DEVELOPING A TECHNIQUE FOR MEASURING THICKNESSES OF THIN FILMS, FROM 5 TO 15 NANOMETERS, USING ATOMIC FORCE MICROSCOPY

by

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GRADUATE COMMITTEE APPROVAL

of a thesis submitted by

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As chair of the candidate's graduate committee, I have read the thesis of Guillermo Acosta in its final form and have found that (1) its format, citations, and bibliographical style are consistent and acceptable and fulfill university and department style requirements; (2) its illustrative materials including figures, tables, and charts are in place; and (3) the final manuscript is satisfactory to the graduate committee and is ready for submission to the university library.

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Contents

List of Figures

Chapter 1

Introduction

1.1 Standard techniques for measuring film thickness

Thin film research typically involves the study of several samples of a certain material, each of different thickness. In order to study the optical properties of thin films at energies above the visible spectrum (in the extreme ultraviolet), it is paramount that the thickness of each sample be known. However, in this field of study, the thin films are very thin, usually between 6 and 50 nm thick, and standard measurement techniques become difficult to apply successfully.

Of the characterization methods available, ellipsometry offers by far the easiest method of determining film thickness. It is very powerful and has the potential to provide a wealth of information about a sample being studied. Ellipsometric data analysis involves building a model to describe the sample, and allowing a computer to vary the parameters of the model until the calculations closely resemble the experimental data. However, it has been found that often the optical constants available do not provide an accurate description of very thin films, as they are intended for use with bulk materials. A solution to this would be to experimentally determine appropriate optical constants for each material to be used in the samples, but doing so would require the thickness of the sample be known. In short, ellipsometry can be used to provide the thickness of a film if its optical properties are known; or it can find the optical properties of a film if the thickness is known—but it is unable to report a reliable answer for either if both are unknown [1].

Another characterization method available is x-ray diffraction (XRD), which is

also an optical characterization method, but of a much higher energy regime. At 1.54 Å, the wavelength of the Cu II α photon used in XRD, the optical constants of most materials are experimentally known, and the distinction of whether the sample is bulk or a thin film does not need to be made. However, there are issues which prevent XRD from being thin film friendly. For example, XRD of thin films involves using near-grazing angles of incidence. At these angles, any error regarding the measured angles, either describing the position of the source or the position of the detector, will lead to difficulties determining the true characteristics of the sample. Also, since this method is optical, there are potential complications that come from the optical properties of the material being studied. At this wavelength the optical constants of most materials are known, but some materials have optical constants very similar to that of the substrate (for example, scandium films on silicon). In this case, there will be a lack of optical distinction between film and substrate, such that it becomes difficult to identify features in the data due to the presence of the film from those due to the substrate [2].

A very powerful tool in solid state physics is the transmission electron microscope (TEM), which has both diffraction and imaging sensitivities that allow for sub-angstrom resolution. However, the preparation of thin film samples for the purposes of film thickness determination with a TEM is a very time intensive and expensive endeavor. Also, the procedures by which samples are prepared for cross sectional imaging compromise the structure and purity of thin films, as it is typical for a thin film sample to be epoxied, polished, and released—all of which involve the use of aqueous solution [3]. Handling of a thin film in any of these manners will affect its physical structure, and thereby its optical performance.

Lastly, Atomic Force Microscopy (AFM) could be used to measure the thickness of a sample. An AFM has a pointed probe at the end of a long arm, similar to that of a record player, which can be used to "scan" a surface. A laser is bounced off the back of the probe, into a detector, as the probe rasters across the surface. Signals at the detector are used to provide information about the height of features on the surface, and this information can be used to assemble an image showing the

topography of the sample. AFM is also well suited to thin film studies since it offers sub-angstrom resolution [4]. Notice, data collected using this technique is purely a physical measurement, without the optical sensitivities of previously described methods. Analysis of the data does not require knowledge about the chemical composition of the sample—the only requirement is that the surface of the sample have the kind of physical features from which desired data could be collected. Much of the technique reported here involves producing such a "scannable" feature.

1.2 Earlier Techniques for Physical Measurement of Film Thickness

The characterization methods previously mentioned are by no means exhaustive as far as what is available to determine thin film thickness. They do, however, represent those to which the thin film group at Brigham Young University has access. As mentioned above, there are complications to studying thin films with the optical methods, suggesting that a physical measurement would be a welcome alternative. There has been other work done which involves physical measurements of surface features, at the nanoscale.

Profilometry is a measurement technique very similar to AFM, as it uses a pointed probe, or stylus, lightly dragged across the surface of a film, over an edge where the film ends, and down to the substrate [5]. However, a profilometer's range of measurement does not cover the typical thickness of our films: profilometers can measure surface features from 20 nm to several microns. Also, profilometers do not have the sensitivity that an AFM does: typical profilometers have a resolution of 1 nm, while an AFM has sub-angstrom resolution. Also, AFM has the added advantage of providing an image of the surface from which data was collected. This serves to confirm to the quality of the surface, which, given the precision we hope to achieve in our studies, can be crucial.

Optical profilometry, despite having a similar name, is a non-contact characterization methood which uses white light interferometry to develop an image of a surface. Under optimal conditions, this technique has angstrom resolution [6]. However, the area of the scan is several millimeters square, and achieving ultimate

resolution requires that the sample be flat over the entire region. This, unfortunately, is not true for all samples, as quartz and silicon substrates are not perfectly flat. The consequence is that the actual resolution of an optical profiler with typical thin films samples would most likely be several angstroms, possibly even a nanometer.

The technique reported in this thesis does not mark the first use of AFM to measure stepheights. AFM has long played a role in determining the thickness of films in the semiconductor industry. Among the methods and processes for producing features of interest for AFM study, "Shadow masking" (simply covering part of the substrate so that it does not get coated, and removing the cover afterward) and "lift-off" (by having a resist on a portion of the substrate, depositing the film, and removing the resist to leave a step feature) allow coatings as thin as 200 nm to be measured [7]. However, as stated earlier, the thickness of the films of interest are far thinner than that, typically less than 15 nanometers.

Chapter 2

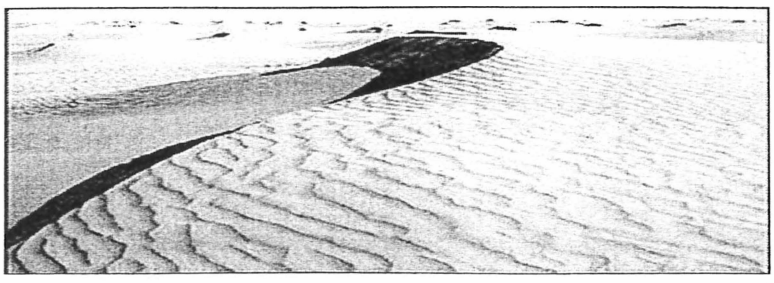
Abruptor Design

To be able to measure film thickness with an AFM, there must be (as mentioned in Section 1.1) a "step" on the sample over which the AFM probe can be scanned. This step is similar to a stair or step on a sidewalk, where the top of the step is the surface of the thin film, and the bottom of the step is the surface of the substrate. AFM scan sizes are on the order of, at most, tens of microns, so for a step between a film's surface to substrate to be useful, the transition between the two must take place over no more than a micron or two. An exaggeration of this concept is shown in Figure 2.1.

Originally, step fabrication was attempted after deposition, by scraping away the film to expose the substrate below. This approach failed repeatedly—the scraping process introduced vast amounts of nano-debris (which clings to the AFM tip, making data collection nearly impossible), as well as distorting the sample surface so that data collected did not reflect the thickness of the original film.

Attempts at masking the substrate during deposition using microscope slides and razor blades glued or taped to the substrate (shadow masking) prior to deposition were made. These masks were removed afterward with hopes that part of the shadow left behind would provide the desired step. With each sample, the transition region was much too broad, such that the thicknesses of the films could not be determined.

Masking the substrate during film deposition was attempted again. However, instead of simply laying a mask on the substrate, a razor blade was placed with only its sharpened edge in contact with the substrate. A rigid support for the razor blade was machined, and the device is called the Abruptor (pictured in Figure 2.2). There



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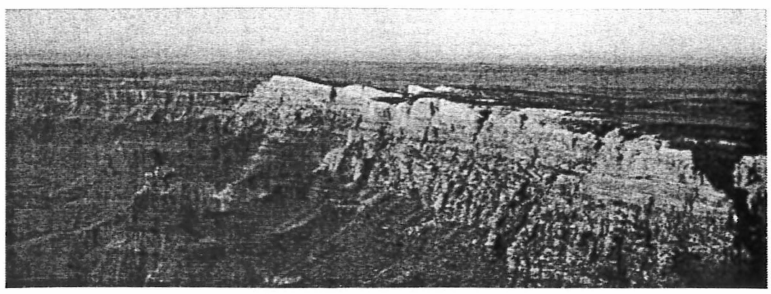
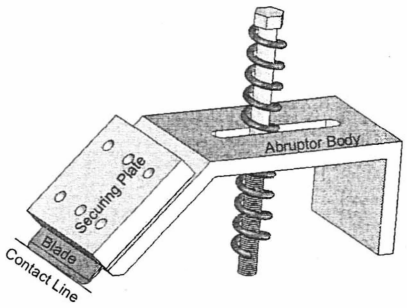


(b)

Figure 2.1: Imagine the above images as atomic topographies. The first shows a gentle transition from the top of the dune to the bottom, while the second shows and immediate and ***abrupt*** transition between being on top of the cliff to being on the bottom of the canyon.

was concern over whether or not the presence of the Abruptor would influence the coating of the surface locally. However, the angle with which the razor tilts back when in contact with the substrates surface is such that atoms drifting towards the surface do so completely unimpeded; the space immediately above the contact line will not produce a "shadow" during the sputtering process. A bolt, passing through two springs, one above and one below the Abruptor body, attaches the Abruptor to the platform on which the substrate sits during deposition. Tension provided by the springs allows for the blade to be placed in contact with substrate with moderate control and precision. As discussed in the next chapter, placing the blade in contact with the substrate is a delicate and crucial step when highest quality measurements are desired.



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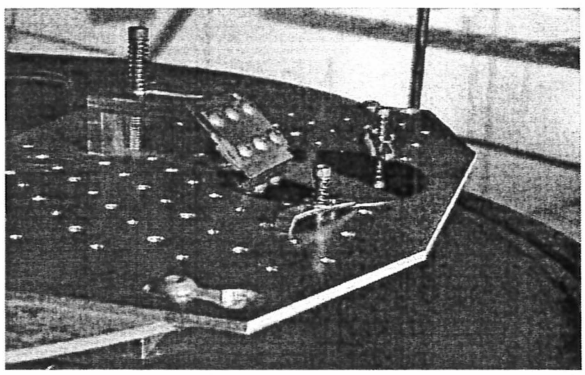

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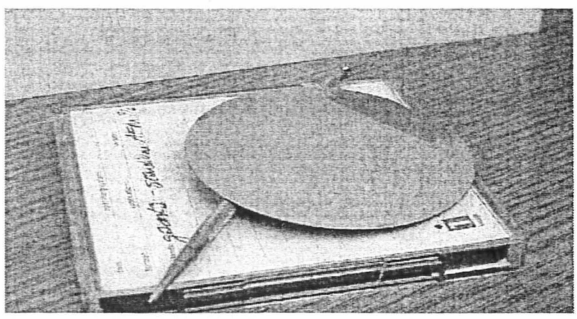

(c)

Figure 2.2: First, we see an illustration of the Abruptor design with its parts identified, followed by a photo showing an Abruptor installed over a substrate before being loaded into the deposition chamber. Lastly, a thin film sample is shown next to a Zip disk for scale. Notice the "shadow" on the sample where the substrate did not get coated. This is where the Abruptor was positioned during depostion.

Chapter 3

Measurements

3.1 Initial Results

Success was found with the first use of the Abruptor, which was with a thin film of vanadium (220 s deposition from a DC magnetron sputtergun onto a 3" silicon wafer). There was less than three hours between removal of the sample from the deposition chamber and data collection at the AFM. Using the scope in tapping mode and silicon nitride tip, the image in Figure 3.1 was one of several collected during the AFM session. The repeated appearance of a rounded triangle throughout the image is an artifact that probably comes from the substrate being ⟨111⟩ Si (future films were deposited on ⟨100⟩, which were perfectly flat). Silicon crystals with this orientation will not provide a perfectly flat surface. Instead, the surface will have pits, shown here as the depression inside the triangular features. Note that the triangle shape in the image appears with the same orientation, with the same frequency and prominence on both the film and substrate sides. The approximate sputter rate was known to be 0.04–0.05 nm/s, meaning the expected film thickness was 8.8–11 nm. A film this thin would translate any features the substrate might have without distortion, such as these from the ⟨111⟩ Si. Analysis of the image using the Digital Instruments software Nanoscope reported the film to have a thickness of 10.3 nm, seen in Figure 3.2, with the step transition occurring over less than a micron.

Following the initial AFM session, the vanadium sample was placed in an oven at 100 C for three days, to accelerate the oxidation of the film, after which it was remeasured with the AFM. It is routine for samples to be annealed in this way, and

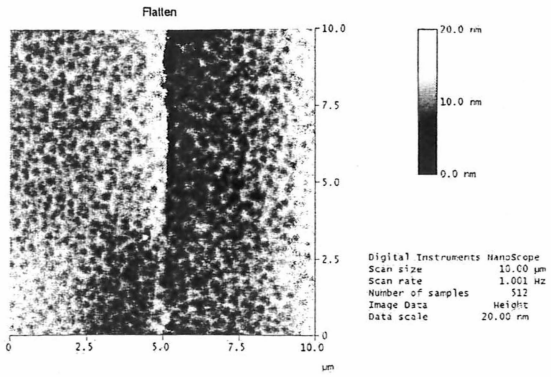

Figure 3.1: From the first AFM session using a sample having an Abruptor-produced step, this was the first image collected. To interpret the image, imagine that it is what an observer above the surface sees when looking straight down, where the brighter features are taller than those that are darker. On the right is a scale, in nanometers. The size of the scan is 10 microns square.

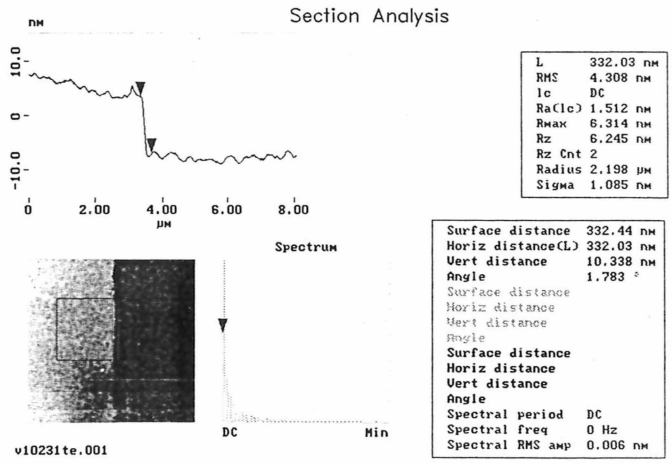

Figure 3.2: Using the Nanoscope software from Digital Instruments, the height of the step is shown to be approximately 10.3 nm. The software displays an average cross section in the top right portion of this figure, with which two cursors appear for determining distances between points along the cross section. Distances are reported in the data box the lower right— here, the "vert distance" is reported to be 10.3 nm, calculated from the the selected region of the image. It is difficult to see in a black and white rendering, but there is selection box (appearing as a black rectangle) in the image portion of this figure. When using this software, the selection box is used to choose the portion of the scan which will be used to generate the average cross section.

for the Abruptor to truly provide AFM measurable steps, the steps should be robust enough to provide thickness data following such an annealing period. Oxide formation will generally result in the film swelling, as oxygen is incorporated into the film's structure, similar to a sponge absorbing water. Analysis of the new images showed the thickness of the sample was now 14.8 nm, shown in Figure 3.3 Assuming that the densities of the deposited film and the resulting oxide—presumably divanadium pentoxide—are the same as their bulk counterparts, a swelling factor of 3.25 was predicted assuming all of the deposited vanadium is consumed in the oxidation process. The measured swelling factor of the vanadium thin film in this case approximately 1.4, assuming that during the initial AFM session the amount of oxide present was negligible. With such a difference between predicted and measured values, it seems likely that the oxidation of the thin film was incomplete, which is in agreement with our experiences with thin films oxidizing. The mobility of ions in the film is rarely great enough to oxidize all of the virgin material. Regarding thin films of this nature, ion transport will be dominant along grain boundaries. Such transport is unable to expose every deposited vanadium atom to oxygen.

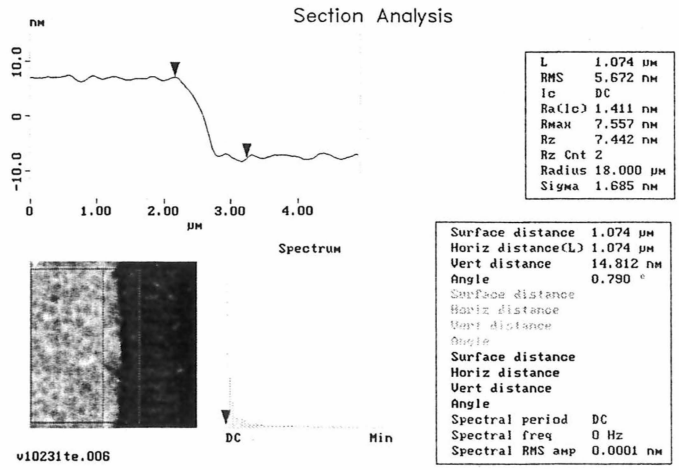

Figure 3.3: Following an annealing period, the height of the step is found to be approximately 14.8 nm.

There was concern over whether or not different positions along the step produced by the Abruptor would measure consistently. Admittedly, the films produced in the sputter system are not perfectly uniform (the degree of which was studied using AFM, and is reported in the next section of this chapter), but it was expected that several measurements along a single step would be in moderate agreement. Measurements at different positions along the step, in both pre- and post-annealing sessions, do indeed report consistent values. As an example, Figure 3.4 offers a second measurement of the annealed vanadium film, taken at a different position along the step. Notice, the measurements of Figure 3.3 and Figure 3.4 match the nicely, despite the two locations of the data collection being separated by millimeters.

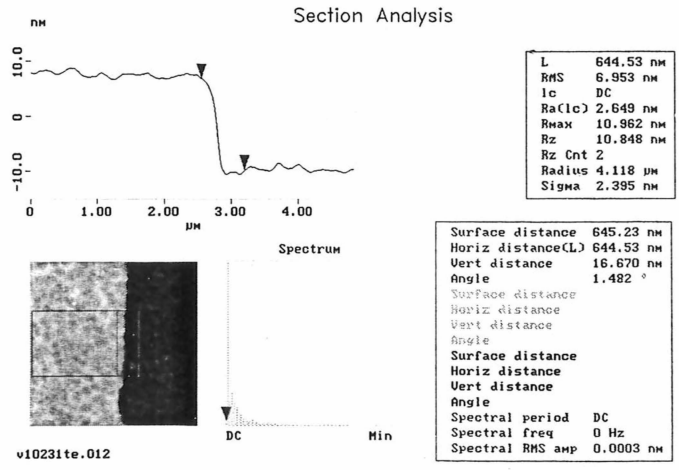

Figure 3.4: Subsequent scans of the annealed vanadium film at different positions along the step report similar thickness values. Here, the film thickness is approximately 16.6 nm, which is reasonably similar (within 10%) to what was reported by Figure 3.3

Several samples of vanadium thin films, representing a thickness range of 6–15 nm, were studied in this way. Repeatedly, the step produced by the Abruptor provided consistent results.

3.2 Thickness Uniformity

AFM offers substantial control to the user to select the region of the surface to be measured. The operator has the ability to choose not only the size of the scan (for thin films, scan sizes of 10 and 20 microns were found to work best when determining film thickness), but also the position on the surface of where the scan is taken. The sample is positioned under the AFM probe with a combination of stepper motors (smallest step: 2 microns) and piezo electronics (smallest displacement: 0.1 micron). This ability to make site specific measurements made an investigation into film uniformity possible.

A characteristic of our samples that had yet to be addressed completely was their uniformity. When samples are made in the sputter system, the substrate onto which the films are to be deposited is passed through a coating region, as illustrated by Figure 3.5. In the coating region, the density of the atoms that form the film decreases with increasing distance from the gun. This is illustrated by analogy with Figure 3.6. Since the substrate's surface is flat, the film will not be uniform.

The uniformity of the coating was studied with the Abruptor positioned so that the step produced would reveal the cross section of the film *perpendicular* to the path the sample takes through the coating region (to produce steps for film thickness measurements, the Abruptor was positioned *parallel* to the path), clarified by Figure 3.7. In this position, it was necessary to stop sputtering as the Abruptor contact line approached the center of the coating region. If sputtering continued as the Abruptor passed though the coating region, local shadowing would occur as the body of the Abruptor would block atoms drifting to the surface.

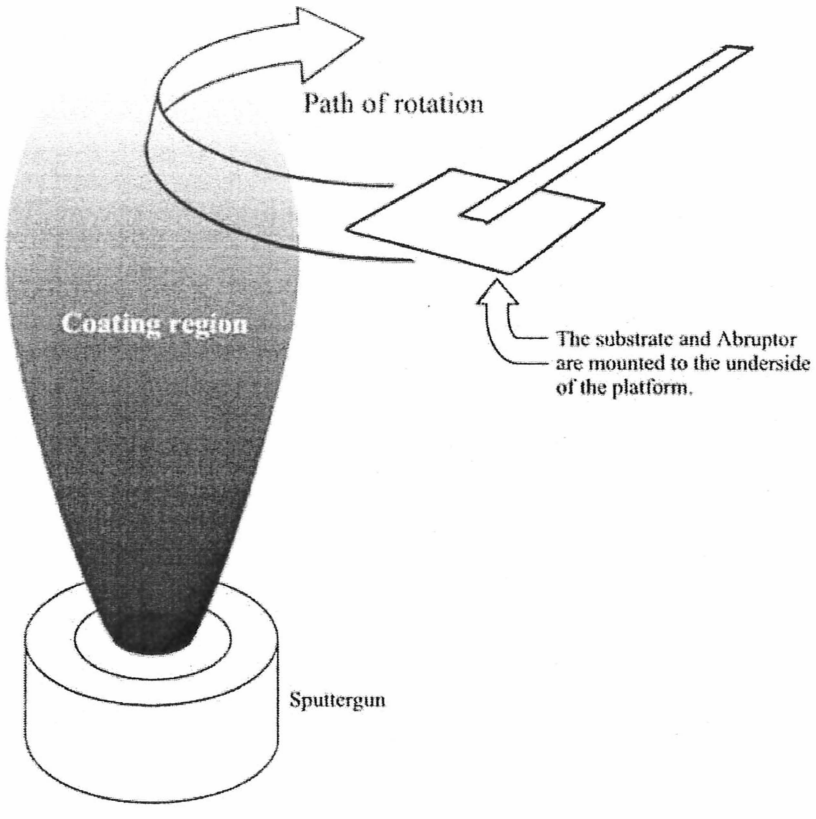

Figure 3.5: Film depostition is performed in a high vacuum chamber, inside of which there is a sputtergun which produces a "mist" of atoms to make the thin film. The space where the "mist" of atoms exists is called the coating region. A platform carrying the substrate is passed through the coating region, and emerges coated with a thin film of atoms from the "mist".

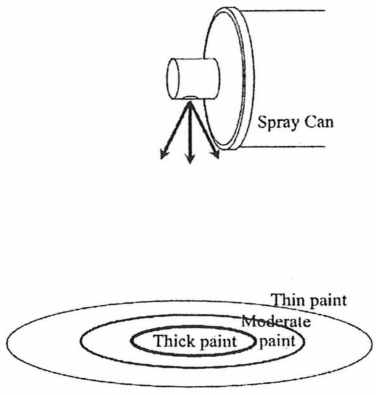

Figure 3.6: The density of the atoms in the coating region described by Figure 3.5 is not uniform. Consider a spraypaint can to develop an analogy. A dot painted on the ground will have the thickest paint at the center of the dot, with less paint in any direction. This shows that with less distance from the source, the larger the density of the "mist" (either paint molecules or atoms), while being farther from the source will result in the "mist" being less dense.

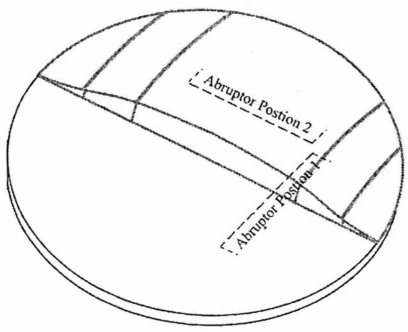

Figure 3.7: Here, we see a thin film on a substrate. Half of the film has been removed, to show the profile of the film we are interested in. The variation in the film thickness across the substrate has been exagerrated. There is a slight "curve" to the coating, due to the path it took through the coating region (see Figure 3.5). By placing the Abruptor so that the contact line is tangent to this curve (Abruptor Position 1), the step produced will have a more or less uniform height across its entire length. Placed perpendicular to this curve (Abruptor Position 2), the Abruptor will produce a step that will not be uniform across its length, but will show the degree to which this nonuniformity is present.

With the Abruptor set as described, a film of vanadium was deposited on silicon, then placed into the annealing chamber for three days. Twelve AFM scans were taken along the edge, approximately every three millimeters along the Abruptor produced step. Figure 3.8 plots the thickness vs. position, showing the coating is thinner with increasing distance from the center of the coating region as expected. Though the films are not absolutely uniform, the measurements show that the samples can be considered sufficiently uniform when probed by beams of other characterization methods, such as XRD and ellipsometry.

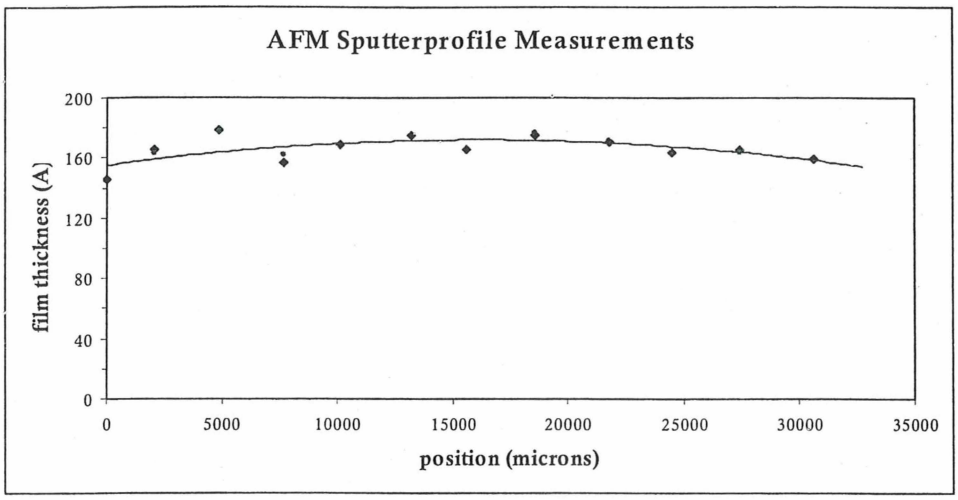

Figure 3.8: Spaced approximatly three millimeters apart, twelve thickness measurements were made along the step. The step was produced with the orientation such that the profile it offered would show the uniformity of the film.

3.3 Unexpected Surface Features

Following the confirmation of the utility of the Abruptor, its use became standard on samples being grown in the high vacuum sputter system. Though imaging sessions were usually routine, occasionally features would appear in the images that were without immediate explanation. Figure 3.9 shows four examples of strange structures on sample surfaces. Each of the images in Figure 3.9 is that of a step between film and substrate. On the surface of the film in each image, there is a number of small ridges which run parallel to each other, intersecting the step at various angles. In each instance, the family of ridges have approximately the same height, but do not have uniform spacing. Also, the ridges do not intersect each other, suggesting that all of them are the consequence of a single physical movement or action. These features were not consistently present among the samples. There is little doubt that these features appear because the use of the Abruptor, but their exact origin is unclear. Were these features placed on the surface during Abruptor engagement, or during its removal? Apparently, the Abruptor acted as a plow, but it could not be said for certain that if what was being "pushed" was the deposited material. If the removal of the Abruptor was indeed responsible for the features, the process would most likely have the deposited film buckle/or and tear, thereby introducing rip marks and nano-debris. However, when these unusual features would appear in the image, the scan area would not be unusually dusty.

It seems more credible that the features were in place before deposition occurred. With films as thin as these, between 4–15 nm, any nanoscale structure on the substrate would translate through the film perfectly. The thin film group has previously considered the issue of contaminants on surfaces, and has found that the exposure of any clean surface to ambient conditions will ultimately result in the surface being coated by a hydrocarbon layer. The presence of this contaminant is easily overlooked by most analysis, as it is on the order of 1 nm thick, organic, amorphous, and largely transparent in the visible spectrum. Experiments using X-Ray Photoelectron Spectroscopy have confirmed its presence [8]. Regarding the strange features showcased in Figure 3.9, there is a possible explanation that involves hydrocarbon

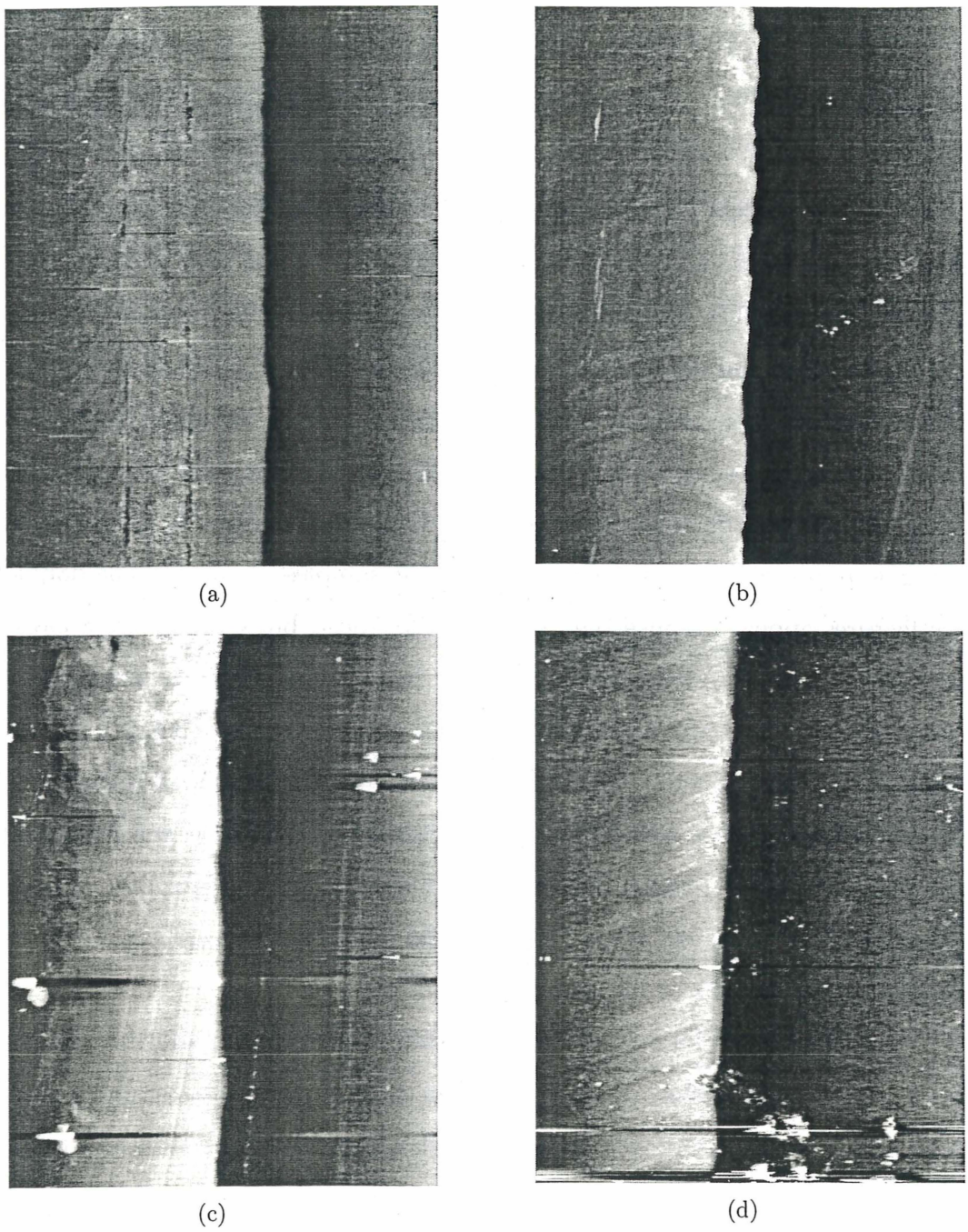

Figure 3.9: Four examples of the puzzling surface features are shown, each from different samples. Each image is that of a step between film and substrate. On the film side of each step, there appear to be ridges, which are not seen on the substrate side. Between the images, the families of ridges have little in common, while ridges of a specific family follow a common contour. Two of the images here show some nanodust, which is responsible for a few bright scan lines, but this is not unusual to see on the sample surfaces.

contaminants and use of the Abruptor.

The inspiration for the explanation came one February morning while resolving a natural deposition in the driveway. If, during the initial contact of the Abruptor with the surface, some of the hydrocarbon layer were "plowed" in front of the blade (similar to that shown in Figure 3.10), a film deposited afterward would report this on its surface.

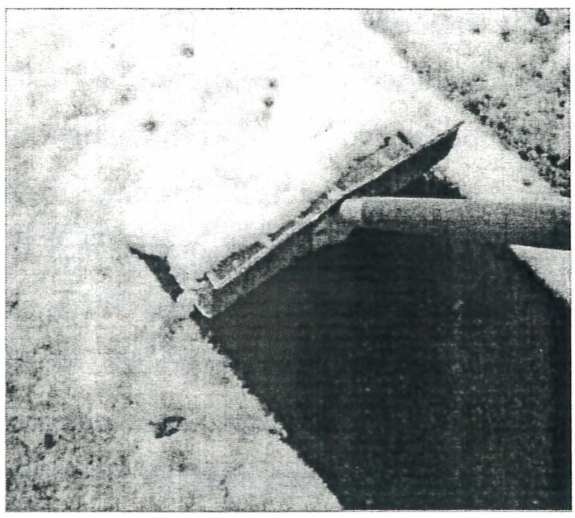

Figure 3.10: Not exactly at the nanoscale, but the buildup of snow in front of a shovel is believed to be similar to what is occuring with the blade of the Abruptor as it engages the surface before film deposition.

Standard Abruptor engagement is done as delicately as possible, such that there is no sliding of the blade across the surface— it was found early that the blade sliding across the surface would "pile up" nanodust along the step, complicating image collection.

Instead, during initial engagement, the Abruptor body is placed on the platform and held so that the contact edge of the blade is off the surface (refer to Figure 2.2). Then, it is secured in place by the bolt (through the two springs) into the sample platform, *while still holding the body so that the blade is not in contact with the*

surface. The body is then *gingerly* let down so that the razor's edge meets the wafer surface, at which point the compressed springs hold the Abruptor in place.

Even though Abruptor engagement is made by letting the blade down onto the surface, there is still potential for hydrocarbon "plowing". As initial contact is made, the portion of the blade that is not sandwiched between the securing plate and support surface (between the edge of the razor and the Abruptor body) will bow under the applied force. With experience, a "feel" for the proper bolt setting can be developed to minimize "blade-bow", but in can never be absolutely eliminated. When blade-bow does occur, the contact edge is not absolutely fixed to one position, and can, very minutely, slide along the substrate surface. As the blade slides along the surface, it could plow some of the hydrocarbon contaminant in front of the blade, at isolated points of contact, discussed in Figure 3.11. Images made with the AFM report angstrom-scale features, in areas 10 microns square, such that **any** hydrocarbons plowed during blade-bow will appear in the images. Being organic and amorphous, hydrocarbons are usually transparent to AFM probes: an AFM probe would push through the hydrocarbon until the surface below is encountered, practically unaffected by the hydrocarbon contaminant. However, here the plowed hydrocarbons are coated with a thin film, whose surface will match exactly the surface over which it was deposited. Subsequently, thickness measurements will report not only the thickness of the film plus the hydrocarbon below.

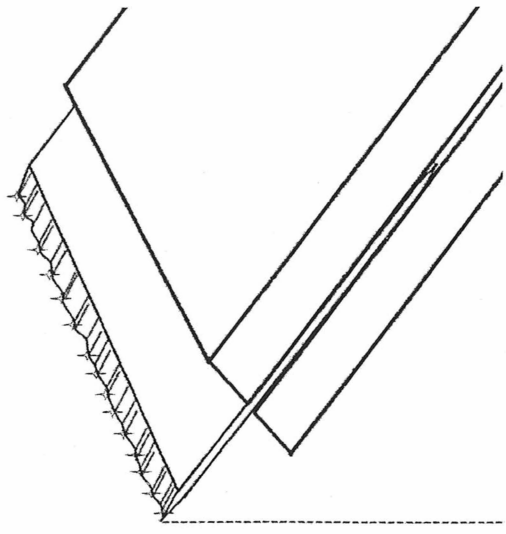

Figure 3.11: At the nanoscale, the edge of the blade will not be uniform, but instead jagged. This will mean that true blade contact with the surface of the substrate will be made at isolated points. Considering blade-bow and the "plowing" of hydrocarbons, this would explain why the strange features appear only occassionally.

3.4 Measurements Made for an Oxidation Study

Having become familiar with the use and performance of the Abruptor, it was employed in a study of thin films that focused on developing the ability to determine the amount of oxide on thin films exposed to atmosphere. In these experiments, thin film samples would be annealed for an extended period to accelerate oxidation to the point of inactivity, such that subsequent room temperature oxidation would be slight. The samples would then be measured at multiple angles with a spectroscopic ellipsometer, followed by film thickness measurements using the AFM. Ellipsometric data can be analyzed to determine the optical constants describing the oxide, provided that the thickness of the sample is a known parameter. The thicknesses of films as thin as these have proved difficult to determine by other methods, however, with steps produced using the Abruptor, thickness measurements are straightforward. Also, determining the thickness of the films using AFM would allow for multi-sample

analysis of the ellipsometric data, which is very powerful in determining the most accurate description of the films.

Three thin film samples were deposited, separately, as a multiple of a single sputter time (133 s, 266 s, and 399 s) to produce samples that would have thicknesses similarly related. A graph of the thickness measurements is shown in Figure 3.12, with a trendline showing the results of a linear fit. The equation of the trendline shows the thickness vs. time would extrapolate to 1.4 nm for a 0 s deposition (instead of to 0 nm for a 0 s deposition), which is cause for distress. There are two possible explanations for this.

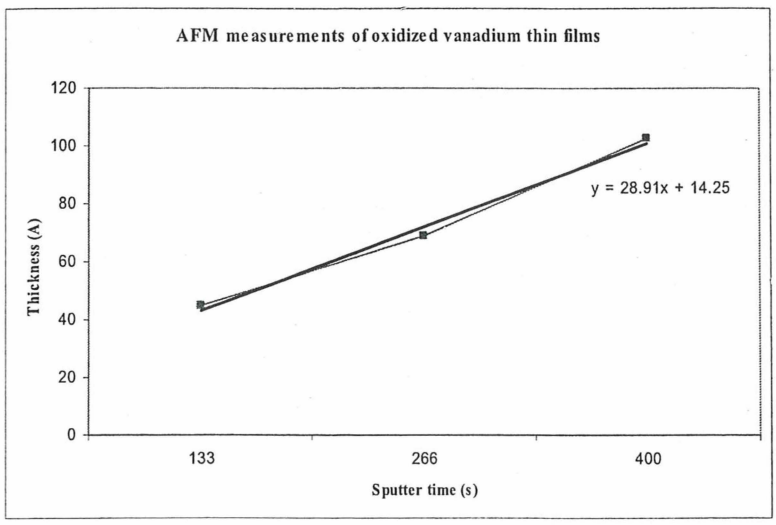

Figure 3.12: Films with sputter times that are multiples of each other are expected to have thicknesses which are multiples of each other. Here, though, we see that the film that has the 266 s sputter time is not twice as thick as the film with the 133 s sputter time, nor is the 399 s film three times as thick as the 133 s film.

The first involves the presence of hydrocarbons on the substrate surface, such that the thickness measurements are not solely that of the film, but that of the film *plus* the hydrocarbon underneath. Based on recent experience with hydrocarbon contamination, the hydrocarbons present on the substrate before film deposition could

be between 0.1–1.5 nm thick, depending upon the amount exposure to ambient conditions.

A second explanation for the nonlinearity of the film thicknesses comes from the possibility that original films are not completely oxidized. It is not unusual among metal thin films for a small amount of the original material to remain un-oxidized. The percentage of the virgin material that does not get consumed during oxidation, though, could be thickness dependent. Perhaps thinner films are more completely oxidized, while thicker films have more of their original material remaining inside the oxidized film. This would lead to swelling factors of thinner films being larger than those in thicker films, since a larger percent of the original material has oxygen atoms incorporated into it following the annealing process.

Further study of the oxidation of thin films is needed to determine the relative validity of the above explanations. However, it was data collected using an AFM on the steps of the thin films that recognized the existence of this issue, showing again the value of this technique for producing steps in thin films.

Chapter 4

Data Analysis

Extended use of the Abruptor and analysis of the steps produced shows that the success rate of producing a measurable step is about 70% when a step of the highest quality is needed, and nearly 90% for work where only a thickness estimate or confirmation is sought. As described in Chapter 3.3, bringing the razor's edge into contact with the substrate is a delicate procedure that requires a skilled hand. Similarly, removal of the Abruptor once a film has been deposited has the potential for ruining the sample. Since measurements made of the surface will report nanoscale features, it must be acknowledged that the engagement or removal of the Abruptor will never be an action without consequence. On the nanoscale, the "macro" action of placing or removing the Abruptor could be as cataclysmic as an earthquake.

Originally, the Abruptor was simply unbolted from the platform, and, if care was not taken, the Abruptor might slip, ever so slightly, into the film, and scar portions of the step. Now, removal is instead done by sliding the Abruptor away from the step, then unbolting it from the platform. This dragging of the blade across the substrate is considered benign since during blade engagement blade-bow will plow hydrocarbons in the step region anyway. The quality of the initial images is attributed to luck, as they represent literally the first use of the Abruptor.

Transitions between film and substrate vary, but steps typically have slopes on the order of 8 nm over 0.7-0.9 microns. Thinner films (less than 6 nm) have proven more difficult to produce a high quality transition with, where the transition from film to substrate may have an acceptable slope only in isolated regions along the length of the edge. The best transition observed has been a drop of 12.5 nm

over a mere 0.12 microns, while the thinnest measured film has been 3.6 nm. It is believed that thinner films could be measured, provided that the appropriate surface position for engaging the AFM could be seen with the "guidance" optics. A majority of the extremely thin films studied have oxides present that are essentially transparent in the visible, making identifying the step while cruising the surface prior to probe engagement very difficult. Being able to determine where to engage the probe with the sample is crucial, otherwise collecting an image of the step is akin to taking shots in the dark.

Quantitative analysis is done with the Nanoscope software provided by Digital Instruments. Raw AFM data is first made flat using the *Flatten* function, since the scan of the probe over the surface resembles the path of a pendulum (which is more evident in larger scans). When scanning a step with the AFM, the region shown in the image is not flat, but instead two flat surfaces of different heights. *Flatten* is appropriate for use on a image where the entire scan is a single, flat surface. Samples with steps introduce a complication that *Flatten* does not accommodate well. As a result, use of *Flatten* will yield an image with flat, but sloping surfaces, seen in Figures 4.1 and 4.2. Despite this shortcoming, from here the measurement of the step can be taken several ways.

Originally, the *Stepheight* function was used to determine film thickness after *Flatten* was applied. However, it was later found that this approach is not ideal for use with our samples. Using *Stepheight* would make use of **all** the data in the image area, and nearly all scans bear the presence of non-ideal features: typically nanodust, or peaks along the step's ridge. Also, *Stepheight* prompted the uses to select a region of both the film and the substrate for weighting data in the actual stepheight calculation. This film/substrate area selection proved to be very influential over has with the reported value, in addition to the undesirability of having **all** data in the image included in the thickness measurement.

A better method for analyzing data was found. It is flexible enough to account for the inherent imperfections of surfaces and allow for user discretion in which portions of a scan are favored over others. Following the use of the *Flatten*(which,

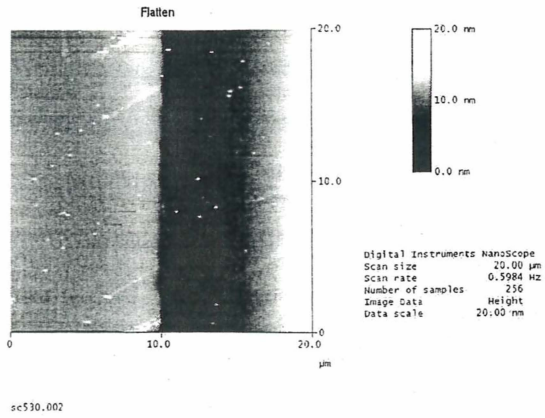

Figure 4.1: Raw AFM data of steps in thin films is rarely able to be used directly to give the desired measurements, so some manipulation is required. Here, the *Flatten* function in the Nanoscope software package has been applied to an image of a step in a scandium thin film. Notice the trend the image has (from left to right): the image of the film gradually goes from dark to light, then the step appears, and the substrate also goes from dark to light. According to the image, neither the film nor the substrate are on the horizontal plane. This sample used a Si ⟨100⟩ Si substrate, which is smooth— note the absence of the rounded triangle features of Figures 3.1 thru 3.4

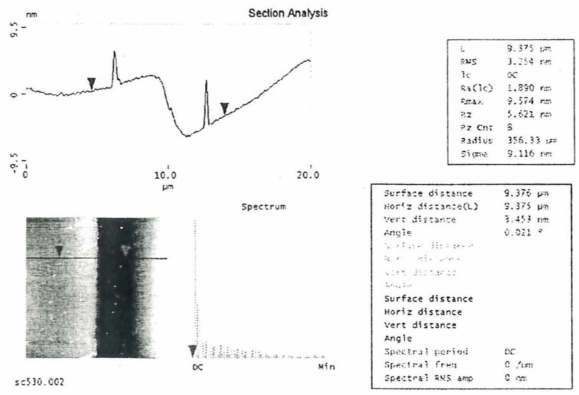

Figure 4.2: To illustrate the effects of *Flatten*, the *Section Analysis* function is used to show the profile of the image along the line selected. The profile shows the trend decribed in the caption of Figure 4.1—the surface of the film and substrate are flat, but not horizontal. The profile displayed is a cross section of the image along the selected line, and here it includes some nanodust, shown as spikes. There are also two cursors in the profile which can be positioned to points of interest on the profile. Distance between the two cursors are reported in the data box to the lower right.

despite its shortcomings, is a necessary first step to "reference" all the raw data to one plane), *Plane Fit Auto* is used to establish a portion of the image as being truly flat; typically the substrate half of the image is chosen. At this point, the quality of the scan can be roughly gauged—if, following the use of *Plane Fit*, both the film and substrate regions of the image each have a uniform coloring, then the image is showing each region to be horizontally flat. Should the image instead report that one region (the usual suspect is the film) to not be flat, but rolling slightly, it will be necessary to accommodate this in the next step.

Now, a cross section analysis of the image is conducted. The *Section Analysis* function offers several options for how a profile of the image will be reported. Among them is an average profile, with which the user is first prompted to select an axis, perpendicular to which the cross section will be calculated. Next, a rectangular region of the image (with two sides parallel to and two sides perpendicular to the chosen axis). This rectangular selection box determines what portion of the image will be used in calculating and average profile. With this ability to select data, it is possible to exclude portions of the scan which are not appropriate to a proper film thickness measurement (such as dust, local depressions in the film, or isolated peaks in the step's ridge). Following the calculation, the average cross section will be displayed, along with two cursors that "ride" along the profile. By positioning the cursors to appropriate positions along the profile will provide a film thickness measurement in the data box showing the surface, vertical and horizontal distances between the cursors. Figure 4.3 shows this method of analysis applied to the image used in Figures 4.1 and 4.2.

Despite the advantages of this approach, there still may be issues with the reported numbers. With the exception of the highest quality steps, the average profile displayed using *Section Analysis* will exhibit a slightly rounded corner where the profile transitions from the surface of the film to the slope of the step, instead of a sharp, crisp corner (see Figure 4.3). In other cases, the film side of the profile may not be perfectly flat, but instead slope down to the transition. The horizontal and vertical distance between the cursors in the data box are simply computed as the

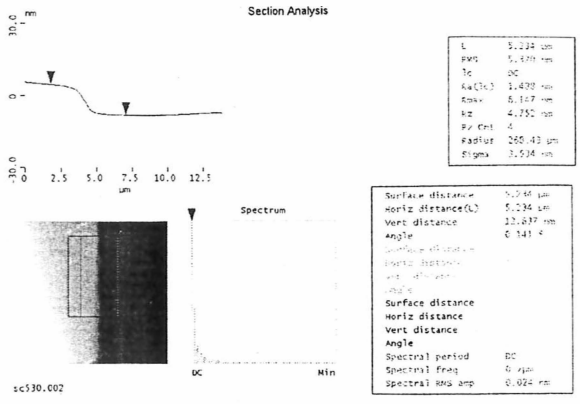

Figure 4.3: A region of the image of the scandium thin film has been selected, marked by a black rectangular ouline in the image area to the lower left (this may be difficult to see in a black and white rendering). The data in the selected region is used to determine an average profile of that region, which is displayed to the top left. Included with the displayed profile are two cursors, which may be placed along the profile for determining distances, which are reported in the data box to the lower right.

difference between coordinate pairs on a cartesian grid. Should the profile be shaped either of these ways, it becomes difficult to choose a cursor position which represents the true height of the film.

Chapter 5

Conclusion

The technique developed to create a step in a thin films allows for film thickness to be determined with an AFM. Films ranging between 6–15 nm can be measured with relative ease, and it has been shown that films as thin as 4 nm can be measured as well. Anomalous features along the film side of steps are attributed to the presence of a layer of hydrocarbon, 1–10 nm thick, on the substrate's surface prior to deposition. AFM measurements of metallic thin films before and after an annealing period show a nonlinear relationship among film thicknesses, despite a linear relationship among sputter times. To resolve this issue further study is required.

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