pristine and electron exposed graphene without the intercalated oxygen. The graphene with intercalated oxygen was investigated using x-ray photoelectron spectroscopy (XPS).

3 Methods

3.1 Scanning Tunneling Microscopy

The working principle of a scanning tunneling microscope (STM) is that a tip is raster-scanned over the sample while a voltage is applied between the tip and surface.

3.1.1 Electron Tunneling

In a classical view electrons can only travel through a vacuum barrier if their kinetic energy is higher than the barrier height determined by the sample and tip work functions. Due to quantum mechanical tunneling there is, however, a non-zero possibility that the electron travels through the gap. This is illustrated in figure 1 where the wave function of the electron is shown to partially pass through the barrier.
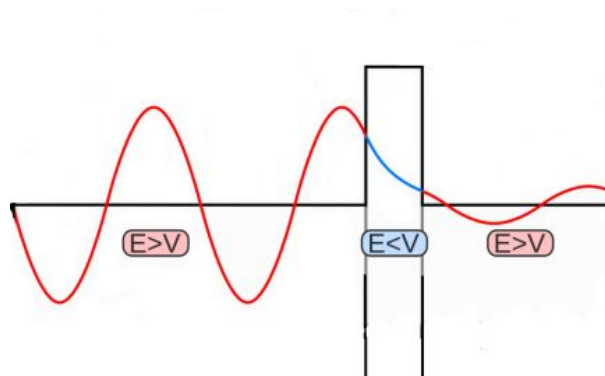

Figure 1: This image shows the amplitude of an electrons wave function decreasing as it travels through a shifting potential object.

In an STM experiment, a bias voltage difference is applied between the surface and the tip. Therefore, the surface must be conductive for the STM to function.

3.1.2 The Local Density of States of Solid Surfaces

The local density of states (LDOS) describes the electron density at a particular position inside or outside the surface at a certain energy. The LDOS plays a crucial role in determining the tunneling probability since a larger electron density results in a greater tunneling probability. The LDOS vary when moving between individual atoms allowing for the STM to produce images with atomic resolution.

3.1.3 Scanning In practice

By raster scanning a sharp conductive tip across a surface, a current can be created from electrons tunneling from the surface to the tip. The contrast of the image is then generated by changes in the local density of states. This can be considered a distinction to other imaging techniques that create a direct topological image of the surface while an STM generates a topological image of the LDOS of the surface.
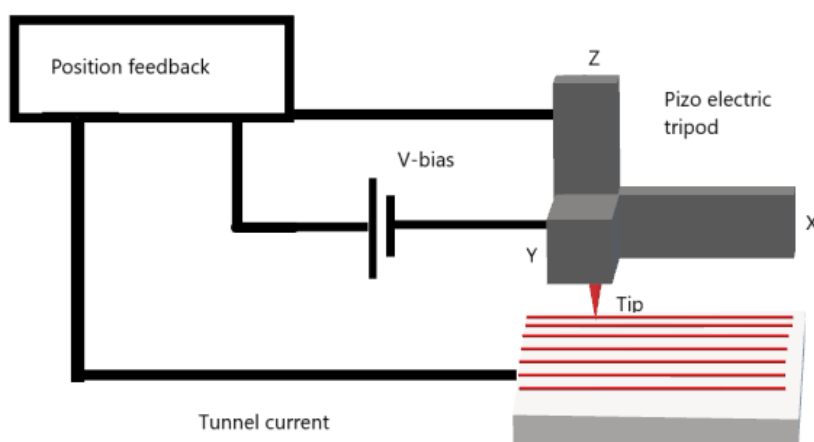

Figure 2: Illustration of an STM setup.

STM can work in two modes using the concept above. The first mode constant current mode attempts to keep the current stable while adjusting and measuring the extension of the tip. The second being constant height mode works by keeping the tip at a stable level above the surface while measuring the current. In my studies I used the constant current mode. In order to move in a precise enough manner to achieve an atomic resolution the tip of the STM is moved using piezo electric materials, which are materials that changes their dimensions when a voltage difference is applied to them. This allows the tip to scan areas that are only a couple of nano-meters wide.

To ensure that the sample is kept clean it is important to conduct the STM experiment at an extremely low pressure typically around 10^{-10} mbar. Additionally, it is also important that the sample is vibrationally isolated, since the tip is moving ~1 nm above the surface. This is because small external vibrations coupling to the tunneling microscope will disturb or might even cause the tip to crash into the surface.

3.2   X-ray Photoelectron Spectroscopy

X-ray photoelectron spectroscopy (XPS) is based on the photoelectric effect. The technique uses monochromatic light in the x-ray regime for photoemissions of core electrons. The emitted electrons are collected and their kinetic energy is measured. Since the initial photon energy is known the binding energy of the electron can be determined by subtracting its kinetic energy.

$$BE = h\nu - E_{kin} \tag{1}$$

The binding energy of the measured electrons provide both elemental and chemical information about the surface studied. The elemental information comes from the different core level energies of different elements. For example, the O 1s level is around 530 eV, while the C 1s level is around 285 eV. The chemical information comes from the so-called chemical-shifts due to core levels being slightly perturbed by the surrounding atoms. As an example, the C 1s of graphene will have a binding energy of 283 eV when oxygen has been intercalated. This 2 eV shift can in a simple electrostatic view be understood from the highly electronegative O-atoms, which will lower the C 1s level of carbon, as valence electrons are removed from the C-atom.

3.2.1   The Mean Free Path of Electrons in a Solid Material

The mean free path (MFP) of an electron is the average distance that an emitted electron can travel before it scatters. This distance depends heavily on the kinetic energy of the emitted electron. In my experiments the x-ray was tuned so that the electrons were given a kinetic energy of approximately 100 eV. At this energy the MFP is as low as 5 Å in solid materials and only electrons from the two topmost atomic layers are able to escape without losing energy by scattering processes.

3.2.2   Synchrotron Based X-ray Photoelectron Spectroscopy

When generating the x-ray radiation, the best method is to use a synchrotron. In a synchrotron, accelerated electrons kept in a storage ring are used to emit photons. The photon energy can be tuned by changing how the particle travels in order to maximize the intensity for a desired energy. The light emitted does however come out at several energies and is therefore filtered in order to produce only the desired energy. This possibility of selecting any energy to produce is what makes synchrotron-based sources ideal for XPS studies.

3.2.3 The Background in Photosmission Spectra

In XPS spectra all peaks will be observed together with some background. This background originates from electrons that have lost energy before being detected. As these electrons are no longer characteristic for the core level they originate from they are usually removed from the spectra.

3.2.4 Gaussian Fits for Spectra

The simplest way to fit a photoemission peak after the background has been removed is to use a Gaussian curve shape described by:

$$\frac{1}{\sigma\sqrt{2\pi}}exp\left(-\frac{1}{2}\frac{(x-\mu)^2}{\sigma^2}\right) \tag{2}$$

The reason it takes on a Gaussian shape is due to the used X-rays also being produced with a Gaussian distribution around the desired energy. The detector furthermore also has a minimum resolution for the detected electrons.

3.2.5 Voigt Approximation

If the experiment where to be perfect with an X-ray source with a single wavelength and a detector capable of resolving the electrons perfectly there would still be a broadening occurring. This broadening is due to the lifetime of the core hole generated when the electron is emitted. This broadening takes on a Lorentzian shape.

To include both broadening effects discussed above it is necessary to create a convolution of a Gaussian and Lorentzian curve shape. Such a convolution is called a Voigt curve shape. An analytical approximation to the real numerical convolution of the Voigt shape is called a pseudo-Voigt. For all XPS spectra in this thesis a pseudo-Voigt shape was used for deconvolution.
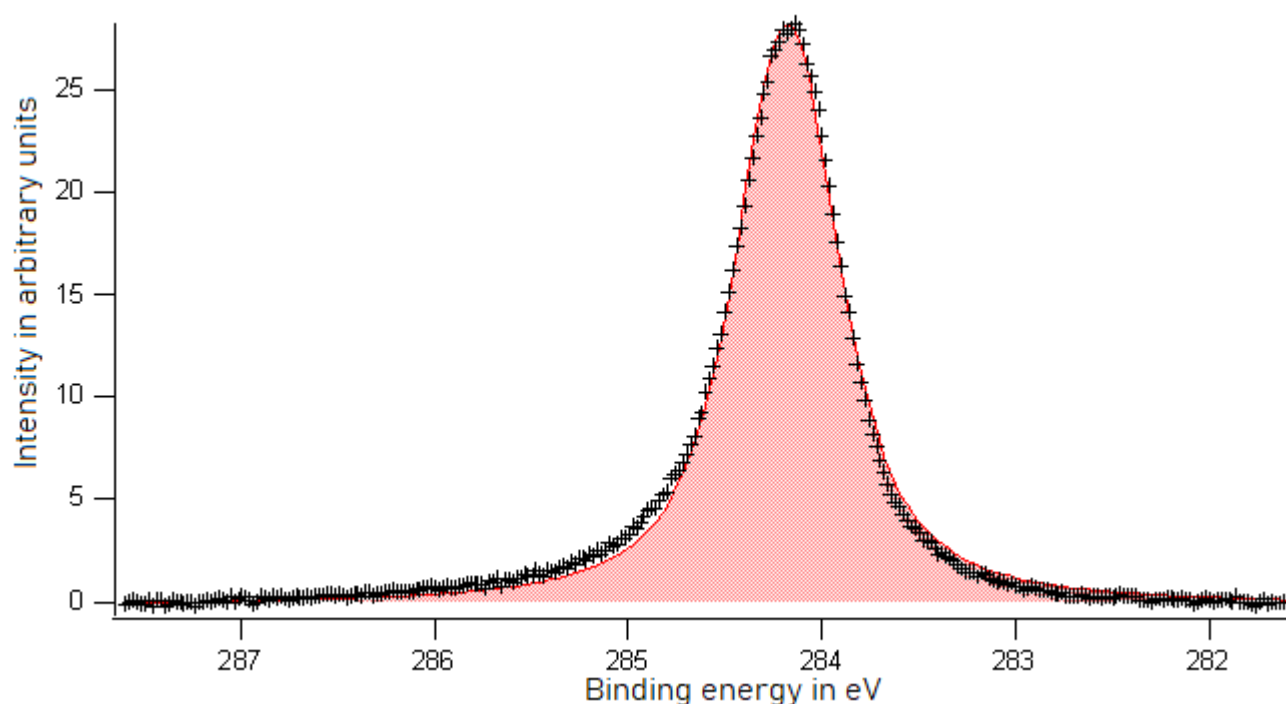

Figure 3: An image of a C 1s peak where the data have been marked with crosses while the fit has been coloured red.

4   RESULTS AND DISCUSSION

My studies consist of two parts. The first part focuses on STM characterization of pristine and electron exposed graphene. The second part focuses on the analysis of previously taken XPS data of pristine and O-intercalated graphene before and after electron exposure. This section is organised by first introducing the graphene growth and STM characterization. Following this, XPS is used to characterize pristine graphene, then O-intercalated graphene, and finally O-intercalated and electron exposed graphene.

4.1   The structure of graphene on Ir(111)

In my experiments, I grew graphene on a Iridium(111) surface. Iridium has a face centered cubic (FCC) crystalline structure. Its (111) surface is hexagonal shaped with an in-plane lattice constant of 2.72 Å [5]. In my experiments it was important to achieve a coverage of around 50%. The reason is that O_2 needs bare Ir(111) patches to dissociate before the atomic and chemisorbed oxygen atoms can diffuse and intercalate under the graphene patches. Figure 4 shows a representative image graphene grown onto Ir(111). The bottom left corner of figure 4 shows a patch of bare iridium.
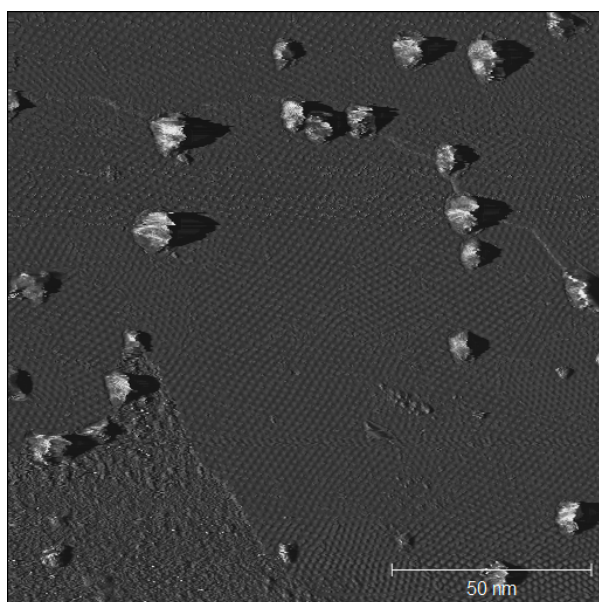

Figure 4: Image of graphene layer on iridium. This image will be discussed in further detail in section 4.3.

The lattice constant of graphene is 2.46 Å [6] which in combination with the lattice constant of iridium being 2.57 Å means that the graphene does not line up perfectly. However due to both being crystalline in nature they form a repeated hexagonal pattern called a Moiré pattern. This pattern is the large honeycomb structure seen in figure 5b).
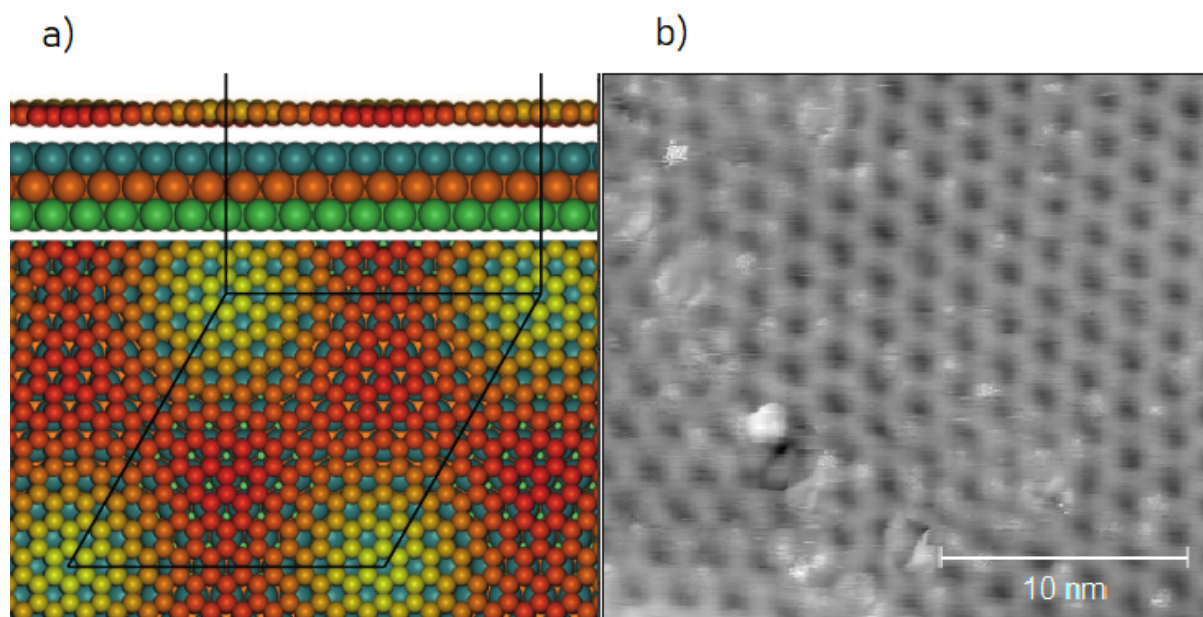

Figure 5: Image shows the comparison between the theoretical structure in figure 5 a) and an STM image in figure 5 b).

Figure 5a) shows the combined structure of graphene and iridium having a honeycomb and hexagonal structure, respectively. The unit cell of this structure is formed with 2 carbon atoms and 1 iridium atom.

4.2   Graphene Growth

Before growing graphene, the surface of the crystal was prepared by several cycles of argon sputtering and annealing to high temperatures. These treatments allow us to achieve an atomically clean surface.

In my experiments the graphene flakes were grown using two different methods. The first of these is temperature programmed growth (TPG). When using this method, the substrate is dosed with C_2H_4 near room temperature until the sample has been fully

saturated. Subsequently the sample is heated to elevated temperatures. This method as described in [1] is well suited to grow graphene flakes with the same rotation (R0). Smaller flakes can more easily be rotated on the surface which lowers the risk that one traps graphene flakes in local minimum and rotated structures. The second method is chemical vapor deposition (CVD). In contrast, to the TPG method one doses the precursor onto the sample at elevated temperature. This method has the benefit that one can control the coverage by the pressure of the precursor and the dosing time. In my STM experiments TPG was used to grow graphene onto Ir(111). In more detail ethylene was dosed at a pressure of 10^{-6} mbar for 100 s before the surface was brought to high temperatures (1000 – 1100 K). Due to the high temperature ethylene disassociates immediately when in contact with the heated Ir(111) and H_2 desorbs leaving carbon on the surface. While the system is kept heated the carbon atoms remain mobile and react with one another forming graphene islands.

Figure 6 shows this process with the iridium atoms depicted as grey circles and the carbon atoms as blue ones. The movements of the carbon atoms are drawn as arrows. In figure 6d) a pair of malformed graphene rings marked by 5 and 7 are shown to have formed due to the presence of the iridium edge.
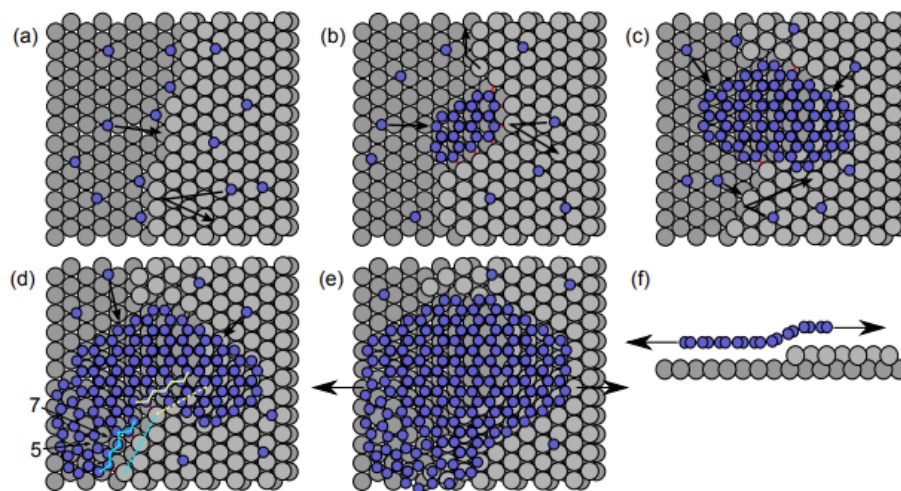

Figure 6: A top view of graphene on Ir(111) visualized as blue and grey circles respectively. The arrows show the movements of the carbon atoms as graphene is being formed. The points marked with 5 and 7 show points where graphene has not formed properly. This image has been reproduced from [7].

With the carbon free to move over the iridium surface the result of this reaction is the creation of small crystalline carbon flakes. Ideally, the graphene will be produced in a way where the entire coverage is oriented in the same way. While growing the graphene

there may appear to be separate levels. These levels are the different Ir(111) terraces. The graphene growth stops once the surface is covered by a single layer of graphene, since the ethylene does not dissociate on graphene. Therefore, no additional layers of graphene are formed.

4.3   STM data

Figures 7 and 8 show STM images (1300 × 1450 Å^2) acquired after TPG growth using three iterations of dosing ethylene at 10^{-6} mbar for 100 s before heating to 1000 K. After the third time the sample was flashed to 1300 K.

4.3.1   Pristine graphene Characterization

Figure 7 shows a representative image of the pristine graphene grown using the parameters above with a number of features marked. The red circles show the location of protrusions. The bare iridium is coloured blue. The edges between Ir levels are shown in yellow. A number of large rotational domains can be found in the image. Intersections of such domains are coloured green.
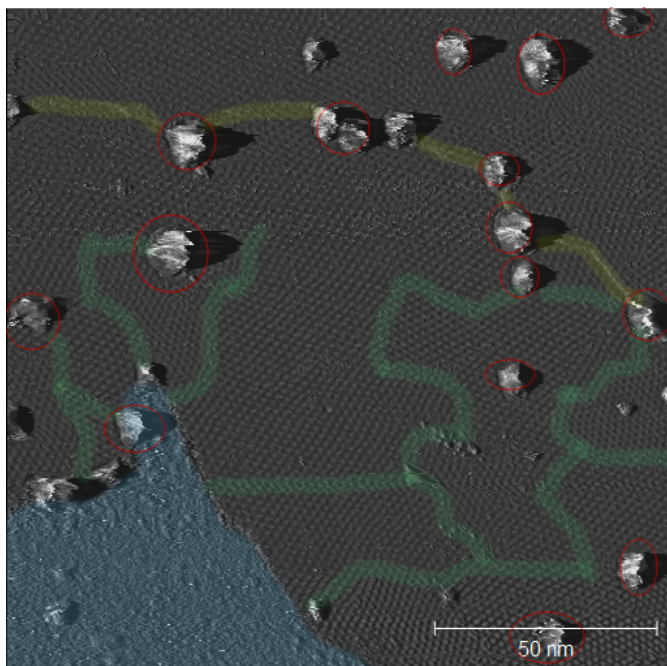

Figure 7: STM image (1300 × 1450 Å^2) of graphene grown on Ir(111). Beyond the graphene there are four things marked. In red are protrusions, in blue is an area of iridium, in yellow is graphene grown over steps in the iridium and intersections of rotational domains are marked in green.

The features described above can often be observed in STM images of graphene on Ir(111) [7]. The protrusions marked in red are most likely caused by the presence of contaminants on the surface of the iridium. The different rotational domains occur when flakes with different orientations grow into one another. The reason the flakes have different orientation is because the sample was not flashed to a high enough temperature.

4.3.2   Effect of Electron beam on Pristine Graphene

After electron exposure the sample was again imaged with the STM. Figure 8 shows a representative STM image. In this image the same type of protrusions as in figure 7 can be seen marked in red. This image also shows the presence of iridium levels marked in yellow. The bare iridium is also here marked in blue and the intersections of rotational domains are marked in green.
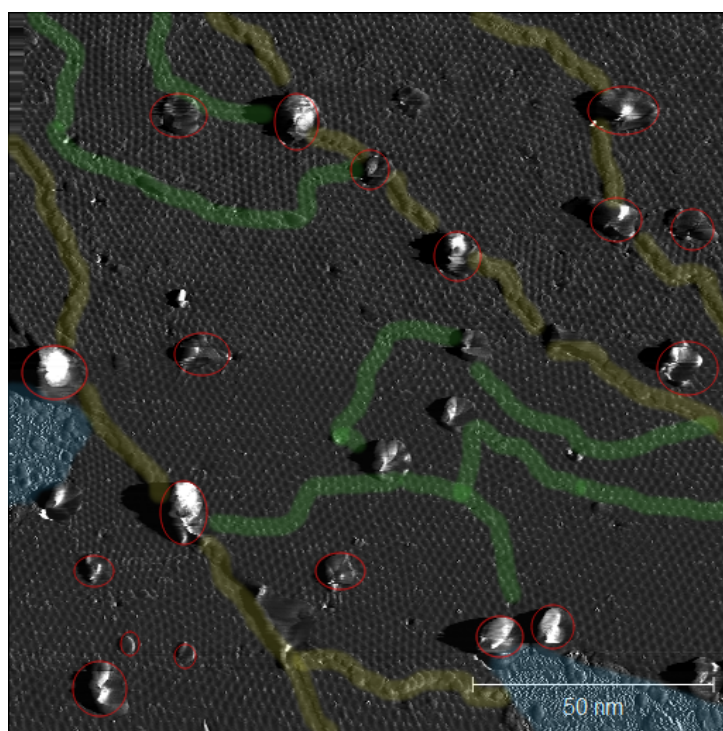

Figure 8: Image of the graphene after being exposed to the electron beam. In red are protrusions, In blue is an area of iridium, in yellow are graphene grown over steps in the iridium and intersections of rotational domains are marked in green.

Figure 9 is a closer look at the sample after electron exposure where the domain intersections have been coloured green, the iridium levels have been coloured yellow and the protrusions are circled in red.
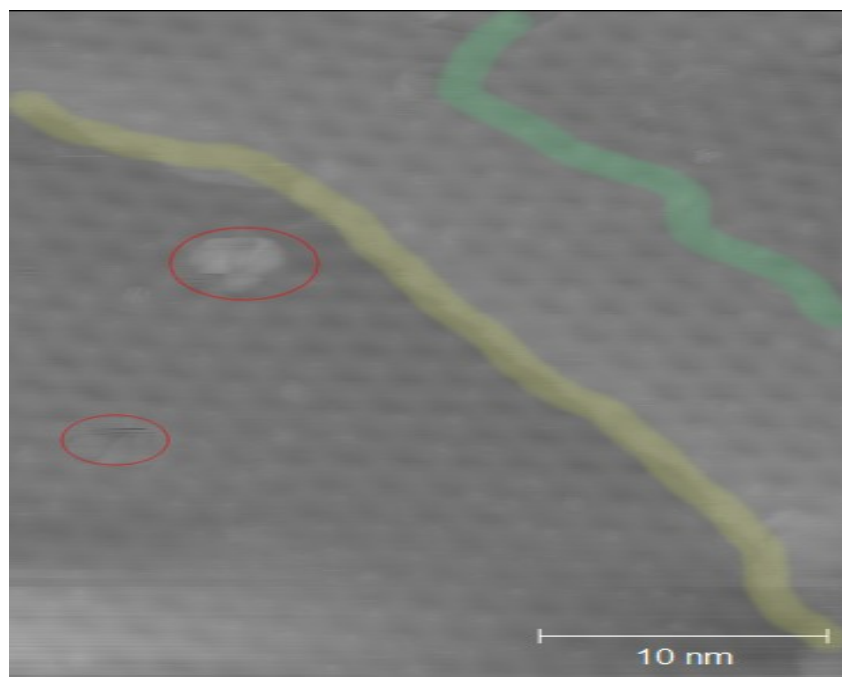
Figure 9: A closer look at the surface of the graphene after electron exposure

The images shown in figure 8,9 show no evident changes when compared to figure 7. This suggests that the electron exposure has had no effect on the pristine graphene.

4.4 XPS Data

This section covers the analysis of previously taken XPS data analyzing graphene flakes before and after oxygen intercalation, and after subsequent electron exposure. The structure of this part will follow the process of growing graphene on Ir(111), intercalating oxygen, exposing the sample to an electron beam, and finally flashing the sample.

4.4.1 Graphene Flakes Grown on Iridium(111)

Graphene was grown onto iridium(111) using three cycles of TPG dosing ethylene at a pressure of 10^{-7} mbar for 100 s at a temperature of 400 K before flashing to 1129 K. The resulting coverage estimated from the XPS (not shown) was around 90%.
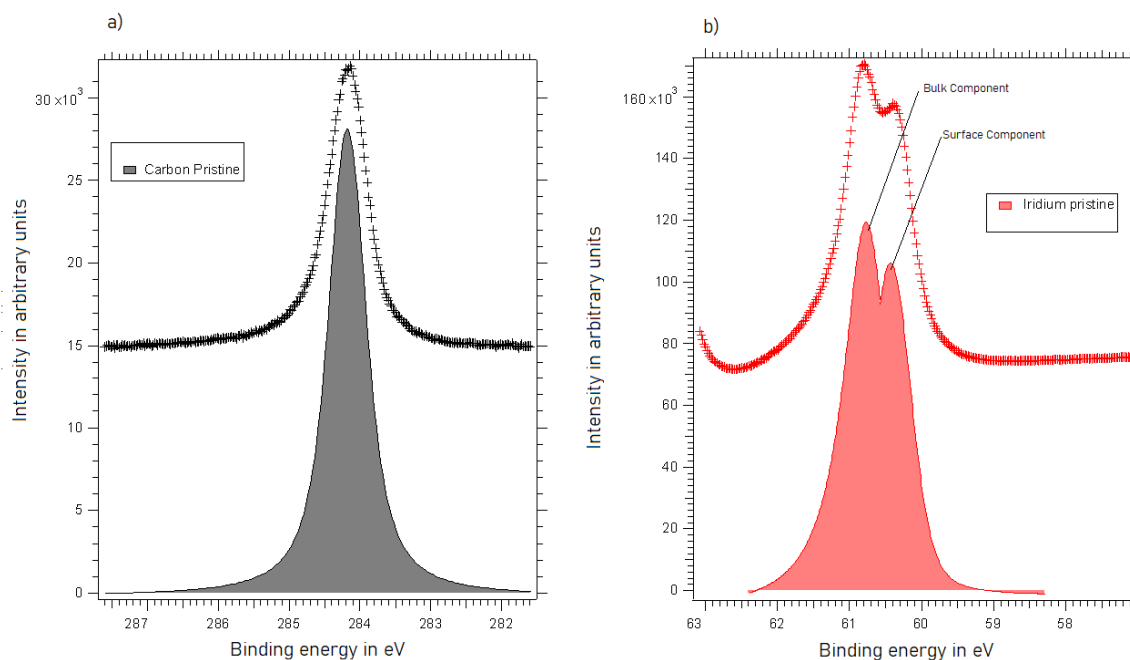

Figure 10: XPS spectrum of pristine graphene grown using CVD.

The graphs in figure 10 show C 1s and Ir $4f_{7/2}$ spectra acquired from pristine graphene flakes. In figure 10a) a single component is observed at an energy of, 284.2 eV. Grånäs *et al.* [8] found that the C 1s peak for graphene grown on iridium has an energy close to 284.1 eV.

In figure 10b) two peaks can be found at 60.8 eV and 60.4 eV. Grånäs *et al.* [8] found two peaks for Ir with BE at 60.81 eV and 60.31 eV matching the ones observed here. The peak found at 60.8 eV corresponds to the atoms found in the bulk of the Ir while the other peak shows the binding energy of the atoms at the surface. The peak found at 60.4 eV has a lower binding energy due to the surface atoms breaking the crystalline structure of the bulk which means they are not bound to their surrounding atoms in the same way as the bulk.[8]
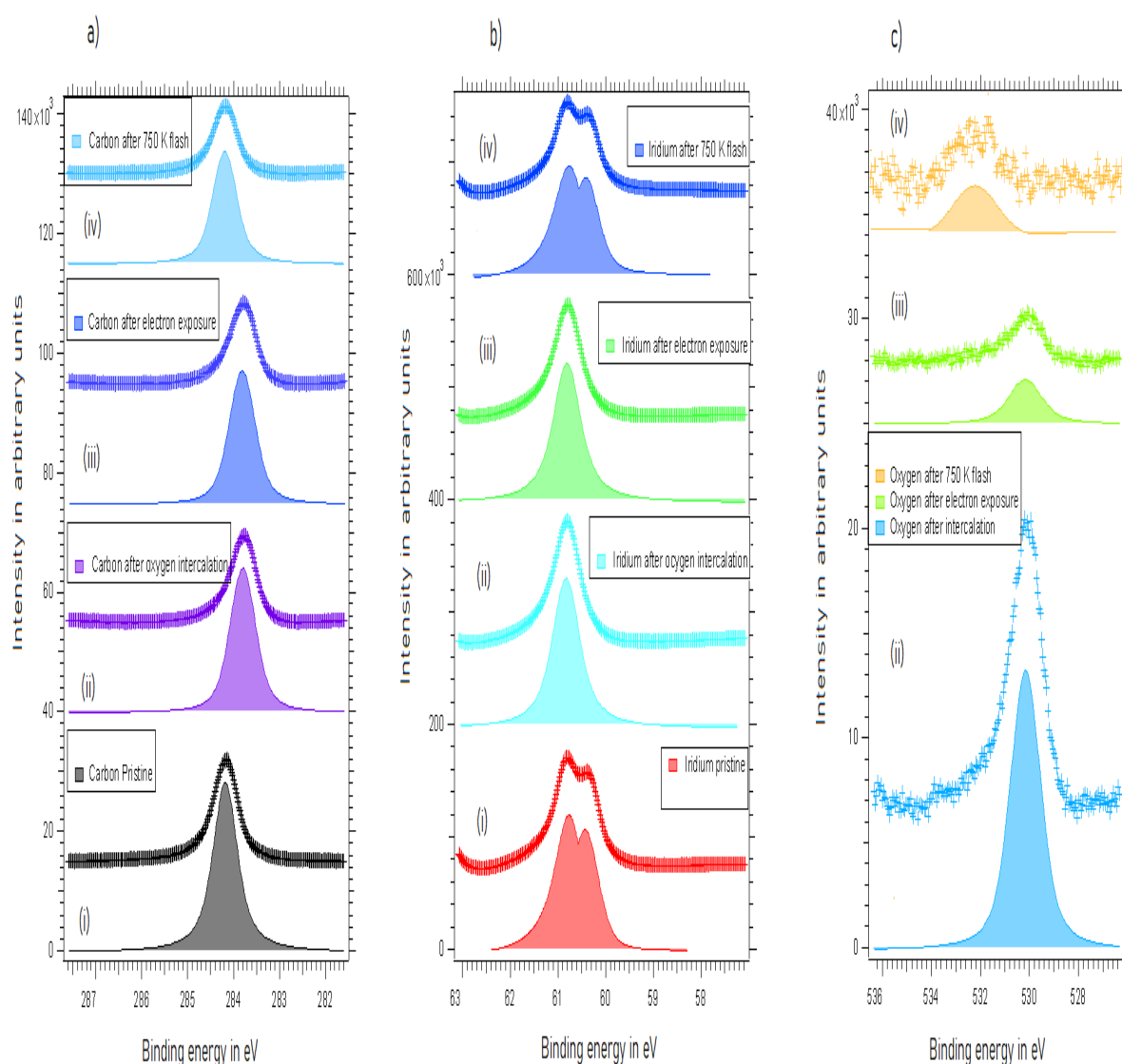

Figure 11: Spectra of C 1s (a), Ir 4f$_{7/2}$ (b) and O 1s (c) at all stages of the experiment. From the bottom the spectra show the pristine state (i), after oxygen intercalation (ii), after electron beam exposure (iii) and finally after a 750 K flash (iv).
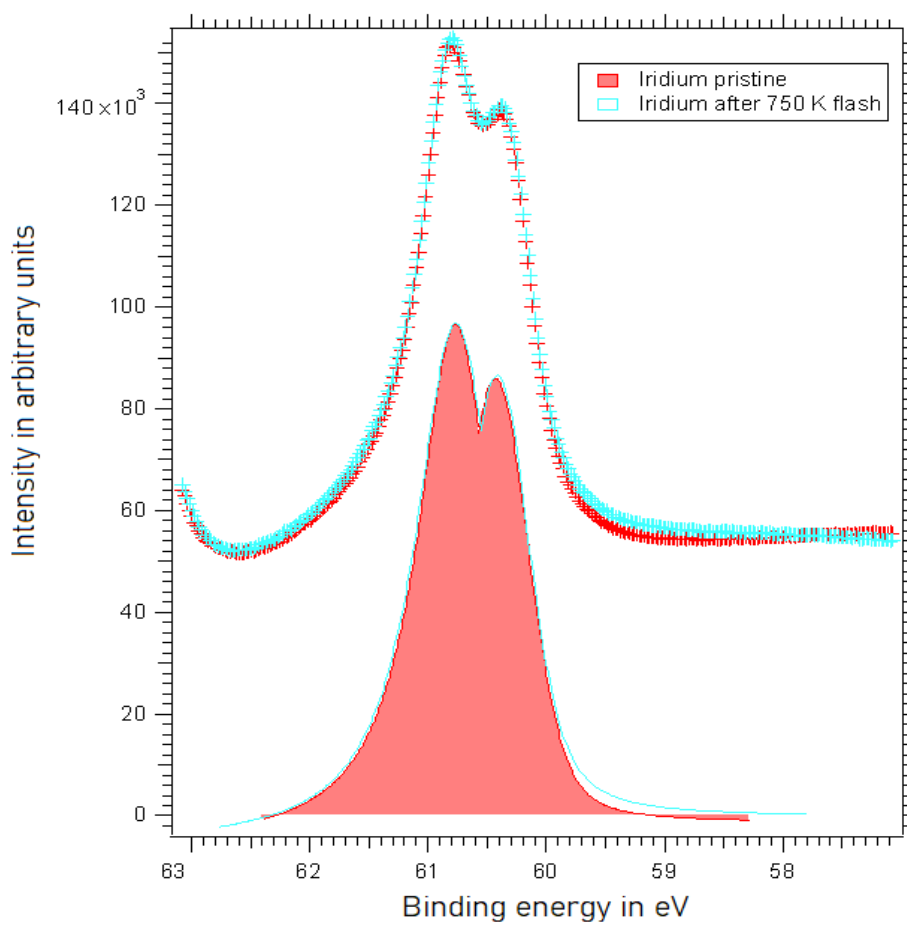
Figure 12: Comparison of the pristine iridium and the scaled up iridium after the 750 K flash.

4.4.2 Intercalating Oxygen

After having characterized the pristine sample the graphene flakes was placed in a chamber with oxygen dosed at a pressure of 10^{-5} mbar for 100 s at a temperature of 450-470 K. This was done in order to intercalate oxygen underneath the graphene flakes.

Figure 11a)ii) does not have the same C 1s component seen in the pristine case. Instead a new peak with a -0.4 eV shift relative to the pristine graphene is seen. This shift is due to the graphene being doped by the intercalated oxygen layer below. This shift is comparable to the -0.45±0.02 eV observed by Grånäs *et al.* in [8] if experimental errorbars are considered.

To compare the intensity of the surface Ir component, the spectra in panel (b) of figure 11 have been normalized to the bulk component. In the Ir spectrum (figure 11b)ii)) the surface peak has vanished. This means that all of the iridium atoms on the surface bind to oxygen. From this observation the conclusion can be drawn that the graphene flakes have been fully intercalated by the oxygen. Figure 11c)ii) shows an oxygen component at a binding energy of 530 eV. Comparing this component, the ones found by Johansson *et al.* [9] it seems to match the BE of chemisorbed oxygen.

4.4.3 Electron Beam Exposure

The O-intercalated graphene flakes were exposed to an electron beam. After electron exposure figure 11a)iii) shows no change in the binding energy of the carbon peak remaining at the position characteristic of O-intercalated Gr flakes (283.8 eV). The amplitude of the peak has however decreased by 10%. The iridium peak in figure 11b)iii) shows little change when compared to its appearance after the oxygen intercalation. The amplitude of the oxygen peak observed in figure 11c)iii) has dramatically decreased when compared to before the electron exposure. From these observations it is concluded that carbon atoms in the graphene film reacts with oxygen to produce CO_2 which desorbs. The conclusion that the gas produced is CO_2 is made based on the observation that the intensity of the O 1s decrease by an amount close to 2 times that of the C 1s. The surface peak of the Ir remains missing as the defects generated by the electron exposure generate C-atoms that bind downward to the underlying Ir atoms.

4.4.4 Flashing the Sample

The spectra in figure 11a)iv), b)iv), c)iv) shows the C 1s, Ir $4f_{7/2}$, and O 1s spectra acquired after flashing the sample to 750 K. Panel (a) shows that the C 1s binding energy has returned to its original binding energy of 284.2 eV. Furthermore, its amplitude has decreased to 65% of its initial value. The iridium spectrum shown in figure 11b)iv) is identical to the pristine spectrum with surface component having returned completely. Figure 12 shows this comparison where the two spectra in figure 10b) and figure 11b)iv) have been normalized to the bulk component. The oxygen component observed at 530 eV as seen in figure 11c)iii) is absent in 11c)iv), but a new component can now be observed at 531 eV. The binding energy of this component matches that of CO adsorbed to the surface as described by Johansson *et al.* [9].

5 CONCLUSIONS AND OUTLOOK

5.1 Conclusions

The first conclusion from my studies is that pristine graphene is unaffected by electron exposure at the conditions that I used. This conclusion was made based upon the lack of evident changes between the pristine graphene in figure 7 and the graphene after electron exposure in figures 8,9.

The second conclusion is that graphene flakes with intercalated oxygen are etched if exposed to an electron beam at my experimental conditions. This was concluded from the changes found using XPS shown in figures 10,11,12. The main changes were the reduction of the C 1s and O 1s intensities.

Etching is only observed in the second experiment where the graphene was intercalated with oxygen. This in combination with the lack of etching when the pristine graphene was exposed to the electrons suggests that what occurs during the etching is a reaction between the carbon and oxygen. It is likely that C-C bonds are broken by electron collisions. Without O-intercalation the graphene quickly heals, but with an intercalated O-layer below the graphene defects O-atoms, are grabbed from below and forms CO or CO_2, which desorbs.

5.2 Outlook

The first and most interesting thing to test would be to expose O-intercalated graphene flakes to electrons and characterize the surface with subsequently with STM. Such studies could for example show if the etching occurs at the edges or all over the flakes. This was actually a planned experiment for my bachelor thesis work, but due to experimental problems with the film growth I did not have time for this.

As shown oxygen is capable of etching the graphene surface. In future experiments it would be interesting to test other gases such as nitrogen and or chlorine. The main interest of such experiments would be if the etching only occurs when the carbon forms simple molecules such as CO and CO_2 or if it is possible that the etching can produce more complicated molecules such as carbon tetrachloride (CCl_4).

As a comparison to the previous idea, it would be useful to attempt to reproduce the same results on graphene grown on another or several other substrates. The first question is if the etching of the graphene with intercalated oxygen only occurs when grown on certain substrates. It could be that the coupling strength of graphene to its substrate affects the electron induced etching such that graphene on some substrates is easier to etch by electron exposure. This could for example be studied in experiments using copper which is also interesting for the commercial production of graphene

With the possible end goal of partially etching graphene in order to produce usable structures it would be useful to know if the etching process affects the graphene. In order to investigate this an experiment focused on etching graphene in order to produce some segment of graphene after which its properties such as band gap and overall conductivity would be investigated.

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