**Note: Written using a slightly different grading rubric. Pay attention to the overarching feel of the report. You may also check out actual journal articles.**

Atomic Force Microscopy:  
**Binary Coding of Compact Discs**  
Author and Partners  
513 Instrumental Analysis  
Date experiment done:  
Date Experiment submitted:

Abstract:  
Written for a different format. A paragraph summary of the key points in the report should be included here.
Introduction:

Optical microscopy gives a researcher the ability to observe features of a sample that are too small to be seen by the human eye. However, optical diffraction limits the researcher to a resolution of a few micrometers. Atomic force microscopy (AFM) forgoes this limit by probing the surface of the sample with a microscale contact tip. The instrument shines a laser on the tip while a detector measures the deflection of the laser from the tip. As the contact tip moves across the surface, the deflection of the laser changes based on the physical features of the sample that the tip crosses over. This results in a sensitive form of quantitative microscopy that can detect surface features with nanoscale resolution.

AFM’s capacity to scan nanoscale images of solids is utilized in several fields of chemistry, particularly materials chemistry. Films of nanoparticles can be analyzed using AFM to determine the sizes and shapes of the nanoparticles. The development of new computer technology can also be aided by AFM by allowing technicians to observe surface features of microscopic silicon chips. In this experiment, the binary encoding of compact discs (CDs) will be observed by AFM and the spaces between indentations (pits) and non-indentations (lands) on the CD will be measured.

Experimental:

For this experiment, an Agilent 5400 Scanning Probe Microscope was calibrated for a scan rate of 1.00 line/sec. Picoview software was used for all data acquisition. Environmental noise was minimized by suspending the instrument on a granite slab inside a soundproofed chamber. A contacted tip was loaded and the helium/neon laser was aligned with the detector to make the deflection about 1 V and the friction about 0 V.

First, the CD was prepared for analysis. In order to expose the indented plastic portion of the disc, the top of the CD was etched with a box cutter. Then, the CD was placed in 100 mL of aqua regia (3:1 of 12M HCl to 6M nitric acid) for approximately 1-2 hrs. Once the metal plating of the CD had completely dissolved, the CD was removed from the aqua regia and dowsed with water. The CD was then cut into small, coin-sized pieces that could be placed on the sample tray.

Prior to scanning the CD, the optimum parameters of the instrument were tested using a chip with standardized grooves on the surface. The chip was brought close the tip using a manual approach followed by full contact using a computer-assisted approach. A 90 μm × 90 μm region of the surface was scanned while the integral gain, proportional gain, setpoint voltage, scan rate,
and scan angle were adjusted. Then, two different scan sizes were tested to inspect the change in resolution. The tip was then disengaged from the standard chip and the chip was removed from the sample tray.

The prepared portion of CD was then attached to the sample disc using double-sided tape and placed onto the sample tray. The CD was brought into contact with the probe using a similar approach method as with the standard. A 20 μm × 20 μm region of the surface of the CD was scanned with a scan speed of 1 line/sec, setpoint of -1.0000 V, angle of 0.0⁰, integral gain of 6.100% and proportional gain of 2.000%. Region sizes of 50 μm ×50 μm and 5 μm × 5 μm were also tested using these settings. The distance between the pits and lands was determined using topography vs. distance traces of two sections of the CD calculated with the provided Picoview software.¹

Results and Graphs:

The measured lengths of the pits and lands ranged from 392nm to 3068nm based on the scans of the surface of the CD and the diagonal cross-section of pits and lands. Using these values, the bit length was determined to be 78 ± 1 nm. A detailed description of how this bit length was determined, as well as the scan images, can be found in Data and Sample Calculations section of this report.

This bit length could then be used to determine the binary coding sequence that makes up the tested trace of the CD. A “1” correlates to a change in the level of the surface in a bit length, No change is registered as a “0.” Figure 1 shows the result of this binary encoding.

Figure 1: Binary encoding of the yellow trace shown in the image on the left. The topography vs. distance graph for this cross-section was labeled with the number of 78±1 nm bits that fit evenly within each pit and land. These values were then used to encode the topography into binary.
Discussion:

As has been shown by this experiment, AFM is a powerful technique for quantitatively analyzing nanoscale features of solid surfaces. The instrument was able to detect and image with high resolution features of a CD that could be less than 1 micrometer in length and width. These features would be difficult to analyze using optical microscopy and impossible to see with the human eye. Not only that, but the scan gave a quantitative output that allowed for the calculation of the bit size for this CD.

The incremental length of the bit was determined to be 78±1 nm, which does not correlate well to literature values. Dana, Hammouri, and Sunar report the bit length to range from 833 nm to 3563 nm in increments of 278 nm to 324 nm. These increments would be the bit size, the calculated value from this experiment does not fall within this range. Digital resources outside of journals also report a similar range as Dana, Hammouri, and Sunar, and go on to state that there are only 9 fundamental lengths for pits and lands. The calculations from this experiment indicated that there were at least 17 unique lengths for the pits and lands.

There may be several reasons from this large deviation from what is the common consensus of bit length. The first may be the calibration of the instrument. The instrument may have assigned a 20 μm × 20 μm size to a region that may have actually been larger. This would have made the pits and lands seem smaller than they actually were, resulting in a smaller bit length. However, this potential error does not account for the 17 different pit and land lengths that were observed. It may also be that this CD has dynamic bit lengths that are not constant throughout the entire CD. This would explain the different pit and land sizes and may account for why the calculated bit length is much smaller than the expected range (if the lengths are made up of dynamic increments, they would be more likely to be uniformly divisible by a smaller bit size than a large one). It may also be true that these values for the pit lengths and bit size are correct and that the CD that was tested was not 14-bit but instead 48 or 64-bit. The large binary code shown in Figure 1 would be compatible with a 48-bit system.

Regardless of these potential sources of error, AFM still provides some interesting insight about CD production. Once tabulated, the data shows that the pits were usually shorter than the lands, barring a few outliers. It may be the case that developers limit the size of the pits to decrease the chances that the pits will be malformed and thus provide incorrect information once read. Another interesting development strategy can be seen in Figure 6 of the Data section. As
can be seen in this image, the change from pit to land is not instantaneous, but instead occurs over 0.4 μm of distance. The diffraction limit for optical microscopy (the highest possible resolution an optical microscope can obtain due to the nature of light as an electromagnetic wave) is typically cited as 0.2 μm. This may simply be a coincidence, but it possible that CD developers have this limit in mind and ensure that the laser can still resolve the change from pit to land by making this change occur over twice the distance of the optical diffraction limit.

References:
1. Lab handout from Professor Ruthanne Paradise of the University of Massachusetts, Amherst for CHEM 513 entitled: Atomic Force Microscopy. 2012
Data and Calculations:

Figure 2: Changes in scan quality of standard based on changes in a.) setpoint voltage, in which the blank area of the image is indicative of a setpoint voltage that was too low and resulted in the tip losing contact with the surface, b.) scan rate, in which smoothing occurred by increasing the scan speed, and c.) scan angle, which shows straighten at the expense of resolution.

Figure 3: Scan of standard with (left) 20 μm × 20 μm and (right) 5μm ×5μm scan sizes.
Figure 4: Images of 20 μm × 20 μm region of the surface of CD with a sample topography vs. distance graph that correlates to the yellow line in the top-right image.

Figure 5: Image of surface of CD with (left) 50 μm × 50 μm and (right) 5 μm × 5 μm scan region.
Figure 6: Topography vs. distance graph of the 5 μm × 5 μm region with multiple features identified to indicate the start and end of the lands and pits.

Sample Calculations:

Figure 7: Topography vs distance graph for two different diagonal cross-sections of the 20 μm × 20 μm images of the surface of the CD. The x-axis was set as a threshold to use the x-intercept to calculate the transitions between pits and lands.
Length of pits and lands:
Using the labeled x-intercepts in Table 6, the difference between transition points was calculated to give the length of the pit or land.

\[
\text{Ex. } 2.276 - 0.0785 = 1.491 \, \mu m
\]

Length of one bit:
The difference of two segments was calculated and used as a preliminary bit length.

\[
\text{Ex. } 942 \, \text{nm} - 865 \, \text{nm} = 77 \, \text{nm}
\]
The bit length was assumed to be constant, so if this is the true bit length, it should divide evenly into all other pit and land lengths to provide a number of bits that make up the segment.

\[
\text{Ex. } 1020 \, \text{nm} / 77 \, \text{nm} = 13.24
\]
This value was subtracted from the nearest whole integer.

\[
\text{Ex. } 13.24 - 13 = 0.24
\]
The average of these deviations from the nearest integer was taken for every pit and land length of both trials. Dividing all of the pit and land lengths by the tested bit length must provide whole-number integers for all of the pits and lengths, and this deviation tracks how closely the tested bit length fit into the pit and land lengths. A pit length of 78.5 nm minimized this deviation, which indicates that it is the bit length for this CD. A sample of this tabulated method of guess-and-check is shown in Table 1.

**Table 1: Segment of Deviation Optimization of Bit-Length**

<table>
<thead>
<tr>
<th>Segment Length (nm)</th>
<th># of Bits (Bit-size = 77 nm)</th>
<th># of Bits (Bit-size = 78.5 nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>706</td>
<td>9.1688</td>
<td>8.9936</td>
</tr>
<tr>
<td>1570</td>
<td>20.3896</td>
<td>20.0000</td>
</tr>
<tr>
<td>1020</td>
<td>13.2468</td>
<td>12.9936</td>
</tr>
<tr>
<td>785</td>
<td>10.1948</td>
<td>10.0000</td>
</tr>
<tr>
<td>942</td>
<td>12.2338</td>
<td>12.0000</td>
</tr>
<tr>
<td>3061</td>
<td>39.7532</td>
<td>38.9936</td>
</tr>
<tr>
<td>1256</td>
<td>16.3117</td>
<td>16.0000</td>
</tr>
<tr>
<td>1256</td>
<td>16.3117</td>
<td>16.0000</td>
</tr>
<tr>
<td>1573</td>
<td>20.4286</td>
<td>20.0382</td>
</tr>
<tr>
<td>1416</td>
<td>18.3896</td>
<td>18.0382</td>
</tr>
<tr>
<td>Avg. Dev. from Nearest Integer</td>
<td>0.197</td>
<td>0.014</td>
</tr>
</tbody>
</table>
Error Analysis:

*Error in bit length:*

Using the deviation method in Excel, the deviation was multiplied by the tested bit length to provide a standard range within which the true bit length may fall.

Ex. 78.5 nm × 0.014 = 1.01 nm