

Determining the Distance between Gold Nanodots using Atomic Force Microscopy

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An atomic force microscope (AFM) was used to obtain high-quality images of gold nanodots. Image processing filtering and 2D Fast Fourier Transform were applied to decrease the effect of primary artefacts associated with probe-sample interaction and to reduce external noise effects. Using the optimised images, surface properties such as the distance between nano double dots was calculated. This was given to be (141 ± 7) nm and does coincide with the expected reference value.

I. INTRODUCTION

Atomic force microscopy was first developed in 1986 by Binning, Quate and Gerber [1]. This form of microscopy relies on atomic interactions between a probe and a sample and allows imaging of both conducting and isolating materials in the order of nanometers. Probe microscopy was one of the key instruments that caused the rapid development of nanotechnology in the 1980s. Nanodots have a wide range of applications from energy and information storage to optical devices such as laser tweezers [2]. AFM can be used for both fabricating nanopatterns as well as analysing the dimension of nanodots [3].

This experiment mainly focuses on analytical methods to improve the image quality to obtain the correct dimensions of gold nanodots.

II. APPARATUS AND THEORY

A. AFM Operation

The fundamental basis of AFM is the interaction between a tip and the substrate which is monitored and controlled through a feedback loop while scanning the surface. These interactions are caused by Van-der-Waal forces and atomic repulsion. As depicted in Figure 1, a cantilever with a sharp tip scans the sample along its surface. As the tip approaches the sample the attractive and repulsive forces at close-ranges cause the cantilever to bend towards the sample and bend away from the sample respectively. This bending is described by hook's law:

$$F = -k\Delta x. \quad (1)$$

The changes in the position of the cantilever are being recorded through the reflected laser beam on the quadrant detector[1].

AFM operates in many different modes. Two of these are contact and non-contact mode. While operating in contact mode the tip is always in contact with the sample whereas in non-contact mode the tip oscillates at a

frequency near the resonance frequency of the cantilever and does not touch the sample. Here, any change in amplitude and phase of the oscillation will be monitored.

Quadrant detector

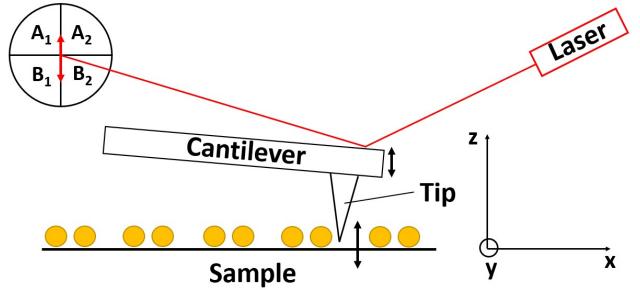


FIG. 1. **AFM set-up.** The cantilever scans the surface along the x-y plane. Changes in the z-axis are being recorded and scanned as the topology of the sample.

B. PID Feedback Loop and Artefacts

The deflection of the cantilever is being used in a Proportional-Integral-Derivative (PID) feedback loop. With the help of a piezoelectric metal, this is used to maintain the set-point at a constant value. Depending on the mode of operation, the set-point is either the force between the tip and the sample (contact) or the percentage of the initial frequency of the cantilever (non-contact). The integral gain has the highest impact on the image since it accounts for the error over time. The derivative gain is primarily useful for samples with tall edges. Tuning the PID values is an important step to acquiring a high-quality image. For this, different automatic and semi-automatic methods can be used [4].

The AFM images are the result of tip-sample interactions and very often involve artefacts. Due to the small size of nanodots relative to the tip, the most common artefact is convolution as shown in Figure 2. Furthermore, the system requires some calibration so that the measured values correspond to real physical values.

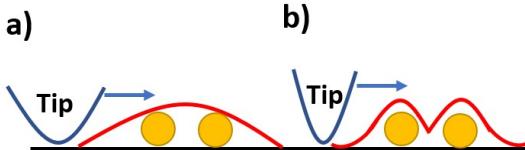


FIG. 2. Convolution as an artefact. The red line describes the resulting image. Since nanodots are small objects, the scanned image is convolved with the shape of the tip. **a)** Blunt tip or a tip further away from the sample. **b)** Sharp tip or a tip very close to the sample. It is notable that even with a sharp tip in contact mode, the effect of convolution cannot be neglected.

III. EXPERIMENTAL PROCEDURE AND RESULTS

As shown in Figure 3a, the expected literature value for the distance between the double dots is 139 nm. Images with different modes and with the variation of PID values were taken. Best raw results were obtained with contact mode as expected. Using the software Gwyddion, plane levelling was applied such that all image points were used to compute a plane which is then subtracted from the data. These results are shown in Figures 3b and 3c.

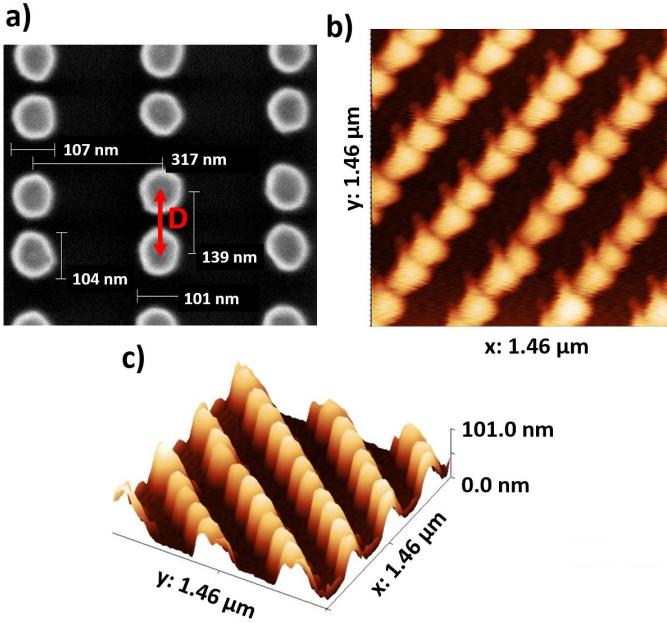


FIG. 3. Images of gold nanodots. **a)** Is the literature image of gold nanodots taken by SEM [2]. The red arrow depicts the distance between double dots, denoted as D. **b)** Image taken by AFM, PID = 10000, 1000, 0 with a set-point of 20 nN. **c)** 3D image of nanodots taken by AFM.

The effect of convolution is present in image 3b. Furthermore, there is some additional noise, reducing the quality of the image. Before determining the distance D between the double dots, some data processing filtering

was applied.

2D Fast Fourier Transform was done to identify the high-frequency noises in the image and extract them accordingly. Again, this was done using the software Gwyddion. The results are shown in Figure 4.

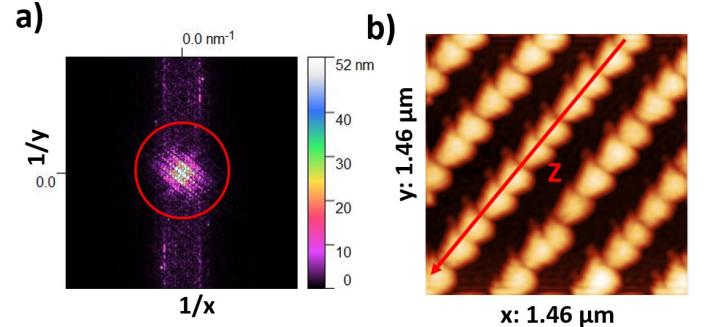


FIG. 4. 2D FFT results. **a)** The Fourier Transform of the image is depicted in k-space. The frequencies highlighted by the red circle correspond to the noise and were deleted from the raw image. **b)** The resulting image after removing the high frequencies. The red line Z is along the nanodot rows and is used for extracting the surface profile.

Subsequently, a surface height profile along the line Z was drawn, as shown in Figure 4b. The resulting height curves are shown in Figure 5. These were used to extract the distance between nano double dots.

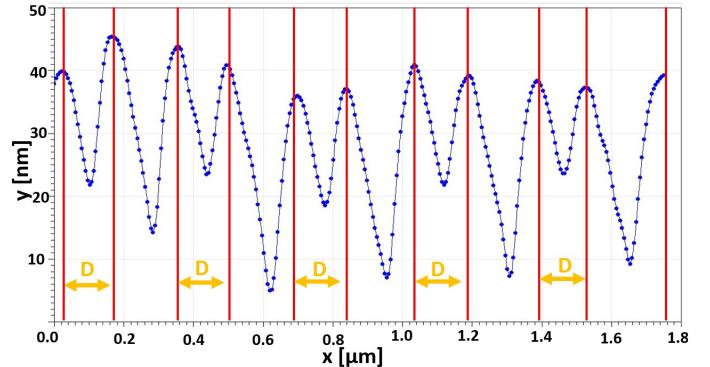


FIG. 5. Surface height profile The peaks are highlighted by the red lines and describe the position of the nanodots on the image. The smaller distance between the peaks, depicted with orange arrows, describe the distance D between the nanodots.

Multiple measurements along the rows on the optimised image were made. The small distance between the peaks was calculated and averaged. The standard deviation of the results was taken to be the random error on these measurements. The distance between the gold nanodots was calculated to be:

$$D = (141 \pm 7) \text{ nm},$$

which is consistent with the given literature value.

IV. DISCUSSION AND FURTHER ANALYSIS

A. Mode of Operation

AFM contact mode seemed to deliver the most accurate images for the nano sample. This is due to the fact that the tip provides a stronger feedback signal as it moves in a highly repulsive force regime. A typical disadvantage of contact mode is possible damages to soft samples [5]. However, since the nanodots are not soft samples, no damage to them was observed. A further expected disadvantage while operating in contact mode is that the tip is more likely to break when it approaches sharp edges. Again, while imaging the nanodots, no such damage was observed.

B. Error Analysis

The main source of random error in the experiment were the environmental perturbations such as the short high-frequency noises. These could be mostly due to the electrical equipment used in the set-up of AFM. One candidate is the quadrant photo-detector. The randomness of incoming photons of the laser beam on the detector could be a possible explanation [6]. 2D FFT noise filter has been applied to remove the high-frequency noises. Comparing the image obtained after applying the 2D FFT filter, shown in Figure 4b, with the original raw image depicted in Figure 3b, some changes are notable. While the resulting image clearly has fewer noises present, it also has a lower degree of resolution. Additionally, some distortion at the bottom edge of the filtered image is observed. This could be due to the fact that in addition to the noise signal also some other signals have been removed which are of interest.

The main sources of systematic error are the calibration of AFM and the artefacts due to the tip-sample in-

teraction. The main artefact present in the images is convolution as discussed in Figure 2. Comparing the true SEM image of the nanodots, depicted in Figure 3a, with the surface height obtained in Figure 5, the convoluted double-peaks can be clearly observed which correspond to double dots. In order to improve the quality of results, more measurements on the peaks have been made. Moreover, the surface height of multiple rows in the image were extracted and taken into account for the calculation of standard deviation. A further source of systematic error that seemed to be underestimated in the experiment was the calibration of the AFM. As discussed in the theory section, AFM and probe microscopy are not true imaging and rely on the interaction between a probe and the sample. Therefore, it is questionable if measured height profiles on the images correspond to absolute real physical values. In order to compensate for this, prior to imaging the gold nanodots, a reference sample with known physical dimensions has been imaged and the measured height profiles and dimensions were compared to determine the calibration error of the equipment.

V. CONCLUSION

The distance between double dots was obtained using the AFM images in contact mode. The dominant source of error seemed to be the artefacts from convolution and calibration errors. However, the applied optimisation filters that reduced the noise seemed to introduce other artefacts. It is clear now that image optimisation by filtering can be very useful but it requires great caution. Every filter can possibly introduce more artefacts if not used correctly. Especially, the 2D FFT filtering only works when the signal of interest is greater than the signal of noise. If this is not the case, the filtering removes parts of the signal of interest and reduces the resolution of the image dramatically. Using a vacuum chamber for the AFM could improve the initial quality of the images.

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- [1] G. Binnig, C. Quate, and C. Gerber, The atomic force microscope, *Physical Review Letters* **56**, 930 (1986).
 - [2] A. Grigorenko, N. Roberts, M. Dickinson, and ZhangY, Nanometric optical tweezers based on nanostructured substrates, *Nature Photonics* **2**, 365 (2008).
 - [3] Z. Liu, N.-D. Jiao, K. Xu, Z. Wang, Z. Dong, and L. Liu, Nanodot deposition and its application with atomic force microscope, *Journal of Nanoparticle Research* **15** (2013).
 - [4] D. Abramovitch, S. Hoen, and R. Workman, Semi-automatic tuning of pid gains for atomic force microscopes, *Asian Journal of Control* **11**, 188 (2008).
 - [5] M. Marrese, V. Guarino, and L. Ambrosio, Atomic force microscopy: A powerful tool to address scaffold design in tissue engineering, *Journal of Functional Biomaterials* **8**, 7 (2017).
 - [6] U. A. Javid, Implementation of closed loop feedback control of piezoelectric disc based atomic force microscope (2016).