On the 1/f Noise of Atomic-Layer-Deposition Metal Films

by

Xiawa Wang

Submitted to the Department of Electrical Engineering and Computer Science
in Partial Fulfillment of the Requirements for the Degree of Master of Engineering in Electrical Engineering and Computer Science at the MASSACHUSETTS INSTITUTE OF TECHNOLOGY

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Abstract

This thesis presents the measurement techniques and results of low-frequency noise for atomic-layer-deposition Pt films. Atomic-layer-deposition has been developed as an approach to make ultra-thin and conformal films. It has been employed to make detectors of bolometers. 1/f noise is a fundamental limit to the resolution. The experiments are designed to characterize the 1/f noise of the ALD fabricated Pt films. The measurement results show that for 7nm and 13nm ALD fabricated Pt films, 1/f noise is about two orders of magnitude larger than reported for continuous Pt films in literature. The thin film is also very likely to suffer from electromigration damage.

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Chapter 1

Introduction

1.1 Background and Motivations

The main goal of the $1/f$ noise measurement is to characterize the resolution of an infrared camera. As its name indicates, the camera is an image forming device using infrared radiation. Similar to a common camera that takes pictures in the daylight, infrared cameras can shoot images in a dark environment. Due to the specialties of the camera, it has been widely used in military, medical and commercial applications to provide night vision. The pictures in 1-1 compare the images using regular camera and infrared camera in a dark environment.

The core component of a hand-held infrared camera is a micro-machined bolometer. Infrared radiation is absorbed by the detecting material, causing a temperature rise. The temperature rise will change the resistance of the detector and form an electrical signal. Materials that have high temperature dependence of resistance (TCR), low thermal conductance and low $1/f$ noise are most ideal for bolometer detectors. Current materials explored include metals, amorphous silicon and vanadium oxides. Thin film platinum is also the studied material for bolometer detectors for its high TCR and low impurity levels [1]. Recently, atomic-layer-deposition (ALD) is incorporated in the fabrication of these platinum detectors. Due to the self-limited reactions of ALD, the platinum films as thin as 7nm have been demonstrated to be continuous and electrically conducting.
Low noise is a key figure of merit in the design of bolometers. The signal in the thermal detector is demonstrated by the resistance change induced by the temperature rise. 1/f noise is the intrinsic resistance fluctuation of the detector material. Therefore, a high noise level will obscure the measured signal and reduce the resolution of the image. For thin metal films, two types of noise, thermal noise and 1/f noise are visible. In the low frequency domain, 1/f is dominant. This thesis aims to investigate the 1/f noise of these platinum films.

1.2 1/f Noise in Thin Metal Films

1/f noise is an intrinsic fluctuation phenomenon that widely occurs in nature.Electronic 1/f noise is observed in all kinds of electronic systems, such as resistors, transistors and amplifiers. The term 1/f refers to the shape of the noise power spectrum. The noise power scales linearly with frequency. As the frequency goes higher, the
noise power decreases and becomes negligible in comparison to thermal noise.

Toward the lower frequency, the flat-off of the power spectrum is not observed experimentally. Many models have been proposed to explain the $1/f$ power spectrum. However, no universal explanation can be found to explain all the observations.

1.3 Thesis Organization

In this thesis, Chapter 2 mainly talks about atomic-layer-deposition, including its principles, limitations and current applications. The deposition parameters for the samples used in this research are also presented. Chapter 3 includes the background about electronic noise. The major properties of noise and the current modeling of $1/f$ noise are summarized. The setup and measurement details are discussed in chapter 4. The calibration of the system is shown with carbon resistors. Chapter 5 includes all the measurement data of ALD samples. The results are analyzed and compared with existing data in literatures. Measurement limitations and further improvements are further discussed.
Chapter 2

Atomic Layer Deposition

2.1 Introduction

2.1.1 Principles

Atomic-layer-deposition (ALD), originally called Atomic-layer-epitaxy (ALE), is a thin film deposition method that is widely used in micro-fabrication. It is firstly introduced by Dr. Tuomo Suntola in 1974 for the growth of Zinc Sulfide. ALD is a modification of the widely used Chemical-vapor-deposition (CVD) method. In ALD, the precursor gases and vapors are pulsed into the reaction chamber alternatively. The gases are absorbed onto the reaction surface. In order to produce continuous and conformal film, the precursor doses must be enough so that surface saturation is achieved and the process is insensitive to small changes in the precursor doses. Between each precursor pulsing, inert gases are used to purge the chamber. The cycle of the ALD process looks as figure 2-1.

The self-limiting mechanism facilitates the growth of conformal films with atomic-level control of thickness. These high quality films have high strength and low impurity level. However, due to its low deposition rate, ALD is only suitable for depositing films in the nanometer scale.
2.1.2 Major Applications

ALD is firstly invented for the electroluminescent displays. Since then, it has aroused much interest in both academia and industry. In the 1990s, ALD is taken into the IC industry for producing the next generation high-k dielectric MOSFETs. With the decreasing thickness of the high-k materials, the slow deposition rate of ALD becomes less significant. Moreover, ALD is also used to produce anti-reflective coating, nanotube formation, tungsten nitride (WN) metal barrier for Cu interconnects and recently for bolometer detectors. The pictures 2-2 and 2-3 show the nano-scale structures fabricated using ALD.

Figure 2-2: $\text{TiO}_2$/Ni/$\text{TiO}_2$ Nanotubes
(Source from [2])
2.1.3 Limitations

As mentioned above, the low deposition rate is one of the major limitations of ALD. The graph 2-4 shows the relation between the film thickness and the deposition cycles.

Each reaction cycle can take up to minutes to accomplish. Therefore, ALD is not practical to make thick films.

In addition, non-idealities in the film growth often complicate the ALD process [4]. Firstly, the film growth doesn’t happen layer-by-layer as desired. Rather, a limited number of reactive surface sites and the steric hindrances between ligands in the chemisorption layer only allow a fraction of a monolayer to be formed in each cycle.
The very first cycles convert the surface from the substrate to the film material, the reactive sites and growth rate may also change. Moreover, the temperature is often kept below 400C. Since the reaction process is often reversible, high temperature will increase film decomposition. As a compromise, increasing the exposure time will help to complete the reaction, but the deposition rate is further reduced.

2.2 Sample Platinum Films Preparation

2.2.1 ALD Al₂O₃

The Al₂O₃ adhesion layer is firstly grown using ALD on thermal oxides of silicon substrate. Trimethylaluminium(TMA) and H₂O are used as two precursors to grow the Al₂O₃. The reaction follows [6]:

\[
\begin{align*}
(A) & \quad \text{AlOH}^* + \text{Al(CH}_3)_3^- \rightarrow \text{AlOAl(CH}_3)_2^* + \text{CH}_4 \quad (2.1) \\
(B) & \quad \text{AlCH}_3^* + \text{H}_2^- \rightarrow \text{AlOH}^* + \text{CH}_4 \quad (2.2)
\end{align*}
\]

Asterisks denote the surface species. The overall reaction is

\[
2\text{Al(CH}_3)_3 + 3\text{H}_2\text{O}^- \rightarrow \text{Al}_2\text{O}_3 + 3\text{CH}_4 \quad (2.3)
\]

The Al₂O₃ deposition process for each cycle is as follows

1. Open the pump valve and let the Ar flow for at least 60s

2. Close the valve, close the Ar and pump the Trimethylaluminium(TMA) for 0.25s

3. Close the TMA valve, open the H₂O valve for 0.25s

The pressure in the chamber varies by time as shown in figure 2-5
2.2.2 ALD Pt

MeCpPtMe₃ and clean dry air (CDA) are used as two precursors for the Pt growth. Initially, the pump valve is closed. The deposition process is as follows.

1. Close the Ar valve and close the pump valve

2. Open the MeCpPtMe₃ valve for 0.35s and then wait for 2s

3. Open the Ar valve, open the pump valve and purge the chamber for 60s

4. Close the Ar valve, and open the air valve for 0.25s and wait for 2s

5. Open the Ar valve, open the pump valve and wait for 60s

The pressure in the chamber varies by time as shown in figure 2-6
The TEM pictures of the fabricated 14nm samples are shown in 2-7. The lower limit of the sample thickness is approximately 7nm. The film is discontinuous and doesn’t conduct beyond that limit. From the TEM pictures in 2-8, it can be shown that discontinuity occurs in the 7nm Pt films.

2.3 Fabrication Process

ALD Pt samples of thickness 7nm and 13nm are fabricated. The fabrication process is as follows

1. 100nm or 500nm thermal oxide is grown on the silicon wafer substrate

2. The $Al_2O_3$ ALD films are deposited on the oxide as adhesion layer
3. The Pt ALD films are deposited on the $Al_2O_3$ layer

4. The Pt films are patterned using plasma reactive ion etching

5. 30nm Ti and 200nm Au contacts are deposited using liftoff
Figure 2-13: Au Contact Liftoff
Chapter 3

Noise Theory

1/f noise, also called "flicker noise", "pink noise", or "excess noise" is a ubiquitous phenomenon in electrical systems. When current passes through a simple metal film, 1/f noise causes the voltage across it to fluctuate randomly around the mean value. 1/f noise is intrinsic to the material, but is affected by many external factors such as temperature, stress, substrates and fabrication methods. Therefore, the noise level is an important indicator for the quality of the material and the fabrication process. The origin of 1/f noise remains debatable even with decades of research. This chapter introduces the 1/f noise in general, describes experimental results about the noise and includes several theoretical approaches that try to model the noise.

3.1 General Properties of Noise

3.1.1 Noise Power Spectral Density

Power Spectral Density

Electronic noise is a random process and is often described by its power spectral density (PSD). For any arbitrary electrical signal, PSD is the distribution of the signal power throughout the frequency spectrum, which is defined as:

\[ S(f) = \lim_{\Delta f \to 0} \frac{\Delta P(f)}{\Delta f} \]  

(3.1)
If the Fourier transform of a signal exists, the PSD is just the square of the magnitude of the transform. However, the Fourier transform of the noise doesn’t exist because noise can be treated as an aperiodic stochastic signal that lasts forever. It is neither square integrable nor absolutely integrable over an infinite interval. The Wiener-Khinchin theorem provides an alternative way that relates the PSD to the autocorrelation function as long as the noise is wide-sense-stationary:

\[ S_{xx}(f) = \int_{-\infty}^{\infty} R_{xx}(\tau) e^{-j2\pi f \tau} d\tau \] (3.2)

where \( S_{xx}(f) \) is the noise PSD and \( R_{xx}(\tau) \) is the autocorrelation function. If the random process is wide-sense-stationary, the autocorrelation function only depends on the time difference between the sampling rather than the absolute time.

\[ R_{xx}(\tau) = E[x(t)x(t+\tau)] \] (3.3)

**Resistance Fluctuation**

1/f noise is now believed to originate from resistance fluctuation. \( R_0 \) is the average resistance and \( \Delta R(t) \) is the fluctuation that centers around 0.

\[ R(t) = R_0 + \Delta R(t) \] (3.4)

The biasing current doesn’t cause the fluctuation, but only makes the resistance fluctuation visible. Based on Ohm’s law:

\[ V(t) = R(t)I_{bias} \] (3.5)

The power spectrum follows as:

\[ S_V = S_{R}I_{bias}^2 = S_R \frac{V^2}{R_0^2} \] (3.6)

\[ \frac{S_V}{V^2} = \frac{S_R}{R_0^2} \] (3.7)
The fact that 1/f noise originates from resistance fluctuation is verified by the experiments of Voss and Clark [30] with no current biasing. Since thermal noise also contains information about the resistance, the experiment is designed to observe the 1/f noise in the thermal noise. The equilibrium noise of the resistor is amplified and passed through a bandpass filter from $f_0$ to $f_1$. The signal is squared and averaged over time $t > 1/f_0$ to obtain $P(t)$. $P(t)$ is a slowly varying signal proportional to thermal noise, but is also sensitive to slow resistance fluctuations. In figure 3-1, Voss and Clark demonstrate the validity of 1/f noise measurement using current source, voltage source as well as the thermal noise method.

![Figure 3-1: 1/f Noise Measurement Thermal Noise](Source from [30])

**Stationarity**

Before applying the Wiener-Khinchin Theorem, the noise random process needs to be wide-sense-stationary, which means that the first and second moments of a noise process only depend on the relative sampling time, rather than the absolute time.
Ensemble mean:

\[ E[x(t_i)] = E[x(t_j)] = constant \tag{3.8} \]

Autocorrelation function:

\[ E[x(t_i)x(t_i + \tau)] = R_{xx}(\tau) \tag{3.9} \]

In addition, 1/f noise is Gaussian. For Gaussian process, wide-sense-stationary means strict-sense-stationary. Therefore, all information is revealed in the power spectral density. Stationarity of flicker noise was verified by Stoisiek and Wolf [11]. In their experiment, the 1/f noise of a carbon resistor \( \xi(t) \) is amplified, bandlimited, squared and exponentially averaged. The resulting data \( \eta(t) \), as well as the original noise data \( \xi(t) \), are passed through an amplitude analyzer to obtain the probability density distribution (PDF) of the amplitude. The variance of \( \eta(t) \) as a function of \( \xi(t) \) is obtained as \( \sigma^2_\xi(t) \). The experimental result is in full agreement with theoretical calculation if \( \xi(t) \) is a normally distributed stationary random process.

Even though the 1/f noise is stationary, the total stationarity of the noise measurement will be affected by some other mechanisms within the specimen.

- **Resistance Drift**

  For newly prepared samples, macroscopic resistance drift is often observed. The drift is mainly caused by the slow annealing of the unstable defects. According to Matthiessen’s rule, as the number of unstable defects decreases, the resistance will also decrease. Koch [7] investigated the effect of resistance drift on the spectrum of noise. If the drift is approximately linear with time, the power spectrum will appear as \( 1/f^2 \) rather than \( 1/f \). Moreover, different windows will also give out different exponent for the cross-over of 1/f noise and drift-induced noise. In order to eliminate resistance drift, the specimen typically undergo heat treatment for the defects to reach equilibrium.

- **Electromigration**

  Even though 1/f noise is only seen when there is a current passing through the
samples, the current doesn’t cause the noise but only makes it visible. However, when the current is sufficiently large, the high current density will cause material transport due to the collisions between electrons and lattice atoms. The metal films are often damaged due to Joule heating or electromigration. Shingubara and Kaneko [13] showed that a film that suffers from electromigration damage often shows abrupt changes in the resistance. The resistance change in a series of discrete steps will also result in a $1/f^2$ spectrum [7].

![Figure 3-2: Electromigration Damage of Solder Joints](image)

**Ergodicity**

While it is very hard to prove whether a random process is ergodic or not, ergodicity is generally assumed for stationary noise. Under this assumption, the probability density function across the ensemble of the value obtained at a particular sampling time is no longer necessary to compute the first and second moments of the random process. Rather, the mean and autocorrelation function can be obtained from time-averaging of a single realization. Ensemble mean:

$$E[x(t)] = \frac{1}{2T} \lim_{T \to \infty} \int_{-T}^{T} x(t)dt$$

(3.10)

Autocorrelation function:

$$R_{xx}(\tau) = E[x(t)x(t+\tau)] = \frac{1}{2T} \lim_{T \to \infty} \int_{-T}^{T} x(t)x(t+\tau)dt$$

(3.11)
Since ergodicity involves time-averaging of a noise signal, a non-stationary process with a time-varying mean cannot be ergodic. Therefore, the assumption of ergodicity requires stationarity.

### 3.1.2 Noise in Metal Films

**Thermal Noise**

Thermal noise is a well-understood phenomenon that is caused by Brownian motion of charge carriers inside a conductor. It shows up as voltage fluctuations regardless of the applied bias. For a sample with a given resistance $R$, the thermal noise power spectral density is flat across the entire frequency range:

$$S_{th} = 4k_BTR$$  \hspace{1cm} (3.12)

**1/f Noise**

1/f noise is a low frequency phenomenon. Caloyannides recorded the 1/f noise spectrum down to $0.5\mu Hz$ in operational amplifiers [8] and no rolloffs are observed. These low frequency experiments are very hard to implement and the waiting time becomes unbearable. The higher ends of the 1/f noise are buried in the thermal noise. The frequency where the 1/f noise PSD meets the thermal noise is often called "corner frequency".

There are many factors that affect the magnitude and shape of 1/f noise. It has shown dependence on temperature, defects, stress and substrate materials. Even specimens prepared by the same technology and having similar electro-physical parameters often have noise levels differ by an order of magnitude or larger. 1/f noise is believed to be of a completely different origin from thermal noise. The total noise level in the sample is equivalent to the sum of the noise power.

$$S_{total} = S_{th} + S_{flicker}$$  \hspace{1cm} (3.13)
Electromigration Noise

For high-current density, the current induced noise is observable. Early stage electromigration occurs when the current density is larger than $10^6 \text{A/cm}^2$; An empirical expression for the voltage spectrum is [33]

$$S_v(f) = \frac{I^3 C}{f^\gamma T} \exp\left(\frac{-E_a}{kT}\right)$$

(3.14)

where $\beta \geq 3$ and $\gamma \geq 2$. One obvious difference between $1/f$ noise and electromigration noise is the power exponent. Typically $\gamma \sim 0.8 - 1.3$ for $1/f$ noise in metals while $\gamma$ exceeds 2 for electromigration noise.

3.2 Summarization of Experimental Results

3.2.1 Empirical Relation

In 1969, Hooge provides an empirical formula for homogeneous layers. The empirical formula takes the form as:

$$\frac{S_v(f)}{U^2} = \frac{S_R(f)}{R^2} = \frac{\alpha}{N f^\gamma}$$

(3.15)

Here $U$ denotes the voltage across the sample. The symbol $N$ is the total number of free electrons. The value $\alpha$, also called Hooge parameter, is an empirical, dimensionless constant. It takes an average value of $\alpha = 2 \times 10^{-3}$. The power exponent $\gamma$ describes the shape of the $1/f$ noise and can take a value from 0.8-1.3.

This empirical relation is widely used in literatures as an estimate of $1/f$ noise. The Hooge parameter is used to compare noise levels of different materials. Because there are many factors that affect the noise level and shape, the underlying cause of $1/f$ noise is very hard to predict. It is very possible that the observed noise has several independent origins.
3.2.2 Temperature Dependence

Several independent works on the temperature dependence of 1/f noise have been proposed. However, the experimental results are not fully consistent. Both strong and weak temperature dependence have been observed.

1. Hooge [25] investigated the dependence of noise level on the temperature for continuous gold films. The samples were immersed in liquid He, liquid $N_2$, $CO_2$ in alcohol and room temperature. The lines connect points of the same sample. From their data, a rough conclusion is made that 1/f noise has a temperature dependence no stronger than $T^{1/2}$.

2. Eberhard and Horn [22] measured the temperature dependence of 1/f noise in silver and copper thin films. The power exponent $\alpha$ changes from 1.03 to 1.20 when the temperature drops from 400K to 150K for both the silver and copper films of various thicknesses. The temperature dependence of the noise level and the 1/f exponent at 20Hz in the Ag film is shown in figure 3-4 and 3-5. The noise level

![Figure 3-3: Temperature Dependence of 1/f Noise in Au Films (Source from [25])](image-url)
decreases rapidly below room temperature. The peak occurs at around 410K for the silver films and at around 490K for the copper films. Below room temperature, the dependence can be ascribed as

\[
S_v(20Hz) = N_0 + N'exp\left(\frac{-E_g}{k_BT}\right)
\]  

(3.16)

\(E_g\) increases as the properties of the film become more bulk-like.

3. Cottle and Chen [26] measured the temperature dependence of 1/f noise on aluminum and Al-Si films at 1Hz. The temperature dependence of the noise level has similarities with the figures obtained by Eberhard and Horn. The noise level also has a peak value at 350K and 370K respectively. The experimental results suggest that there might exist more than one factors that give rise to 1/f noise. Some have strong temperature dependence while others have weak temperature dependence.
3.2.3 Stress and Strain Dependence

Zhigal’skii [31] measured the dependence of 1/f noise on mechanical stress for Al, Cr and Mo films. Controlled external mechanical stress was applied to the film by deflecting a cantilever mounted substrate. The results show that both the magnitude and shape of the noise change with applied stress.

From his measurement results, it has been shown that the PSD of 1/f noise is proportional to $exp(-\sigma V/kT)$, where $\sigma$ is the stress and $V$ is the activation volume, that is close to the atomic volumes of bulk metals. The exponent $\gamma$ also has a dependence of the mechanical stress as shown in figure 3-7 and 3-8.

On the other hand, Fleetwood and Giordano [21] made a qualitative description of the dependence of 1/f noise on the strain of metal films. They measured 1/f noise in the platinum films with applied stress to the substrate. Their measurement results are reproduced here ($\alpha$ and $\gamma$ are switched in their notation). The noise of the original film is denoted at point A. Stress was applied to the film at point B, removed at point C and re-applied at point D. From the graph 3-9, it can be shown that the noise level is quite sensitive to the strain in the film and a process with strain relaxation might contribute to the 1/f noise.
3.2.4 Substrate Dependence

Dutta, Eberhard and Horn [27] compared the noise level and its temperature dependence for silver and copper films on sapphire and quartz substrate respectively. The thermal conductivity of quartz is an order of magnitude lower than sapphire at room temperature, and is relatively constant while the conductivity of sapphire is nearly two orders of magnitude higher at 100K than at 300K. The measurement results are reproduced here in figure 3-10 and 3-11. The substrate doesn't affect the noise for silver film over the whole temperature range. For copper film, the noise is also the same for both substrates above room temperature. However, the level of the noise changes significantly below room temperature. From these results, Dutta and his coworkers raise a hypothesis that the $1/f$ noise might be composed of two parts. Type A noise is dependent on the substrate and has a weak temperature dependence. This type of noise is dominant for copper films below room temperature. Type B noise is independent of the substrate but has a strong temperature dependence. This type of noise is dominant for copper films above room temperature and over all temperature
regime for silver films.

3.2.5 Defect Dependence

Pelz and Clarke [29] studied the dependence of 1/f noise on defects. They measured 1/f noise on polycrystalline copper films with different doses of electron irradiation. Both the resistivity and noise level increase with the induced defects. The result is shown in figure 3-12. The noise level increases by more than an order of magnitude and the slope steepens by about $9\% \pm 2\%$. The samples are then annealed. The 1/f noise and resistivity both reduce. The noise level recovers partially over the annealing temperature range $200K < T_A < 300K$ where most resistivity recovers. The noise level further exhibits a strong recovery at $T_A < 135K$ where resistivity recovery is weak. The recovery curve of noise vs. resistance looks as figure 3-13. The experiments show a clear indication that the introduced defects are responsible for the added 1/f noise.
3.3 1/f Noise Models

Given the ubiquity and complication of 1/f noise, the origin of it remains a mystery for today. The section below tries to list some existing modelings that are supported by experimental data. However, as mentioned before, none of the theories are capable of explaining all the experimental results.

3.3.1 Lattice Scattering

Based on the empirical relation, Hooge hypothesizes that lattice scattering causes the 1/f noise [24]. The resistance of a sample is given by \( \rho = \frac{1}{qn\mu} \). The fluctuations in carrier number \( n \) and carrier mobility \( \mu \) can both result in resistance fluctuations. Kleinpenning [23] has investigated the 1/f noise in thermal voltage, hall voltage, hot electrons and concluded that the data matches better with mobility fluctuation. Mobility characterizes how fast an electron moves when pulled by an electric field and is determined by how long it takes for an electron to collide with scattering centers. Three scattering mechanisms are present in the homogenous metal films: lattice scattering, impurity scattering and surface scattering. However, only lattice scattering is found to cause resistance fluctuations. The total mobility is given by

\[
\frac{1}{\mu} = \frac{1}{\mu_{latt}} + \frac{1}{\mu_{imp}}
\]  

(3.17)
The Hooge parameter $\alpha$ is modified as

$$\alpha = \alpha_{latt} \left( \frac{\mu}{\mu_{latt}} \right)^2$$

(3.18)

where $\alpha_{latt} = 2 \times 10^{-3}$. In Hooge’s modeling, 1/f noise clearly has a bulk origin. However, the description fails to describe the dependence of 1/f noise on external conditions, such as temperature, stress and defects.

### 3.3.2 Temperature-fluctuation Model

Voss and Clarke proposes the temperature-fluctuation model of 1/f noise, in which case, the equilibrium temperature fluctuations modulate the resistance [15]. The modeling starts with the well-known diffusion model for energy fluctuations. Even though, the diffusion model doesn’t give out 1/f spectrum, they included an empirical 1/f region and normalized the resulting spectrum. The resulting prediction matches the data surprisingly well. Therefore, it is hypothesized that spatially correlated
Figure 3-10: Substrate Dependence of 1/f Noise in Cu Films
(Source from [27])

Figure 3-11: Substrate Dependence of 1/f Noise in Ag Films
(Source from [27])
Figure 3-12: Dependence of 1/f Noise on Defects in Cu Films
(Source from [29])

Figure 3-13: Changes of 1/f Noise and Resistivity for Different Annealing Temperatures
(Source from [29])
temperature fluctuations give rise to the 1/f shape of the noise PSD.

\[ S_v(f) \propto \frac{\beta^2 k_B T^2}{C_v[3 + 2\ln(l/w)f]} \]  

(3.19)

Here \( \beta \) is the TCR and \( C_v \) denotes the total heat capacity of the sample, which is approximately \( 3NK_B \) at room temperature. Parameters \( l \) and \( w \) are the length and width of the metal films. This model explains the shape of 1/f and has excellent agreement with many experimental results. It has been shown experimentally that manganin, an alloy that has a TCR close to zero has non-observable 1/f noise. In addition, the noise PSD is linearly proportional to the square of TCR \( \beta^2 \) and is inversely proportional to the number of atoms \( N \). Moreover, the frequency dependence of noise spatial correlation is also experimentally demonstrated.

Even though the temperature-fluctuation model achieves considerable success in explaining the observations, it fails to predict the temperature dependence of the noise as shown in figure 3-4.

### 3.3.3 Dutta-Dimon-Horn Model

Eberhard and Horn [22] are the first to propose that vacancy and interstitial diffusion might be another cause of 1/f noise. The magnitude of noise is dependent on the number of diffusing vacancies. As the temperature decreases, the number of diffusing vacancies becomes very small, and the noise level decreases correspondingly.

In 1979, Dutta, Dimon and Horn [28] developed a theory that thermal-activated random fluctuations over barriers height near 1eV give rise to the 1/f noise in metal films. Since the temperature dependence of 1/f noise in many metal films has similar shapes as in figure 3-4, a distribution of activation energies \( E \) is extracted as shown in 3-14. The activation energies have a narrow distribution near 1eV. This distribution of energy has successfully predicted the dependence of the noise power exponent on temperature as shown in figure 3-5.

Based on the Dutta-Dimon-Horn model, F.N.H.Robinson [34] further described a more concrete picture of the activated process and how it couples to the resistance.
fluctuations. In his hypothesis, the thermally activated random process near 1eV comes from the frozen-in lattice defects of a sample’s thermal and mechanical history. Those defects are mobile and diffuse with an activation energy between 0.1eV and 1eV as required by the Dutta-Dimon-Horn model. In addition, the resistance fluctuation comes from the release and trap of the conduction electrons of an activation center. The magnitude of the fluctuation is related to the number of activation centers and the scattering cross section. When two or more mobile defects occur in a region within which the electronic wavefunction is coherent, the scattering amplitudes can interfere and modulate the scattering cross section, which then results in the resistance fluctuation. The Dutta-Dimon-Horn model qualitatively explains the dependence of 1/f noise on defects as shown in figure 3-12. The electron irradiation introduced defects in the sample that cause both the resistivity and the 1/f noise to increase. A large fraction of 1/f noise recovers at $T < 135K$ because a subpopulation of mobile defects are readily annealed at low temperatures. The majority of static defects that are responsible for resistivity change do not contribute to 1/f noise.
Chapter 4

Measurement System

4.1 System Overview

4.1.1 Measurement Setups

Two measurement setups: DC and AC measurements are used to measure 1/f noise. DC measurement is very straightforward. Nevertheless, AC setup is more practical for measuring the noise of ALD Pt samples because of its low noise floor in the measurement system. In DC measurement, a battery provides the current that passes through the ALD platinum samples so that 1/f noise shows up as voltage fluctuations. The noise voltage is then amplified through the low-noise preamplifier and analyzed by a spectrum analyzer to obtain the power spectral density. The DC measurement setup is shown in the figure 4-1.

![DC Measurement Block Diagram](image)

Figure 4-1: DC Measurement Block Diagram

Since 1/f noise is ubiquitous in all electronic systems, the one coming from the
preamplifier adds up to the noise coming from the samples. Therefore, this measurement technique has the drawback that the noise voltage from the sample needs to be at least equivalent to the input noise from the amplifier. In order to increase the noise voltage from the sample, the biasing current can be increased. However, the high current density within the metal film has the potential to cause electromigration and ultimately damages the Pt samples. An alternative AC measurement setup is used in most of the measurements. AC measurement of 1/f noise is introduced by Scofield [18] in 1987 and is then widely used in low frequency, small signal measurements. In this method, an AC current is passed through the sample, which modulates the noise voltage to the carrier frequency. After amplification near the carrier frequency, the voltage is demodulated using a lock-in amplifier. The output voltage is low passed and analyzed using the same spectrum analyzer. The block diagram is shown in figure 4-2.

![AC Measurement Block Diagram](image)

Figure 4-2: AC Measurement Block Diagram

The advantage of AC measurement is that the noise voltage from the sample is amplified near the carrier frequency. The 1/f noise region of the amplifier is avoided. The validity of the AC method will be verified both theoretically and experimentally.
4.1.2 AC Measurement Signal and Noise Path

Signal Path

1. Stage 1 - Modulation

1/f noise exhibits a resistance fluctuation, which can be written as

\[ R(t) = R_0 + \Delta R_i(t) \quad \text{where} \quad i = 1, 2, 3, 4 \quad (4.1) \]

\( \Delta R \) is centered around 0 and is much smaller than \( R_0 \). For a Wheatstone consisting of four identical resistors, the total voltage fluctuation when current \( i_0 \) is used to pass through the resistors is

\[ v_{\text{bridge}} = \frac{1}{2} i_0 \cos(\omega_c t)(\Delta R_1(t) + \Delta R_2(t) + \Delta R_3(t) + \Delta R_4(t)) \]

\[ = \frac{1}{2} (v_{r1} + v_{r2} + v_{r3} + v_{r4}) \quad (4.2) \]

2. Stage 2 - Pre-amplifier

The bridge voltage is amplified differentially by the preamplifier with a gain of \( G_{\text{preamp}} \)

\[ v_{s2} = v_{\text{bridge}} G_{\text{preamp}} \quad (4.3) \]

3. Stage 3 - Lock-in Amplifier

The output from the preamplifier is further amplified by the lock-in amplifier. The signal is demodulated by the same driving current with no phase difference.

\[ v_{s3} = G_{\text{lockin}} v_{s2} \cos(\omega_c t) \quad (4.4) \]

\[ = \frac{1}{2} i_0 G_{\text{lockin}} G_{\text{preamp}} \cos(\omega_c t) \cos(\omega_c t)(\Delta R_1(t) + \Delta R_2(t) + \Delta R_3(t) + \Delta R_4(t)) \]

\[ = \frac{1}{4} i_0 G_{\text{lockin}} G_{\text{preamp}} (1 + \cos(2\omega_c t))(\Delta R_1(t) + \Delta R_2(t) + \Delta R_3(t) + \Delta R_4(t)) \]

4. Stage 4 - Signal Spectrum

After passing through a low pass filter, the higher frequency components are
gone. The voltage signal after stage 3 is therefore

\[ v_{s4} = \frac{1}{4} G_{\text{lockin}} G_{\text{preampl}} (\Delta R_1(t) + \Delta R_2(t) + \Delta R_3(t) + \Delta R_4(t)) \] (4.5)

The autocorrelation function is

\[ R_{vv}(\tau) = E[v_{s4}(t)v_{s4}(t + \tau)] \]
\[ = \frac{1}{16} i^2 G_{\text{lockin}}^2 G_{\text{preampl}}^2 E[\sum_{i,j} R_i(t)R_j(t)] \] (4.6)

The 1/f noise from different resistors are orthogonal because noise has zero mean, and is also independent from each other.

\[ E[R_i(t)R_j(t + \tau)] = E[R_i(t)]E[R_j(t + \tau)] = 0 \text{ for } i \neq j \] (4.7)

\[ R_{vv}(\tau) = \frac{1}{16} i^2 G_{\text{lockin}}^2 G_{\text{preampl}}^2 (R_{rr1}(\tau) + R_{rr2}(\tau) + R_{rr3}(\tau) + R_{rr4}(\tau)) \] (4.8)

The resulting PSD of the signal is therefore

\[ S_{vv}(f) = \frac{1}{16} i^2 G_{\text{lockin}}^2 G_{\text{preampl}}^2 (S_{rr1} + S_{rr2} + S_{rr3} + S_{rr4}) \] (4.9)

For a Wheatstone bridge with four identical resistors, the resulting PSD of the 1/f noise is

\[ S_{vv}(f) = \frac{1}{4} i^2 G_{\text{lockin}}^2 G_{\text{preampl}}^2 S_{zz}(f) \] (4.10)

**Noise Path**

1. **Stage 1 - Components**

   The background noise of the measurement system is composed of the thermal noise of the bridge resistors and the input noise of the preamplifier. Neither of the two noise sources comes from resistance fluctuations, so they will not be modulated by the AC current. In addition, both noise sources are independent and spread over the whole frequency spectrum. The power spectral densities of
the noises are denoted as $S_{\text{thermal}}$ and $S_{\text{preamp}}$. Assume that the four resistors in the Wheatstone bridge are identical. The total background noise voltage is

$$
v_{\text{background}} = v_{\text{thermal}} + v_{\text{preamp}} \quad (4.11)
$$

$$
v_{\text{background}} = \frac{1}{2}(v_{\text{th}1} + v_{\text{th}2} + v_{\text{th}3} + v_{\text{th}4}) + v_{\text{preamp}} \quad (4.12)
$$

2. Stage 2 - Pre-amplifier

The background noise voltages are passed into the pre-amplifier.

$$
v_{n2} = v_{\text{background}}G_{\text{preamp}} \quad (4.13)
$$

Stage 3 - Lock-in Amplifier

The background noise is also modulated and amplified by the lock-in amplifier.

$$
v_{n3} = v_{n2}G_{\text{lockin}}\cos(\omega_c t) \quad (4.14)
$$

$$
v_{n3} = v_{\text{background}}G_{\text{preamp}}G_{\text{lockin}}\cos(\omega_c t)
$$

3. Stage 4 - Noise Spectrum

After low pass filtering, only the fluctuation voltages that are originally close to the carrier frequency are now shifted to the low frequency regime and are preserved. Autocorrelation of the background noise is:

$$
R_{nn}(\tau) = E[v_{n3}(t)v_{n3}(t + \tau)] \quad (4.15)
$$

$$
R_{nn}(\tau) = G_{\text{preamp}}^2G_{\text{lockin}}^2(\cos(\omega_c t)\cos(\omega_c t + \omega_c \tau) + \cos(2\omega_c t + \omega_c \tau))
$$

The output noise from the lock-in amplifier is no longer wide-sense-stationary. Since the auto-correlation function is only sinusoidal, by taking the time average
of the auto-correlation function, we obtain

$$\overline{R_{nn}(\tau)} = \frac{1}{2} G_{lockin}^2 G_{preamp}^2 \cos(\omega_c \tau) R_{nn}(\tau) \quad (4.16)$$

The PSD of the output noise is

$$S_{nn}(f) = \frac{1}{2} G_{lockin}^2 G_{preamp}^2 \int_{-\infty}^{\infty} \overline{R_{nn}(\tau)} \cos(\omega_c \tau) \cos(2\pi f \tau) d\tau \quad (4.17)$$

$$= \frac{1}{4} G_{lockin}^2 G_{preamp}^2 \left( \int_{-\infty}^{\infty} R(\tau) \cos(2\pi (f_c + f) \tau) d\tau + \int_{-\infty}^{\infty} R(\tau) \cos(2\pi (f_c - f) \tau) d\tau \right)$$

$$= \frac{1}{4} G_{lockin}^2 G_{preamp}^2 (S(f_c + f) + S(f_c - f))$$

Since the carrier frequency is selected so that the 1/f region of the preamplifier is avoided, both of the background noise sources have a white spectrum in the frequency range of interest.

$$S(f_c + f) = S(f_c - f) = S(f)$$

$$= S_{thermal}(f) + S_{preamp}(f) \quad (4.18)$$

The total PSD coming from the background noise is then

$$S_{nn}(f) = \frac{1}{2} G_{lockin}^2 G_{preamp}^2 (S_{thermal}(f) + S_{preamp}(f))$$

$$= \frac{1}{2} G_{lockin}^2 G_{preamp}^2 \left( \frac{1}{4} (S_{th1} + S_{th2} + S_{th3} + S_{th4}) + S_{preamp} \right)$$

$$= \frac{1}{2} G_{lockin}^2 G_{preamp}^2 (S_{thR} + S_{preamp})$$

### 4.1.3 DC and AC Measurement Comparison

Carbon resistors are used as verification tools for the measurement systems. The noise is measured at different dc biases. The 1/f shape and the dependence of the noise PSD on $V^2$ are seen from the measurement results. The 1/f noise levels are also in agreement with literature values for carbon resistors. Four 10kΩ resistors form a Wheatstone bridge. A 2.95V biasing voltage supplies the DC current through a
$10k\Omega$ biasing resistor, which is in series with the bridge. The biasing voltage is then doubled. The $1/f$ noise PSD is shown in figure 4-3.

![Figure 4-3: 1/f Noise of Carbon Resistors Under Different Biases](image)

The validity of AC measurements is also verified using the carbon resistors. Both DC and AC measurements are performed for a $12k\Omega$ Wheatstone bridge. Carrier frequency is selected at 150Hz. A $4.5V$ DC source and a $4.5V_{rms}$ AC source are used to supply current to the resistors respectively. Figure 4-4 shows that the measurement results from AC and DC setups fully agree with each other.

### 4.2 Detailed Measurement Stages

#### 4.2.1 Measurement Circuits

A Wheatstone bridge is used in all of the measurements for the samples since it can effectively reject noise from power supplies and biasing resistors. The bridge also makes the measurement insensitive to the bath temperature fluctuation. The output noise is the average noise of the four resistors. It is ideal to use a fully balanced bridge with four identical sample resistors. However, the mismatch of bridge resistances due to fabrication tolerance can be very large. Therefore, half-bridges are also
Comparison between DC and AC Noise Measurement Setup

Figure 4-4: AC and DC Measurement Comparison

used here, where two of the sample resistors are replaced with wirewound resistors. The wirewound resistor values are carefully picked so that the bridge is maximally balanced. Wirewound resistors have very low 1/f noise. In our measurements, they do not show up any 1/f noise down to the lowest frequency measured. Figure 4-5 shows an example of an 8kΩ wirewound resistor under 30µA biasing current. The biasing condition is very typical for producing a current density on the order of $10^5 \, A/cm^2$ in Pt samples. The wirewound resistor only shows thermal noise.

4.2.2 Low Noise Preamplifier

The SR560 low-noise preamplifier from the Stanford Research Systems is used in the first stage noise amplification. The preamplifier provides a gain of 2000 and a bandpass filter from 30Hz to 1kHz with 6dB roll off on each side. Even though a higher gain is desired for noise measurement, the bridge voltage caused by the mismatch of the bridge resistors will be so large that the input of the next stage lock-in amplifier will be overloaded. Moreover, the preamplifier is powered by batteries rather than power lines so that spurious pickups are much lower. The amplifier input is shorted
to obtain the equivalent input noise. The result is shown in figure 4-6.

A carrier frequency of 150Hz effectively avoids the 1/f noise from the preamplifier and the ubiquitous 60Hz signals have no significant impact on the measurements. The gain flatness and the bandpass filter response are characterized by a simple attenuator circuit as in figure 4-7. The gain is very flat within the frequency range of interest as shown in the frequency response 4-8.

### 4.2.3 Lock-in Amplifier

**Lock-in Settings**

The SR510 lock-in amplifier from the Stanford Research Laboratory is used perform the signal lock-in. This amplifier is shown in figure 4-9. The lock-in amplifier uses a single-ended input. The sensitivity is set to 500mV. The output of the low-noise preamplifier often has a bias in the range of 0V-500mV due to the mismatch of bridge resistors. The offset of the lock-in amplifier is carefully adjusted so as to zero out the DC biasing as much as possible. The two build-in low-pass filters are both deactivated.
Gain Characterization

Since the input of the lock-in amplifier is a high-frequency signal while the output of it is a low frequency one near DC, the gain of the lock-in amplifier in the measurement is defined as follows. Suppose the input to the lock-in amplifier is

\[ v_{input} = v_{in} \cos(\omega_c t) \]  

(4.19)

The reference channel takes the driving source and the phase difference is set to 0.

\[ v_{reference} = \cos(\omega_c t) \]  

(4.20)
The output of the lock-in amplifier is then

\[ V_{\text{output}} = G_{\text{lockin}} v_{\text{in}} \cos(\omega_c t) \cos(\omega t) \]

\[ = \frac{1}{2} G_{\text{lockin}} v_{\text{in}} (1 + \cos(2\omega_c t)) \]  

(4.21)

Since the high-frequency components are filtered out by the low-pass filter, the output to input ratio is

\[ \frac{v_{\text{output}}}{v_{\text{in}}} = \frac{1}{2} G_{\text{lockin}} \]  

(4.22)

The gain is defined as

\[ G_{\text{lockin}} = \frac{2v_{\text{output}}}{v_{\text{in}}} \]  

(4.23)
In our measurement, the lock-in amplifier is operated in the low gain mode, with a sensitivity of 500mV. The specification means that with a 500mV\textsubscript{rms} input, the output of the amplifier will be 10V if the input is in phase with the reference signal and -10V if the input is out of phase with the reference. Therefore,

\[
G_{\text{lockin}} = \frac{2 \times 10}{0.5 \times \sqrt{2}} = 20\sqrt{2}
\]  

(4.24)

The gain flatness of the lock-in amplifier is characterized by two Agilent 321120A signal generators synchronized with no phase difference. One signal generator is internally modulated with a low frequency signal. The modulated signal is fed into the input of the lock-in amplifier while the other signal is fed into the reference channel. The output of the lock-in is monitored through an oscilloscope. The gain of the amplifier measured up to 20Hz is very flat and equal to the DC gain.

**Low Pass Filter**

A second order low pass filter with a cut-off frequency at \( f = \frac{1}{2\pi RC} = 194Hz \) is constructed to reject the high frequency components. The circuit is shown in 4-10 and the frequency response of the filter is shown in 4-11.

![2nd Order Low Pass Filter](image)

Figure 4-10: 2nd Order Low Pass Filter

**4.2.4 Dynamic Signal Analyzer**

The HP35665A Dynamic Signal Analyzer (DSA) is used to estimate the power spectral density of noise. The DSA samples the input voltage, performs a Fast Fourier
Transform and squares the result to obtain a periodogram of the noise spectrum.

**Frequency Span and Sampling**

When the amplified noise voltages are fed into the DSA, it first passes through an analog anti-aliasing filter, which sets its cutoff frequency at 154112Hz. The sampling frequency of the analog to digital converter (ADC) is 308224Hz. Since the sampling frequency is much higher than twice the frequency that the measurements are taken, aliasing will not occur. The block diagram of the DSA looks as figure 4-12 [35]. The multiplication by a sinusoidal signal is used to measure non-baseband zoomed-

![Block Diagram of Dynamic Signal Analyzer](image)

**Figure 4-12: Block Diagram of Dynamic Signal Analyzer**

in frequency range, which is not used in our measurement. The frequency span of 0.390625Hz is mostly used for the noise measurements of Pt samples.
Periodogram

Periodograms are used to estimate the PSD of a random process [10]. Suppose a random realization of the noise process $x_c(t)$ is sampled with a sampling period of $T$ for a total of $N$ samples. The relationship between the PSD of the noise process and the sampled sequence is then:

$$S_{xx}(e^{j\Omega}) = S_{x_c x_c}(j\omega)|_{\Omega = \frac{\pi}{T}}$$  \hspace{1cm} (4.25)

The sequence within a finite time interval is

$$v[n] = x_c(nT)w[n] = x[n]w[n]$$  \hspace{1cm} (4.26)

where $w[n]$ is a window function that is only non-zero for $n \in [0, N-1]$. $v[n]$ is a finite length sequence with a well-defined Fourier transform most of the time. The periodogram, which is an estimator of PSD, is defined as follows:

$$I(e^{j\Omega}) = \frac{1}{N}|V(e^{j\Omega})|^2$$  \hspace{1cm} (4.27)

Size of Sampled Sequence

The periodogram can be further expanded to

$$I(e^{j\Omega}) = \frac{1}{N}F(v[n] * v[-n])$$  \hspace{1cm} (4.28)

$$= \frac{1}{N}F(\sum_{m=-\infty}^{N-1} x[m]x[m+n]w[m]w[m+n])$$

In order to verify whether the periodogram is an unbiased estimator, the expected value of the periodogram is compared with the PSD.

$$E[I(e^{j\Omega})] = \frac{1}{N}F(\sum_{m=-\infty}^{\infty} E[x[m]x[m+n]w[m]w[m+n]])$$

$$= \frac{1}{N}F(R_{xx}[n]\sum_{m=-\infty}^{N-1} w[m]w[m+n])$$  \hspace{1cm} (4.29)
As seen in 4.29, the bias of the estimator depends on the window function. When the length of window is small, the estimator is smeared by the spectrum of the window. However, as \( N \to \infty \), the power spectrum of the window \( |W(e^{i\Omega})|^2 \to \delta \). Thus, \( E[I(e^{i\Omega})] \to S_{zz}(e^{j\Omega}) \). The bias approaches 0.

In the 1/f noise measurement, \( N \) is taken to be 2048, which is the maximum number of data points for each run. 2048 data points are used to compute 1024 points in frequency domain. The lowest frequency points are often discarded since the time length is not long enough to provide accurate information.

**Periodogram Averaging**

Another important figure of merit in evaluating the performance of an estimator is the consistency. When the variance of the estimator is equal to zero, the estimator is a consistent one and vice versa. For the single periodogram estimate, as \( N \to \infty \), the variance of the periodogram is.

\[
\text{var}(I(e^{i\Omega})) \approx S_{zz}(e^{j\Omega})
\]  \hspace{1cm} (4.30)

Therefore, single periodogram is not a consistent estimator. In order to improve the estimation of noise PSD, a new estimator is introduced by averaging multiple independent periodogram estimates.

\[
I_{zz}(e^{j\Omega}) = \frac{1}{K} \sum_{k=1}^{K} I(e^{j\Omega})
\]  \hspace{1cm} (4.31)

The new estimator \( I_{zz}(e^{j\Omega}) \) is also unbiased since \( E[I_{zz}(e^{j\Omega})] = E[I(e^{j\Omega})] = S_{zz} \). The variance of the estimator is now

\[
\text{var}(I_{zz}(e^{j\Omega}) = \frac{1}{K} \text{var}(I(e^{j\Omega})) = \frac{1}{K} S_{zz}(e^{j\Omega})
\]  \hspace{1cm} (4.32)
As $K \to \infty$, $I_{xx}(e^{j\Omega}) \to 0$. Therefore, increasing the number of realizations over which the averaging is done will reduce the noise in the estimate. However, taking more averages will significantly increase the measuring time, which makes the waiting time of the measurements even worse. Therefore, 10 averages are used to produce a relatively smooth estimate of the $1/f$ noise PSD.

**Windowing**

In the discrete signal processing of sampled noise data, the window function $w[n]$ is used to multiply with the input signal and repeat the resulting sequence to make a periodic signal. In the time domain, a uniform window preserves the exact shape of the sampled sequence. Other commonly used windows, such as Hann and Flattop, are smoother and weights the edge data less than the ones in the middle. The window function is often analyzed in the frequency domain in order to analyze its effect on the spectrum. In the time domain, the window function $w[n]$ is multiplied to the sampled data $x[n]$

$$y[n] = x[n]w[n]$$  \hspace{1cm} (4.33)
Table 4.1: Window Parameter Summary

<table>
<thead>
<tr>
<th></th>
<th>Uniform</th>
<th>Hann</th>
<th>Flattop</th>
</tr>
</thead>
<tbody>
<tr>
<td>Main Lobe Width [bins]</td>
<td>0.89</td>
<td>1.44</td>
<td>3.72</td>
</tr>
<tr>
<td>Maximum Side Lobe [dB]</td>
<td>-13</td>
<td>-31</td>
<td>-93</td>
</tr>
<tr>
<td>Side lobe roll-off Rate [dB/decade]</td>
<td>20</td>
<td>60</td>
<td>20</td>
</tr>
<tr>
<td>Coherent Gain</td>
<td>1.00</td>
<td>0.50</td>
<td>0.22</td>
</tr>
<tr>
<td>Noise Power Bandwidth [bin]</td>
<td>1.00</td>
<td>1.50</td>
<td>3.77</td>
</tr>
<tr>
<td>Worse-case Amplitude Error [dB]</td>
<td>-4</td>
<td>-1.5</td>
<td>-0.005</td>
</tr>
</tbody>
</table>

This corresponds to the periodic convolution in the frequency domain.

\[
Y(e^{j\Omega}) = \frac{1}{2\pi} X(e^{j\Omega}) \ast W(e^{j\Omega})
\]  

(4.34)

Therefore, the windowing of the signal is equivalent to putting a series of bandpass filters centered on each frequency bin. It is desirable that the bandpass filter preserves the signal amplitude and is as narrow as possible. Therefore, the optimal window is selected based on the main lobe width, the side lobe height and the side lobe roll-off rate. Table 4.1 is a summarization of the three commonly used windows.

Noise is a broadband signal with contents of similar amplitudes over a wide frequency range. In order to resolve two different frequency components, a low side-lobe height and a fast roll-off rate are required. The flattop window offers the best amplitude accuracy but its frequency resolution is too poor for noise measurement. The uniform window has the best frequency resolution. However, it has a lower side lobe attenuation ratio, which means that many frequency components near the center frequency will all contribute to the reading of the center frequency. In addition, the high amplitude uncertainty will result in inaccurate data. For the noise measurement, the Hann window is chosen because of its relatively good resolution, high side-lobe attenuation to reject adjacent interference and the relatively good amplitude accuracy. The coherent gain is already being normalized by the DSA.
4.2.5 Oven Temperature Control

In order to minimize the bath temperature fluctuations, the samples are placed inside a laboratory oven, which has a temperature stability within 0.2°C. The temperature of the oven is monitored through a commercial thermistor HSRTD-3-100-A-40-E. The well-calibrated thermistor has a resistance of 100Ω at 0°C and a TCR of 0.00385Ω/°C. Therefore, the temperature information can be extracted based on the resistance of RTD thermistor according to

\[ T = \frac{R_{\text{rtd}} - 100}{100 \times 0.00385} \]  

(4.35)

The thermistor provides an accurate value of the bath temperature at any given time point. It is placed near the Pt samples as close as possible. The resistance value of the RTD is measured by circuit 4-14

![Temperature Measurement Circuit](image_url)

Figure 4-14: Temperature Measurement Circuit

The noise voltage from the samples and the temperature information from the RTD are fed into channel 1 and channel 2 of the DSA respectively and they are sampled at the same time. The approach is to make sure that the measured noise comes from the samples, rather than from the fluctuations of the bath temperature. In fact, some of the measured noise voltages do have a strong correlation with the bath temperature, which will be shown in the next chapter.
Chapter 5

Results and Discussion

5.1 Sample Resistivity

For the Pt samples in this work, the average resistivity is $28.3 \mu \Omega \cdot cm$ for 7nm films and $23.4 \mu \Omega \cdot cm$ for 13nm films, both higher than the $10.6 \mu \Omega \cdot cm$ bulk resistivity. By Matthiessen’s rule, the resistivity of a metal film is composed of two parts. Part $\rho_L$, also called the bulk resistivity, is caused by the scattering of the electrons by phonons in the lattice. The contribution of $\rho_i$ to the resistivity is induced by the presence of imperfections in the lattice. The resistivity often increases as the film thickness decreases because the contribution of the defects is much more noticeable.

$$\rho = \rho_L + \rho_i \quad (5.1)$$

In addition to ALD, the platinum thin films are most commonly fabricated by Chemical Vapor Deposition (CVD) and sputtering. The resistivity of the ALD fabricated films is generally smaller than the resistivity of the films fabricated by CVD or sputtering. Table 5.1 compares the resistivities of Pt films fabricated by different methods. The symbol $t$ refers to the thickness of the film.
Table 5.1: Comparison of Pt Film Resistivities Fabricated by Different Methods

<table>
<thead>
<tr>
<th></th>
<th>ALD</th>
<th>CVD</th>
<th>Sputtering</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\rho$ [$\mu\Omega \cdot cm$]</td>
<td>$t$ [nm]</td>
<td>Source</td>
</tr>
<tr>
<td>18</td>
<td>10</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>50</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>110</td>
<td>36</td>
<td></td>
</tr>
</tbody>
</table>

5.2 Noise of ALD Pt Films

5.2.1 1/f Noise

1/f noise is observed in the Pt samples under a low biasing current. Figure 5-1 shows a typical noise PSD from a Wheatstone bridge consisting of two samples under test and two wirewound resistors. The sample under test is a 7nm Pt film of dimension $7nm \times 50\mu m \times 5000\mu m$ under a biasing current density of $1.1 \times 10^4 A/cm^2$. The noise voltage is sampled at 2Hz, 16Hz and 32Hz and averaged 50 times. The resulting periodograms are overlapped together. Thermal noise is subtracted from the total noise.

![1/f Noise of ALD Pt Sample Films](image)

Figure 5-1: Total Noise of 7nm Pt Sample Films
noise PSD. The resulting 1/f noise is shown in figure 5-2. The solid line drawn has exactly the shape of 1/f.

![Extracted 1/f Noise PSD](image)

**Figure 5-2: 1/f Noise of 7nm Pt Sample Films**

Figure 5-3 shows the total noise of a 13nm samples of dimension $13nm \times 5\mu m \times 10000\mu m$. The noise is measured under three different current densities as shown in the legend. The bridge voltages are sampled at 1Hz, 8Hz and 32Hz and averaged 10 times. The dependence of the noise PSD on the square of voltage is observed.

Based on the Hooge empirical formula in equation 5.2, the noise PSD is normalized by the voltage drop $U^2$ and the total number of charge carriers $N$ in the samples.

$$\frac{S_V(f)}{U^2} = \frac{S_R(f)}{R^2} = \frac{\alpha}{Nf^\gamma} \tag{5.2}$$

The number of charge carriers in a sample is directly proportional to the number of atoms, which is readily calculated. However, the exact relation between these two numbers is quite complicated. For simplicity, $N$ is taken to be the number of atoms
in the sample, which is

\[ N = \frac{V}{V_a} \times Av \]  

(5.3)

The symbol V is the volume of the sample and \( V_a \) is the atomic volume of Platinum, which is 9.1cm\(^3\)/mol. The constant Av is the Avogadro number 6.022 \( \times 10^{23} \) mol\(^{-1}\).

Table 5.2 summarizes the measurement results and compares them with the reports in literatures. The results are accurate within ±20%.

The 1/f noise in the ALD Pt films generally falls on the same order of magnitude. However, sample-to-sample variation of the noise magnitude and power exponent is also observed. In comparison to other continuous Pt films, the noise in the ALD Pt films is about two orders of magnitude larger. Referring to the defect-induced-noise experiments in [29], the ALD fabricated films might have a small amount of mobile defects that give rise to the additional 1/f noise. On the other hand, the concentration of static defects is small in comparison to the other films because of the relatively low resistivity observed.
Table 5.2: Summarization of 1/f Noise in Pt Thin Films

<table>
<thead>
<tr>
<th>Hooge Parameter $\alpha$</th>
<th>Power Exponent $\gamma$</th>
<th>Thickness [nm]</th>
<th>Resistivity [$\mu\Omega\text{cm}$]</th>
<th>Fabrication Method</th>
<th>Substrate Material</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0156</td>
<td>0.9338</td>
<td>7</td>
<td>29.7</td>
<td>ALD</td>
<td>$A_2O_3$ on $SiO_2$</td>
</tr>
<tr>
<td>2</td>
<td>0.0159</td>
<td>1.194</td>
<td>7</td>
<td>26.0</td>
<td>ALD</td>
<td>$A_2O_3$ on $SiO_2$</td>
</tr>
<tr>
<td>3</td>
<td>0.0401</td>
<td>1.249</td>
<td>7</td>
<td>28.9</td>
<td>ALD</td>
<td>$A_2O_3$ on $SiO_2$</td>
</tr>
<tr>
<td>4</td>
<td>0.013</td>
<td>1.016</td>
<td>13</td>
<td>24.4</td>
<td>ALD</td>
<td>$A_2O_3$ on $SiO_2$</td>
</tr>
<tr>
<td>5</td>
<td>0.085</td>
<td>1.279</td>
<td>13</td>
<td>22.5</td>
<td>ALD</td>
<td>$A_2O_3$ on $SiO_2$</td>
</tr>
<tr>
<td>6</td>
<td>0.05</td>
<td>1.242</td>
<td>13</td>
<td>24.4</td>
<td>ALD</td>
<td>$A_2O_3$ on $SiO_2$</td>
</tr>
<tr>
<td>7</td>
<td>0.00064</td>
<td>1.35</td>
<td>unspecified</td>
<td>40-60</td>
<td>Ion Beam Sputtering</td>
<td>Glass or Sapphire</td>
</tr>
<tr>
<td>8</td>
<td>0.00026</td>
<td>1.15</td>
<td>8 - 20</td>
<td>40-60</td>
<td>Ion Beam Sputtering</td>
<td>Glass or Sapphire</td>
</tr>
<tr>
<td>10</td>
<td>0.0001-0.003</td>
<td>1.08-1.23</td>
<td>8-20</td>
<td>40-60</td>
<td>Ion Beam Sputtering</td>
<td>Glass or Sapphire</td>
</tr>
<tr>
<td>11</td>
<td>0.00002</td>
<td>1.10</td>
<td>142</td>
<td>12.0</td>
<td>Electron Beam Evaporated</td>
<td>Sapphire</td>
</tr>
<tr>
<td>12</td>
<td>0.00007</td>
<td>1.06</td>
<td>125</td>
<td>10.7</td>
<td>Electron Beam Evaporated</td>
<td>Sapphire</td>
</tr>
<tr>
<td>13</td>
<td>0.00012</td>
<td>1.18</td>
<td>164</td>
<td>13.5</td>
<td>Electron Beam Evaporated</td>
<td>Sapphire</td>
</tr>
<tr>
<td>14</td>
<td>0.03</td>
<td>1</td>
<td>1.8</td>
<td>7200-28800</td>
<td>Laser Electrodispersion</td>
<td>$SiO_2$</td>
</tr>
</tbody>
</table>
5.2.2 Other Noise Spectrum

$1/f^2$ Noise

Most of the Pt samples exhibit $1/f^2$ noise PSD under high current densities. The $1/f^2$ spectrum is most probably caused by early stage electromigration. However, the current density that induces electromigration is generally smaller than $10^6 \text{A/cm}^2$, which is commonly reported in the literatures. For the 7nm sample films, the noise shape changes to $1/f^2$ even at a current density as low as $2 \times 10^4 \text{A/cm}^2$. For the 13nm films, the shape of the noise also changes at a current density of approximately $10^5 \text{A/cm}^2$. It is very likely that the thickness of the samples is not as uniform as desired. There are some points in the resistor that are much thinner than elsewhere. When the current increases, the current density through those points reaches the critical value and induces electromigration noise. One $1/f^2$ noise PSD is shown in figure 5-4. The power exponent $\gamma$ is approximately 2.

![Figure 5-4: 1/f Noise of Pt Sample Films](image)

When the current continues to increase after electromigration noise occurs, the magnitude of the noise increases and the power exponent also increases. Figure
5-5 shows a 13nm Pt film under electromigration damage. The power exponent increases from 1.8 to 2.2 when the current density changes from $1.7 \times 10^5 \text{A/cm}^2$ to $2.9 \times 10^5 \text{A/cm}^2$.

![Dependence of Electromigration Noise on Biasing Current](image)

**Figure 5-5: Dependence of $1/f^2$ Noise on Biasing Current**

The temperature dependence of the $1/f^2$ noise is recorded for the same sample in figure 5-6. The points in the figure can be fitted as an activated process as in equation 5.4. The activation energy $E_a$ is approximately 0.562eV.

$$S_{\nu \nu} = Aexp\left(-\frac{E_a}{k_B T}\right)$$

(5.4)

**$1/f^{3/2}$ Noise**

Some of the sample films also exhibit a noise PSD of power exponent close to 1.5. The $1/f^{3/2}$ noise has been reported in Ag films with electromigration damage [40] and in Pt films with metallic adhesion layers [38]. According to Swastik [40], long range diffusion in the form of resistance fluctuations will result in the $1/f^{3/2}$ spectrum. Ag films that suffer from electromigration show up the $1/f^{3/2}$ PSD because more diffusion pathways are opened up by the damage. The Pt films with metallic adhesion layer
also show up $1/f^{3/2}$ noise because of the long range diffusion of oxygen atoms along the interface.

For the ALD fabricated Pt samples of 7nm, $1/f^{3/2}$ noise is observed under a biasing current density of $1.2 \times 10^5 A/cm^2$. For the 13nm samples, the noise shows up $1/f^{3/2}$ fluctuations under a biasing current density of $1.7 \times 10^5 A/cm^2$. However, for other samples of the same thickness and under similar biasing conditions, $1/f^2$ noise normally shows up instead of $1/f^{3/2}$. For the ALD samples that show up $1/f^{3/2}$ spectrum, it is possible that a long range diffusion process involving hydrogen or oxygen is also present. Since the $1/f^{3/2}$ noise is mostly observed under biasing current close to the electromigration threshold, the diffusion process might also involve pathways created by the damage.
5.3 Limitations and Future Work

5.3.1 Temperature Correlation

For some samples, the bridge voltage is strongly correlated to the bath temperature fluctuation. In figure 5-7, it can be seen that the bridge voltage closely follows the oven temperature fluctuation. The temperature correlation is caused by the resistance mismatch in the Wheatstone bridge. The resistance of the sample Pt films range from $3.7k\Omega$ to $34k\Omega$. With a 1% mismatch of the resistance caused by fabrication imperfections, the resistance mismatch ranges from $10\Omega$ to $393\Omega$.

An attempt has been made to subtract the temperature influence from the noise voltage. The TCR of the samples are measured by Shingo Yoneoka in the previous work and the result is shown in figure 5-8.

The temperature compensation doesn’t have any impact on the thermal noise regime. However, in the $1/f$ regime, the power exponent $\gamma$ reduces with the compensation as shown in figure 5-9.

However, the estimates of the noise PSD are not trustworthy even with tempera-
ture compensation. Applying temperature compensation for very weakly correlated samples will cause stronger correlation. In addition, the temperature compensation will introduce more artifacts if there is temperature delay between the thermistor and the bridge voltage.

5.3.2 Future Work

Matching Resistors

In the future, it is desirable to devote more effort to better matching the bridge resistors. For the resistors used in this experiments, the length ranges from $1000\mu m$ to $10000\mu m$. The long resistors can be divided into small segments and the segments from the two resistors can be interweaved so that the variations due to fabrication non-uniformity will be canceled out.
Consistent Estimator

The accuracy of the noise PSD estimation depends on the number of averages of the periodograms. In this measurement, the periodograms are only averaged 10 times due to time limitations. When the thermal noise is subtracted from the total noise, the 1/f noise PSD will vary on both shape and magnitude depending on where the thermal noise floor is taken. In the measurements above, the 1/f noise is extracted for the frequency range where its power is equal or larger than the thermal noise. However, this method will leave too few data to correctly extract the noise PSD when the biasing current is small. In the future, it would be better to take more averages of the periodogram to generate smoother curves.

More Complete Assessment of Film

In addition, it would be helpful to have a more complete assessment of the films through other measurements. Since the 1/f noise is dependent on so many factors, there are many possibilities that contribute to the large noise observed. Information about the film residual stress and the grain size will be helpful. It would also be useful
to observe the temperature dependence, substrate dependence, thickness dependence and stress dependence of the noise in the samples. These observations would give more insights about the origin of the $1/f$ noise in ALD fabricated thin films.
Bibliography


