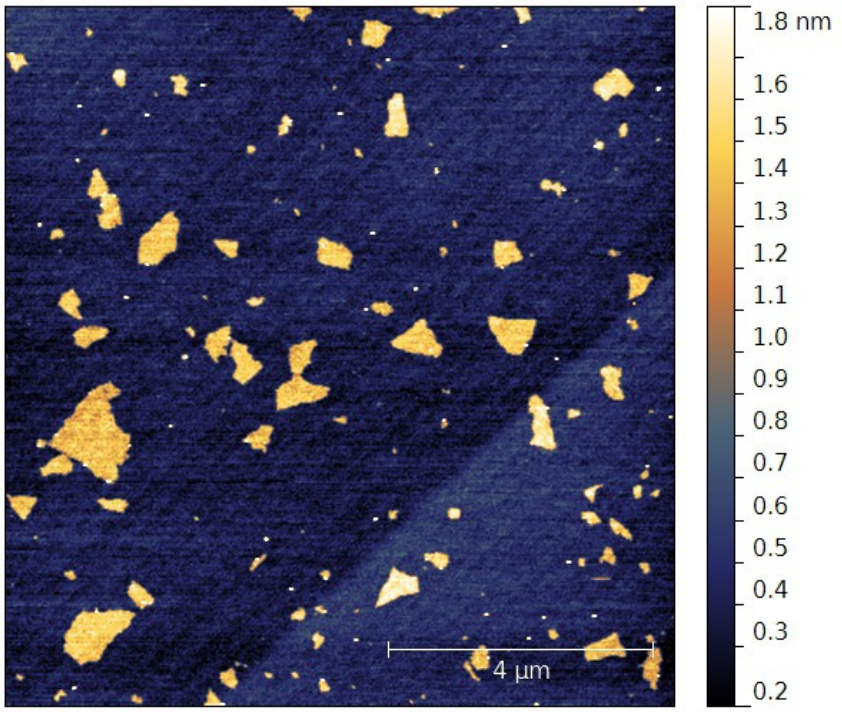
Atomic Force Microscopy Studies

of

Two-Dimensional Materials



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1. Introduction

Atomic Force Microscopes are ideally suited for creating 3-D images and measurements on 2-D materials. This is because AFMs have extreme contrast on flat samples and can magnify surface heights by factors of millions to billions. Standard reference samples that illustrate the extreme contrast of an AFM are SiC and Si(111). Each of these samples has well characterized surface features that are in the same range as 2-D materials such as graphene.

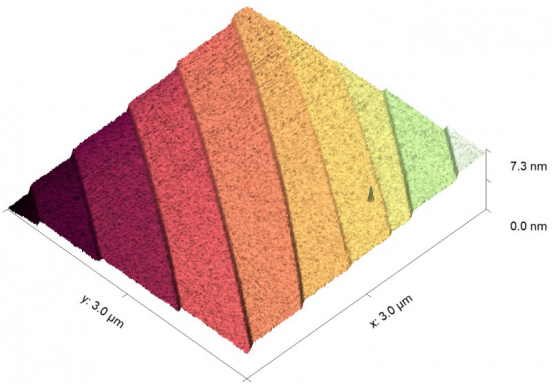

Figure 1a: Three dimensional color scaled image of SiC. The steps on this sample are 750 pico-meters

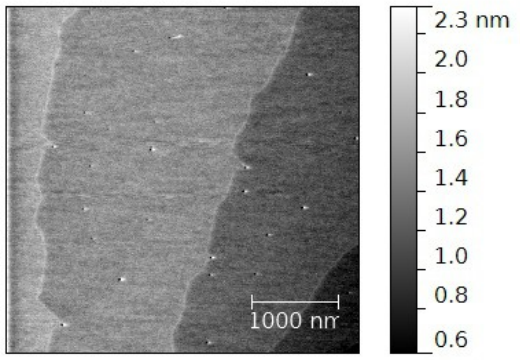

Figure 1b: Gray scale image of Si(111) atomic steps. The steps on this sample are 300 pico-meters in height.

Besides illustrating the power of an AFM, these types of samples serve as calibration samples for microscopes used for imaging 2-D materials.

Two dimensional materials are currently under development with potential to gain enormous importance in electronics, sensing, optics and other areas[1]. Such materials, despite facile production methods in many cases, can display radically different properties compared to 3D or bulk materials. These new and enhanced properties come about due to nanoscale confinement effects, meaning they are generally accessible only when a material is limited to 1, or at most to a few atomic layers. For this reason, research and development in 2D material and 2D materials-based devices relies crucially on the ability to characterise such materials at the nanoscale, including the observation of atomic steps. AFM is unique in its ability to measure sample heights with resolution in excess of 0.1 nm. This explains why AFM has become a key tool in the arsenal of researchers studying 2D materials.

2. Discussion

Although graphene is a single atom thick sheet, it is not typically found to be perfectly flat. Indeed, some nanometre scale corrugations, are commonly observed and may increase the stability of the 2D lattice[2]. Despite its great strength, graphene is also a highly flexible material, and typically takes on the form of the underlying substrate. So, for example, on Si/SiO_2 wafers, graphene can exhibit a considerable roughness due to the underlying substrate[3]. Thus, a considerable texture, dependent on the SiO_2 structure at the wafer surface, can be seen in the CVD graphene flakes shown in the left image in figure 2 below.

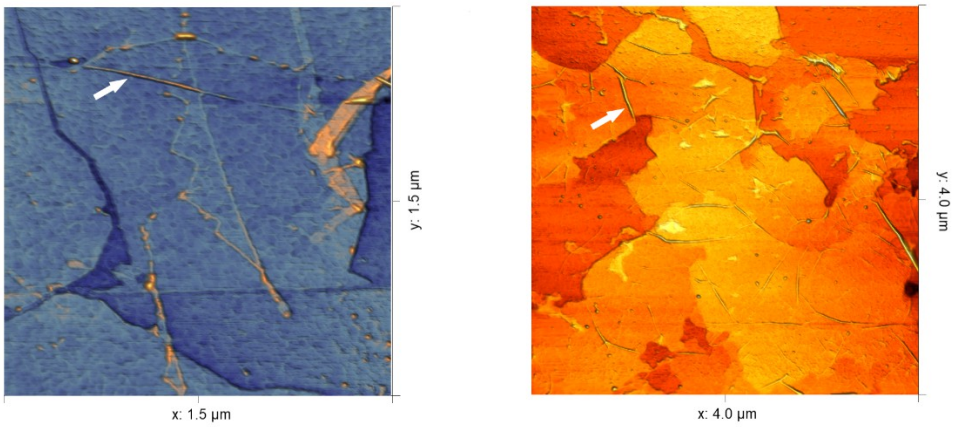

Figure 2. Examples of AFM images of CVD graphene deposited on Si/SiO2 wafers. Left: Single-layer graphene on a silicon wafer. In this example, the effect of the underlying texture on the graphene sheet is clearly seen. Right: Multilayer graphene of a silicon wafer. Arrows highlight some defects, typical of CVD graphene.

For many applications, graphene is grown on one substrate and transferred to another. This can lead to considerable changes in surface coverage. In order to understand data from transferred graphene, it can be useful to measure surface coverage.

Figure 3 shows images of multi-crystalline nickel metal surface coated with CVD graphene. In this sample, the coverage was almost 100% on the nickel surface. After transfer to a silicon wafer, it could be seen that coverage was decreased. During CVD growth of graphene, a number of defects can be introduced, including grain boundaries, as well so-called “wrinkles”. These defects can alter the properties of the graphene layers, and in some cases can be useful, for example, grain –boundaries show increase chemical reactivity, potentially useful in sensing applications[4]. It has been shown that AFM is a highly useful technique to map and even distinguish different defects on graphene. Some of these defects are highlighted in figure 2.

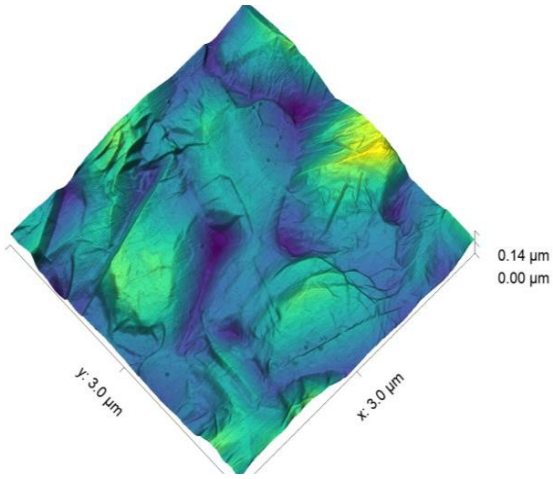

Figure 3. Image of multi-crystalline nickel film with graphene deposited on top: This image shows the surface had almost 100% coverage; However after transfer to another substrate much lower coverage was found.

Imaging of graphene by AFM can be carried out in either contact or vibrating mode using AFMWorkshop instruments. However, it should be noted that the method chosen can change the height measured for the graphene monolayers. In fact here have been quite a variety of different heights reported from 0.3 to 1.7 nm for single-layer graphene[2]. These differences have been attributed to complex tip-sample interactions, which can depend on the mode used, and nature of substrate and scanning environment. An image of single graphene flakes is shown in figure 4, along with a height profile. In this case, the flakes were measured using vibrating mode, and the height profile in figure 4 shows that the height measured was 1.3 nm. In order to achieve very low noise levels, such as those shown in figure 4, it is important to optimise vibration and acoustic isolation. It is also highly recommended to use a small z height scanner (such as the AFM Workshop 15×15×7 µm scanner, and ensure low gain setting are used. The measurements environment can also affect the results. In some cases, where surfactant or transfer agents contaminate the graphene surface, measuring in a liquid environment can result in a cleaner image.

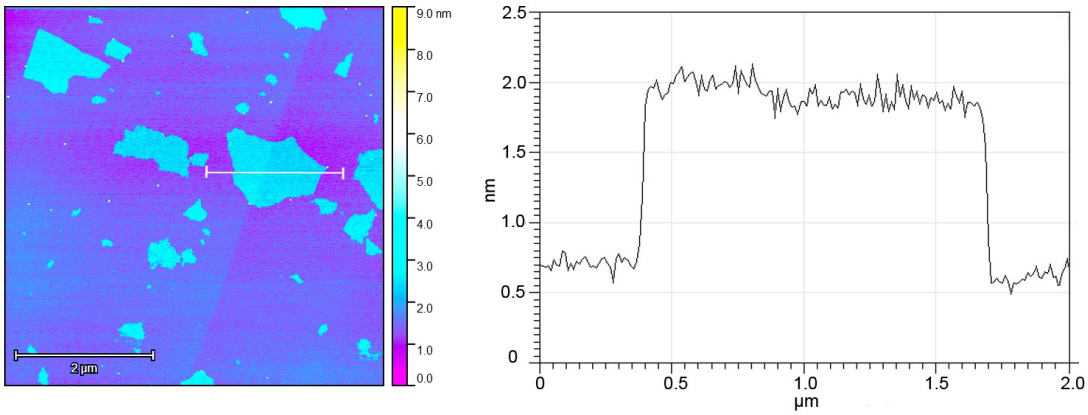

Figure 4. Height image of single graphene flakes on a well-polished silicon wafer sample, and height profile across the indicated flake.

In addition to imaging applications, AFM can be very useful for directly measuring the unique properties of 2D materials. For example, methods such as conducting AFM, electrostatic force microscopy (EFM), and Kelvin probe microscopy have proven useful in measuring the electrical properties of 2D materials such as graphene, graphene oxide, or MoS_2[5]. A unique advantage of using AFM for these kind of experiments, in addition to the measurement of very small features, is the ability to directly compare the electrical properties to the material structure in two dimensional materials. Similarly, magnetic force microscopy (MFM) can be useful to measure magnetic fields associated with 2D magnetic materials such as graphene, MoS_2, *etc.*[6]. Finally, AFM can be extremely useful to measure mechanical properties of 2D materials. For such experiments, a substrate with many holes can be coated with the material to be investigated, and the mechanical resistance probed directly by measurement of force-distance curves[7].

Overall, AFM is a vital tool for nanotechnologists who require characterisation and development of 2D materials and devices, combining ultra-high resolution size characterisation with the ability to characterise a number of other key properties of these fascinating materials.

All images in this report were measured with the AFMWorkshop AFMs.

3. References

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