Design and Implementation of DC Magnetron Sputter Deposition System and Hall Effect System Via LabView

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Jason Wright
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This thesis titled
Design and Implementation of DC Magnetron Sputter Deposition System and Hall Effect
System Via LabView

by

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ABSTRACT

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Design and Implementation of DC Magnetron Sputter Deposition System and Hall Effect System Via LabView

Director of Thesis: Savas Kaya

Integrated software controlled systems are necessary for accelerating academic research and process development. This allows a tremendous increase with the accuracy, productivity and reliability levels in instrument operation and research development. Software controlled fabrication equipment and characterization systems allow researchers and process engineers a wide range of advantages over manual analog controlled systems of the past including improving safety, reducing operator errors, increasing the rate of data collection/monitoring capacity and ease of use. The focus of this thesis is the software development and accompanying hardware implementation for two systems: DC Sputter system and Hall Effect system. The DC Sputter system, which was re-designed ground-up, will be compatible with operation in two modes. The first operational mode is completely automated, with simple push button commands for execution of programmed deposition routines. In contrast, the second operational mode allows the full manual control of the system, digitally, over a touch screen interface, thus significantly improving productivity and user friendliness. In addition to a LabView interface implemented, the DC Sputter system also has custom circuitry and hardware interlocks in parallel with the advance software controls. The Hall Effect system differs slightly, in the software design; it has only one mode of operation that is fully automated. The Hall
Effect system, built from the ground up, has a custom test bed and I/O connection platform developed to allow for testing of varied sized samples. Finally, each system has been tested for proper functionality to confirm reliable system operation.
DEDICATION

I would like to first dedicate this thesis to my parents, Erin and Bob, without their constant sacrifice, I would never have had these opportunities. They have gone without so that I could have a chance at making a better life for myself.

I would also like to dedicate this thesis to my fiancée, Ashley. She has provided me with the encouragement and support to strive to be the best person I can be.
ACKNOWLEDGMENTS

I would first like to sincerely acknowledge my two advisors Dr. Savas Kaya and Dr. Wojciech Jadwisienczak, without their guidance and leadership the work for this thesis could not have happened. They have exponentially increased my knowledge during my time as their student. In addition they have both became my mentors and friends.

I would also like to acknowledge Dr. Faiz Rahman. Although I have worked a shorter time with Dr. Rahman he has provided me with the benefit of his wisdom and experience. He has also become a mentor and friend.

Lastly, I would like to thank Nicholas Montgomery, an undergraduate researcher who helped me during the testing and debugging of the DC Sputter system.
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CHAPTER 1: INTRODUCTION

The ongoing nanotechnology revolution can offer solutions to fundamental problems humanity must deal with in the 21st century including energy, environment, health, security and poverty. The ever-increasing invention-to-product cycles in modern economies requires higher productivity and precision, for researchers and manufacturing systems. Many advanced scientific and engineering tools cannot be operated safely and reliably without the embedded controllers and computers that act as interfaces between the human user and the instruments. In recent years, with the advent of ever cheaper and more capable touch-screen enabled modern microcomputers, instrument control and instrument engineering is advancing to new heights at a phenomenal pace. In particular, the use of National Instrument’s LabView graphical programming interface has become almost an industrial standard in developing user-friendly virtual instrument controls with data monitoring, collection and presentation capabilities. This thesis exemplifies two such efforts. The first is a LabView-based control of a re-vamped from the ground-up DC magnetron sputtering system and a ground-up Hall Effect characterization system. This work was carried out in the nanofabrication and characterization labs run jointly by Drs. Kaya and Jadwisienczak. As a result of this work, we significantly improve productivity, user experience and accuracy of two instruments crucial for development of novel nano-scale optical and electronic devices and sensors.

The need for increased productivity is not only a market reality. In today’s modern academic research it is paramount to have the ability to quickly generate new research. This puts academic researchers in an awkward position. Should they sacrifice
the other ideals of academia for higher research throughput? Regardless of which side you are on, pro or con, there is no denying the dramatic increase of research throughput. The total number of scientific paper publication has been shown to be exponentially growing every year for the past several decades [1-5], shown in figure 1. It is shown that every year a researcher must publish 4 – 8% more publications than the year before [2-4].

Figure 1: Left - Number of Papers Published Every Year. Right - Ratio of the Number of Papers Published Year to Year [2]

So how does a researcher meet this demand? The first option is to forgo everything else and solely focus on research. This is not a realistic solution. The next option is to hire, recruit, an army of graduate students to perform the research. This too is not an option for majority of universities, so the integration of ‘smart’ software-controlled systems and computer-enabled automation for higher productivity in research labs appear to be the most practical solution, which we pursue here.
1.1 Overview

This thesis explores the development of two smart software controlled systems. The first system is a DC Sputter Deposition system, while the second system is a Hall Effect system. The two systems, while being vastly different, share the same development process. The first step, in development, is to determine the required instrumentation. This step is the essential building block for subsequent steps. This requires the developer to not only understand the desired functionality of the system but also the capabilities and compatibilities of existing instruments. This step is followed by the initial hardware development, the “bones”, of the system. This requires in-depth knowledge of each instrument and excellent “hands on” skills. The ensuing step is the software development, the “brain”, of the system. This entails the developer too not only understand the operational, functional and performance requirements but also ensure the safety and ease of use. The next step is to do extensive testing and debugging, during which the development can cycle back to any of the previous steps necessary. Once proper functionality and safe operation of the system is obtained, rounds of calibration and quality monitoring occur. This step requires the use of several other measurement systems to independently verify the results of from the system being developed. This includes Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM), Ellipsometry, X-ray Diffraction (XRD) and Source Measuring Unit (SMU). Each system must pass repeatability, reliability and reproducibility standards before the design can be finalized. In this thesis, work on all above steps to implement the two systems mentioned will be presented.
1.2 Motivation for Research

The general motivation for this research is to enable our laboratories with the ability to improve productivity, reliability and an enhanced user experience. The addition of automated software controlled systems has many advantages over traditional analog, manually, controlled systems. Academia has a large turnover rate of researchers hence; training of new users is a constant need. The automated software controlled system allows a new user to be trained in a matter of minutes or hours rather than days or weeks. This alone can speed up the output of a lab. In research, one of the largest problems is repeatability, reliability and reproducibility from user to user. In an analog, manually, controlled system this requires every user to operate and run the system in exactly the way each time and every time. This is almost impossible to realistically accomplish, given the fact that many users will be new and open-loop controls cannot guarantee the same performance. However, with a software-controlled system this becomes a rather easy task. The last major reason, especially being in academia, is the safety of users. Many users may not be aware of potential safety hazards of the system. This can cause potential accidents that harm the system, or worse, the user. A software-controlled system can mitigate many of these potential situations.

1.3 Applications

It is important to point out the range of applications relevant for each system being designed in this work. In the two following subsections, each system will be discussed independently.
1.3.1 DC Sputtering

Magnetron sputtering is a flexible deposition method, which makes it used over a multitude of different industries. These industries vary from electronics, micro-electrical-mechanical systems (MEMS), data storage, optics, bio-medical and surface coatings. These critical industries all have different needs/uses that sputtering is employed to solve, especially when high-temperature employed by traditional thermal evaporation cannot be used. While there are too many applications to name them all, it is important to remember that in micro/nano technologies surfaces determine/dominate the observed response of many materials and devices. Since sputtering can deposit a broad range of thin films of metal, insulator or semiconductor materials, its’ application has grown considerably in recent years. The most common uses for sputtering are interconnects in electronics, magnetic disks in data storage, anti-reflective coatings in optics, anti-corrosion coatings in surface coatings, and anti-rejection coatings in medical applications [6-8].

1.3.2 Hall Effect

Hall Effect is a powerful characterization tool. It offers the ability to precisely measure numerous fundamental material/device parameters from one test, most commonly for semiconductor materials. It allows direct electrical measurement of majority carrier type and concentration. It also allows for the calculation of carrier mobility. This means that it is used in electronics, solar cells, lasers, LEDs and many material-based industries [9-11].
CHAPTER 2: BACKGROUND

This chapter will discuss the theory and background that relates to the two systems and the measurement techniques used in this thesis. The theory will be discussed only in a general overview, for a more detailed description, refer to the provided references. If the reader is unfamiliar with these subjects, then it is highly recommended to read the references before continuing to further chapters.

2.1 Theory

DC Sputtering and the Hall Effect will be described first. This is followed by the theory behind Atomic Force Microscopy, Ellipsometry and X-Ray Diffraction. In the sections covering the measurement systems, key parameters used later in the analysis will be covered. Lastly, LabView will be introduced, which is the software used to develop the code for the system.

2.1.1 DC Sputtering

Sputtering is a form of physical vapor deposition. Physical vapor deposition is the process by which a film is formed by the physical transport of atoms directly from the source to the substrate through a plasma phase. This process takes place inside of a vacuum chamber. The target/source and the substrate/sample are placed on two parallel electrodes. The chamber is then filled with an inert working, gas. In most systems, this gas is Argon. A DC voltage is applied between the two parallel electrodes. The target has the negative side of the voltage applied, making it the cathode. The substrate has the positive side or ground of the voltage applied, making it the anode. The applied voltage for this system is up to 1k Volt. The application of a high voltage generates an electric
field, which accelerates the free electrons present in the chamber. There is a stochastic probability that these accelerating free electrons will collide with the Argon atoms present. If a free electron collides inelastically with an argon atom then one of two outcomes is likely. First, the electron will cause excitation of the Argon atom; this is the cause for gas atoms to glow. This electron will then either cause an outer-shell electron to be ejected or will be re-ejected, thus having one electron hit the Argon atom and one electron leaving the Argon atom. Second, if the electron has enough kinetic energy when it collides with the Argon atom it will cause the ionization of the atom, shown in figure 2. This means the electron that collides with the atom will cause two electrons to be ejected. This other ejected electron is known as a secondary electron. This now creates a cascading effect, with one electron producing two electrons and an ionized Argon atom. If a large enough number of Argon atoms undergo this process, then plasma will be formed. These newly formed ions will be attracted to the cathode, which is held at negative voltage. These ions will slam/collide into the target surface. This collision will provide enough energy to break the bonds between atoms of the target material and physically eject/sputtered target material atoms. These atoms will move in more or less a line of sight path, scattering is mostly due to collisions with Argon atoms in the chamber. A percentage of these sputtered atoms will strike the substrate. If the energy/momentum carried by these sputtered atoms is not too low or too high, it will cause these atoms to adhere/stick to the substrate surface.
During this repeating process near the cathode, a positive charge will build up. This is due to the electrons having a much higher velocity than the Argon ions. This will cause the area around the cathode to become depleted of electrons. This depletion then causes fewer collisions, of electrons and Argon atoms, and in turn causes this area to be void of plasma. This area is called the dark zone because of this effect.

This effect can be minimized by using a magnetron sputtering head. This differs from the normal sputtering head by the addition of magnets. The head is normally in the form of an outer-ring and inner-ring/circle of alternated magnets located parallel, behind, the target. This causes a magnetic field to be created around the target. The magnetic field will then confine/trap electrons close to the target surface, shown in figure 3. This
will then decrease the depletion in this zone and effectively shrink the dark zone to a much smaller area.

![Schematic of DC Magnetron Sputtering](image)

**Figure 3: Schematic of DC Magnetron Sputtering** [13]

This is the general overview of DC magnetron sputtering, for more information refer to [12-15].

### 2.1.2 Hall Effect

The Hall Effect can be observed when applying a magnetic field perpendicular to the surface of a sample and a current along the length of the sample, parallel to the surface. This creates a force perpendicular to both the magnetic field and the current, which in turn creates a transverse voltage that is perpendicular to both the magnetic field and the current, shown in figure 4. This is because of the transverse force the magnetic
field exerts on the moving charged carriers [16]. The underlying principle behind this transverse force is the Lorentz force.

\[ F_L = q \cdot (v \times B) \]  

(1)

\[ F_y = -q \cdot v_{Dx} \cdot B_z \]  

(2)

Figure 4: Schematic of Forces for Hall Effect [16]

The Lorentz force is most commonly known as the right hand rule, which allows us to determine the direction of the force on a charge carrier based on the direction of the current, more specifically the drift velocity, and the direction of the applied magnetic field. An example for both electrons and holes is shown in figure 5.

Figure 5: Flow-lines of the Charged Carriers [17]
This means that the Hall Effect measurement, in the simplest form, is to apply a current and a magnetic field, then measure the resulting voltage. The Hall Effect measurement can be used to determine a variety of parameters. The first parameter is measured directly and is the Hall voltage. The next parameter is calculated directly from the measured voltage, along with the applied current and voltage, which is the Hall coefficient [16].

\[
V_H = \frac{l_x B_z}{q p t}; \quad V_H = \frac{l_x B_z}{q n t} \tag{3}
\]

\[
R_H = \frac{V_H + t}{I_x + B_z}; \quad R_H = \frac{1}{q p}; \quad R_H = \frac{1}{q n} \tag{4}
\]

To find many of the other material parameters, first the resistivity of the sample must be measured. This is similar in method to the Hall Effect, with the exception that no magnetic field is applied. Once the resistivity is measured then the carrier mobility, carrier concentration and the conductivity type, N or P, can all be calculated from the Hall coefficient and the resistivity. For more information, refer to references [16,17].

2.1.3 Atomic Force Microscopy

The Atomic Force Microscope is designed to measure local properties such as height, friction, and magnetism, with a probe. A sharp probe is brought into close proximity with the sample to be analyzed. The probe is a cantilever with one fixed end and a pyramidal tip located on the underside on the opposite end. The probe is moved in a raster pattern relative to the sample, and measurements are taken in a serial fashion at discreet locations.

Here only “Tapping Mode” will be covered because this was the mode with which the imaging was done. In tapping mode, the probe is driven to the natural resonance frequency using a piezoelectric motor. The tip is then brought close to the
sample surface. In this mode, direct force on the sample surface is not measured. Instead, the force is constructed by adding the short-range repulsive Coulomb and long-range attractive Van der Waals forces, shown in figure 6. The desired set point is maintained by processing the resulting error signal by a proportional-integral-differential (PID) feedback controller that drives the piezoelectric motor to minimize the error signal. For more information, refer to references [18-20].

Figure 6: Left – AFM Tapping Mode. Right – Tapping Mode Forces [18]
2.1.4 Ellipsometry

Ellipsometry is a non-invasive technique to measure the optical properties of a material through the polarization of reflected electromagnetic waves. The electric field of an electromagnetic wave is always orthogonal to the direction of propagation. In the case of Ellipsometry, there are two main classifications for electromagnetic waves, polarized or unpolarized. If the electromagnetic wave consists of random spatial characteristics, frequencies and/or phases, it is un-polarized. If a wave consists of sinusoidal components of one particular direction, frequency and phase, it is polarized. When two orthogonal waves are in-phase, the result will be linearly polarization. If the orthogonal waves are 90° out-of-phase and equal in amplitude, the result will be circular polarization. The most common polarization is elliptical, which results from orthogonal waves of arbitrary amplitude and phase. The three types of polarization are shown in figure 7.

<table>
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<td>$E_x = 1.00$</td>
<td>$E_x = 1.00$</td>
</tr>
<tr>
<td>$E_y = 0.00$</td>
<td>$E_y = 1.00$</td>
<td>$E_y = 1.00$</td>
</tr>
<tr>
<td>$\Delta \phi = 0.00^\circ$</td>
<td>$\Delta \phi = 90.00^\circ$</td>
<td>$\Delta \phi = 45.00^\circ$</td>
</tr>
</tbody>
</table>

Figure 7: Types of Polarization [21]
Maxwell’s equations must remain satisfied when an electromagnetic wave interacts with a material/boundary. Electric fields parallel and perpendicular to the plane of incidence are considered p- and s- polarized. The conditions at the boundary allow solving for different solutions for p- and s- polarized electric fields. Fresnel described the amount of light reflected and transmitted at an interface between materials, shown in figure 8. Thin film and multilayer structures involve multiple interfaces. This gives Fresnel reflection and transmission coefficients at each interface.

Figure 8: Top – Snell’s Law. Bottom – Fresnel’s Law [22]
Ellipsometry is primarily interested in how p- and s- components change upon reflection or transmission in relation to each other. This means that the reference beam is part of the experiment. A known polarized electromagnetic wave is reflected/transmitted from the sample, and the output polarization is measured, shown in figure 9. For more information, refer to references [21-26].

Figure 9: Typical Ellipsometry Setup [23]

2.1.5 X-Ray Diffraction

X-ray diffraction states that crystalline solids produced intense peaks, Bragg peaks, of reflected X-rays at specific wavelengths and incident angles. Bragg diffraction is explained as a crystal with a set of discrete parallel planes separated by a constant distance, d, with atoms periodically spaced. This is true if an X-ray at a specific incident angle will produce a Bragg peak. This happens only if the reflections off the various planes interfered constructively, shown in figure 10. The interference is constructive when the phase shift is a multiple of $2\pi$. 
The Bragg diffraction occurs when the incident X-ray produces electromagnetic radiation with wavelength comparable to the atomic spacing between atoms of the unit cell. These waves are then scattered in a specular fashion by the atoms in the crystal, and undergo constructive interference. Constructive interference is defined as waves remaining in phase with each other since the path length of each wave is equal to the wavelength times an integer multiple. Now it is easy to see the verification that Bragg's law describes the condition for constructive interference from successive crystallographic planes, which are commonly noted in Miller Indices, examples are shown in figure 11. This explains the basic physics principles behind X-ray diffraction, for a deep explanation refer to [29,30].
As stated above the diffraction pattern is obtained by measuring the intensity of scattered waves as a function of the scattering angle. It is important to discuss here the difference between simple cases for explaining the theory and cases of real samples. First, we should look at the simple case that if only two planes of atoms were diffracting, as shown in figures above, then the transition from constructive to destructive interference would be gradual as the angle is varied. However, for the case of a real sample, many atomic planes are present and contribute to the X-ray interaction/interference. This causes very sharp transitions from constructive to deconstructive interference, which gives sharp Gaussian peaks. The spectra will also contain a bremsstrahlung signal, which is a continuous signal that is considered as the background for the X-ray collection.

The collected intensity spectrum will allow the material to be classified based on the location and intensity of the peaks. Each peak will correspond to a specific set of Miller Indices. The combination of these Miller Indices will allow the user to categorize
the material’s unit cell, class of crystal structure, and the Bravais lattice. This is possible due to the fact that only certain combinations of Miller indices are allowed for each type of unit cell. For more information, refer to references [27-30]

2.2 LabView

This section will give a brief description of the software LabView. LabView is a graphical programming language. It was developed by National Instruments. This software allows graphical user interfaces to be developed for various purposes. LabView is a parallel operation language, unlike most sequential operation languages. The figure 12 shows an example of the front interface panel and the back programming panel.

![Example of LabView Front Panel and Back Panel](image)

2.2.1 Front Panel

The front panel is what LabView calls the graphical user interface. This is the screen that users see when the program is running. The front panel allows the user to pass inputs and outputs from the user to the back panel/block diagram. The interface is created by placing visual controls and indicators on the front panel, the user can also place other
visual components to customize the application, this includes various decorations. The user can also develop custom controls and indicators if the standard types do not fit the users need.

Controls are various types in LabView. The first type are buttons, these can be flip, slide, push or dial. The next type is numerator; these can be slide, knob or text entry. The last main type is string; these can be ring, enumerator or text entry. There are various other types of controls, but there are too many to list here.

Indicators come in just as many varieties as controls. The indicators come in the same forms as the controls above with the addition of LED’s, charts and graphs.

The front panel can also be customized in a variety of ways. The text can be changed by color, text size, font type and style. Controls and indicators can be changed by size, color and what parts of the control/indicator is visible.

This is a very brief overview, for more information refer to [31-33].

2.2.2 Back Panel

The back panel, or more commonly the block diagram, is where the user actually constructs the code. The code is a graphical source code, also known as G code. This is what replaces traditional text based code. The code being graphical blocks makes it easier for new users to develop code. The block diagram code uses these graphical blocks to control the front panel controls/indicators. Front panel controls/indicators appear as icon terminals on the block diagram. The back panel uses wires to connect graphical blocks; this is how LabView passes data. The movement of data through the wired paths on the
block diagram determines the execution order of the graphical blocks/functions in the code. This is known as dataflow programming.

This is a very brief overview; for more information refer to [31-33].
CHAPTER 3: DC SPUTTER

This chapter will cover the development of the DC Magnetron Sputter system, including the hardware, the software and the results from successful depositions. The sputter system required extensive repair and testing of all instruments before the upgrade was implemented. The original system was not in working order when purchased. The original control circuitry was analog based and did not perform the necessary steps for a successful deposition. In addition it had only two safety interlocks, which were the water meter and the lid switch. The system relied on the user manually watching the front panel of the instruments, then determining if it was safe to proceed. The vacuum lines were also improperly designed, with no roughing line being present. Lastly, the system’s chassis ground was improperly connected; this made the system unsafe to operate. First in this chapter, the hardware development is covered, which includes the instruments used and the custom circuitry implemented. This is followed by the software development and lastly the results from successful depositions. These results have two purposes. The first is for calibration of the deposition parameters and to calibrate the thickness meter. The second is to examine the quality of the deposited films.

3.1 Hardware

This section will cover the hardware development for the system. It starts by giving a description of the instruments used and their functionality. It will then move to describing the custom circuitry used in the system. A general system diagram showing the connections between different instruments and the chamber is shown in figure 13.
3.1.1 Instruments

In this section, the work done on the instruments in the system will be described.

The table below gives an overview of the model, type and manufacturer.

![Figure 13: Hardware Schematic Diagram of DC Sputter System](image)
Table 1: List of Instruments and Components in Redesigned DC Sputter System

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Model</th>
<th>Function</th>
</tr>
</thead>
<tbody>
<tr>
<td>Advanced Energy</td>
<td>MDX 1K Magnetron</td>
<td>Deposition Gun Source</td>
</tr>
<tr>
<td></td>
<td>Drive</td>
<td></td>
</tr>
<tr>
<td>Sycon</td>
<td>STC-200</td>
<td>Deposition Rate Controller</td>
</tr>
<tr>
<td>Leybold</td>
<td>TMP 1000</td>
<td>Turbo Pump</td>
</tr>
<tr>
<td>Leybold</td>
<td>NT 1000</td>
<td>Turbo Pump Controller</td>
</tr>
<tr>
<td>Granville Phillips</td>
<td>307</td>
<td>Vacuum Measurement Controller</td>
</tr>
<tr>
<td>Granville Phillips</td>
<td>275 &amp; 274</td>
<td>Convectron Gauge &amp; Ion Gauge</td>
</tr>
<tr>
<td>Proteus</td>
<td>100</td>
<td>Water flow switch</td>
</tr>
<tr>
<td>Clippard</td>
<td>ET-3M</td>
<td>Pneumatic Valves</td>
</tr>
<tr>
<td>Kurt Lesker</td>
<td>KF40</td>
<td>Pneumatic Vacuum Valves</td>
</tr>
<tr>
<td>Leybold</td>
<td>D16B</td>
<td>Mechanical Pump</td>
</tr>
</tbody>
</table>

The instruments will not be described in detail in this section. However, if a brief description is need for the reader to understand the functionality and the purpose in the system, refer to the appendix and references [34-42]. The redesigned system is a ground up rebuild from the previous system, a picture of both systems is shown in figure 14.

The instruments and hardware were recalibrated and repaired, this was due to the instruments all needing some type of repair. The mechanical pump had a complete rebuild done, including valves, seals, gaskets and O-rings. All of the pneumatic vacuum valves were also all rebuilt. The cables for the Convectron gauges, Ion gauge and pressure controller were all rewired. The pressure controller also had a full calibration performed. The turbo controller cable was rewired, as well as the seals were checked and the pump was leveled. The deposition controller cable was rewired and the chassis ground was reconfigured. The deposition controller, pressure gauge controller and thickness meter all had communication cables fabricated.
There were several additions to this system that the previous version did not have. The first was the addition of a roughing line. The previous setup used the foreline/turbo line to rough the chamber; this is less effective and hard on the turbo pump. This addition of the roughing line also required adding an additional pneumatic vacuum valve. There was also the addition of the deposition rate controller. This allows for better control and more precise knowledge of the deposited film thickness. The vacuum lines were reconfigured so that the distance from the mechanical pump to the turbo was as short as possible. In addition, the vacuum lines were also standardized to KF 40 connections and size. The last change was reconfiguring the chamber ports to include a roughing port and a port for the thickness monitor.

There was also the addition of several additional safety interlocks. These include three interlocks from the pressure controller; two interlocks are based on the pressure of the chamber and one based on the pressure of the foreline. There was one interlock added based on the turbo reaching normal operation.
3.1.2 Circuitry

This section will cover the custom circuitry that was developed for this system. First, the chips used will be described. Next, the typical connections will be shown. Lastly, the custom printed circuit board will be described.

The two types of chips used are the ULN2803, Darlington array, and the PB134005, relay, these are shown in figure 15. These chips are used for the control of the binary functioning operations for the system. First, the ULN2803 chip is described. The inputs are labeled “B”, example “1B”. The outputs are labeled “C”, example “1C”. Then input/output pairs have same number, example “1B” is paired with “1C”. Next, the PB134005 chip is described. The A1, A2 are the input or Coil. The output is from 11 to 14.
Next the typical configuration for using these chips will be discussed. The ULN2803 is the main chip used on the printed circuit board. First, the connections to the ULN2803 are shown for driving a single device in figure 16. Second, the connections are shown for driving multiple devices, as depicted in figure 17. The wiring of the chip may seem strange to those not familiar with a Darlington transistor array. The Darlington Array chip allows current to flow through the device. While the circuit is completed via the ground pin on the chip.
The printed circuit board controls the pneumatic valves, deposition gun, turbo pump and interlocks. The PCB has two planes for the connecting paths, one on top and the other on the bottom. The circuit diagram and path layout for the PCB is shown in figure 18. The detailed description of the connectivity on this circuit board is provided in the Appendix.
Figure 18: Custom Printed Circuit Board

The printed circuit board has several paths that are laid out on the board. In the figure above the ground path is in Green, the Common (24V) path is in Purple and the Common (5V) path is in Blue. In the figure above the pink is just re-colored because it was green so there is no confusion in what is ground path.

The specific arrangement of connections on the printed circuit board will not be discussed here; instead this is left to the appendix. The detailed information on connections are listed in the Appendix in the same order that they are numbered in figure 18.
3.2 Software

This section will describe the LabView software and the software to hardware interfaces. The functionality and operation will be described first. This will cover the two operational modes, manual and automatic, along with the subroutines in automatic mode. Due to very large number of complex functions developed as well as the ‘tabbed’ approach we used to implement these functions under a single ‘GUI window’, the graphical details (so called LabView back-panel) are not provided in this document. The first reason is that they will appear to be unreasonably small and complex even in a page size figure. Secondly, the ‘tabbed’ approach means that there are a large number of nested ‘back-planes’, which make navigation unreasonably complex and difficult in ‘static’ printed format. As a result, we only provide the general description of the software at the highest abstraction level, as shown in figure 19.
Figure 19: Highest Level Software Flow Chart
3.2.1 Functionality

The software is broken down visually into two major components. The first is the system diagram. This displays the current state of the system. This is depicted in the figure 20.

![Software User Interface with System Diagram Circled](image)

The vacuum lines will be green if under vacuum, or white if at atmosphere. The pneumatic valves, Turbo pump and mechanical pump will be grey if closed/off, or they will be green if open/on. The deposition gun will be grey if off, or will be orange if on. The working gas line will be white if no gas is flowing, or it will be orange if gas is flowing. The vent gas line will be white if no gas is flowing, or will be blue if gas is flowing. The figure 21 shows a depiction of just the system diagram.
The tabs to the left, shown in figure 22, of the system diagram are the control tabs. These tabs allow the user to control operation of the system.
Now each tab will be described. This will include what options the user can select and what that option does. The first tab that will be looked at is the “User Mode”, this is the tab users will most commonly use. The user mode tab is shown in figure 23.
This tab has four main areas. This includes the operation selection, deposition gun levels, termination of deposition criteria and number of times to purge. First, the operation selection will be discussed. This has six selectable options: Rough Chamber Down, Turbo Pump On, Turbo Pump Chamber Down, Run Recipe, Vent to Atmosphere, Purge and Abort (not shown in figure above).
Rough Chamber Down – If this is selected the system will vacuum the chamber down to the user set point. This button will only be visible if the chamber pressure is higher than the set point.

Turbo Pump On – If this is selected the system will open the fore-line, start the turbo pump and allow it to reach normal operation. The gate valve will remain shut during this process. This will only be visible if the turbo pump is not already on.

Turbo Pump Chamber Down – If this is selected the system will run the “Rough Chamber Down” and “Turbo Pump On”, then open the gate valve. This will only be visible if the turbo is off or the gate valve is closed or the chamber is above base pressure.

Run Recipe – If this is selected the system will begin a full run, which includes all the steps necessary to deposit target material. The deposition will be terminated once the desired thickness or time has been met. The system will then park under vacuum.

Vent to Atmosphere – If this is selected the system will use the vent gas to raise the chamber pressure to atmosphere. This is only visible if the chamber is under vacuum.

Purge - If this is selected the system will use the vent gas to purge the chamber for the user set time. This is only visible if the chamber is under vacuum.

Abort – If this is selected the system will abort the currently selected routine and return the system to a safe state. This is only visible if a routine is in process.

The deposition gun levels control allows the user to set the voltage level and the ramp rate. An indicator shows the current valve in relation to the full scale for each control. The user has the ability to change the voltage level during a run, so if the user is
unsure of what level to use start with a lower value and increase. It is important that the user see a stable plasma is formed.

There are two options for specifying the deposition amount, a time limit or setting a thickness limit. For the time limit option, the user sets the time duration that the deposition will take place. When the deposition process starts a timer will start, when the time reaches the limit the deposition is terminated. For thickness limit option, the user sets a desired thickness. When the deposition process starts, the deposited thickness is monitored via the thickness monitor. When the deposited thickness reaches the limit the deposition is terminated.

The second tab is the “User Mode – Setting”, this allows the user to set the set points used in the routines from the “User Mode” tab, shown in figure 24.
The controls on this page allow the user to set the values that are used as the set points in the subroutines. These set points are the values to which the inputs to the software must reach to satisfy the ending criteria, on a particular subroutines. The set points are limited in the value ranges that can be entered. The set-point controls that are set on the “Configuration Mode – Settings” set the upper value that can be set on this tab.
page. Only advanced users can set controls on the “Configuration Mode – Settings”, this is to ensure the system will not be harmed.

The first two tabs are accessible to normal users. The last two tabs are only accessible to advanced users. To ensure this these last two tabs require a password, shown in figure 25. This is because the user can potentially harm the system in these modes.

![Software Password Prompt](image)

**Figure 25: Software Password Prompt**

The password only needs entered once per session. Once the password has been correctly entered, the user is free to go to any tab and not re-enter the password. If the software is closed or restarted the password must be re-entered.

The third tab is the “Manual Mode”, shown in figure 26, this allows the user to manually operate all of the system components. In this mode, the user has the ability to
potentially harm the system; however there are still both software and hardware interlocks in place to prevent catastrophic system failure.

![The User Mode Tab](image)

**Figure 26: The User Mode Tab**

Each button either turns on/opens or turns off/closes the corresponding component. The user must set the Voltage level and Ramp level on this page if using
deposition gun in manual mode. The values from the “User Mode” are not used, when on this page.

The fourth tab is “Configuration Mode – Settings”, this allows the user to set the upper limits for the system, shown in figure 27.
The set points on this tab allow the user to control what values are entered on the “User Mode – Settings” tab. The values entered here will be set as the upper limit, for pressure set points. While the value entered for the wait, time will be set as the lower limit. This ensures that the basic user cannot enter values that are incorrect for they system. Once the software is started, the only way to stop the software is with the stop button. This is located in figure 28.

![Figure 28: Shows the Location for the Button to Stop the Software](image)

Click on the button and hold for a second, the software will then stop or display a pop-up box telling the user that the system has went into cool down. If the system is in cool down the software will close once cool down is completed.
3.2.2 Software to Hardware Integration

The software controls integration to the hardware of the system is discussed in this section. In the previous section, the functionality/operation of the code was described. However, the software needs a way to control the system’s hardware in order to operate as desired. This was accomplished through a variety of interfaces. This will be discussed by breaking down the system into interface categories. The first category is data acquisition unit digital outputs, the second category is data acquisition unit analog outputs and the third is RS232 communication.

First, the interfaces that are connected through the data acquisition unit digital outputs will be discussed. The digital outputs control all of the binary operating instruments/components of the system. This is done through several different methods. The first method is through using the digital outputs to act as the gate for the uln2803 chips, this allows it to turn on and off when a digital output is either high or low. When the digital output is high the uln2803 connects its’ output to common, this allows it to supply 24 volts to the clippard pneumatic valves. When the clippard valve has 24 volts applied it then allows 80 psi flow of nitrogen to propagate. This flow is then applied to the Kurt Lesker pneumatic vacuum valve. When 80 psi is applied to the Kurt Lesker valve it causes the valve to open. This allows for the vacuum lines to be turned on/open or off/closed. This method is used for the roughing valve, foreline valve and the gate valve. This same method is used for the gas flow lines, with the only difference being the Kurt Lesker valves are MKS gas flow valves. This method is used for the working gas and the vent gas. The next two items in this category are controlled in a different method.
It starts the same with either a digital high or low being applied to an uln2803. This time the uln2803 chip is connect to a 5 volt source. When a digital high is applied to the uln2803 this then causes 5 volts to be applied to the input of the PB134005 Chip. This allows the output of the PB134005 Chip to connect to each other, causing the chip to act as a closed circuit. The turbo pump controller and the deposition gun power supply are turned on and off by the contacts between two pins being either a closed or open circuit. The last method is the simplest. This is a straight connect between the data acquisition unit digital input/output to several interlock relays. The relay status is read directly from the data acquisition unit. This is used for the water flow meter, the lid switch, the turbo failure and the turbo steady state.

Next, the data acquisition unit analog outputs will be discussed. This method controls fewer items than the previous digital output methods. The analog outputs are used by only one instrument, the deposition gun power supply. The connection is simple. The outputs on the data acquisition unit are connected directly to the inputs on the gun power supply. This allows for control of the power/voltage level and the ramp rate. The only analog input is also from the deposition gun power supply, it is the power/voltage level monitoring.

The last method is RS232 communication. This method controls the pressure controller and the thickness monitor controller. These two instruments take custom RS232 dependent to each device. The RS232 are used for both control of the instruments function, as well as reading off data from the instruments. This is how the pressure is read
off for the foreline and the chamber, also how the thickness of the deposition is read
during deposition.

3.3 Measurements

The measurements performed on these films were to verify the system produced a
high quality film. In addition, the measurements were used to look at the repeatability and
reproducibility of the system, from deposition to deposition. These films were measured
by various techniques including Ellipsometry, AFM and XRD.

3.3.1 Calibration and Testing of System

The calibration/testing process took place in three distinct phases, each phase was
necessary to complete before the next phase could be proceeded too. The first phase was
the calibration/testing of the system components/instruments. The second phase was the
calibration/testing for proper system parameters. The third phase was the
calibration/testing of the thickness meter.

The first phase of calibration/testing will be described now. This phase took the
longest of the three phases, because it did not only require calibration/testing but also
repair. The order of steps described is also the order in which these steps occurred. First,
the Leybold dual vane rotary pump was tested for base level vacuum. The vacuum could
only achieve 5 torr; to fix this a full rebuild was done on the pump. This allowed the
pump to reach the proper base level of 1 mtorr. This step was required to test the turbo
pump, because the foreline pressure must be lower than 50 mtorr. The turbo pump
required breaking in, because it was not used for an extended amount of time. The break
in process requires the turbo pump to be turned and allowed to speed up to a given
percentage of the top rate, then turned off and allowed to spin down. The steps are 10%, 25%, 40%, 60%, 80% and then 100%. Once the vacuum could reach the correct vacuum level, the pressure controller and pressure sensors were calibrated. The pressure controller controls two different types of sensors, convectron and ion gauges. The convectron gauges must each be calibrated to read the proper pressure, this is done by calibrating the gauge at atmosphere, 760 torr, and at the base for the gauge, 1E-4 torr. Then the ion gauge is calibrated differently, the emission current is monitored and adjusted so that it matches the expected emission current output. Next, the vacuum valves were all checked for proper seal leakage. Several of the valves were rebuilt because they exceeded the recommended leak rate 1E-8 mbar*l/s. At this point the vacuum system is calibrated. Next, the Deposition gun power supply was tested. The voltage and current were checked to ensure that the specified output was reached within 5%. The final items were the water flow meter and manual gas flow regulators. This completed the testing and calibration of the components.

From this point, all of the system components operate correctly. The parameters for deposition must be determined for proper operation. This set was the shortest of the three. This step was aided by research, [46, 47]. The research gave good starting values, from this a set of simple DOE experiments were used to determine the good ranges for parameters for this system. It was determined that the most stable plasma occurred when the gas flow caused the pressure to stabilize at 1-3 mtorr. This is well within the suggested range of 0.1 – 50 mtorr. The deposition controller has three operating modes, voltage control, current control or power control. It was determined that the most stable
plasma, with fewest arcing events, occurred in the voltage control mode. While the plasma could be maintained anywhere between 250 – 600 volts, above 600 volts arcing events were observed. The target to substrate spacing was set to the minimum allowable distance. This distance is based on the geometrical path that the lid must travel in order to close. Lastly the issue of film contaminates was examined, since this is an issue of base pressure and operational parameters. It was found that a base pressure of at least 1E-6 torr must be reached for 30 minutes and the system must always be backfilled with pure nitrogen when raising pressure. This kept oxygen and water vapor migration into the chamber walls to a minimum, while also allowing adequate time for diffusion out of the chamber walls [48,49].

The last phase is the calibration of the thickness meter. The thickness meter calibration required a series of deposition then measurement cycles. First, a deposition of 1000 Å was done, this is the recommended amount of deposition for proper calibration. After the deposition, the film thickness was measured via Dektak. Then a comparison from the measured thickness and the thickness recorded from the thickness meter a correction factor could be calculated. This correction factor was then entered into the thickness meter. Then the series was repeated. This continued until the thickness meter readout matched the measured thickness from the Dektak.

3.3.2 Ellipsometry

There are a total of two samples analyzed via Ellipsometry. The two samples came from two different deposition runs. The second sample tested is also the same
sample that was tested via the AFM, in chapter 3.3.3, and was the called the fast deposition sample.

It is important to first set up the model that will be used to fit to the experimental data. The model that is used is a three-layer model, shown in figure 29. The bottom layer is the substrate, the middle layer is the thin film deposited and the top layer is the roughness layer [50, 51]. The substrate layer is crystalline silicon, this is because the film is deposited on a silicon wafer. This substrate layer is also a reference file instead of a dispersion file. The middle layer is an aluminum dispersion file. The equations for this dispersion file are drude equations. This is because drude equations describe the dispersion of light in a metal. The top layer is two files, one is the same aluminum file as the middle layer and the other is a void reference file. The void file mimics air. This is why the top layer is 50% void and 50% aluminum, this replicates a rough surface. This gives the representation of a RMS roughness layer. The spectra was collected under the same conditions for each sample. The range was 1.50 – 4.70 eV, this is where the system produces the most reliable data. The increment was 0.025 eV.

![Figure 29: Ellipsometry Model for Aluminum Film](image)

The data collected is summarized in the table below. The measurements were confirmed using the Dektak. The confirmation could only be done on spots that were
taken around where the magnet that was used to hold the sample. This was because the magnet acted like a mask, allowing the silicon surface to be protected during deposition.

### Table 2: Overview of Ellipsometry Data

<table>
<thead>
<tr>
<th>Sample</th>
<th>Location</th>
<th>Closeness of fit</th>
<th>Thickness</th>
<th>Roughness</th>
<th>Percent Difference vs Dektak</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>Spot 1</td>
<td>0.348402</td>
<td>103.806 nm</td>
<td>16.698 nm</td>
<td>NA</td>
</tr>
<tr>
<td>2</td>
<td>Spot 2</td>
<td>0.347426</td>
<td>100.170 nm</td>
<td>14.528 nm</td>
<td>NA</td>
</tr>
<tr>
<td>2</td>
<td>Hole Spot 1</td>
<td>0.332536</td>
<td>120.000 nm</td>
<td>12.871 nm</td>
<td>14.35 %</td>
</tr>
<tr>
<td>2</td>
<td>Hole Spot 2</td>
<td>0.415861</td>
<td>140.000 nm</td>
<td>11.108 nm</td>
<td>8.03 %</td>
</tr>
<tr>
<td>1</td>
<td>Spot 1</td>
<td>0.278354</td>
<td>117.876 nm</td>
<td>17.392 nm</td>
<td>NA</td>
</tr>
</tbody>
</table>

It can be seen that there was an increased thickness around the magnet. This was expected because of the enhanced magnetic field at this local point. The increase in thickness around the magnet was first seen visually. This was hypothesized to be an increase in thickness, which was then later confirmed by the ellipsometry and Dektak measurements.

3.3.3 Atomic Forces Microscopy

There are three samples analyzed via AFM. The first sample is a bare silicon wafer, which is single side polished. This was the substrate used for all depositions. It is important to analyze this silicon wafer so it can be used as a reference. This sample had two separate locations investigated with analysis done at each location. Then the averaged analyzed data taken from the two locations was used as the reference roughness. First, the silicon wafer spot 1 will be examined, shown in figure 34 and 35.
Figure 30: Reference Sample Spot 1– Left – 3-D plot. Right – 2-D plot.

Figure 31: Reference Sample Spot 2– Left – 3-D Plot. Right – 2-D Plot.

Table 3: Reference Sample - Analyzed Parameters for Silicon Wafer Spot 1 and Spot 2

<table>
<thead>
<tr>
<th>Scan Size (1.00 × 1.00 µm)</th>
<th>Spot 1</th>
<th>Spot 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average value</td>
<td>-0.022 nm</td>
<td>0.008 nm</td>
</tr>
<tr>
<td>Minimum Height</td>
<td>-0.585 nm</td>
<td>-0.506 nm</td>
</tr>
<tr>
<td>Maximum Height</td>
<td>0.670 nm</td>
<td>0.586 nm</td>
</tr>
<tr>
<td>Median Value</td>
<td>-0.024 nm</td>
<td>0.006 nm</td>
</tr>
<tr>
<td>Roughness Average Ra</td>
<td>0.099 nm</td>
<td>0.097 nm</td>
</tr>
<tr>
<td>Root Mean Square Roughness Rq</td>
<td>0.125 nm</td>
<td>0.123 nm</td>
</tr>
<tr>
<td>Root Mean Square Roughness (grain-wise) Rq</td>
<td>0.125 nm</td>
<td>0.123 nm</td>
</tr>
<tr>
<td>Skew</td>
<td>0.142</td>
<td>0.139</td>
</tr>
<tr>
<td>Kurtosis</td>
<td>0.276</td>
<td>0.231</td>
</tr>
</tbody>
</table>
**Reference sample:** It is seen from figures 30 and 31 and the data in table 3 that the silicon wafer has a low surface roughness. This was expected since this was an industry-fabricated wafer. To verify that this low roughness was common to the entire wafer a second spot is chosen and analyzed. Figure 31 and the data in the table above confirm that this second spot showed the same low surface roughness. This verifies the data found at spot 1. Only two spots were used because of the general expected standards of industry wafers, if this wafer came from other sources additional measurements at multiple spots would be needed to state the same conclusion. The table below gives an average of the two spots and will be used as the reference data for the silicon wafer/substrate. This reference value will be subtracted from the roughness of the sputtered films in order to better characterize the roughness from the deposited films.

<table>
<thead>
<tr>
<th>Scan Size</th>
<th>1.00 × 1.00 µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Roughness Average Ra</td>
<td>0.098 nm</td>
</tr>
<tr>
<td>Root Mean Square Roughness Rq</td>
<td>0.124 nm</td>
</tr>
<tr>
<td>Root Mean Square Roughness (grain-wise) Rq</td>
<td>0.124 nm</td>
</tr>
</tbody>
</table>

The first deposited sample, sample 1, is an aluminum deposition with a slow deposition/growth rate. This sample was grown at a rate of approximately 0.3 – 0.6 Å/s. Similar to the reference sample, this Al deposited sample will have two spots analyzed then the data averaged.
Figure 32: Sample 1 Spot 1 - Left – 3-D Plot. Right – 2-D Plot.

Figure 33: Sample 1 Spot 2 - Left – 3-D Plot. Right – 2-D Plot.

Table 5: Sample 1 - Analyzed Parameters for AL Film Slow Deposition Spot 1 and Spot 2

<table>
<thead>
<tr>
<th></th>
<th>Spot 1</th>
<th>Spot 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scan Size (9.000 × 9.000 μm)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average Value</td>
<td>-0.02 nm</td>
<td>-0.03 nm</td>
</tr>
<tr>
<td>Minimum Height</td>
<td>-22.09 nm</td>
<td>-36.41 nm</td>
</tr>
<tr>
<td>Maximum Height</td>
<td>20.86 nm</td>
<td>55.24 nm</td>
</tr>
<tr>
<td>Median Value</td>
<td>0.09 nm</td>
<td>-0.04 nm</td>
</tr>
<tr>
<td>Roughness Average Ra</td>
<td>4.15 nm</td>
<td>5.37 nm</td>
</tr>
<tr>
<td>Root Mean Square Roughness Rq</td>
<td>5.19 nm</td>
<td>6.99 nm</td>
</tr>
<tr>
<td>Root Mean Square Roughness (grain-wise) Rq</td>
<td>5.19 nm</td>
<td>6.99 nm</td>
</tr>
<tr>
<td>Skew</td>
<td>-0.072</td>
<td>0.022</td>
</tr>
<tr>
<td>Kurtosis</td>
<td>-0.0923</td>
<td>1.96</td>
</tr>
</tbody>
</table>
Sample 1: It is seen from figures 32 and 33 and table 5 that the deposited film has a significantly higher roughness than the reference. While the roughness is larger than the reference this roughness is low compared to other sources of AL sputtered films [52]. The data for spot 2 is similar to the data found at spot 1. There was a 1.2 nm difference in the roughness observed between the two spots. This not only shows that the film has a low surface roughness but also shows good uniformity across the sample. The difference in roughness between spot 1 and spot 2 is a 22% difference, comparatively. This large percent difference is acceptable because of the small roughness value.

The table below gives an average of the two spots and will be compared to the reference data for the silicon wafer/substrate. The total increase in roughness, when compared to the reference sample, is 4.662 nm and the RMS roughness is increased 5.966. This amount of total roughness is very good for DC Magnetron Sputtering [52].

<table>
<thead>
<tr>
<th>Table 6: Sample 1 - Averaged Analyzed Parameters for AL Film Slow Deposition</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Scan Size</strong></td>
</tr>
<tr>
<td>Roughness Average Ra</td>
</tr>
<tr>
<td>Root Mean Square Roughness Rq</td>
</tr>
<tr>
<td>Root Mean Square Roughness (grain-wise) Rq</td>
</tr>
</tbody>
</table>

The second sample, sample 2, that was analyzed was deposited at a faster deposition rate. This sample’s deposition rate was 1.5 – 2.0 Å/s. It is important to note here that this deposition rate is not the fastest that could be obtained with the system. This deposition was preformed after full calibration of the thickness meter had taken place, because of this data was collected for both 5.0 x 5.0 µm scans, figures 38 and 39, and 1.0
x 1.0 µm scans, figures 40 and 41. This sample will have two spots analyzed, with the two scans sizes taken at both spots.

Figure 34: Sample 2 Spot 1 5.0 µm Scan - Left – 3-D Plot. Right – 2-D Plot.

Figure 35: Sample 2 Spot 1 1.0 µm Scan - Left – 3-D Plot. Right – 2-D Plot.

Figure 36: Sample 2 Spot 2 5.0 µm Scan - Left – 3-D Plot. Right – 2-D Plot.
Table 7: Sample 2 - Analyzed Parameters for AL Film Fast Deposition Spot 1 and Spot 2

<table>
<thead>
<tr>
<th>Scan Size</th>
<th>5.0 × 5.0 µm</th>
<th>1.0 × 1.0 µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spot Location</td>
<td>Spot 1</td>
<td>Spot 2</td>
</tr>
<tr>
<td>Average Value</td>
<td>-0.3 nm</td>
<td>-0.02 nm</td>
</tr>
<tr>
<td>Minimum Height</td>
<td>-27.1 nm</td>
<td>-25.52 nm</td>
</tr>
<tr>
<td>Maximum Height</td>
<td>81.4 nm</td>
<td>68.75 nm</td>
</tr>
<tr>
<td>Median Value</td>
<td>-2.8 nm</td>
<td>-2.48 nm</td>
</tr>
<tr>
<td>Roughness Average Ra</td>
<td>9.5 nm</td>
<td>8.77 nm</td>
</tr>
<tr>
<td>Root Mean Square Roughness Rq</td>
<td>12.4 nm</td>
<td>11.25 nm</td>
</tr>
<tr>
<td>Root Mean Square Roughness (grain-wise) Rq</td>
<td>12.4 nm</td>
<td>11.25 nm</td>
</tr>
<tr>
<td>Skew</td>
<td>1.24</td>
<td>1.1</td>
</tr>
<tr>
<td>Kurtosis</td>
<td>2.53</td>
<td>1.65</td>
</tr>
</tbody>
</table>

Sample 2: It is seen from the previous four figures and the data in the table above that the data for both scan sizes at spot 1 and spot 2 are in close agreement. There is a 1.5 – 2 nm difference in the roughness calculations between the two scan sizes at spot 1 and a 0.2 – 0.5 nm difference at spot 2. This gives us a 15% difference at spot 1 and a 15% difference at spot 2, comparatively. Just as in sample 1, this is an acceptable percentage difference because of
the small roughness values. It can be seen in the 1.0 µm scans of sample 2 that the grain size is much larger than what was seen in sample 1. This is expected due to the faster deposition rate. The data for spot 2 showed slightly more uniform gain size than what was seen at spot 1. However, it still showed a large variation in grain sizes. Just as in Sample 1, the deposition has good uniformity across the sample. This also confirms that the surface roughness is a function of deposition rate, this verifies the results found in [52].

| Table 8: Sample 2 - Averaged Analyzed Parameters for AL Film Fast Deposition |
|-----------------------------|------------------|------------------|
| Scan Size                  | 5.0 x 5.0 µm     | 1.0 x 1.0 µm     |
| Roughness Average Ra       | 9.14 nm          | 8.51 nm          |
| Root Mean Square Roughness Rq | 11.83 nm       | 11.17 nm         |
| Root Mean Square Roughness (grain-wise) Rq | 11.83 nm  | 11.17 nm         |

The table above gives an average of the two spots and will be used compared to the reference data for the silicon wafer/substrate. The total increase in roughness is 9.042 nm and the RMS roughness is increased 11.706 nm. The increase in the roughness between sample 1 compared to sample 2 is roughly doubled. This large percent difference is expected because of the higher deposition rate used in the deposition of sample 2.

The measurements, for sample 1 and sample 2, from Ellipsometry, AFM and Dektak all closely agree with each other. The differences that are seen between the roughness measurements in the AFM and Ellipsometry are most likely due to two factors. The first factor is that ellipsometry averages over a much larger spot size than the AFM.
The AFM averages over several microns, where ellipsometry averages over several hundred. If there are scratches or other surface abnormalities, this can skew the roughness values found by ellipsometry. The second factor that could affect the roughness value is the method in which the ellipsometry software solves for thickness values. The software tries pseudo-random values as initial starting points then tries to force the model parameters to fit the measurement data. The software will use any value that provides a closer fit and the software only reports what the best fit is. Therefore, this means that the final values provided by the ellipsometry software may not be the most correct values but what provides the best fit.

3.3.4 X-ray Diffraction

There were two samples that were tested. These samples came from two separate depositions. This was done to check the repeatability from deposition to deposition. The samples were tested several weeks after deposition, shown in figures 38 and 39. Due to the fact that this was a sputtered deposition the spectra shows the characteristics of an amorphous Aluminum layer. The largest peaks are from the substrate, the explanation for this change in penetration depth can be seen in [53].
It can be seen from the two previous figures that the two depositions match each other almost exactly. This shows that the system operates very uniform from deposition to deposition. This is the goal for this system. The full analysis of a deposited film is left for later. This is because the system will be used to deposit various materials and the deposition parameters will have to be optimized for that specific material.
CHAPTER 4: HALL EFFECT

This chapter will cover the development of the Hall Effect system, which will include sections discussing the hardware, the software and the results. This system required extensive repair and testing of the instruments used. The repairs will be mentioned only briefly, as in they do not define the development process. This chapter will focus on the two main processes necessary to the system development in addition to the results. The first section will cover the hardware development, which includes the instruments used and the custom test bed. The second section is the software development. Then lastly, the results from manufactures suggested resistor squares and the comparison between NIST’s test results and our system’s test results.

4.1 Hardware

The section will cover the hardware development for the system. It will start by giving a description of the instruments used and their functionality. It will then move to describing the custom test bed used in the system.

4.1.1 Instruments

In this section a list of the instruments used in the system will be given. The work done to these instruments will be briefly discussed. The table below gives an overview of the model, type and manufacturer.
Table 9: Summary of Instruments in the Hall Effect System

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Model</th>
<th>Function</th>
</tr>
</thead>
<tbody>
<tr>
<td>Keithley</td>
<td>236</td>
<td>Source Measure Unit</td>
</tr>
<tr>
<td>Keithley</td>
<td>2000</td>
<td>Digital Multimeter</td>
</tr>
<tr>
<td>Keithley</td>
<td>7001</td>
<td>Switching Mainframe</td>
</tr>
<tr>
<td>Keithley</td>
<td>7065</td>
<td>Hall Effect Card</td>
</tr>
<tr>
<td>Keithley</td>
<td>487</td>
<td>Picoammeter</td>
</tr>
<tr>
<td>LakeShore</td>
<td>455</td>
<td>Gauss Meter</td>
</tr>
<tr>
<td>Agilent</td>
<td>E3634</td>
<td>DC Power Supply</td>
</tr>
</tbody>
</table>

A description of the instruments will be given in the appendix. If a more detailed account of each instrument is required follow the reference for that instrument. The system diagram is shown in figure 40. This diagram shows how the instruments are connected together, how the test bed is connected to the instruments and how the instruments connect to the computer. If it is not immediately clear, the Hall Effect card is the central hub. This card will be described in further detail in the next section.
The system had all of the cables, triax, BNC and banana, custom made, while several of the cables were not standard and required a custom design and fabrication, these will be discussed in later sections. The system is depicted in figure 41. This picture shows everything but the water chiller which is required to cool the electromagnet.

Figure 40: Hardware Schematic of Hall Effect System
4.1.2 Connections to 7001 Hall Effect Card

This section will cover the connections that are made to the Hall Effect card. This is the general overview of how the instruments in the system are connected, shown in figure 50. The central unit to this system is the Hall Effect card, which integrates the SMU, DMM, Picoammeter and test bed.

The SMU is connected to the current source input, two-lug triax, on the Hall Effect card. In this system, the SMU is used in place of a programmable current source. The SMU triax output connection had to be modified to work with the Hall Effect card. The inter-shield on an SMU triax is guard, while the inter-shield on a Current Source triax is force low, because of this the two instruments cannot be freely interchanged. In order to make this compatible, a custom connector was fabricated for the SMU so that the inter-shield could be connected to force low.
The DMM is connected to the screw terminal connector. This allows the DMM to monitor the voltage, when appropriate cross-point is closed. The force high from the DMM is connected to the screw terminal force high terminal. The force low from the DMM is connected to the screw terminal force low and ground.

The Picoammeter is connected to the current monitoring BNC connector. A custom BNC to triax cable was made to allow the picoammeter to connect to the Hall Effect card.

A diagram showing a detailed depiction of the connections made between the Hall Effect card and the other instruments is shown in figure 42.

![Diagram of Connections to the Hall Effect Card](image)

**Figure 42: Schematic of Connections to the Hall Effect Card**

4.1.3 Connections to Test Bed

The test bed is connected through four triax connectors, shown in either figure 42 or 43. One sample triax connector from the Hall Effect card is connected directly to one
triax connector on the test bed. This triax connection on the test bed is connected to one tungsten probe. This allows the Hall Effect card to switch configuration of which probes the current is applied between and which probes the voltage is measured between.

On the test bed the triax connector is a triax jack. The backside of the jack connector has three soldering locations, one of the center connector, one for the inter-shield and one to the body for ground. In this setup, the center connector is soldered to a small insulated flexible wire. The other end of the wire is soldered to a pogo pin receptacle. The inside diameter of the pogo pin connector is the same as the outside of the tungsten probe. Then the tungsten probe is inserted into the pogo pin.

Figure 43: Picture of the Test Bed
4.2 Software

This section will describe the software. The functionality and operation will be described first. If additional information is required to understand the instrument commands then refer to the instrument references, this will provide in-depth explanations for each command.

4.2.1 Functionality

The software is visually set up with one section of tabs, each tab has a general function for the system. This makes it easier for the user to run the system. The tabs are arranged out so that the user moves from the first tab to each successive tab in order to set up the system correctly.

The first tab is the “System Settings”, this tab contains the controls for many system setting or configuration options, shown in figure 4.4. First the GPIB, General Purpose Interface Bus, addresses are given for each instrument. The address entered in the controls on this block must match the address saved in the instrument, as well each address must be unique. This tab also allows the user to bring up a pop-up window of the configuration page for each instrument. This block is not commonly used, because the system runs an automatic configuration of each instrument when the software is turn on. This block is only used if an instrument is turned off after the software is started or if the instrument is malfunctioning. This tab also controls the meter configuration. This block differs from the instrument reset in the fact that it is user definable options. These option do not change the functionallity of the instrument. This tab is set-up to serve as the configurations for the system.
The initial configuration is a subroutine that runs during the first cycle of the main routine, shown in figure 45. This ensures that the instruments are properly set, thus eliminating any improper functions set by users on the instruments front interface.
The second tab is the “Test Set Up” this, shown in figure 46, controls the parameters that must be set for the software to run a successful test. This tab has several features of the code that are controlled from here.

First is to define the material parameters. This is where the user enters the thickness of the sample. This thickness is needed for when the calculations are done after the measurements. It also defines the stabilization threshold. This is the threshold value that the delta of the voltage measurement must reach for the software to record that voltage during a measurement. This is done to ensure the measurement taken is once the system has achieved steady state, and not during the transient stage of the instruments.

This tab also allows the user to set the Hall Effect card for one of two impedance ranges. There is a low resistance mode and a high resistance mode, for further details on the ranges these modes support refer to [57]. The source parameters are also set on this tab. This allows the user to select one of three source/excitation options, either optical, electrical or both optical and electrical at the same time. It also allows the current level to be set. It allows the user to set parameters for the magnetic level and calibration for the gauss meter. Lastly, this is where the user selects the destination for the file to be saved at and the root name for the file. This root name will have the time and date appended, so that it is easier to track when an experiment was done. It also allows the user to select what type of file format, either excel, word, plain text or matlab.
The third tab is the “Run-Time Test Controls”, this tab has the controls needed at the time the test is started, shown in figure 47. It also displays the results from the current test. First, the measurement start control is on this tab. It also contains the selection for what type of test to perform. The user can select one of three measurements to run, either ohmic test, resistivity test or Hall Coefficient test. The user can select either a single run or a sweep of runs. If sweep is selected it allows the user to specify the number of runs to perform. There are also controls to clear the saved data and to clear to Hall Effect card.
As well the abort control, this control is only visible once the run has started and will abort the current run if pressed. This tab also displays the data from the current run in progress or the total data from a sweep. The data is updated for either resistivity or Hall Effect, this is determined by which is currently selected. Last, the white square in the bottom left corner, is where the current test bed configuration is displayed.

Figure 47: Run-Time Test Controls Tab Page

Resistivity Routine

The resistivity routine is the subroutine that measures the resistivity of the sample. This routine is considered the single measurement, since it gives a single resistivity value when the routine is over. This sample measurement orientation steps is shown in figure 48.
Figure 48: Resistivity Test Probe Orientations for each Step
**Resistivity Sweep**

This subroutine is the same as the Resistivity subroutine except it is run for a specified number of times. The results are stored in arrays where the new data is appended to the array.

**Hall Coefficient**

This routine is the subroutine that measures the Hall Coefficient of the sample. This routine is considered the single measurement, since it gives a single Hall coefficient value when the routine is over. This sample measurement orientation steps is shown in figure 49.

**Hall Coefficient Sweep**

This is the same as the Hall Coefficient routine, with the exception that the value of the magnetic field is increased by the user defined step size ever iteration. The results are stored in arrays where the new data is appended to the array.
Figure 49: Hall Coefficient Test Probe Orientations for each Step
Ohmic Sweep

The ohmic sweep routine is the subroutine that measures the current and voltage then calculates the resistance. This routine uses the step 1 pin configuration from the resistivity routine. The current is increased by the step value every iteration of the loop.

The next tab is the Calculations, shown in figure 50, it displays the results from the calculations ran on the date from either the Resistivity or Hall Effect test. It has one block that displays the calculations for a sweep and a separate block for a single run. It has a control to save the data to the user defined file location.

![Figure 50: Calculations Tab Page](image)
This final tab is the Graphs, it displays the calculated data in the form of a graph rather than in form of arrays, shown in figure 51. It has a control located at the top to allow the user to display Resistivity, Hall Coefficient or Ohmic sweep data.

![Graph Tab Page](image)

**Figure 51: Graph Tab Page**

### 4.3 Results

The measurements performed were to verify the system was properly functioning. In addition, the measurements were used to look at the repeatability and reproducibility. This testing was suggested from the senior testing engineer from Keithley. The calibration methods were developed in accordance with the manuals for each instrument. This ensures that the calibration meets manufactures standards.
4.3.1 Test Bed

This section will contain the results from the testing of the test bed connections. First, the results from the first test bed will be shown. This first test bed fabricated was not functional. The probes were gold plated and soldered to in order to make ohmic contacts. However, the connections to the tungsten probes were not good/stable ohmic contacts. Several redesigns were done until a function test bed was developed. The partial gold plated tungsten probes are shown in figure 52.

Figure 52: Gold Plated Tungsten Probe

Table 10: Overview of the Data from First Attempt at Fabricating Test Bed Connections

<table>
<thead>
<tr>
<th>Probe</th>
<th>Probe Resistance + gold plating</th>
<th>Probe + wire</th>
<th>Probe + wire + triax connector</th>
<th>Probe + wire + Triax + holder</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.57 Ω</td>
<td>0.8 – 30+ Ω</td>
<td>0.9 – 30+ Ω</td>
<td>0.9 – 50+ Ω</td>
</tr>
<tr>
<td>2</td>
<td>0.43 Ω</td>
<td>2.3 – 30+ Ω</td>
<td>2.5 – 30+ Ω</td>
<td>2.5 – 50+ Ω</td>
</tr>
<tr>
<td>3</td>
<td>0.72 Ω</td>
<td>1.1 – 40+ Ω</td>
<td>1.4 – 40+ Ω</td>
<td>1.4 – 50+ Ω</td>
</tr>
<tr>
<td>4</td>
<td>0.32 Ω</td>
<td>1.0 – 30+ Ω</td>
<td>1.3 – 30+ Ω</td>
<td>1.3 – 50+ Ω</td>
</tr>
</tbody>
</table>
It can be seen from the table above that the test bed connections were very unstable. Unfortunately, this was not known when the probes were first made. This was only found out while testing, during development.

The second attempt produced high quality connections. The table below shows the results. The figure 53 shows the new connections that are on the test bed and the new longer tungsten probes.

**Figure 53: Left - New Connections on Test Bed. Right- New Tungsten Probes**

**Table 11: Overview of the Data from Second Attempt at Fabricating Test Bed Connections**

<table>
<thead>
<tr>
<th>Probe</th>
<th>Probe Resistance</th>
<th>Probe + pogo connector</th>
<th>Probe + pogo + wire + triax</th>
<th>Probe + pogo + wire + Triax + holder</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.098 Ω</td>
<td>0.102 Ω</td>
<td>0.201 Ω</td>
<td>0.202 Ω</td>
</tr>
<tr>
<td>2</td>
<td>0.100 Ω</td>
<td>0.102 Ω</td>
<td>0.202 Ω</td>
<td>0.202 Ω</td>
</tr>
<tr>
<td>3</td>
<td>0.101 Ω</td>
<td>0.102 Ω</td>
<td>0.200 Ω</td>
<td>0.201 Ω</td>
</tr>
<tr>
<td>4</td>
<td>0.097 Ω</td>
<td>0.101 Ω</td>
<td>0.201 Ω</td>
<td>0.202 Ω</td>
</tr>
</tbody>
</table>
It is seen from the data that the updated configuration made very stable and high quality connections. These connections were used for the testing in the following sections.

4.3.3 1k Resistor Square

This section contains the results from the testing of the 1k ohm resistor square. The sample is a square of four 1 k resistors soldered to a printed circuit board, shown in figure 54. This sample was tested a total of 4 times, on four separate days. The additional testing on separate days was done to ensure the repeatability and reproducibility of the system.

![Figure 54: 1kΩ and 10kΩ Samples](image)
Table 12: Summary for Testing of the 1k Ohm Resistor Square via Wire

<table>
<thead>
<tr>
<th>Pin Pair</th>
<th>Resistance (kΩ)</th>
<th>Measured Voltage (V)</th>
<th>Measured Current (mA)</th>
<th>Calculated Resistance (kΩ)</th>
<th>Percent Error (%)</th>
<th>Percent Difference run to run (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 – 2</td>
<td>0.746</td>
<td>0.74625</td>
<td>0.99918</td>
<td>0.7469</td>
<td>0.1156</td>
<td>0.6103</td>
</tr>
<tr>
<td>2 – 3</td>
<td>0.746</td>
<td>0.74599</td>
<td>0.99918</td>
<td>0.7466</td>
<td>0.1807</td>
<td>0.8757</td>
</tr>
<tr>
<td>3 – 4</td>
<td>0.746</td>
<td>0.74591</td>
<td>0.99915</td>
<td>0.7465</td>
<td>0.0730</td>
<td>0.5683</td>
</tr>
<tr>
<td>4 – 1</td>
<td>0.746</td>
<td>0.74604</td>
<td>0.99911</td>
<td>0.7467</td>
<td>0.0944</td>
<td>0.7341</td>
</tr>
<tr>
<td>1 – 3</td>
<td>0.995</td>
<td>0.9955</td>
<td>1.00006</td>
<td>0.9954</td>
<td>0.0442</td>
<td>0.8452</td>
</tr>
<tr>
<td>2 – 4</td>
<td>0.995</td>
<td>0.9955</td>
<td>1.00004</td>
<td>0.9955</td>
<td>0.0462</td>
<td>0.7642</td>
</tr>
</tbody>
</table>

This testing shows the system is calibrated correctly and the connections are low noise. This test is a verification that the system connections for the current source, the ammeter and the voltage meter are all connected correctly. This also verifies that the system grounding and the guarding are also correctly connected.

4.3.4 10k Resistor Square

This section contains the results from the testing of the 10k ohm resistor square. The sample is constructed the same as the 1k sample however four 10k resistors are used.
in place of the 1k resistors. This sample also tested the same as the 1k sample. This was done in addition to the 1k sample to ensure that the testing is correct for multiple samples of different resistance.

Table 14: Summary for Testing of the 10k Ohm Resistor Square via Wire

<table>
<thead>
<tr>
<th>Pin Pair</th>
<th>Resistance (kΩ)</th>
<th>Measured Voltage (V)</th>
<th>Measured Current (µA)</th>
<th>Calculated Resistance (kΩ)</th>
<th>Percent Error (%)</th>
<th>Percent Difference run to run (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 – 2</td>
<td>7.48</td>
<td>0.7495</td>
<td>99.958</td>
<td>7.498</td>
<td>0.2426</td>
<td>0.6851</td>
</tr>
<tr>
<td>2 – 3</td>
<td>7.48</td>
<td>0.7494</td>
<td>99.952</td>
<td>7.497</td>
<td>0.2352</td>
<td>0.6947</td>
</tr>
<tr>
<td>3 – 4</td>
<td>7.48</td>
<td>0.7496</td>
<td>99.922</td>
<td>7.500</td>
<td>0.2921</td>
<td>0.7358</td>
</tr>
<tr>
<td>4 – 1</td>
<td>7.48</td>
<td>0.7492</td>
<td>99.951</td>
<td>7.495</td>
<td>0.2095</td>
<td>0.5945</td>
</tr>
<tr>
<td>1 – 3</td>
<td>9.98</td>
<td>0.9997</td>
<td>99.990</td>
<td>9.998</td>
<td>0.1803</td>
<td>0.7711</td>
</tr>
<tr>
<td>2 – 4</td>
<td>9.98</td>
<td>0.9996</td>
<td>99.983</td>
<td>9.997</td>
<td>0.1773</td>
<td>0.6115</td>
</tr>
</tbody>
</table>

Table 15: Summary for Testing of the 10k Ohm Resistor Square via Probe

<table>
<thead>
<tr>
<th>Pin Pair</th>
<th>Resistance (kΩ)</th>
<th>Measured Voltage (V)</th>
<th>Measured Current (µA)</th>
<th>Calculated Resistance (kΩ)</th>
<th>Percent Error (%)</th>
<th>Percent Difference run to run (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 – 2</td>
<td>7.48</td>
<td>0.7495</td>
<td>99.955</td>
<td>7.498</td>
<td>0.2456</td>
<td>0.8774</td>
</tr>
<tr>
<td>2 – 3</td>
<td>7.48</td>
<td>0.7496</td>
<td>99.956</td>
<td>7.499</td>
<td>0.2580</td>
<td>0.8515</td>
</tr>
<tr>
<td>3 – 4</td>
<td>7.48</td>
<td>0.7496</td>
<td>99.923</td>
<td>7.501</td>
<td>0.2911</td>
<td>0.5655</td>
</tr>
<tr>
<td>4 – 1</td>
<td>7.48</td>
<td>0.7496</td>
<td>99.953</td>
<td>7.497</td>
<td>0.2342</td>
<td>0.5444</td>
</tr>
<tr>
<td>1 – 3</td>
<td>9.98</td>
<td>0.9998</td>
<td>99.991</td>
<td>9.999</td>
<td>0.1893</td>
<td>0.7925</td>
</tr>
<tr>
<td>2 – 4</td>
<td>9.98</td>
<td>0.9997</td>
<td>99.989</td>
<td>9.998</td>
<td>0.1813</td>
<td>0.7835</td>
</tr>
</tbody>
</table>
4.3.4 Silicon Sample

This section contains the results from testing of a silicon, p-type, sample. This sample was sent to NIST for testing and was sent back with the results. The measurements from NIST and this system’s measurements will be compared.

Table 16: Results from System and from NIST before Factory Calibration

<table>
<thead>
<tr>
<th>Step</th>
<th>Measurement 0° Rotation</th>
<th>Measurement 90° Rotation</th>
<th>NIST Measurement</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Voltage (mV)</td>
<td>Current (mA)</td>
<td>Voltage (mV)</td>
</tr>
<tr>
<td>1</td>
<td>-52.37</td>
<td>1.001</td>
<td>-95.72</td>
</tr>
<tr>
<td>2</td>
<td>52.41</td>
<td>1.002</td>
<td>95.89</td>
</tr>
<tr>
<td>3</td>
<td>-43.51</td>
<td>1.001</td>
<td>-43.41</td>
</tr>
<tr>
<td>4</td>
<td>95.81</td>
<td>1.003</td>
<td>52.45</td>
</tr>
<tr>
<td>5</td>
<td>-52.75</td>
<td>1.004</td>
<td>-96.13</td>
</tr>
<tr>
<td>6</td>
<td>52.82</td>
<td>1.001</td>
<td>96.09</td>
</tr>
<tr>
<td>7</td>
<td>-43.45</td>
<td>1.007</td>
<td>-43.76</td>
</tr>
<tr>
<td>8</td>
<td>95.78</td>
<td>1.007</td>
<td>52.45</td>
</tr>
</tbody>
</table>

It is seen in the table above that the measurements taken with the system closely match those taken at NIST. The minus sign on some measurements should be ignored for the purpose comparison. The minus sign is present because in the NIST configuration the pins that the voltage are measured are reversed when the current is reversed so that the voltage measurement is always positive. In this system’s set-up the pins are not reversed so when the current is reversed the sign on the voltage measurement will be reversed as well.
Table 17: Resistivity Results from System and from NIST after Factory Calibration

<table>
<thead>
<tr>
<th>Step</th>
<th>Voltage (mV)</th>
<th>Current (mA)</th>
<th>Voltage (mV)</th>
<th>Current (mA)</th>
<th>Vs. NIST</th>
<th>Run to Run</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-52.11</td>
<td>1.002</td>
<td>52.73</td>
<td>1.0</td>
<td>1.175</td>
<td>3.234</td>
</tr>
<tr>
<td>2</td>
<td>52.23</td>
<td>1.001</td>
<td>52.54</td>
<td>1.0</td>
<td>0.590</td>
<td>3.135</td>
</tr>
<tr>
<td>3</td>
<td>-95.27</td>
<td>1.002</td>
<td>94.94</td>
<td>1.0</td>
<td>0.347</td>
<td>1.365</td>
</tr>
<tr>
<td>4</td>
<td>95.11</td>
<td>1.002</td>
<td>95.53</td>
<td>1.0</td>
<td>0.439</td>
<td>1.363</td>
</tr>
<tr>
<td>5</td>
<td>-52.13</td>
<td>1.001</td>
<td>52.17</td>
<td>1.0</td>
<td>0.076</td>
<td>2.873</td>
</tr>
<tr>
<td>6</td>
<td>52.19</td>
<td>1.002</td>
<td>52.51</td>
<td>1.0</td>
<td>0.609</td>
<td>3.021</td>
</tr>
<tr>
<td>7</td>
<td>-95.18</td>
<td>1.003</td>
<td>95.03</td>
<td>1.0</td>
<td>0.157</td>
<td>1.278</td>
</tr>
<tr>
<td>8</td>
<td>95.31</td>
<td>1.004</td>
<td>94.98</td>
<td>1.0</td>
<td>0.347</td>
<td>1.197</td>
</tr>
</tbody>
</table>

Table 18: Hall Coefficient Results from System and from NIST after Factory Calibration

<table>
<thead>
<tr>
<th>Step</th>
<th>Voltage (mV)</th>
<th>Current (mA)</th>
<th>Voltage (mV)</th>
<th>Current (mA)</th>
<th>Vs. NIST</th>
<th>Run to Run</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>48.55</td>
<td>1.003</td>
<td>49.67</td>
<td>1.0</td>
<td>2.306</td>
<td>2.598</td>
</tr>
<tr>
<td>2</td>
<td>47.87</td>
<td>1.002</td>
<td>48.78</td>
<td>1.0</td>
<td>1.900</td>
<td>2.710</td>
</tr>
<tr>
<td>3</td>
<td>36.54</td>
<td>1.002</td>
<td>35.82</td>
<td>1.0</td>
<td>1.970</td>
<td>2.931</td>
</tr>
<tr>
<td>4</td>
<td>36.68</td>
<td>1.001</td>
<td>36.32</td>
<td>1.0</td>
<td>0.981</td>
<td>3.083</td>
</tr>
<tr>
<td>5</td>
<td>36.87</td>
<td>1.001</td>
<td>37.48</td>
<td>1.0</td>
<td>1.654</td>
<td>3.141</td>
</tr>
<tr>
<td>6</td>
<td>36.23</td>
<td>1.001</td>
<td>36.58</td>
<td>1.0</td>
<td>0.966</td>
<td>2.879</td>
</tr>
<tr>
<td>7</td>
<td>47.64</td>
<td>1.002</td>
<td>48.03</td>
<td>1.0</td>
<td>0.818</td>
<td>3.357</td>
</tr>
<tr>
<td>8</td>
<td>48.01</td>
<td>1.003</td>
<td>48.53</td>
<td>1.0</td>
<td>1.083</td>
<td>2.451</td>
</tr>
</tbody>
</table>

It can be seen from tables 40 and 41 that the calibration of the hall effect card solved the errors on steps three and seven. The system was able to come within 1.7% of the NIST measurements for resistivity and 2.3% for hall coefficient. The system also performed very well with run-to-run repeatability. The system was able to show lower than 3.4% discrepancy between runs on either the resistivity or hall coefficient. This falls
well below the typical 5% measurement discrepancy accepted as normal. This testing was the last verification that the system is fully operational and functional.
CHAPTER 5: CONCLUSIONS & FUTURE WORK

This section will discuss the conclusions drawn about the outcomes of each system. A general overview will be given for each system; this includes the performance and functionality. The specific problems encountered for each system will be addressed; these problems may not necessarily be mentioned in the previous chapters.

5.1 DC Sputter Conclusion

The final DC Magnetron Sputter system is fully functional and operational. The system meets both operational and performance markers. The system is fully functional in both operating modes, manual and automatic. The system was able to produce reproducible and high quality films from run to run.

For completeness and guidance, it is necessary to mention that several problems were encountered during the development and the calibration of the system. These problems will be discussed in the following paragraphs. The first paragraph comprises hardware/instrument as well as software problems, while the second is related to operational problems.

5.1.1 Hardware and Instrument Problems

The first of these problems was the improper functioning of the ion and convectron gauges. The convectron gauges were reading the wrong pressure values and the ion gauge was not turning on when the commands were being given. The convectron gauges required not only a recalibration, this was executed manually from the front panel of the pressure controller, but also the I/O cable required rewiring. The connections were loose in the connecting head, once the heads were rewired this fixed the problem. The
nonfunctioning ion gauge was a difficult problem to diagnosis. The ion gauge was checked for proper resistance between collection/supply pairs, these resistances fell within 5% of manufactures specifications. Then the power supply was examined to ensure proper power levels were being output, this also met manufactures specifications. Then the ion gauge module was checked. This finally led to the source of the problem, a current leak in the monitoring circuitry. This leakage was large enough to trip the open circuit protection. A replacement module was ordered which fixed the problem.

The next problem encountered was the deposition gun creating a weak unstable plasma. This was found to be caused by two sources. The first source was the operational mode of the selected from the deposition gun power supply. It was found that in power regulation mode the unit would drop the voltage under the required amount to sustain the plasma. This was fixed by switching the unit to voltage regulation mode. In this mode, the voltage is fixed and the current is varied to sustain that voltage. The second source was the working gas flow rate. The flow was too low, so there were not enough argon atoms to ionize to sustain the plasma, presumably due to the evacuation rate from the sizable turbo pump. This was fixed by increasing the gas flow rate.

5.1.2 Operational Problems

There was a problem of excess oxygen and water vapor migration into the chamber walls and subsequently into the deposited film, leading to oxidation of the Al films deposited. This was first noticed visually, by the color patterns on deposited films. This problem was caused by two sources, first back filling the chamber from the room and the second not allowing the chamber adequate time under vacuum to reach base
pressure. The problem was solved by backfilling the chamber with nitrogen during venting. This allowed the nitrogen to migrate into the chamber walls first during backfill and create a barrier blocking oxygen and water vapor species when the chamber was opened at atmosphere. To ensure film quality the chamber was also allowed to set at base pressure for an additional 15 minutes. To further ensure that this would not continue to be a problem a dummy run is done whenever the system has be inactive for more than one week. This dummy run allows the deposited aluminum to act as a getter. This gettering action allows the oxygen and water vapor to be attracted to this sacrificial aluminum layer on the chamber. This allows the subsequent deposition to produce films with significantly less oxygen/water vapor contamination.

5.1.3 Performance Enhancements

Once the problems had been fixed, the system produced high quality films. The film thickness uniformity was stable, with less than a 5% change in thickness between runs. The surface roughness was very low, on average less than 10nm roughness. This roughness is very low when compared with other sputtered samples [52].

The system also took considerably less time to perform a deposition when compared with the previous version. The original set-up of the system did not have a separate roughing line. The chamber was roughed through the foreline/turbo. This allowed only one deposition to occur before the turbo had to be allowed to spin down so that the chamber could be roughed down again. This was mitigated by adding a separate roughing line, allowing multiple deposition runs as well as cutting out the time needed for the turbo to spin down. The original system also vented with regular atmospheric air,
this problem is discussed in the section above. When the chamber was redesigned to vent with nitrogen, the time required to reach base pressure was decreased by 60%-70%. Combining these factors, the overall time for the system to make multiple successive runs decreased by over 90%, as compared to the original manual/analog design. This shows a significant improvement for productivity and repeatability.

The final enhancement is the time it takes for training. The training was given to two students, one was familiar with sputtering deposition and vacuum systems the other student was not familiar with either. The student familiar with vacuum systems only took roughly 10-15 minutes to train. The other student that not familiar with either with vacuum systems took only slightly longer to train, at about 35 minutes. This is considerably fast than the training given to student for the other analog systems in the laboratory.

5.2 Hall Effect Conclusion

The Hall Effect system is also a successfully designed system. The system was developed to be fully functionally, meeting both operational and performance markers. The system is fully functional in the automatic operating mode, with all the sub functions fully operational. The system was shown to be properly designed, from a hardware standpoint, and fully operational, from a software standpoint. The performance of the system fell within 3%, when compared to the NIST measured reference sample.

As before, for completeness and guidance, it is necessary to mention that several problems were encountered during the development and the calibration of the system. These problems will be discussed in the following paragraphs. The first paragraph
comprises hardware/instrument as well as software problems, while the second is related to operational problems.

5.2.1 Hardware and Instrument Problems

The first problem that was encountered was the incompatibility of using the source measuring unit with the switching matrix. This problem took a long time to diagnose. It required a joint discussion with the head engineers for the SMU and the switching matrix to find the problem. The problem was the inter shield on the triax connector. The switching matrix assumes the inter shield to be similar to a current source instrument, where the inter shield is the force low. The SMU has the inter shield connected as the guard. This causes the two instruments to see a potential of zero and thus no current flow. This was remedied by making a custom triax connector on the SMU, where the inter shield was bypassed from connecting to the guard and instead connected to force low. This eliminated the problem.

The second problem encountered was the accuracy of the digital multimeter. This problem also took a long time to diagnose. The problem was found to be the calibration of the instrument. Several attempts were made to recalibrate the unit. This did improve the accuracy but did not improve it enough to meet manufactures specification. The unit had to be sent back to the manufacturers for a full calibration. This solved the problem.

The next problem encountered was poor connection at the test bed, this took the longest to diagnose. The first test bed used tungsten probes that were soldered to wire that was soldered to the triax connector. Tungsten is a hard metal to solder to; it may form a mechanical connection but does not always form an electrical connection. It was found
that the contacts to the probes did not form good ohmic contacts. The first attempt at solving this problem was to deposit nickel then gold plate the end of the tungsten probes. This would allow the solder to form a good ohmic contact to the gold. This improved the contacts but two contacts were still found to be poor connections. The solution was found by ordering pogo pin connectors that the inside diameter was the same as the outside diameter of the probe. This proved to provide both a strong mechanical connection and a good ohmic connection, which solved the problem.

The last hardware problem could only be diagnosed after the above problems were solved. There was a discrepancy, in the voltage readings, between steps when measuring resistivity. This problem was one that was seen in the system since the beginning of development. This problem was difficult to diagnose because it could be due to several factors. Therefore, each possible source of error had to be eliminated sequentially, in order to find the actual source. The problems discussed above were listed in the order they were solved, this was also the order of probability that they were the source of the problem. The last step in this sequence was the Hall Effect card itself. To verify that the error was coming from the card all other possible sources had to be eliminated. Once this was done and the error was still present the only source left was the card. After examining the steps on which the error occurred it was found that this corresponded to a set of two particular steps. When the steps 3 and 7 were used then we saw the error. This means that something in the circuitry associated with these matrix crosspoints had failed. This diagnosis was confirmed by the lead test engineer for the
Hall Effect card at Keithley. The card was sent in for calibration. This calibration fixed the errors that were seen.

5.2.2 Operational Problems

There were only two operational problems however; these took several attempts to fix. The first problem was with the process of zeroing out the meters during experiments. The software only zeroed the meters before the start of the experiment. This caused the noise bias to be incorrect, referenced to the current level or voltage level. This was eventually fixed; the correct procedure is outlined in chapter 4.2. This corrected some discrepancy that was seen from run to run. The second problem was also related to the internal communication of the software. The problem was between two sub routines not always communicating with each other. This would cause either the code to be stuck waiting for an update or to update with the wrong information. This was solved by restructuring the method in which the two sub routines passed data. After this restructuring, the code showed no further problems in communication.

5.2.3 Performance Enhancements

Once the problems had been fixed, the system was able to pass the recommended testing specified by the manufacturers. The system was able to make uniform and stable measurements from run to run. The system was able to test samples with under a 3% error on the measurements. The system also significantly decreases the time to take measurements this cuts the time to take a single measurement by roughly 50%. It decreases the time to take sweeps by an exponential amount.
5.3 Future Work

This section is dedicated to suggesting what future work should be done to the two systems. The first system that will be covered is the DC Magnetron Sputter system, this will be followed by the Hall Effect system.

The DC Magnetron Sputter system has several additions that would make it more flexible. The first suggestion is to replace the manual gas line regulator, on the working gas line, with a mass flow controller. This would allow the gas flow to be regulated by the software. It would also allow the addition of more precise control over the amount of flow. The second suggestion is to add an additional two working gas lines and a manifold. This would allow the addition of one line for active gasses and one line for passive gasses, greatly expanding the system’s capability to deposit a wider variety of materials. It is also assumed that if these lines were added then they would be fitted with mass flow controllers as well. The next suggestion is to replace the current substrate holder. The current holder requires the sample to be held by magnets, this causes an uneven flow around where the magnet is placed. The last suggestion is to replace the current backing pump with a pump that has a larger flow rate. The current pump is undersized for the system. If this were to be replaced it would cut down on roughing time and place less strain on the turbo pump during initial pump down periods.

The Hall Effect system also has several updates that could be made to it. The first suggestion is to replace the current source, with the Keithley model 6220. The system currently uses the source measuring unit as the current source. If this SMU were to be replaced with a programmable current source the system would operate more stable. The
second suggestion is to add an optical source for stimulation in addition to the electrical source. The code currently has this option, but it is not yet implemented in the hardware. The last suggestion would be to add the addition of an Ammeter, Keithley model 6487. This would allow the measurement of higher currents. One of the current limitations to the system is that the current can only be raised to 2 mA. If the meter was changed, the current could be set too much higher levels, improving the S/N ratio significantly. Lastly, a process for developing reliable, stable and repeatable contacts needs to be developed, for future users. The placement, size and quality of the contacts greatly influences the error introduced into the measurement. This process is not trivial and requires in-depth knowledge involving many aspects of deposition.
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APPENDIX: REFERENCE MATERIAL

Hardware Details For The DC Magnetron Sputter System

The DC Magnetron Sputter system during the initial design had several 3-D CAD drawings done for potential configurations. This helped to finalize what design choice for the vacuum lines would be chosen. The final choice is shown in figure 55.

Figure 55: 3-D CAD Drawings of Vacuum Components

The deposition gun power supply is the MDX 1K Magnetron Drive by Advanced Energy. This is a regulatory power supply, which can supply up to 1k watt. It has the
ability to protect from arcing and short circuits. It also has remote interface, RS232, and interlock capabilities. The unit can output 0 – 1000 volts and 0 – 1 amps [34].

The deposition rate controller/thickness monitor is the STC-200 by Sycon. This unit allows the monitoring of up to 2 channels. In this system we currently only use one. It allows the user to input the target material parameters, this allows for more precise calculations of thickness. It has the ability to store up to 8 material profiles, and has one profile active. This unit can measure with the resolution of 0.1 angstrom per second and up to the range of 5000k angstroms. It uses a 6 MHz crystal as the sensor. This unit has remote interface, RS232, and interlock capabilities [35].

The turbo pump and controller are the TMP 1000 and the NT 1000 by Leybold. The turbo pump, TMP 1000, can achieve a base pressure of 7.5E-10 torr. The rotational speed is 36,000 rpm when at full speed. It has a maximum flow rate of 850 liters/second. The controller, NT 1000, has a frequency output of 595 Hz, when at normal operation. The controller uses a series of steps to slowly increase the drive frequency from 0 – 595 Hz [36, 37].

The vacuum controller is the 307 by Granville Philips. The vacuum controller has the six modules. These modules are the power supply, the ion gauge electrometer, the remote input/output, the Convectron gauge, the process control and the RS232. These modules will be discussed in the order listed above. The power module regulates the power for the controller and for the ion gauge. The ion gauge module can read up to two ion gauges sequentially. It also regulates the input and output current from the ion gauge. Currently the unit only uses one ion gauge, the 274 ion gauge from Granville Philips. The
remote input/output module is not currently used. The Convectron gauge module allows up to two Convectron gauges to be read simultaneously. It has the ability to automatically turn on/off the ion gauge; this feature is disabled in the current system. Currently the unit uses two Convectron gauges; both are 275 convectron gauges by Granville Philips. The process control module allows up to six single pole double throw relays to be set. These relays are set from the pressure readings off the two Convectron gauges and ion gauge. In the system currently, three relays are being used. The RS232 module allows the controller to be fully controlled via RS232 commands. This is currently how the controller is operated [38].

The water flow meter is the 100 by Proteus. This flow meter is an adjustable set point flow meter relay. The flow rate set point can be set for 0.06 to 60 gallons/minute. The relay closes a contact when the flow rate greater than the adjustable set point [39].

The mechanical pump is the D16B by Leybold. It is a dual vane oil rotary pump. It has a base pressure of 1E-4 torr. The flow rate is 11.6 cfm [40].
Now the Data acquisition unit will be described. The figure below shows the output connection on the data acquisition unit.
Hardware And Measurement Details For The Hall Effect System

The Keithley 236 source measure unit, is an instrument that allows either a current or voltage to be sourced and then as the source is being applied a measurement of either a voltage or current from the same connection is made. This unit can also output a DC bias current or voltage. This bias function is what is used in this system. The SMU is a widely diverse and sophisticated instrument, for more information refer to [54].

The Keithley 2000 digital multimeter unit, is an instrument that allows either a current or voltage to be measured. This unit has the ability to measure voltage over several ranges and resolutions. In this system, the DMM is used to measure voltage. For more information, refer to [55].

The Keithley 7001 switching mainframe unit, is the hub for the 7065 hall effect card. This card is the central unit for the system. It allows the programmable switching of card connections, based on which matrix crosspoints are closed or open. It allows a current to be sourced into one input. This sourced input is then applied between two of the four outputs. The other two outputs are used for the system to monitor a voltage. This voltage is output through two screw terminal outputs, which is connected to the DMM. There is also a connection that allows the leakage current to be monitored, which is connected to the Picoammeter. For more information refer to [56, 57].

The Keithley 487 Picoammeter unit, allows the monitoring of a current. This unit has the ability to measure current over several ranges and resolutions. In this system, the Picoammeter is used to measure leakage current. For more information, refer to [58].
The system is also uses a large electromagnet. This magnet has a large power supply that is controlled from the Agilent power supply. The system is pictured in figure 49.

The resistor square ideal layout and equivalent circuit is shown in figure 65.

![Resistor Square Diagram](image)

**Figure 58: Depiction of Resistor Square**

\[ R = \frac{1}{\frac{1}{R} + \left(\frac{1}{R} + \frac{1}{R}\right)} \]

Resistivity Formulas from Keithley 7065 card
\[ \rho_A = \frac{1.1331 \cdot f_A \cdot t_S}{I} \cdot (V_2 + V_4 - V_1 - V_3) \]

\[ \rho_B = \frac{1.1331 \cdot f_B \cdot t_S}{I} \cdot (V_6 + V_8 - V_5 - V_7) \]

\[ Q_A = \frac{V_2 - V_1}{V_4 - V_3} \]

\[ Q_B = \frac{V_6 - V_5}{V_8 - V_7} \]

\[ \frac{Q - 1}{Q + 1} = \frac{f}{0.693} \cdot \cosh^{-1} \left( \frac{1}{2} \cdot e^{\frac{0.693}{f}} \right) \]

Hall Coefficient Formulas from Keithley 7065 card

\[ R_{HC} = \frac{2.5 \cdot 10^7 \cdot t_S}{B \cdot I} \cdot (V_2 - V_1 + V_5 - V_6) \]

\[ R_{HD} = \frac{2.5 \cdot 10^7 \cdot t_S}{B \cdot I} \cdot (V_4 - V_3 + V_7 - V_8) \]
This subroutine sets the following parameters for each instrument.

Switching Mainframe (7001)
- Set slot for slot 1

Source Measuring Unit (237)
- Set output to current
- Set trigger to continuous
- Set trigger origin to immediate only
- Set function to DC
- Set delay to off
- Set sense to local
- Set trigger function to enable

- Set measurement to DC
- Set range to auto
- Set rate to medium
- Set readings to 1

**PicoAmpmeter (487)**
- Set range to auto
- Set Integration period to line cycle integration

**Power Supply for Magnet (E3634)**
- Set voltage range to high

**Gauss Meter (455)**
- Set measurement mode to DC
- Set resolution to 5 digits
- Set RMS mode to wide band
- Set peak mode to periodic
- Set peak display to positive
Atomic Force Microscopy

The roughness data was calculated with the following formulas.

4.9 One-Dimensional Roughness Parameters

Standardized one-dimensional roughness parameters can be evaluated with the roughness tool.

The one-dimensional texture is split into waviness (the low-frequency components defining the overall shape) and roughness (the high-frequency components) at the cut-off frequency. This frequency is specified in the units of the Nyquist frequency, that is value 1.0 corresponds to the Nyquist frequency.

In the following formulas we assume the mean value of \( r_j \) is zero, i.e. it holds

\[
    r_j = z_j - \bar{z}
\]

Roughness Amplitude Parameters


Arithmetical mean deviation. The average deviation of all points roughness profile from a mean line over the evaluation length

\[
    R_a = \frac{1}{N} \sum_{j=1}^{N} |r_j|
\]

An older means of specifying a range for \( R_a \) is RHR. This is a symbol on a drawing specifying a minimum and maximum value for \( R_a \).


The average of the measured height deviations taken within the evaluation length and measured from the mean line

\[
    R_q = \sqrt{\frac{1}{N} \sum_{j=1}^{N} r_j^2}
\]

Figure 60: Calculations for AFM [59]