12-2020

Data Processing & Analysis for Atomic Force Microscopy (AFM)

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Senior honors thesis submitted to the faculty of the Suffolk University Physics Department in partial fulfillment of the requirements for the degree of

Bachelor of Science

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Suffolk University
December 2020

ABSTRACT

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Scanning Probe Microscopy (SPM) has become a critical tool for characterization of materials in fields such as physics, material science, chemistry, and biology. Atomic Force Microscopy (AFM) is an increasingly useful technique because of its high resolution in three dimensions, the sample does not need to be conductive, and the technique does not need to take place in vacuum. AFM can image a wide variety of topographies and many different types of materials. AFM can deliver 3D topography information from the angstrom level to the micron scale with high resolution. One of the most important aspects of Atomic Force Microscopy that is not often discussed is data processing and analysis. The goal of this report is to demonstrate various data processing and analysis techniques such as studying the coating process, grain analysis, texture analysis and surface roughness.

ACKNOWLEDGENMENTS

I would like to give a huge thank you to my thesis advisor Dr. Perov for his help throughout my years at Suffolk University and his continued support of this project. Dr. Radojev also served as my advisor for this project and assisted greatly with the set up and troubleshooting of the microscope in the lab. I also would like to thank my collaborator Dr. Song Xu for his help bringing the microscope back to working condition and his commitment to helping me become an expert on atomic force microscopy. I also would like to thank Dr. Johnson for his continued support as my academic advisor, thesis advisor, and mentor throughout my past three and a half years at Suffolk University. Without Dr. Johnson's commitment to his students, I do not think Suffolk University's Physics department would be the same type of family that it is today.

TABLE OF CONTENTS

Table of Contents

I. Introduction to Atomic Force Microscopy (AFM)

1.1 Overview

Atomic Force Microscopy is an incredibly useful tool for material characterization and analysis across many disciplines including Biology, Chemistry, and Physics. This project is focused primarily on the applications of AFM for Material Science and Physics. This paper will discuss the background of AFM, how AFM works, data analysis in AFM, and further applications of AFM. Hopefully, this paper will prove as a resource for future Physics majors and further research across all STEM departments at Suffolk University.

1.2 Background

Atomic Force Microscopy (AFM) was invented by IBM scientists Gerd Binnig and Heinrich Rohrer in the 1980s. The atomic force microscope combines properties of scanning tunneling microscopy and stylus profilometry. Binnig and Heinrich later won the Nobel Prize in Physics in 1986 for their invention of the atomic force microscope.

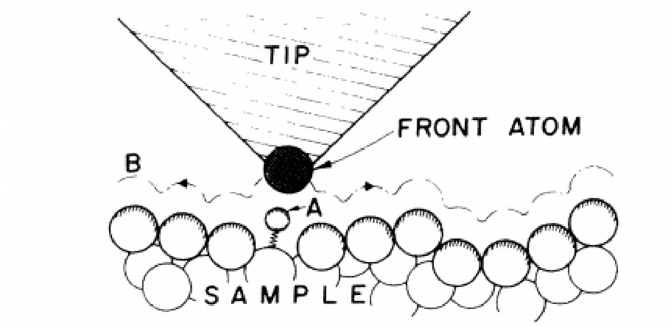


Figure 1: Drawing of AFM tip scanning a sample from Binnig et al. [2]

Typical types of microscopy include light and electron microscopy. These methods are used to essentially take a picture of a sample. These microscopes rely on electromagnetic radiation and magnification to create images. Unlike traditional microscopy, Atomic Force Microscopy uses a probe to create an image of the surface topography of a sample.

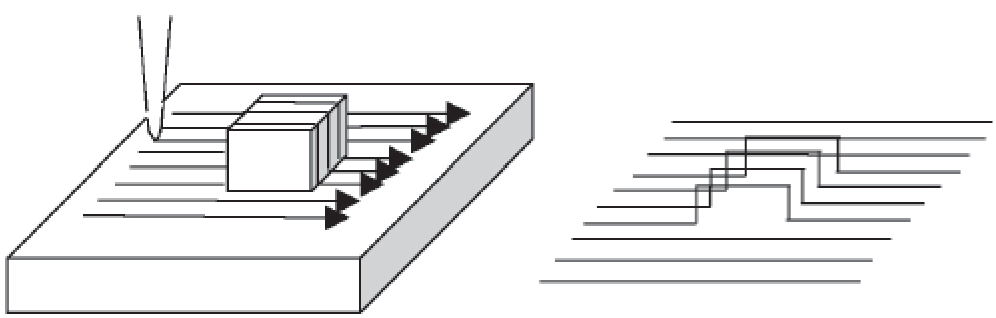
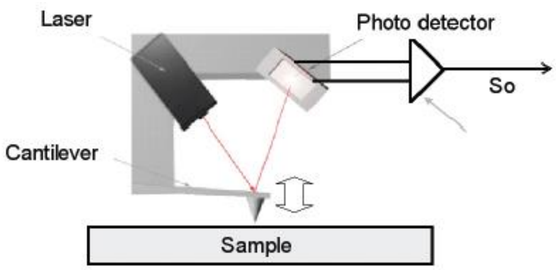


Figure 2: Using a sharp probe to scan across the surface from left to right, the motion of the probe from each pass across the surface is recorded as a 2D line profile, shown on the left. These line profiles are combined to create a 3D image of the surface, shown on the right. [1]

AFM uses a sharp probe (typically < 50 nm in diameter) to scan areas less than 100 microns. The sharpened probe is aligned close enough to the sample such that the probe interacts with the force fields associated with the sample. The probe is then scanned across the surface such that the force remains constant. Then, an image of the surface is reconstructed by monitoring the motion of the cantilever as it is moves across the surface. Scans can last anywhere from seconds to ten minutes, depending on the size of the area being scanned. Typically, magnifications in AFM images are between 100 X and 100,000 X.



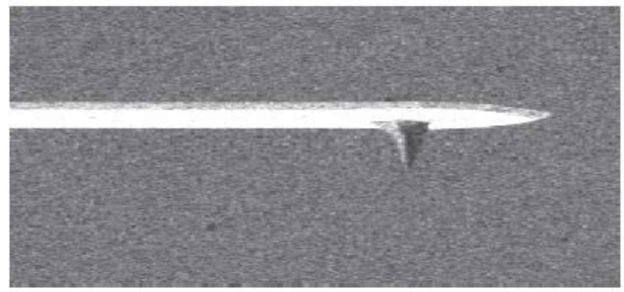


Figure 3: On the left is a schematic of the light lever force system used in AFM, the right is showing a high-resolution image of an AFM cantilever (probe) [1]

In an AFM system, the force between the cantilever and the surface of the sample is measured with a force sensor and the output is sent back to the feedback controller that then drives the Z motion generator. The feedbaAck controller uses the force sensor to maintain a fixed distance between the cantilever and the sample in order to give consistent results and not crash the probe into the sample. Additionally, the system has an XY motion generator, that moves the probe across the surface of the sample. This motion is then monitored and used to produce the image of the surface of the sample.

The force sensor in an AFM is constructed from a light lever (see Figure 3). The output from a laser is focused on the backside of the cantilever and is reflected into a photodetector. The photodetector then compares its outputs in a differential amplifier, which is used to compare the input voltage of the laser and the output backside reflection of the laser from the cantilever . When the probe interacts with the surface of the sample, the cantilever bends, and the light path changes, causing the amount of light that reaches the detector to change. This means that the electronic output of the light lever force sensor is proportional to the force between the probe and the sample.

There are two main types of imaging modes used for AFM, tapping mode and contact mode. This project used contact mode as the mode for data acquisition. In contact mode, the cantilever is scanned across the sample at a fixed deflection.

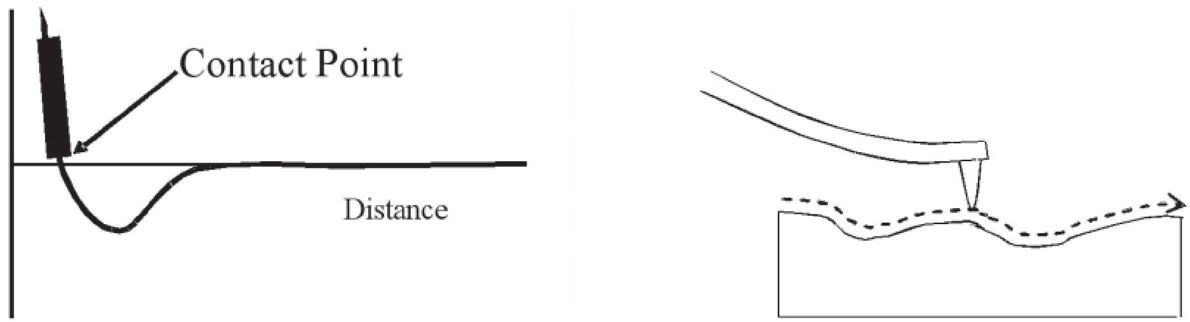


Figure 4: Left shows potential diagram showing the region of the probe while scanning in contact mode. Right shows in contact mode the probe glides over the surface. [1]

Contact mode is typically used for hard samples and for resolutions greater than 50 nm. The cantilevers used for contact mode may be constructed from silicon or silicon nitride

AFMs unique design makes it compact, small enough to fit on a tabletop, while also being high resolution enough to resolve atomic steps and precise enough to fabricate surface structures on the nanometer scale.

1.3 Data Analysis for Atomic Force Microscopy

The main purpose of this project was to (1) repair the microscope within the Suffolk University Physics department to a working condition and (2) determine best uses and explore what can be done with the instrument. The most important part of Atomic Force Microscopy is the breadth and depth of the data analysis that can be done from AFM measurements. There are three main components of data analysis for AFM measurements, processing the data, displaying the data. and analyzing the data.

1.3.1 Processing Data

Processing data is the step where changes are made to the data in the image such as filtering and background subtraction. Often the first step in the data processing is what is called leveling. In all AFM images, there will be some background curvature or slope that must be removed from the image in order to normalize it and make analysis possible. This slope or curvature is often caused by offset between the probe and the surface or by the scanner itself. The primary types of leveling include line by line, three point, and inclusion/exclusion.

Line by line leveling is the most used and the simplest leveling method. This method takes every horizontal or vertical line produced in the AFM image and fits it to a polynomial equation. The polynomial shape is then subtracted from the image line and then the height of each line is set equal to the previous line.

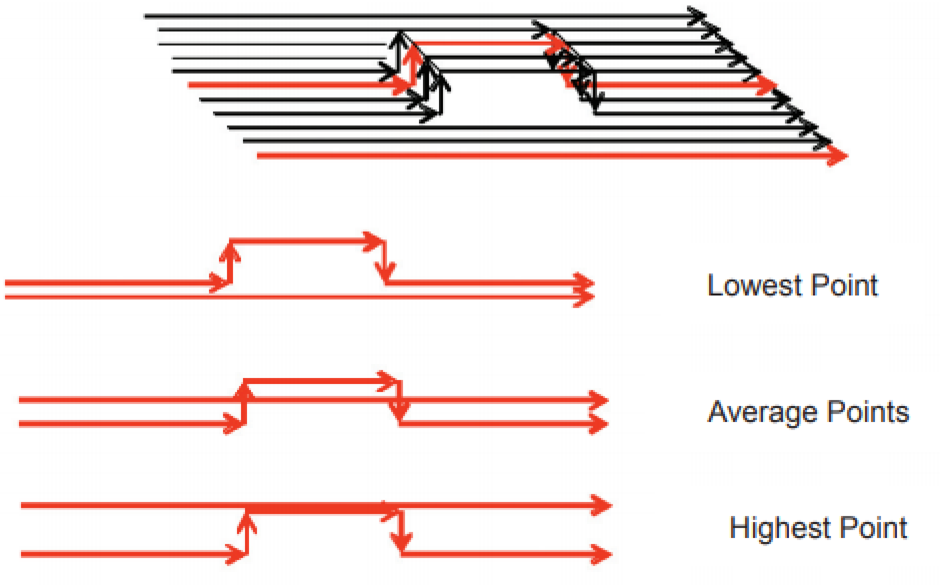


Figure 5: Visual representation of how line by line leveling works [1]

The three-point method uses three user defined points on the image that define a plane that is to be removed from the image. This type of leveling works well for samples that have terraces and the background noise associated with the scanner is much less than the height of the terraces.

Inclusion/exclusion leveling uses user defined areas to remove isolated features on a flat surface. This method is used because in other methods like line by line leveling, the isolated features will leave a streak across the images. The user decides which features are to be included or excluded in line by line leveling. When the leveling is done, the marked features are not used for the calculations for line by line leveling.

Another common method of data processing is the use of histograms to examine feature height. In AFM images, a color bar is used to display the height of features in a sample. The histogram will plot the number of pixels versus the color of the pixels. This is useful for showing surface features that are not visible when accounting for the full range in the Z axis.

By the nature of AFM and it’s use of high voltage electronics to produce images, sometimes there are unwanted high/ low frequency noise that are displayed within AFM output images. This type of noise can be removed by filtering. The most common types of filtering include matrix filtering and fast Fourier transform (FFT) filtering. Matrix filtering averages adjacent points together in an image and then uses that average to sharpen or blur the image. In FFT, the image is taken and then the frequency components are calculated, and then the unwanted frequency components are identified and removed from the image. FFT filtering is useful for images with repetitive patterns.

Lastly, there are many ways to zoom in, scale, and rotate images to look at a particular portion of an image or to avoid showing features that are not of interest to the operator. The place where this technique fails is that it tends to create pixelated images, so operators should always try to remove unwanted features through inclusion/ exclusion or taking smaller scans to avoid features that are not of interest.

1.3.2 Displaying AFM Images

The best way to gain useful information from AFM images is by displaying them, since the images are essentially a 3D map of the surface of the sample. There are several ways to view AFM images, but most commonly they are viewed in 2D or 3D. Additionally, the color scale and contrast of the images can be changed in order to display height changes more dramatically.

1.3.3 Analyzing AFM Images

Since AFM images are representations of a three-dimensional array of numbers, it is possible to make quantitative analysis of AFM images. Though, the quality of the analysis is dependent on the quality of the AFM image. There are several ways to analyze different parts of AFM images, including line profiles, surface roughness calculations, height analysis, particle analysis, and grain analysis. This project focusses primarily on surface roughness, height analysis, and grain analysis.

Surface roughness includes a set of four standard equations that are used to calculate roughness of the sample surface:

Eq. 1. Surface Roughness: $S_a = \frac{\sum_i^N |Z_i - \bar{Z}|}{N}$, $\bar{Z} = \frac{\sum_i^n Z_i}{N}$

Eq. 2. Root Mean Square: $S_q = \sqrt{\frac{\sum_i^N (Z_i - \bar{Z})}{N}}$

Eq. 3. Peak to Peak: $S_r = Z_{max} - Z_{min}$

Eq. 4. Mean Value: $S_m = \bar{Z} - Z_{min}$

Another interesting way to analyze AFM images is through height analysis. There are three main methods for measuring atomic step height from AFM images, histogram analysis, single point at top and bottom of a profile, and a line fit to the top and bottom of the profile. In this project, histogram analysis is used to resolve heights of features on the surface of a sample. Histogram measurements are the most accurate way to resolve step height since the histogram is made up of data from the entire image, allowing for averaging to take effect.

The last type of analysis used in this project is what is known as grain analysis. AFM is a great tool for measuring grain structure on surfaces because AFM has great contrasts on flat samples. Grain analysis is an interesting feature of AFM analysis because AFM images have three-dimensional topography and grain boundaries can be easily identified. Once the grains are identified, the volume, size, area, etc. of the grains can be calculated and displayed.

II. Experimental Set Up

2.1 Atomic Force Microscopy System & Setup

The Atomic Force Microscope used in this project is a 5500 AFM/SPM Microscope from Agilent technologies. It is equipped with two interchangeable scanners, a small scanner and a large scanner. The large multipurpose scanner was used for this project, which has a scan range of 90 microns in XY and 7 in Z. It also has a noise level of less than 5 angstroms in XY and less than 0.3 angstroms in Z.

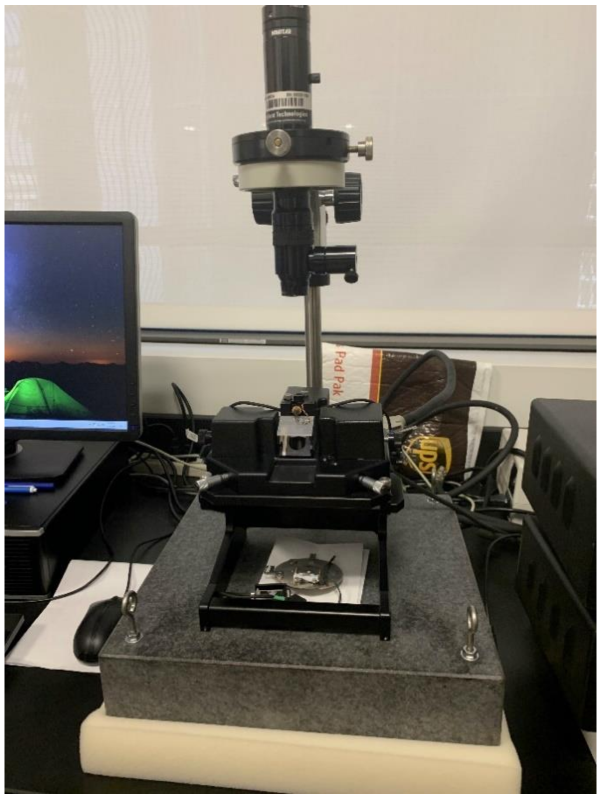


Figure 6: Atomic Force Microscope system with noise isolation slab beneath it. Located on the 5th floor of the Samia building at Suffolk University

Figure 7: Large multipurpose scanner [3]

The imaging mode used was contact mode, and silicon carbide cantilevers manufactured by Bruker were used for the scan.

Figure 8: Standard sample mount [3]

The sputter chrome coated silicon sample was cleaved and mounted on the standard sample mount that was included with the AFM. The scan sizes were 1 micron and 5-micron scans. The sample sits at the center of the disk, and the white bumper keeps the sample stage from getting too close to the scanner.

2.2 Troubleshooting

The purpose of this project was two-fold 1) get the AFM into a working condition where data can be acquired and 2) analyze the data as a proof of concept that the AFM is working properly. In the first phase of the project, several days were spent attempting to troubleshoot issues with the AFM. First, the computer that powers the AFM was replaced. This allowed the AFM to be turned on. After this, troubleshooting was done step by step to eliminate possible issues. Different scanners, cables, and power supplies were tested. Ultimately, the issue was discovered that the AC mode box was causing high frequency disruptions with the high voltage input on the AFM. After removing the AC mode box from the setup, AFM scans were able to be run.

2.3 Data Analysis Software

There are several options for AFM/SPM data analysis software. In this project, Mountains Map premium was used to process, analyze, and display all of the AFM data acquired from this project. [1] This software is capable of every type of processing, analysis, display, and error correction an AFM operator needs. The primary functions used in this project include line by line leveling, step height histogram analysis, roughness analysis, grain analysis, and 3D display.

[1] For anyone interested in being trained on how to use this software, please contact me at mmcdonough4@su.suffolk.edu for a link to a Zoom session with Song Xu on how to use the software for AFM analysis.

III. Results

3.1 Images of Sputter Chrome coated Silicon

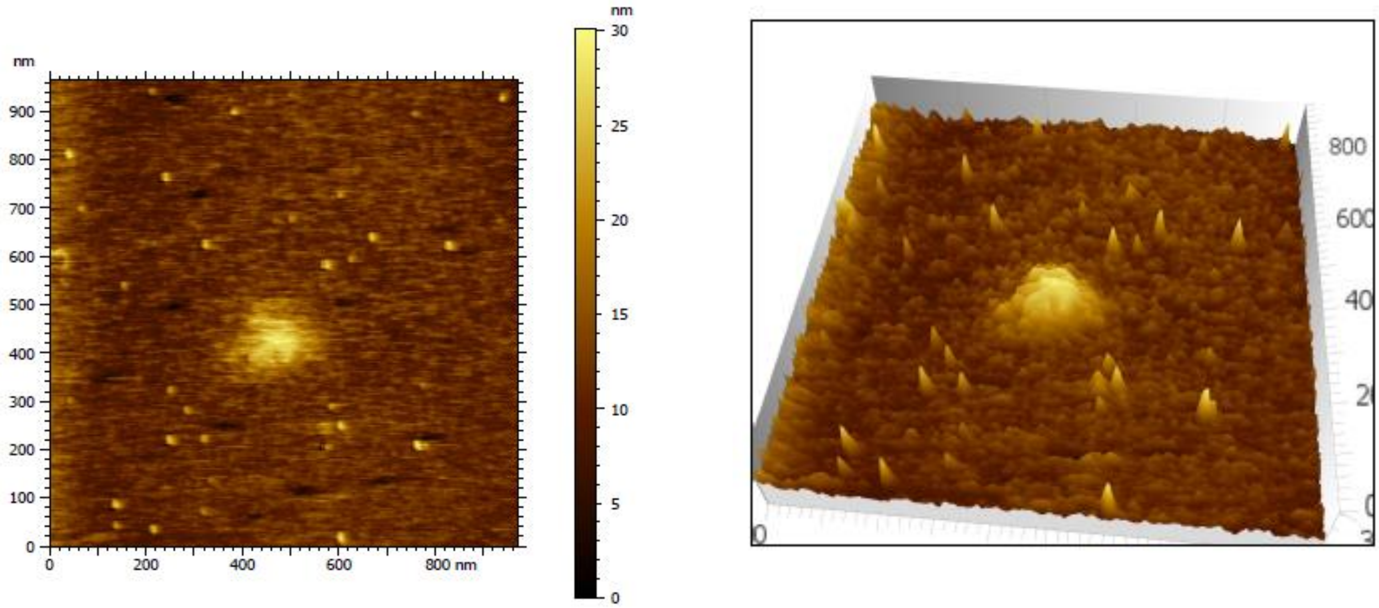


Figure 9: 1micron image of sputter chrome coated silicon (2D on left, 3D on right)

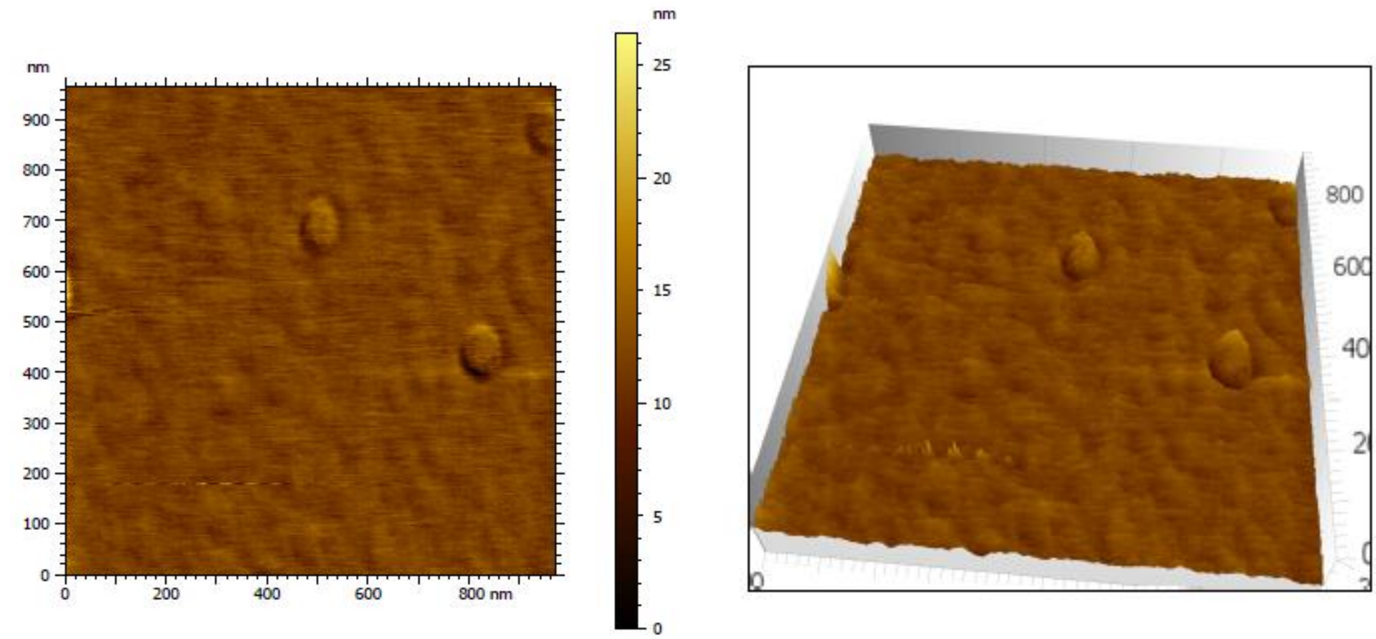


Figure 10: 1 micron image of sputter chrome coated silicon (2D on left, 3D on right)

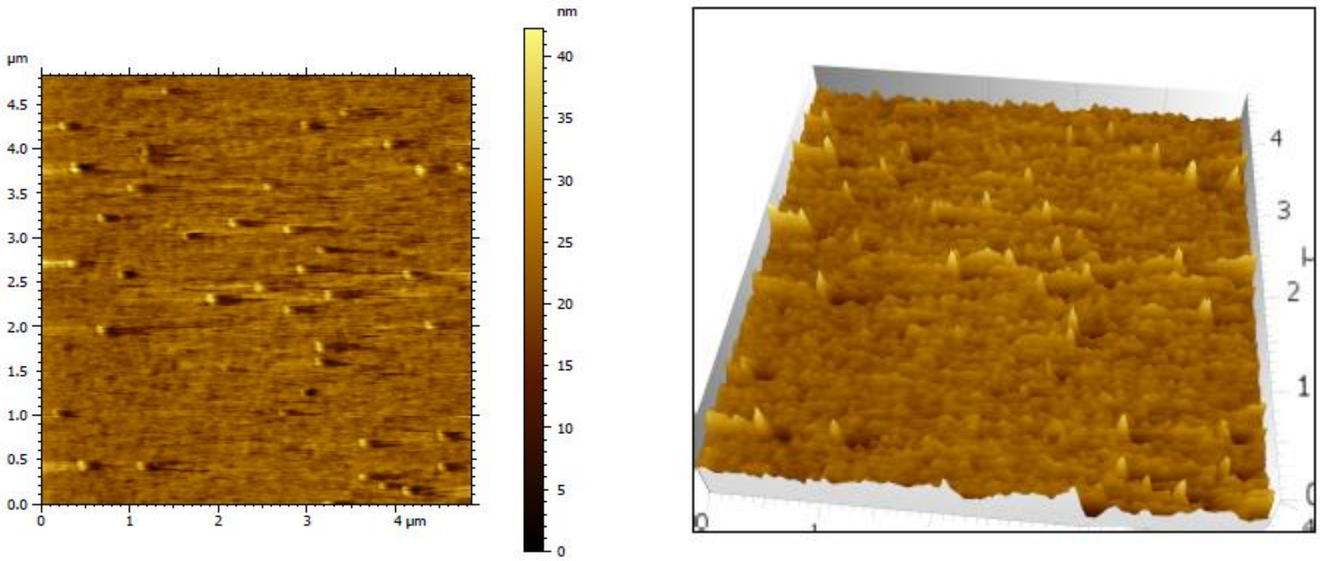


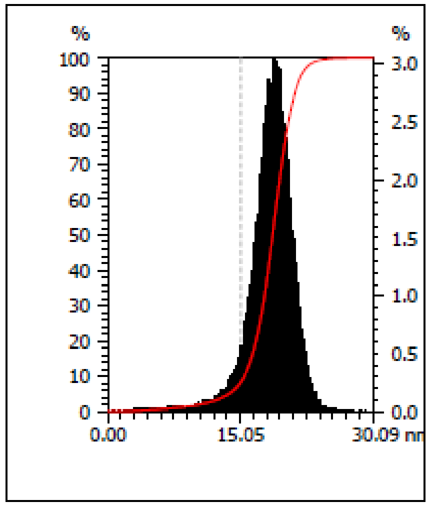
Figure 11: 5 micron image of sputter chrome coated silicon (2D on left, 3D on right)

These three images were taken at various positions on the sample. These images were processed with line by line leveling to normalize the Z range due to the sample stage not being level. In section 3.2, the data from these images will be analyzed and information about roughness, feature height, and grain analysis will be explored. The 3D images show exaggeration in the Z range in order to display the features on the sample. The color change indicates the height of the sample in the Z range from the dark brown being at zero and the bright yellow being the highest point in the sample.

3.2 Interpretation of Results

Definitions: S_q (root mean square roughness), S_{sk} (skewness of sample- degree of distortion from the symmetrical bell curve or the normal distribution), S_{ku} (Kurtosis- measure of whether the data are heavy-tailed or light-tailed relative to a normal distribution) S_p (maximum peak), S_v (valley depth) S_z (maximum height), S_a (arithmetical mean height)

ISO 25178		
Height parameters		
Sq	2.794	nm
Ssk	1.633	
Sku	9.126	
Sp	18.42	nm
Sv	11.68	nm
Sz	30.09	nm
Sa	1.942	nm



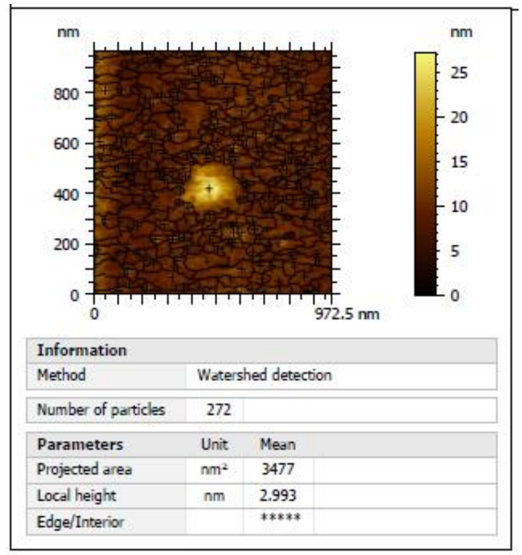
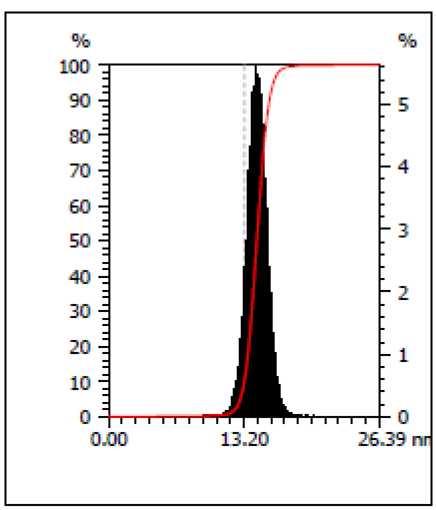


Figure 12: (Left) Shows roughness analysis of the image in Fig. 9, (Center) shows histogram analysis in Fig. 9, (Right) shows grain analysis in Fig. 9

From these three types of analysis, there is a good overall picture of different data about the sample. From the table on the left in Fig. 12, it was calculated that the root mean square roughness (S_q, typically the most accurate measurement of roughness) is 2.794 nm. Additionally, this table tells us the highest peak (S_p) of the sample in Z, which was 18.42 nm. From the histogram in the center of Fig. 12, we notice that there is a large amount of heights that fall in the 16 to 17 nm tall category, but very few larger than 20 nm and smaller than 13 nm. On the right of Fig. 12, there is a grain analysis which calculated that there are 272 distinct particles that can be analyzed.

ISO 25178		
Height parameters		
Sq	1.070	nm
Ssk	0.1762	
Sku	11.00	
Sp	14.45	nm
Sv	11.94	nm
Sz	26.39	nm
Sa	0.7941	nm



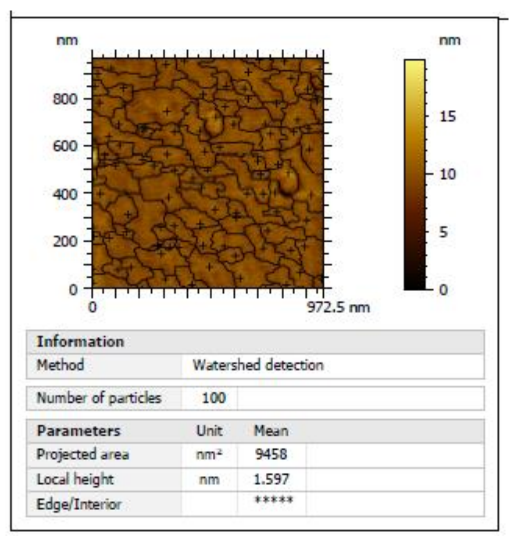
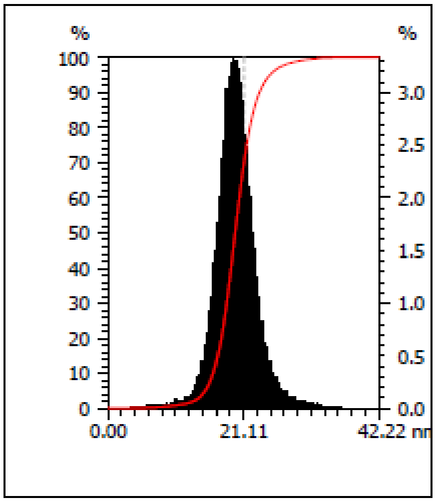


Figure 13: (Left) Shows roughness analysis of the image in Fig. 10, (Center) shows histogram analysis in Fig. 10, (Right) shows grain analysis in Fig. 10

From these three types of analysis, there is a good overall picture of different data about the sample. From the table on the left in Fig. 13, it was calculated that the root mean square roughness (S_q, typically the most accurate measurement of roughness) is 1.070 nm. Additionally, this table tells us the highest peak (S_p) of the sample in Z, which was 14.45 nm. From the histogram in the center of Fig. 13, we notice that there is a large amount of heights that fall in the 13 to 14 nm tall category, but very few larger than 17 nm and smaller than 12 nm. On the right of Fig. 13, there is a grain analysis which calculated that there are 100 distinct particles that can be analyzed.

ISO 25178			
Height parameters			
Sq	3.266	nm	
Ssk	-0.2541		
Sku	6.213		
Sp	19.92	nm	
Sv	22.30	nm	
Sz	42.22	nm	
Sa	2.362	nm	



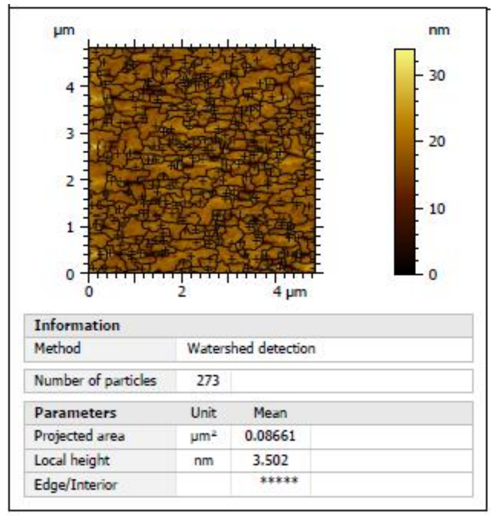


Figure 14: (Left) Shows roughness analysis of the image in Fig. 11, (Center) shows histogram analysis in Fig. 11, (Right) shows grain analysis in Fig. 11

From these three types of analysis, there is a good overall picture of different data about the sample. From the table on the left in Fig. 14, it was calculated that the root mean square roughness (S_q, typically the most accurate measurement of roughness) is 3.266 nm. Additionally, this table tells us the highest peak (S_p) of the sample in Z, which was 19.92 nm. From the histogram in the center of Fig. 14, we notice that there is a large amount of heights that fall in the 19 to 25 nm category, but very few larger than 25 nm and smaller than 18 nm. On the right of Fig. 14, there is a grain analysis which calculated that there are 100 distinct particles that can be analyzed.

3.3 Conclusion and Future Outlook

In conclusion, the Atomic Force Microscope located within the Suffolk University Physics Department has now been restored to full working condition, ready to use for future research projects across disciplines. This tool will be instrumental to future departmental research projects and will serve as a useful tool for discussing interactions at the atomic scale in courses like Advanced Laboratory and Modern Physics. Atomic Force Microscopy has many applications from Material Science, Chemistry, Physics, and Biology, and it is the hope that because of this project the STEM departments at Suffolk University will have a new tool to use in teaching as well as research.

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